



# **Sodium Silicate Stabilization of Collapsible Clayey Calcareous Soils**

**By**

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## **Dedication**

To

The Almighty God who shows me mercy undeserved.

Without His grace, peace and mercy my life could have turned miserable.

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## Abstract

Collapsible soils have caused infrastructural damages resulting in several economic losses and loss of lives in certain cases. The prerequisite for collapse occurring is an open metastable structure; capable of developing in any soil type given the right placement or aging conditions. Natural and anthropogenic (engineered & non-engineered) collapsible soils exist in many regions of the world. In their unsaturated state, these soils exhibit high enough strength (provided by inter-particle bonds of either suction, clay, calcium carbonate or other salts) but upon wetting and/or loading they undergo repacking due to bond softening/weakening. Despite the large body of research in this subject, there is still poor understanding of the process of softening/weakening and the collapse mechanism of certain bond elements, particularly  $\text{CaCO}_3$ , considering its low solubility in water. Because  $\text{CaCO}_3$  is common in natural soils, reaching 1-30 % contents in the most commonly known natural collapsible soil (loess), understanding its influence on the collapse phenomenon is crucial for geotechnical characterization of soils in order to design safe and economic earthworks with their long-term serviceability also in focus.

In this thesis, an innovative method of simulating an open structure supported by calcite and/or clay bonding has been developed to study mainly the effect of calcite and clay content on the collapse potential (CP) and rate of collapse of calcareous silty-clay soils analogous to marls. A relationship between unconfined compressive strength (UCS) and CP was also examined. Collapsibility and UCS were examined using single and double oedometer techniques and unconfined compressive test (UCT), respectively. Additionally, the potential for a single-step application of sodium silicate solution (silicatization) to improve the soils was investigated.

Silicatization was carried out by both impregnation of pre-formed specimens and mixing & kneading prior to specimen formation. Treated oedometer specimens were simply heat-cured ( $45 - 50\text{ }^\circ\text{C}$ ) for 24 hours whereas, some UCT specimens were heat-cured, and others cured under room ambience for 7, 14, and 28 day to assess the effect of curing method on UCS.

CP was estimated by the percentage decrease in height of an oedometer specimen due to wetting. Different wetting fluids (distilled water and a 5% acid solution (AS)) were used in order to investigate the influence of pore fluid pH on CP. Several wetting stresses were adopted but 300 kPa (roughly equivalent to the self-weight of a 15m high embankment) was the main stress studied.

The research found that UCS and stiffness increase with increasing calcite content and clay content, which is possibly due to an increasing in inter-particle bonding. It also reveals that the magnitude and rate of collapse are controlled mainly by the calcite content, pH of wetting

fluid, clay content, dry density, and wetting stress. Both magnitude and rate of collapse decrease with increasing  $\text{CaCO}_3$  content and dry density but CP increases with increasing wetting stress: each sample reaching a maximum defined by their initial strength, beyond which CP decreases. Increasing clay content resulted in higher CP for non-calcareous samples but resulted in lower CP for calcareous samples. Wetting with acidic solution demonstrated higher CP and tends to prolong time to reach complete collapse resulting in long-term collapse known as subsidence settlement.

Silicatization was found to reduce stiffness at early curing with minimal increase in UCS, but at later ages both UCS and stiffness progressively increased with curing time and drying. Silicatization was more effective at increasing strength and stiffness of specimens with higher clay and calcite contents. For each curing period, mixing & kneading produced higher UCS than impregnation by between 140% - 356%, 63% - 193%, and 16% - 71% at 7, 14, and 28 days curing, respectively. At 300kPa wetting stress, silicatization and heat-curing reduced CP by between 59% – 73% under distilled water wetting and 70% – 78% under AS wetting.

**Key Words:** Calcareous, Collapsibility, Unconfined compressive strength, silicatization.

# Table of Contents

DEDICATION .....	I
ACKNOWLEDGEMENT.....	II
ABSTRACT .....	III
LIST OF FIGURES.....	X
LIST OF TABLES .....	XIX
GLOSSARY OF TERMS AND ABBREVIATION .....	XXI
<b>CHAPTER 1 INTRODUCTION.....</b>	<b>1</b>
1.1 GENERAL BACKGROUND.....	1
1.1.1 Strength and stiffness of collapsible soils.....	2
1.1.2 Collapsibility and forms of collapsible soils.....	2
1.1.3 The influence of the carbonate bond on collapsibility .....	4
1.1.4 Economic loses from soil collapse .....	5
1.1.5 Collapse treatment methods.....	6
1.2 PROJECT AIM AND OBJECTIVES .....	8
1.3 SCOPE OF WORK.....	8
1.4 THESIS OUTLINE.....	9
<b>CHAPTER 2 LITERATURE REVIEW.....</b>	<b>10</b>
2.1 INTRODUCTION .....	10
2.2 TYPES AND ORIGIN OF COLLAPSIBLE SOILS .....	10
2.2.1 Manmade (engineered and non-engineered) collapsible soils.....	11
2.2.2 Natural collapsible soils.....	14
2.3 UNDERSTANDING SOIL COLLAPSE MECHANISMS .....	18
2.3.1 Micromechanics (or bond alteration) point of view.....	21
2.3.2 The Mechanical or Effective Stress Viewpoint.....	23
2.3.3 Microstructure of collapsible soils.....	26
2.3.4 Types of bonding in collapsible soils (loess) and structural bond transformation .....	35
2.4 PREDICTING COLLAPSE IN SOILS.....	37
2.4.1 Reconnaissance .....	38
2.4.2 Relationship of fundamental properties .....	39
2.4.3 Field predicting methods.....	42
2.4.4 Experimental predicting methods.....	46
2.5 FORMATION, OCCURRENCE AND GEOTECHNICAL PROPERTIES OF CALCAREOUS SOILS.....	50
2.5.1 Formation and occurrence.....	50

2.5.2	<i>Geotechnical characterization</i> .....	53
2.5.3	<i>Effect of CaCO<sub>3</sub> content on geotechnical properties</i> .....	61
2.5.4	<i>Geochemical characteristics</i> .....	64
2.6	SIMULATION OF COLLAPSIBLE SOILS .....	67
2.6.1	<i>Non-calcareous collapsible soils</i> .....	67
2.6.2	<i>Calcareous collapsible soils</i> .....	68
2.7	SIMULATION OF CALCAREOUS SOILS FOR OTHER GEOTECHNICAL STUDIES.....	68
2.8	OUT-OF-DISCIPLINE ARTIFICIAL PRODUCTION OF CALCAREOUS SOILS .....	71
2.9	COLLAPSE MITIGATING MEASURES: AVAILABLE TECHNIQUES .....	74
2.9.1	<i>Mechanical or physical processes</i> .....	74
2.9.2	<i>Hydraulic processes</i> .....	75
2.9.3	<i>Chemical processes</i> .....	75
2.9.4	<i>Biological processes</i> .....	76
2.9.5	<i>Electro-kinetics (EK)</i> .....	76
2.9.6	<i>Soil Gassing</i> .....	77
2.9.7	<i>Nanotechnology</i> .....	78
2.10	SODIUM SILICATE: A BINDER.....	79
2.10.1	<i>Brief history</i> .....	79
2.10.2	<i>Manufacture of sodium silicates</i> .....	80
2.10.3	<i>Properties of Sodium Silicate</i> .....	80
2.10.4	<i>Sodium silicate - soil reaction mechanism</i> .....	86
2.10.5	<i>Geotechnical application of sodium silicate</i> .....	88
2.11	CHAPTER SUMMARY .....	92
2.11.1	<i>Prerequisite for collapse</i> .....	92
2.11.2	<i>The mechanism and prediction of collapse</i> .....	92
2.11.3	<i>Inter-particle bonding material and collapse potential</i> .....	93
2.11.4	<i>Collapse mitigating measures</i> .....	94
2.12	RESEARCH QUESTIONS AND GAPS IN THE LITERATURE .....	94
<b>CHAPTER 3 METHODOLOGY .....</b>		<b>96</b>
3.1	OVERVIEW .....	96
3.2	SUMMARY OF EACH STAGE OBJECTIVES.....	96
3.3	STAGE 1 – DEVELOPMENT OF A CaCO <sub>3</sub> PRECIPITATION TECHNIQUE.....	98
3.3.1	<i>Preliminary study 1</i> .....	98
3.3.2	<i>Our understanding about lime-carbon dioxide reaction</i> .....	98
3.3.3	<i>Preliminary study 2</i> .....	100
3.3.4	<i>The innovative reaction setup developed in-house</i> .....	100
3.4	STAGE 2 – DESIGN AND PRODUCTION OF MAIN TEST SAMPLES.....	107

3.4.1	<i>Material choices and justification</i> .....	107
3.4.2	<i>Kaolin clay</i> .....	108
3.4.3	<i>Quartz silt</i> .....	109
3.4.4	<i>Hydrated Lime (Ca(OH)<sub>2</sub>)</i> .....	111
3.4.5	<i>Sample constitution and production procedures</i> .....	112
3.4.6	<i>X-Ray diffraction (XRD) analysis</i> .....	124
3.4.7	<i>Micro-fabric analysis</i> .....	129
3.5	STAGE 3 – GEOTECHNICAL TESTING TECHNIQUES .....	134
3.5.1	<i>Tests specimen preparation</i> .....	135
3.5.2	<i>Collapse Testing Apparatus and Procedures</i> .....	136
3.5.3	<i>Unconfined compressive strength testing equipment and protocol</i> .....	140
3.6	GENERAL LIMITATIONS IN THE LABORATORY WORKS .....	143
3.6.1	<i>Scope of work</i> .....	143
3.6.2	<i>Limited number of test samples</i> .....	143
3.6.3	<i>Experimental methodology and conditions</i> .....	143
3.6.4	<i>Quality of SEM images</i> .....	144
3.7	CONCLUSION ON STUDY APPROACH AND METHODOLOGY .....	144
<b>CHAPTER 4 MECHANICAL BEHAVIOUR OF UNTREATED SAMPLES.....</b>		<b>145</b>
4.1	INTRODUCTION .....	145
4.2	UNCONFINED COMPRESSIVE STRENGTH (UCS) TESTING .....	145
4.2.1	<i>Introduction</i> .....	145
4.2.2	<i>Stress-strain relationship of uncemented non-calcareous samples</i> .....	147
4.2.3	<i>Stress-strain relationship of cemented calcareous samples</i> .....	148
4.3	DISCUSSION ON PARAMETERS AFFECTING UCS AND STIFFNESS .....	151
4.3.1	<i>Effect of CaCO<sub>3</sub> content on USC and stiffness, and density related properties</i> .....	151
4.3.2	<i>Effect of clay content on UCS and stiffness</i> .....	156
4.3.3	<i>Summary of UCS behaviour</i> .....	158
4.4	COLLAPSE BEHAVIOUR OF SAMPLES .....	159
4.4.1	<i>Collapsibility of uncemented non-calcareous silty-clays</i> .....	159
4.4.2	<i>Collapsibility of calcite cemented (calcareous) samples</i> .....	163
4.4.3	<i>Collapsibility of uncemented calcareous silty-clay samples</i> .....	184
4.5	DISCUSSION ON PARAMETERS AFFECTING COLLAPSE.....	187
4.5.1	<i>Effect of CaCO<sub>3</sub> content on collapse potential</i> .....	187
4.5.2	<i>Nature of CaCO<sub>3</sub> occurrence - cemented versus uncemented</i> .....	191
4.5.3	<i>Effect of dry density</i> .....	192
4.5.4	<i>Effect of clay content</i> .....	194
4.5.5	<i>Effect of wetting fluid type and rate of collapse</i> .....	200

4.5.6	<i>Comparing single and double oedometer methods</i>	204
4.5.7	<i>Influence of wetting stress on collapse potential</i>	206
4.5.8	<i>Summary on the collapse behaviour of samples</i>	206
<b>CHAPTER 5</b>	<b>SODIUM SILICATE TREATMENT</b>	<b>208</b>
5.1	INTRODUCTION	208
5.2	FORMULATION OF SODIUM SILICATE SOLUTION AND TREATMENT STRATEGY	209
5.2.1	<i>Formulation of solution</i>	209
5.2.2	<i>Main treatment of test specimens</i>	216
5.2.3	<i>Stress-strain behaviour of samples treated by impregnation (capillary rise)</i>	218
5.2.4	<i>Stress-strain behaviour of samples treated by mixing and kneading</i>	224
5.3	FACTORS AFFECTING UCS AND STIFFNESS OF THE TREATED SAMPLES	230
5.3.1	<i>Effect of sample preparation method on UCS and stiffness</i>	230
5.3.2	<i>Effect of calcite content on post-treatment strength and stiffness</i>	232
5.3.3	<i>Effect of curing time and method on post-treatment UCS and stiffness</i>	235
5.3.4	<i>Effect of quantity of solution applied on UCS and stiffness</i>	239
5.3.5	<i>Effect of clay content on UCS and stiffness at different amounts of solution</i>	241
5.4	MITIGATING COLLAPSE POTENTIAL	245
5.4.1	<i>Concluding discussion</i>	249
5.5	CHAPTER SUMMARY	250
<b>CHAPTER 6</b>	<b>GENERAL DISCUSSION</b>	<b>252</b>
6.1	INTRODUCTION	252
6.2	MANUFACTURE OF COLLAPSIBLE/METASTABLE SAMPLES	252
6.3	MECHANISM OF COLLAPSE AND RELATIONSHIP WITH STRENGTH	256
6.3.1	<i>Material property and composition</i>	256
6.3.2	<i>Placement conditions</i>	258
6.3.3	<i>Stress history prior to wetting</i>	259
6.3.4	<i>Relationship between UCS and collapse potential</i>	259
6.4	SODIUM SILICATE IMPROVEMENT FOR EARTHWORKS	261
6.5	IMPLICATION FOR SCALE UP AND APPLICATION IN PRACTICE	261
6.5.1	<i>Scale up</i>	261
6.5.2	<i>Quantifying the amount of silicate for treatment</i>	262
6.6	SUMMARY	263
<b>CHAPTER 7</b>	<b>CONCLUSIONS AND FURTHER RESEARCH</b>	<b>264</b>
7.1	INTRODUCTION	264
7.2	CONCLUSIONS	264
7.2.1	<i>Key characteristic features of collapsible soils and their improvement (objective 1)</i>	264

7.2.2 Development and evaluation of an effective system of production of artificial collapsible calcareous soils (objectives 2 and 3) .....	265
7.2.3 Characteristics of manufactured samples (objectives 4 and 5).....	265
7.2.4 Relationship between UCS and collapse potential (objective 6).....	267
7.2.5 Evaluation of the impact of wetting fluid pH (objective 7) .....	267
7.2.6 Potential of sodium silicate treatment (objective 8).....	268
7.3 FURTHER RESEARCH .....	269
<b>REFERENCES</b> .....	<b>272</b>
<b>APPENDICES</b> .....	<b>299</b>

## List of Figures

Figure 2.1 Main Classes of Collapsible Soils (modified after Rogers (1995)) .....	11
Figure 2.2 The R-size diagram: bond/weight ratio versus nominal diameter of soil particles (after Jefferson and Smalley (1995)) .....	16
Figure 2.3 Distribution of collapsible soils, Kriger, 1986 (adopted from Assallay (1998)) .....	17
Figure 2.4 Capillary suction bonding and clay bonding in collapsible soils suggested by Barden et al. (1973) .....	20
Figure 2.5 Mechanisms of collapse (redrawn after Feda (1995b)) .....	23
Figure 2.6 Collapse caused by wetting or stressing (after Rogers (1995)) .....	23
Figure 2.7 Stress state surface concept, $e$ vs. $(\sigma - u_w)$ vs. $(u_a - u_w)$ (adopted from Delage et al. (2008)) .....	24
Figure 2.8 Oedometer compression on air-dry and saturated silt showing the effect of soaking (Burland, 1965) .....	25
Figure 2.9 Grain column analogy (Burland and Ridley, 1996).....	26
Figure 2.10 Microstructure model of a collapsible loess deposit as suggested by Tan 1988 (after Assallay (1998)).....	27
Figure 2.11 Clean edge-to-edge fabric in angular coarse silt Malan loess (Derbyshire and Mellors, 1988) .....	27
Figure 2.12 Effect of clay content on collapse potential (adopted from Lawton).....	28
Figure 2.13 Clay connector under scanning electron microscope <b>a</b> , UK loess from Kent showing clay bridge (adopted from Derbyshire and Mellors (1988)) <b>b</b> , clay buttress in young polish loess in Grabowska Olszweska 1989 <b>c</b> , loess at 140km North of Paris, in Delage et al. 2006 (adopted from Langroudi (2014) <b>d</b> , clay bridge in loess (adopted from Jackson et al. (2006)).....	29
<i>Figure 2.14 micrographs of collapsible soils with clay bonding a, bentonite compacted dry of Proctor optimum water content b, high clay content of the Tucson alluvial clay c, high concentration of clay in Ford brickearth (after Barden et al. (1973)) .....</i>	30
Figure 2.15 Scanning electron micrographs <b>a</b> , skeletal microstructure of typical loess. Locality Mnesice brick plant near Nove Mesto nad Vahom <b>b</b> , microstructure of loess like sediment from Hajnacka locality. The skeletal microstructure is formed mainly by sand grains <b>c</b> , skeletal-matrix microstructure of clayey loess from Safarikovo locality <b>d</b> , the honey-comb type of microstructure of loess-like sediments from locality Krcava (12m) <b>e</b> , the matrix type of microstructure of loess-like sediment from locality Orechova (8m) (adopted from Klukanova and Frankovska (1995)).....	31
Figure 2.16 <b>a</b> , SEM photomicrograph showing fine needle calcite (x) forming a meniscus film bridging between adjacent silt grains (2.39 m depth). <b>b</b> , SEM photomicrograph showing a meshwork of fine needle calcite forming a meniscus film (x) bridging between adjacent silt grains and forming a	

‘scaffold’ to support clay particles (2.39 m depth). <b>c</b> , SEM photomicrograph of typical macroporous pelletised brickearth fabric, comprising pellets of compact silt grains (with interstitial clay) in an open-packed arrangement. Calcareous brickearth, 2.39 m, Ospringe Pit 1 (adopted from Milodowski et al. (2015)).....	32
Figure 2.17 CaCO <sub>3</sub> nucleation on kaolinite in electron microscope image <b>a</b> , carbonate nodules absorbed by the clay flake surface and overgrowth of carbonate tubular next to the clay laminated aggregate <b>b</b> , carbonates bonding with the clay platelets (adopted from Langroudi (2014)).....	33
Figure 2.18 Carbonate connectors under scanning electron microscope <b>a</b> , authigenic calcite in Melan loess, China (adopted from Derbyshire and Mellors (1988) <b>b&amp;c</b> , pedogenic carbonates in Saskatchewan soils, Canada (adopted from Landi et al. (2003)). .....	33
Figure 2.19 SEM of undisturbed Malan loess, (a) vertical face illustrating the characteristic fabric of Upper Pleistocene loess from the Lanzhou region, western Loess Plateau of north China. The fabric consists of a loosely packed quartz silt skeleton, mainly in face-to-face contact, with only minor amounts of clay mineral and some secondary carbonate coatings on some of the larger grains. (b) showing macropores (1.2 void ratio) with abundant carbonate cementation and the absence of supporting clay matrix (after Derbyshire (2001) and (1995), respectively).....	33
Figure 2.20 Structural bonds transformation in loess under water saturation (redrawn after Osipov and Sokolov (1995)). .....	36
Figure 2.21 Stable and Metastable (collapsible) soil (after Gibbs and Bara 1962) (Assallay, 1998) ...	41
Figure 2.22 (a) Schematic of in-situ collapse testing system using soil boxes on a concrete pad (b) relationship between partial wetting, degree of saturation and matric suction for three silty soils (adopted from Houston et al 2001) .....	44
Figure 2.23 Predicting collapse using single and double oedometer procedures (after Lutenegeger and Hallberg (1988)).....	47
Figure 2.24 Nomograph for determining collapse potential (after Basma and Tuncer (1992)) .....	49
Figure 2.25 Proposed classification for engineering purposes (a), pure carbonate sediments (b), impure carbonate, sandstone and clay sedimentary rocks (adapted from Fookes and Higginbottom (1975))..	55
Figure 2.26 classification of carbonate sediments (adopted from Clark and Walker (1977)).....	55
Figure 2.27 classification of carbonate sediments (90-100% carbonate) (adopted from Beringen et al. (1982)).....	56
Figure 2.28 Proposed system of description (adopted from Datta et al. (1982)) .....	58
Figure 2.29 Variation of compressive index with carbonate content (adopted from Bryant et al. (1974)) .....	63
Figure 2.30 Comparing the impact of different nanomaterials on the collapse potential of Iranian soils (adopted from Iranpour and haddad (2016)). .....	78
Figure 2.31 A ternary of commercial and non-commercial sodium silicate composition (redrawn after Weldes and Lange (1969)). .....	81

<i>Figure 2.32 Viscosities of Na<sub>2</sub>SiO<sub>3</sub> solutions (a) at various weight ratios and Na<sub>2</sub>O percent (b) as a function of temperature (adopted from PQ Corporation 2004 online publication (accessed 14/5/2019)</i>	84
<i>Figure 2.33 Viscosities of Na<sub>2</sub>SiO<sub>3</sub> solutions (a) as a function of density (b) as a function of weight ratio at constant solids (adopted from PQ Corporation 2004 online publication (accessed 14/5/2019)</i>	84
Figure 2.34 polymerization and gelling of silica monomers in (A) alkaline solution, and (B) in acid solution (adopted from Iler (1979))	85
Figure 3.1 Stages of the laboratory methodological framework of the study	97
Figure 3.2 Reaction apparatus for sample carbonation <b>a</b> , empty setup <b>b</b> , stacked with oedometric specimens	101
Figure 3.3 Cylindrical specimens undergoing carbonation	102
Figure 3.4 Colour observation by spraying phenolphthalein solution after carbonation	104
Figure 3.5 Varying carbonation (reaction) periods and conversion level of a clay-lime system prepared differently	105
<i>Figure 3.6 Carbonation success determined by the experimental (Leco) yield and calculated mass gain (a) the effect of initial dry density (b) effect of initial moisture content, before carbonation.</i>	106
Figure 3.7 Particle size distribution of Kaolin (data by supplier)	109
<i>Figure 3.8 Particle size distribution of the Sub 63 microns of the Lanton Alluvium</i>	111
Figure 3.9 Summary of main samples production procedures	113
Figure 3.10 Moisture content – Dry density relationship of primary soils with varying lime contents	117
<i>Figure 3.11 Thermogravimetric and differential scanning calorimetric analysis of samples A1, A2, and A3</i>	118
<i>Figure 3.12 Thermogravimetric and differential scanning calorimetric analysis of samples C1, C2, and C3</i>	119
Figure 3.13 Carbonation success level as related to the theoretical expected CaCO <sub>3</sub> yield (i) moderately calcareous (ii) strongly calcareous (iii) very strongly calcareous samples (iv) percent lime-to-calcium carbonate conversion levels	120
Figure 3.14 Relationship between amount of lime carbonated and moisture retained	123
Figure 3.15 carbonation progressing from compact surface towards core	124
Figure 3.16 Typical mineral composition of initial samples without lime/carbonate treatment, identified by X-ray diffraction analysis	126
Figure 3.17 X-Ray diffraction analysis of manufactured samples (i) initially constituted primary soils (ii) moderately calcareous (iii) strongly calcareous and (iv) very strongly calcareous samples	128
Figure 3.18 SEM images of the individual components of manufactured samples (a) Kaolinite (Polwhite E) – shows clay flakes in a parallel arrangement (b) Lantan alluvium (sub 63 μm) – image	

show a few clay flakes surrounding angular silt-size quartz (c) carbonated hydrated lime – calcium carbonate globules are formed covering entire surface.....	130
Figure 3.19 <b>a</b> and <b>b</b> - SEM micrographs of manufactured samples and their microporosity analysis using PCAS. Black areas represent pores in the PCAS images .....	132
Figure 3.20 SEM micrographs showing EDX mapping.....	133
Figure 3.21 conventional oedometer equipment <b>a</b> , specimen and pot components <b>b</b> , assembled pot with specimen <b>c</b> , setup of test running <b>d</b> , data acquisition system.....	136
Figure 3.22 Instron 5585H series floor model testing systems showing major components (Newcastle University Geotechnical Labs).....	141
Figure 3.23 Estimation of Young's Modulus of elasticity (stiffness).....	142
Figure 4.1 Stress-strain curves of non-calcareous specimens under unconfined compressive stress .	147
Figure 4.2 Failure modes of non-calcareous specimens under unconfined compressive stress .....	147
Figure 4.3 Stress-strain curves of “moderately calcareous” specimens under unconfined compressive stress.....	149
Figure 4.4 Failure modes of “moderately calcareous” specimens under unconfined compressive stress .....	149
Figure 4.5 Stress-strain curves of “strongly calcareous” specimens under unconfined compressive stress.....	149
Figure 4.6 Failure modes of “strongly calcareous” specimens under unconfined compressive stress	149
Figure 4.7 Stress-strain curves of “very strongly calcareous” specimens under unconfined compressive stress.....	150
Figure 4.8 Failure modes of “very strongly calcareous” specimens under unconfined compressive stress.....	150
Figure 4.9 The impact of calcite cementation on UCS and stiffness of low density silty-clays .....	152
Figure 4.10 Relationship between UCS and Stiffness .....	152
Figure 4.11 Effect of CaCO <sub>3</sub> content (chemically precipitated from 3 different sources) on the UCS of sand (plotted from Park et al. (2014) data).....	153
<i>Figure 4.12 Effect of microbially precipitate CaCO<sub>3</sub> content on confined compressive strength (■) and residual strength (□) of sand at 50kPa confining pressure (adopted from Whiffin et al. (2007)) .....</i>	<i>154</i>
<i>Figure 4.13 Dry density relationship with compressive strength.....</i>	<i>154</i>
Figure 4.14 Void ratio relationship with compressive strength .....	155
Figure 4.15 Void ratio relationship with carbonate content .....	155
<i>Figure 4.16 The impact of calcite cementation on strain at failure of low density silty-clays.....</i>	<i>156</i>
Figure 4.17 Relationship between strain and compressive strength.....	156
Figure 4.18 Effect of clay content on the UCS of calcite cemented silty-clays.....	157
Figure 4.19 Effect of clay content on the stiffness of calcite cemented silty-clays. ....	157
Figure 4.20 Effect of clay/silt content on strength (adopted after Teerawattanasuk and Voottipruex (2014)).....	157

Figure 4.21 Single oedometer deformation curves of different uncemented non-calcareous silty-clay samples with two wetting fluids.....	160
Figure 4.22 Double oedometer curves of different uncemented non-calcareous silty-clay samples with two wetting fluids.....	160
Figure 4.23 Single oedometer test results of different low dry-density cemented “moderately calcareous” samples .....	165
Figure 4.24 Double oedometer test results of different low dry-density cemented “moderately calcareous” samples .....	165
Figure 4.25 Single oedometer test results of different low dry-density cemented “strongly calcareous” samples.....	166
Figure 4.26 Double oedometer test results of different low dry-density cemented “strongly calcareous” samples.....	166
Figure 4.27 Single oedometer curves of different low dry-density cemented “very strongly calcareous” samples.....	167
Figure 4.28 Double oedometer curves of different low dry-density cemented “very strongly calcareous” samples .....	167
Figure 4.29 Single oedometer curves of cemented “moderately calcareous” silty-clay samples compacted to their maximum dry densities.....	170
Figure 4.30 Double oedometer curves of cemented “moderately calcareous” silty-clay samples compacted to their maximum dry densities.....	171
Figure 4.31 Single oedometer curves of cemented “strongly calcareous” silty-clay samples compacted to their maximum dry densities.....	171
Figure 4.32 Double oedometer curves of cemented “strongly calcareous” silty-clay samples compacted to their maximum dry densities.....	172
<i>Figure 4.33 Single oedometer curves of cemented “very strongly calcareous” silty-clay sample compacted to their maximum dry densities .....</i>	<i>172</i>
Figure 4.34 Single oedometer curves of cemented “moderately calcareous” silty-clay samples compacted at half their optimum moisture content .....	175
Figure 4.35 Double oedometer curves of cemented “moderately calcareous” silty-clay samples compacted at half their optimum moisture content.....	175
Figure 4.36 Single oedometer curves of cemented “strongly calcareous” silty-clay samples compacted at half their optimum moisture content .....	176
Figure 4.37 Double oedometer curves of cemented “strongly calcareous” silty-clay samples compacted at half their optimum moisture content .....	176
Figure 4.38 Single oedometer curves of cemented “very strongly calcareous” silty-clay samples compacted at half their optimum moisture content.....	177
Figure 4.39 Double oedometer curves of cemented “very strongly calcareous” silty-clay samples compacted at half their optimum moisture content.....	177

Figure 4.40 Collapsibility of cemented sample A1 at different wetting stresses under acid solution wetted single & double oedometer.....	180
Figure 4.41 Collapsibility of cemented sample C1 at different wetting stresses under acid solution wetted single & double oedometer.....	181
Figure 4.42 Collapsibility of cemented sample A2 at different wetting stresses under distilled water wetted single & double oedometer.....	181
Figure 4.43 Collapsibility of cemented sample A2 at different wetting stresses under acid solution wetted single & double oedometer.....	182
Figure 4.44 Collapsibility of cemented sample C2 at different wetting stresses under distilled water wetted single & double oedometer.....	182
Figure 4.45 Collapsibility of cemented sample C2 at different wetting stresses under acid solution wetted single & double oedometer.....	183
Figure 4.46 Single oedometer collapsibility curves of uncemented sample A2 with different wetting fluids.....	185
Figure 4.47 Double oedometer collapsibility curves of uncemented sample A2 with acid solution presoaked.....	185
Figure 4.48 Single oedometer collapsibility curves of uncemented sample C2 with different wetting fluids.....	186
Figure 4.49 Double oedometer collapsibility curves of uncemented sample C2 with acid solution presoaked.....	186
Figure 4.50 Single oedometer collapsibility curves of uncemented sample C3 with different wetting fluids.....	187
Figure 4.51 Relationship between calcite content and collapse potential of series A samples (low-density) under different wetting fluids.....	189
Figure 4.52 Relationship between calcite content and collapse potential of series B samples (low-density) under different wetting fluids.....	189
Figure 4.53 Relationship between calcite content and collapse potential of series C samples (low-density) under different wetting fluids.....	189
Figure 4.54 Extrapolating the impact of calcite content on the collapse potential of all low-density samples (series A, B & C).....	190
Figure 4.55 Influence of cementation and noncementation on the collapse potential of calcareous silty-clay soils.....	192
Figure 4.56 Effect of dry density on the collapse potential of calcareous silty-clay soils under acid solution wetting.....	193
Figure 4.57 Effect of dry density on the collapse potential of calcareous silty-clay soils under acid solution wetting.....	193
Figure 4.58 Effect of clay content on the CP of non-calcareous silty-clay soils with different wetting fluids.....	195

Figure 4.59 Thresholds of collapse potential based on clay content of different collapsible soils (Modified from Langroudi (2014)). M=moderately, S=strongly, VS=very strongly, and C= calcareous. .....	196
Figure 4.60 Effect of clay content and collapse potential (adopted from Basma and Tuncer (1992))	198
Figure 4.61 Effect of clay content on the CP of moderately calcareous silty-clay soils with different wetting fluids.....	199
Figure 4.62 Effect of clay content on the CP of strongly calcareous silty-clay soils with different wetting fluids.....	199
Figure 4.63 Effect of clay content on the CP of very-strongly calcareous silty-clay soils with different wetting fluids.....	199
Figure 4.64 Time rate of collapse of non-calcareous silty-clay soils under different wetting fluid conditions .....	201
Figure 4.65 Time rate of collapse of moderately calcareous silty-clay soils under different wetting fluid conditions.....	201
Figure 4.66 Time rate of collapse of strongly calcareous silty-clay soils under different wetting fluid conditions .....	201
Figure 4.67 Time rate of collapse of very strongly calcareous silty-clay soils under different wetting fluid conditions.....	201
Figure 4.68 Impact of overburden stress at wetting on the time rate of collapse of a moderately calcareous silty-clay soil under acidic solution wetting condition.....	204
Figure 4.69 Impact of overburden stress at wetting on the time rate of collapse of a strongly calcareous silty-clay soil under acidic solution wetting condition.....	204
Figure 4.70 The influence of overburden stress and testing technique on the collapse potential of different calcareous silty-clay soils under acidic solution wetting condition.....	205
Figure 4.71 The influence of overburden stress and testing technique on the collapse potential of different calcareous silty-clay soils under distilled water wetting condition .....	205
Figure 5.1 Solution absorption in oedometer specimens: a, b and c are different stages of the absorption process .....	213
Figure 5.2 Solution absorption in compressive test specimens (a): b, c and d are monitoring stages of the absorption progress.....	213
Figure 5.3 Observed specimens surface consistency after treatment (impregnation by absorption), setting and 2 days oven drying at 45 -50 °C.....	214
Figure 5.4 SEM Micrographs of specimens treated with sodium silicate solution (Si/Na=3, M=4) and oven dried.....	217
<i>Figure 5.5 Stress-strain relationship of moderately calcareous samples impregnated with Si/Na=3, M=4 solution, cured under room ambience for 7, 14 and 28 days, or oven dried 48 hrs under 45-50 °C</i> .....	219

<i>Figure 5.6 Stress-strain behaviour of strongly calcareous samples impregnated with Si/Na=3, M=4 solution, cured under room ambience for 7, 14 and 28 days, or oven dried 48 hrs under 45-50 °C..</i>	220
<i>Figure 5.7 Stress-strain behaviour of very strongly calcareous samples impregnated with Si/Na=3, M=3.5 solution, cured under room ambience for 7, 14 and 28 days, or oven dried 48 hrs under 45-50 °C.....</i>	220
Figure 5.8 Relationship between moisture retained after curing and UCS .....	223
Figure 5.9 Stress-strain relationship of different moderately calcareous samples mixed with varying proportions of a Na <sub>2</sub> SiO <sub>3</sub> solution and cured 7 days .....	225
Figure 5.10 Stress-strain relationship of different moderately calcareous samples mixed with varying proportions of a Na <sub>2</sub> SiO <sub>3</sub> solution and cured 14 days .....	225
<i>Figure 5.11 Stress-strain relationship of different moderately calcareous samples mixed with varying proportions of a Na<sub>2</sub>SiO<sub>3</sub> solution and cured 28 days .....</i>	226
<i>Figure 5.12 Stress-strain relationship of a non-calcareous and moderately calcareous samples mixed with two different proportions of Na<sub>2</sub>SiO<sub>3</sub> solution and cured 14 and 28 days .....</i>	226
Figure 5.13 Relationship between amount of treatment solution and retained moisture content after curing.....	227
Figure 5.14 Relationship between retained moisture content after curing and UCS .....	228
Figure 5.15 The influence of sodium silicate treatment method on unconfined compressive strength .....	231
Figure 5.16 The influence of sodium silicate treatment method on stiffness.....	231
Figure 5.17 Impact of calcite presence on UCS of samples mixed with 30% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days.....	233
Figure 5.18 Impact of calcite presence on UCS of samples mixed with 40% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days.....	233
Figure 5.19 Impact of calcite presence on the stiffness of samples mixed with 30% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days.....	233
Figure 5.20 Impact of calcite presence on the stiffness of samples mixed with 40% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days.....	233
Figure 5.21 Impact of calcite content on the effectiveness of Na <sub>2</sub> SiO <sub>3</sub> treatment (impregnation) of samples.....	234
Figure 5.22 Impact of calcite content on the effectiveness of Na <sub>2</sub> SiO <sub>3</sub> treatment (impregnation) of samples.....	234
Figure 5.23 Impact of calcite content on the effectiveness of Na <sub>2</sub> SiO <sub>3</sub> treatment (impregnation) of samples.....	234
Figure 5.24 Effect of curing method on the UCS of sodium silicate impregnated calcareous silty-clay samples.....	236
Figure 5.25 Effect of curing method on the stiffness of sodium silicate impregnated calcareous silty-clay samples .....	236

Figure 5.26 Effect of curing time on UCS and stiffness of sample A1 .....	237
Figure 5.27 Effect of curing time on UCS and stiffness of sample B1 .....	237
Figure 5.28 Effect of curing time on UCS and stiffness of sample C1 .....	237
Figure 5.29 Relationship between % mass of solution (by mass of dry soil) applied and UCS .....	240
Figure 5.30 Relationship between % mass of solution (by mass of dry soil) applied and stiffness....	240
Figure 5.31 Impact of clay/silt ratio, and presence of carbonate in soil on UCS of samples mixed with 30% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days .....	242
Figure 5.32 Impact of clay/silt ratio, and presence of carbonate in soil on UCS of samples mixed with 40% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days .....	242
Figure 5.33 Impact of clay/silt ratio on UCS of calcareous samples mixed with 50% sodium silicate solution and cured for several days .....	242
Figure 5.34 Impact of clay/silt ratio on the stiffness of calcareous samples mixed with 30% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days .....	243
Figure 5.35 Impact of clay/silt ratio on the stiffness of calcareous samples mixed with 40% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days .....	243
Figure 5.36 Impact of clay/silt ratio on the stiffness of calcareous samples mixed with 50% Na <sub>2</sub> SiO <sub>3</sub> solution and cured for several days .....	243
Figure 5.37 Single oedometer test results of Na <sub>2</sub> SiO <sub>3</sub> treated A1, low dry-density cemented “moderately calcareous” sample .....	246
Figure 5.38 Double oedometer test results of Na <sub>2</sub> SiO <sub>3</sub> treated A1, low dry-density cemented “moderately calcareous” sample .....	246
Figure 5.39 Single oedometer test results of Na <sub>2</sub> SiO <sub>3</sub> treated B1, low dry-density cemented “moderately calcareous” sample .....	247
Figure 5.40 Double oedometer test results of Na <sub>2</sub> SiO <sub>3</sub> treated B1, low dry-density cemented “moderately calcareous” sample .....	247
Figure 5.41 Single oedometer test results of sodium silicate treated C1, low dry-density cemented “moderately calcareous” sample .....	247
Figure 5.42 Double oedometer test results of sodium silicate treated C1, low dry-density cemented “moderately calcareous” sample .....	247
<i>Figure 5.43 Calcareous silty-clay collapse mitigating impact of a sodium silicate solution tested under acidic solution and distilled water wetting conditions of the single oedometer.....</i>	<i>249</i>
Figure 5.44 Calcareous silty-clay collapse mitigating impact of a sodium silicate solution tested under distilled water presoaked (double oedometer).....	249
Figure 6.1 Degree of calcite cementation versus UCS and CP under acidic solution wetted condition .....	260
Figure 6.2 Degree of calcite cementation versus UCS and CP under distilled water wetted condition .....	260

## List of Tables

Table 1.1 Some examples of structural instability caused by collapsible soils.....	6
Table 2.1 Different descriptions of soil collapsibility as in Assallay (1998) .....	18
Table 2.2 Classification of loess bonding types by Yang 1989 (adapted from El Howayek et al. (2011)) .....	35
Table 2.3 classification of structural bonds in loess (adapted from Osipov and Sokolov (1995))......	36
Table 2.4 Comparison of collapse sequences according to Osipov and Sokolov (1995), and Klukanova and Frankovska (1995).....	37
Table 2.5 Characteristics of some Italian calcareous clays (redrawn after Jamiolkowski et al. (1995))	52
Table 2.6 Degree to which calcareous soils meet characteristics of collapsible soils.....	60
Table 2.7 Consolidation undrained triaxial test results (adapted from Demars et al. (1976)).....	61
Table 2.8 Effect of grain size on thermodynamic equilibrium constant for calcite at 25 <sup>0</sup> C (after Chave and Scmalz 1966 (Al-Kaysi, 1983)) .....	67
Table 2.9 silicates with greater alkalinity than SiO <sub>2</sub> :Na <sub>2</sub> O = 1.6 (after Hurley and Thornburn (1972))	82
<i>Table 3.1 Properties of Polwhite E English China clay as supplied by Imerys Performance Minerals ltd, UK</i> .....	108
<i>Table 3.2 Index properties of the sub-63 μm of the Lanton Alluvium</i> .....	111
Table 3.3 Properties of the hydrated lime as presented by Lafarge Tarmac, 2016. ....	112
Table 3.4 designed soil samples composition: degree of calcareousness defined according to Day’s 1983 classification of calcareous soils, cited in Kishchuk (2000).....	114
Table 3.5 Index properties of Primary Soils with varying proportions of lime (uncarbonated) .....	114
Table 3.6 Summary of the degree of lime-to-carbonation conversion as evaluated by the different methods .....	121
Table 3.7 X-Ray diffraction angles of minerals as adapted from the literature .....	125
Table 3.8 Chemical composition of manufacture samples obtained by EDX mapping .....	133
<i>Table 3.9 Collapse percentage as an indication of severity (from Jennings and Knight, 1975 modified after Bell and Culshaw (2001))</i> .....	138
Table 4.1 Physical properties of cylindrical compressive test specimens.....	146
Table 4.2 Mechanical properties of the calcareous silty-clay samples under UCT .....	150
Table 4.3 Physical properties of uncemented non-calcareous test specimens. ....	159
Table 4.4 Collapse potential and degree of severity of uncemented non-calcareous silty-clay samples .....	161

Table 4.5 Physical properties of cemented calcareous oedometric test specimens with low dry densities.....	164
Table 4.6 Specimen compressibility during loading, collapse potentials and degree of severity of low dry-density cemented calcareous samples.....	164
Table 4.7 Physical properties of well-compacted, cemented calcareous oedometric test specimens .	170
Table 4.8 Specimen compressibility during loading, collapse potential at different wetting stresses and degree of severity of well-compacted and cemented calcareous samples.....	173
Table 4.9 Physical properties of poorly compacted (standard Proctor at half OMC), cemented calcareous oedometric test specimens.....	174
Table 4.10 Specimen compressibility during loading, collapse potential at different wetting stresses and degree of severity of poorly compacted cemented calcareous samples .....	178
Table 4.11 Collapse potential at different vertical stress levels under single oedometer.....	179
Table 4.12 collapse potentials at different vertical stresses under double oedometer.....	180
Table 4.13 Specimen compressibility during loading, collapse potential at 300 kPa wetting stress and degree of severity of poorly compacted cemented calcareous samples .....	184
Table 5.1 Sodium silicate solutions formulated and examined for suitability at pilot level (treatment by impregnation).....	211
Table 5.2 Mean percent-absorbed treatment solution (Si/Na = 3, M = 4 (*M=3.5)) for different sets of specimens and their curing time.....	219
Table 5.3 Mechanical properties of the calcareous silty-clay samples under unconfined compressive strength testing .....	221
Table 5.4 Mechanical properties of the treated (by hand mixing) calcareous silty-clay samples under unconfined compressive testing .....	229
Table 5.5 Properties of sodium silicate treated oedometer samples (moderately calcareous) .....	245
Table 5.6 Treated specimens - compressibility during loading, collapse potentials and degree of severity of low dry-density cemented calcareous samples.....	248
<i>Table 6.1 (a &amp; b) Comparable microstructures of SEM images of some present samples and collapsible soils found in the literature.....</i>	<i>253</i>

## Glossary of Terms and Abbreviation

<i>Term or Abbreviation</i>	<i>Definition</i>
CaCl <sub>2</sub>	Calcium chloride
CaCO <sub>3</sub>	Calcium carbonate
CaO	Calcium oxide
Ca(OH) <sub>2</sub>	Calcium hydroxide
Calcareousness	A measure of calcium carbonate content
<i>Non</i>	Zero percent calcium carbonate
<i>Moderately</i>	Approximately twelve percent calcium carbonate
<i>Strongly</i>	Approximately twenty-five percent calcium carbonate
<i>Very strongly</i>	Approximately forty percent calcium carbonate
CaSiO <sub>3</sub>	Calcium silicate
CMG	Calculated mass gain
CO <sub>2</sub>	Carbon dioxide
CP	Collapse potential (measured in percentage height decrease upon wetting)
<i>NT</i>	No trouble
<i>MT</i>	Moderate trouble
<i>T</i>	Trouble
<i>ST</i>	Serious trouble
<i>VST</i>	Very serious trouble
CT	Compressive test
DOT	Double oedometer test
EY	Experimental yield
Leco equipment	Technique for determining organic and total carbon
LEY	Leco experimental yield
m <sub>v</sub>	Coefficient of volume compressibility
Na <sub>2</sub> CO <sub>3</sub>	Sodium carbonate
Na <sub>2</sub> O	Sodium oxide
NaOH	Sodium hydroxide
Na <sub>2</sub> SiO <sub>3</sub>	Sodium silicate

OC	Organic carbon
OPC	Ordinary Portland cement
PCAS	Particles (Pores) and Cracks Analysis System
Polymerization	The process of molecules linking to form a chain (i.e. gel formation)
SiO <sub>2</sub>	Silica
Si/Na	Silica to sodium oxide ratio
Silicatization	The application of sodium silicate in soil improvement
Single-step treatment	Application of a single solution for soil improvement
SOT	Single oedometer test
STA	Simultaneous thermal analysis
TeY	Theoretical expected yield
TC	Total carbon
TG/DSC	Thermogravimetry and diffraction scanning calorimeter
TGAEY	Thermogravimetric analysis - experimental yield
UCS	Unconfined compressive strength
UCT	Unconfined compressive test
USCS	Unified soil classification system

# Chapter 1 Introduction

## 1.1 General Background

In geotechnical engineering, several soils are considered problematic because, they either disperse, expand, collapse, lack strength, undergo excessive settlement, or contain soluble minerals. These characteristics are attributable to their origin and mode of formation, their composition, their mineralogy, the nature of their pore fluids, or their fabric (Briscolland and Chown (2001); Rezaei *et al.* (2012)). Some of the most problematic geomaterials are the moisture sensitive ones, e.g. expansive clays and collapsible soils (Houston *et al.* (2001); Rezaei *et al.* (2012); Vandanapu *et al.* (2016)). The present study focuses mainly on collapsible soils, which are soils with an open metastable structure, whose structure changes (densifies) as a result of water intake with/without additional loading.

Collapsible soils are extremely common globally, presenting structural and geotechnical engineering problems. They form through both natural processes and human activity, and in either cases an open metastable structure (supported by inter-particle bonding) is the main prerequisite to collapse (Dudley, 1970 in Jefferson and Rogers (2012)). However, they are known to be stiff and exhibit high strength in their unsaturated state (Rogers *et al.*, 1994). Huge loses (economic and lives) have resulted from collapsible soils ((Derbyshire (2001); Houston *et al.* (2001); Sun *et al.* (2013); Watts and Charles (2015)) (see section 1.1.4). Therefore, for a geotechnical engineer to be able to prevent such loses, by way of providing economic and efficient engineering solutions, four steps are recommended (Popescu, 1986) to follow: (i) identification – determine the existence of a collapsible soil; (ii) classification – if a collapsible soil exists, what is the significance? (iii) quantification – what is the degree of collapse that will occur? (iv) evaluation – what are the design options? However, Houston *et al.* (2001) recognised that the greatest problems with collapsible soils result from not recognizing the existence and extent of their collapse potential prior to construction.

Collapse is usually triggered either naturally through flooding/groundwater rise and/or loading (e.g. earthquakes), or artificially by human activity. Human activity can be either unintentional via poor drainage control (e.g. broken water or sewer lines, landscape irrigation, poor surface drainage, roof runoff, etc.), or deliberate through ground improvement, e.g. dynamic compaction (Houston *et al.* (2001); Jefferson and Rogers (2012)).

There are several collapse mitigating measures (see Sections 2.9 and 2.10) with each however, presenting some form of limitations. Interestingly, many of the collapse improvement

strategies available have aimed at merely densification and/or strengthening of the soil (Jefferson *et al.* (2005); Iranpour and haddad (2016)). Although these processes can reduce the flow of water into the soil, they are not adequate to prevent bond dissolution from occurring and consequently causing collapse.

### **1.1.1 Strength and stiffness of collapsible soils**

The open metastable structural framework of collapsible soils is supported by cementing forces existing at particle contacts. These forces are usually high enough making collapsible soils strong and stiff in their unsaturated state (Rogers *et al.*, 1994). It is assumed that the stronger the soil the more stable it is, but when net stresses (through wetting or loading) exceed the yield strength collapse occurs (Jefferson and Rogers, 2012).

### **1.1.2 Collapsibility and forms of collapsible soils**

Unlike consolidation where the reduction in volume (or void ratio) is the consequence of time-dependent discharge of excess pore water, the reduction in volume for a collapsing soil is rapid and the result of water ingress into the soil and/or bond breaking caused by increased stresses (Jefferson *et al.*, 2005). Collapsible soils are usually capable of sustaining substantial high-applied vertical stress in their in situ conditions without significant volume change but when wetted, undergo rapid and large reduction in volume (Barden *et al.* (1973); Alan and Robert (1988); Lawton *et al.* (1989); Rogers (1995); Assallay (1998)). The water ingress into the soil is believed to destroy existing inter-particle cement bonds (that effectively hold together a relatively open structure), resulting in densification of the soil through collapse.

Collapsible soils are known to exhibit certain physical and structural features such as an open and potentially metastable structure, an unsaturated state, high void ratio and porosity, low dry density, enough particle-to-particle bonding agent to hold particles together in their unsaturated state (Barden *et al.* (1973); Rogers (1995)). Their mineralogy spans a wide range but particles usually varying between sand and clay sizes. Capillary water, clay minerals, calcium carbonate and other soluble salts have been argued to play a role in the formation of bonding between elements, providing the initial stability in these metastable soils (Rogers *et al.* (1994); (1995); Osipov and Sokolov (1995); El Howayek *et al.* (2011); Jefferson and Rogers (2012); Milodowski *et al.* (2015)). However, wetting, eliminates this bonding and sudden restructuring occurs (Guo, 1958 and Dudley, 1970 in Li *et al.* (2016); Rogers *et al.* (1994); Mansour *et al.* (2008); Peck *et al.* 1974, Clemence and Finbarr 1981 in Ayeldeen *et al.* (2017)).

Both artificially formed (compacted sands, silts and clays) and naturally occurring (loessial, alluvial, and residual) soils have been used in the study of collapsible soils. In natural collapsible soils CaCO<sub>3</sub> seems to dominate the literature as the principal bonding element (Guo, 1958 in Li *et al.* (2016); Demars and Chaney (1982); Feda (1995b); Lamas *et al.* (2002); Mansour *et al.* (2008); Milodowski *et al.* (2015)). Loess, which is the most widely studied of the natural collapsible soils contains CaCO<sub>3</sub> contents ranging between 1 and 30 % (Jefferson *et al.* (2005); Li *et al.* (2016)). Such CaCO<sub>3</sub> contents qualify loess soils as calcareous soils according to the definition of Thompson (2007); Kalra and Maynard, 1991 and Day, 1983 cited in Kishchuk (2000). Day, 1983 define calcareous soils as any soil containing a significant proportion of CaCO<sub>3</sub>, grouping them into *weakly* (<5%), *moderately* (5–15%), *strongly* (15–25%), *very strongly* (25–40%) and *extremely* (>40%) calcareous soils, respectively.

Based on this definition, calcareous (also known as carbonate) soils span a large range of soils widely distributed globally with the arid and semi-arid climates favouring more of their production (Houston *et al.*, 2001). For example, there is extensive occurrence of calcareous soils (mostly clays) in the majority of countries of the Near East (or Middle East) Region (Kadry (1973);). Yong and Ouhadi (2007) cited the works of Guillet 1969; Turner and Seago 1985; Ouhadi 1997; Wust and McLane 2000; Seijmonsbergen *et al.* 2005; Eberhardt *et al.* 2005; and Mathys *et al.* 2005, reporting wetted-state unstable marls occurring in Austria, Canada, U.S., U.K., France, Iran, Switzerland, and Egypt; and Mansour *et al.* (2008) identified collapsible Dead Sea Lisan Marl deposits in Jordan. Marls can be classified as either *very strongly* or *extremely* calcareous soils because they consist CaCO<sub>3</sub> content between 35 – 65 % (Bellair and Pomerol, 1980 in Lamas *et al.* (2002); Pettijohn (1975)). In the UK, sections of the brickearth deposits of south Essex and Kent are also described as possessing some characteristics of calcareous silty-clays (Milodowski *et al.* (2015); Bell and Culshaw (2001) Jefferson *et al.* (2001)).

In addition to natural soils, carbonation has been reported to occur in soils treated with calcium rich substances such as lime, Portland cement or concrete, precipitating significant amounts of CaCO<sub>3</sub> in those soils. For example, a carbonation study carried out in the arid part of Botswana on a road base stabilised with lime confirmed conversion of lime to calcium carbonate to various degrees (Bagoniza *et al.* (1987)). Many other authors including Netterberg (1984) have confirmed the transformation of lime-treated soils to calcareous/carbonate soils.

A further class of material, that increases the distribution of calcareous soils even further are those soils occurring in sites where concrete demolition has taken place. Studies by Renforth

*et al.* (2009) and Washbourne *et al.* (2012) demonstrate precipitation of secondary (pedogenic) CaCO<sub>3</sub> contents reaching 19.9 and 40 % in such sites.

### 1.1.3 The influence of the carbonate bond on collapsibility

Some scholars have questioned whether Calcium Carbonate can readily dissolve to cause a sudden volume reduction in the context of soil collapsibility (e.g. Smalley (1971); Mellors, 1995 in Milodowski *et al.* (2015)). Some researchers have even described it as unreasonable to consider collapse occurring by CaCO<sub>3</sub> dissolution or leaching. It is possible that this view has arisen because distilled or tap water had been used as the wetting fluid in laboratory collapse testing. In such wetting conditions, the dissolution of CaCO<sub>3</sub> is a slow process, which requires several cycles of wetting and drying and therefore cannot explain the phenomenon of sudden reduction in total volume upon wetting (Guo *et al.*, 2004, 2005, 2008 cited in Li *et al.* (2016)). Instead it leads to a process referred to as chemical piping or leaching-and-collapse (Karakouzian *et al.* 1996 in Mansour *et al.* (2008)).

For a more complete understanding of carbonate dissolution as a trigger of collapse, it is necessary to consider both the fact that calcium carbonate has different polymorphs (calcite, aragonite or vaterite), and that each of these behave differently under different environmental conditions, including the chemistry of the wetting fluid.

According to McLaren (2011); Verrecchia (2011), calcium carbonate minerals are most readily soluble in carbon dioxide rich waters (e.g. acid rains) and in cold dilute hydrochloric acid as shown in Equations 1.1 and 1.2 below. Water droplets in the atmosphere react with compounds such as sulphur dioxide, nitrogen oxide, carbon oxides, etc. emitted to the atmosphere by the burning of fossil fuels, and by motor vehicles, intensive agriculture, and other industrial activities to produce weak acids. These acids, some of which are sulphuric, nitric, and carbonic are precipitated during rainfall and can easily dissolve carbonate minerals. Activities leading to the emission of the foregoing compounds are global, though at different rates in different countries. There is the need therefore, to find solutions to their deleterious effects on reacting with earthworks built on or with carbonate soils.



Notwithstanding the preceding arguments, the extension of cities and construction of new earthwork infrastructure all over the world, as well as the concept of sustainable construction has made it almost inevitable for collapsible geomaterials being utilized as either host materials (Mohamed *et al.* (2008); Watts and Charles (2015)) despite warnings, e.g., from *code of practice for foundations (BS 8004:1986)* (Watts and Charles, 2015). Because of the geotechnical challenges and losses arising from these problematic soils, extensive research works on their mechanical behaviours and methods of mitigating these problems have been carried out over many decades.

However, there is a gap in this literature associated with the relative effect of wetting fluid chemistry on collapse of both carbonate soils and their improved counterparts (for example silicate-treated collapsing soils). Also, the literature largely focusses on mechanical behaviour to the expense of chemical properties in defining geotechnical soils which has the potential to lead to misunderstanding of the comprehensive engineering behaviour of certain soils, e.g. calcareous soils whose  $\text{CaCO}_3$  content may dissolve and affect their mechanical behaviour (Lamas *et al.* (2002); (2011)).

#### **1.1.4 Economic losses from soil collapse**

Soil collapse has contributed to serious damage to infrastructure built with or hosted on either calcareous or non-calcareous collapsible soils. This damage appears as cracks or sometimes complete breaking of infrastructure beyond their serviceability limit leading to economic losses and even loss of lives in some instances (Derbyshire (2001); Houston *et al.* (2001); Sun *et al.* (2013)). Houston *et al.* (2001) noted collapsible soils as being the most problematic geomaterial in the context of geotechnical engineering. Considering reports in the literature (Lawton *et al.* (1992); Houston *et al.* (2001); Houston *et al.* (2001); Jefferson *et al.* (2005)), it is possible that globally, economic losses of the order of several billions of dollars may result from these geomaterials each year. For instance, in 1992 (Lawton *et al.*) estimated over US\$100 million of costs resulting from infrastructural damages and litigations caused by collapses in a few cases they reported on in Southern California. Estimates of damage over recent decades exceeding US\$100 million were recorded to result from collapsible loess in Bulgaria (Minkov, 1993 in Jefferson *et al.* (2005)). A commercial building in New Mexico suffered a foundation damage due to soil collapse after heavy watering, causing about US\$0.5 million losses (Houston *et al.*, 2001). Beside these, there are several unquantified losses caused by soil collapse, e.g., Watts and Charles (2015) reported huge damages to numerous infrastructures founded on different kinds of non-engineered fills (summarized under section 2.2.1).

A few examples of infrastructural damages caused by collapsible soils are listed below in Table 1.1.

*Table 1.1 Some examples of structural instability caused by collapsible soils*

<b>Earthwork</b>	<b>Location</b>	<b>Description</b>	<b>Reference</b>
Earth dam	Idaho, USA	Collapse of Teton dam resulting from a permeable loess used in the core.	Smalley (1992)
Compacted fill in a canyon	Southern California	Failure of buildings constructed on compacted fill.	Lawton <i>et al.</i> (1992)
Highway embankments	South Africa	Deformation, cracking and failure of highway embankments resulting from wetting-induced collapsing.	Booth (1977)
Airport Runways	Heathrow, UK	Evidence provided for a calcareous Aeolian loess layer (Langley silt) near the Heathrow airport.	Rose <i>et al.</i> 2000 and Jefferson <i>et al.</i> 2003, in Langroudi (2014)
Buildings and pavements	New Mexico	Damage caused by differential settlement of a commercial building due to watering nearby landscape.	Houston <i>et al.</i> (2001)
Road Embankment	Near Pentalia, southwest Cyprus	Road embankment failure resulting from inundation/sliding of calcareous subgrade.	Hadjigeorgiou <i>et al.</i> (2006)
Industrial installations	Kazakhstan	Occurrence of calcareous loess deposits in Northwest Kazakhstan, South of the River Ural, Karachaganak gas field.	Roohnavaz <i>et al.</i> 2011, in Langroudi (2014)
Houses, Factory, Estates, General Hospital, and Clifford's Tower	Ilkston, West Aukland, Brighton, North Tyneside, and York, respectively (in the UK)	Founded on different kinds of non-engineered fills mainly opencast coal mining backfills, which collapsed following flooding after many years of placement.	Watts and Charles (2015)

### 1.1.5 Collapse treatment methods

There are well-established methods of treating or improving problematic soils encountered at construction sites. Most commonly known include excavating and replacing with alternative

fills, chemical stabilisation and densification. Excavation and disposal raise environmental and economic challenges as in many sites better fills may only be available for haulage from a very far distance. In the case of chemical stabilization, regardless of the huge successes recorded with the use of traditional chemical stabilizers such as ordinary Portland cement (OPC) and lime, recent studies have found them to be detrimental to both the infrastructure they support and the environment. For instance, they may develop deleterious expansive minerals such as ettringite and thaumasite, in the presence of a sulfate material and favourable conditions (Little *et al.* (2005); Little and Nair (2009)). In addition, energy consumption and environmental concerns linked to the production of OPC and lime are other factors diverting attention of researchers to find alternatives for soil treatments. Compaction on the other hand can result in crushing of particles as well as inter-particle bonds, increasing the dissolvability of certain soil minerals (e.g. carbonate in calcareous soil). A situation that can increase the collapse potential of such soil.

While performance is still a critical aspect of soil improvement planning, environmentally friendly materials which are still reasonably priced are most demanded. This demand is what is driving interests towards binder materials such as sodium silicate ( $\text{Na}_2\text{SiO}_3$ ), which poses no known danger to the environment. Rafalko *et al.* (2007) and Moayedi *et al.* (2011) have suggested that silicate ions can react with calcium ions present in significant amounts in soils, to form a calcium silicate gel which hydrates, and hardens, cementing soil particles together. Sokolovich (1965) described this technique as “silicatization” and reported that the reaction through cation exchange, can precipitate insoluble calcium hydroxide with a strongly developed surface on which the anion of silica is adsorbed  $[\text{Ca}(\text{OH})_2.\text{SiO}_2]$ , producing polymers capable of proofing treated soils against infiltration of water as well as increasing strength.

This reaction is utilized in siliceous-based grouts (e.g. colloidal silica) for treatment of predominantly granular soils through polymerization and gelation. However, the reaction of aqueous solutions of  $\text{Na}_2\text{SiO}_3$  with  $\text{CaCO}_3$  in calcareous soils could hold potential for preventing dissolution of  $\text{CaCO}_3$  and reduction of collapse risk (Sokolovich (1965); (1976)). Nevertheless, Hurley and Thornburn (1972) noted that while sodium silicate is able to form silica gel when mixed with any inorganic material, the gel properties such as strength, durability, and permeability will depend on its  $\text{Na}_2\text{O}:\text{SiO}_2$  ratio and concentration, and other factors such as temperature, type of salts, acids, and bases in the reaction.

This research explores the potential benefits of silicate solutions for improving compressive strength and collapsibility of silty-clay-based calcareous soils. Additionally, is investigating the impact of the chemistry of collapse wetting fluid.

## 1.2 Project Aim and Objectives

The aim of this project is to understand how different soil parameters (particularly calcium carbonate bonding and clay content) and wetting fluid type affect collapsibility in calcareous silty-clay soils and the potential of sodium silicate to mitigate collapsibility of these soils. In order to achieve this aim, the following objectives need to be met:

- 1) Determine the key characteristic features and composition of collapsible soils, their mechanism of collapse and treatment measures (with emphasis on the use of sodium silicate ( $\text{Na}_2\text{SiO}_3$ )) from a review of available literature.
- 2) Develop an effective system that can artificially precipitate predetermined amounts of calcite (as bonding element) in artificial open-structured fine-grained soil samples/specimens under laboratory conditions, for collapse and unconfined compressive strength (UCS) testing.
- 3) Evaluate the physical conditions (e.g., density, moisture content, reaction time) that influence the process of artificial precipitation of calcite in the fine-grained soils, to inform the design and manufacture of test samples.
- 4) Characterise the UCS, stiffness and collapse potential of the artificial calcareous silty-clay specimens, manufactured using the method developed in 2 and 3.
- 5) Evaluate the role and interdependency of factors such as calcite content, clay content and other soil physical properties on the UCS and collapse behaviour of calcareous silty-clay specimens tested under different conditions, such as varying pH of wetting fluid and wetting load.
- 6) Determine the relationship between compressive strength (UCS) and collapse potential.
- 7) Evaluate the impact of wetting fluid pH (solvent and nonsolvent) on collapse potential and rate of collapse.
- 8) Assess (using UCS and collapse tests) both the most effective method of application (defined by the post-treatment compressive strength) of  $\text{Na}_2\text{SiO}_3$  solution, and its impact on the reduction in collapse in the calcareous silty-clay specimens.

## 1.3 Scope of Work

This project was restricted to laboratory scale. However, the study presented herein explores the effect of wetting fluid type and tries to simulate field conditions where either a low acidic solution or normal water wets calcareous soils, to understand the influence of these fluids on the soil's collapsibility. The laboratory programme here, adopts a 5% acidic solution and distilled water as wetting fluids (or collapse triggers) to investigate both untreated and silicate-

treated unsaturated calcareous/carbonate silty-clay samples manufactured under laboratory conditions.

The project followed a sequence beginning with manufacture of potentially collapsible calcareous silty-clay soils with varying degrees of cementation, determining collapse potential (CP) by means of the single and double oedometer testing methods as well as determining unconfined compressive strength properties. This was to relate strength with collapsibility. Then, the potential of a well selected (based on  $\text{SiO}_3:\text{Na}_2\text{O}$  ratio and concentration) sodium silicate solution to improve both collapse and UCS characteristics through a non-destructive treatment method (impregnation technique) and mixing and remoulding was investigated.

An innovative technique for simulating calcareous silty-clay soils was developed for manufacturing samples while other laboratory testing methods followed relevant sections of the British Standards Institute (BS1377:1990); ASTM (D 5333 - 03); and other recommendations by authors such as Alan and Robert (1988). Although, in the collapse testing a nonstandard wetting fluid (5% acidic solution) was also adopted. This however, can be related to recommendation by ASTM (D 5333 - 03) that field anticipated pore fluids can be simulated in laboratory testing regime for collapsible soils

#### 1.4 Thesis Outline

This thesis is made up of seven chapters. Chapter 1 introduces the general background and further defines a clear aim and objectives of the research as well as its scope. Chapter 2 reviews existing concepts and characteristics of natural and anthropogenic collapsible soils. Those features and opposing arguments about collapsing soils and available improvement techniques are outlined. Finally, research gaps in the literature are identified. Chapter 3 presents the laboratory methods adopted in the study: methods for precipitating carbonate, selection of soil materials and manufacture of calcareous samples with potential open metastable structure. It also contains systems used to evaluate the physical structure and chemical composition of manufactured samples as well as geotechnical testing methods used. Chapters 4 and 5 present and discuss experimental results from geotechnical testing of untreated and treated samples, respectively. Chapter 5 also proposes procedures for selecting the most appropriate silicate solution for treatment of the samples. Chapter 6 offers integrated discussions of some key contributions of the project, showing the relevance of the investigations carried out. Key aspects included effectiveness of samples manufacturing method, mechanism of collapse and the relationship between compressive strength and collapse potential, as well as applicability of silicization to improve earthworks. Finally, Chapter 7 presents general conclusions reached in the research and suggestions for future research.

## Chapter 2 Literature Review

### 2.1 Introduction

Collapse has been defined as a mechanism of sudden reduction in total volume of a metastable soil structure (i.e. an open structure) under certain loading and/or wetting conditions. These metastable soils, either natural or manmade, usually have high enough bonding strength resulting from either suction, clay,  $\text{CaCO}_3$  or other salts in their natural unsaturated state allowing an open, semi-stable state. Collapse is believed to occur when the particle-to-particle bonding is destroyed by wetting with or without additional loading, thus an initial open structure becomes dense (Osipov and Sokolov (1995); Feda (1995b); Rogers (1995)).

Natural collapsible soils usually form from the natural processes of weathering and wind and water deposition while anthropogenic collapsible soils arise from poorly engineered and non-engineered fills (Rogers (1995); Mansour *et al.* (2008); Watts and Charles (2015)). Although  $\text{CaCO}_3$  is common in natural soils (Mitchell and Soga, 2005) and its presence in significant amounts qualifies such soils as calcareous soils (Thompson, 2007), its role in collapse of collapsible soils has continued to raise concerns.

Furthermore, the development of reliable, environmentally friendly and cost-effective methods to stabilise or reduce collapsibility in collapsible host soils continues to draw attention (Jefferson *et al.*, 2005).

This chapter is dedicated to review available knowledge on the behaviour and features of natural and artificial collapsible soils, the analytical and empirical techniques used to predict and classify collapsibility in unsaturated soils. Available methods of mitigating collapse are also reviewed, with special focus on the use of silicate substances. Other areas considered are laboratory methods for simulating calcareous/carbonate soils that display collapsible features.

### 2.2 Types and Origin of Collapsible Soils

Rogers (1995) broadly classify collapsible soils into manmade and natural types cutting across soils from nearly all origins (see Figure 2.1). Manmade being typically poorly engineered (compacted) or non-engineered soils. The most common types of natural collapsible soils according to the University of Iowa 2002 (Mansour *et al.*, 2008) are: water deposited (alluvial), wind deposited (aeolian), gravity deposited (colluvial), and residual (resulting from extensive

weathering of parent rock) soils. They range from clays, silts, loess, volcanic ash tuffs, and fine sands.

Collapsible open structures form naturally from a combination of particle type, shape, grading, water content, and deposition mechanisms. However, compacting soils (both fine soils and granular soils with angular grains) dry of optimum (Barden *et al.* (1973); Tadepalli and Fredlund (1991); Fredlund and Gan (1995); Rogers (1995); Assallay (1998); Houston *et al.* (2001); Alonso *et al.* (2010b)) or wet of optimum (when moisture reduces in future) (Lawton *et al.* (1989); Lawton *et al.* (1992)) can also yield an open structure collapsible upon wetting.

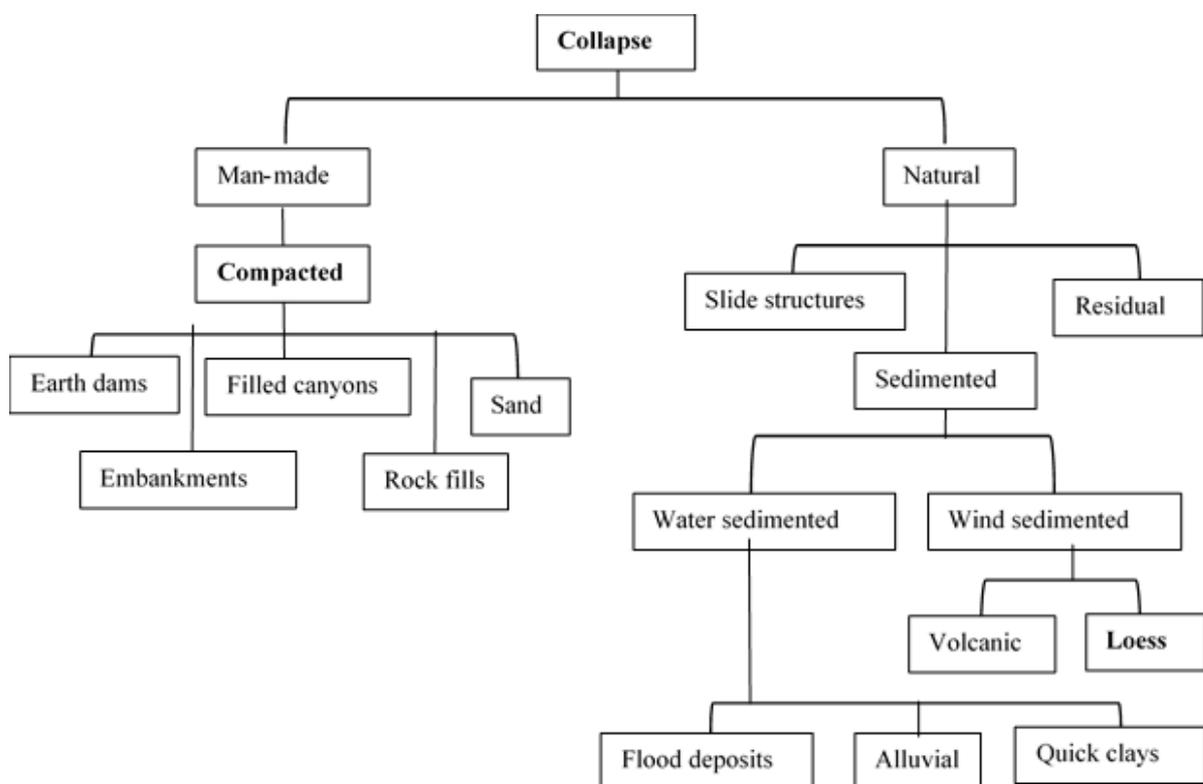


Figure 2.1 Main Classes of Collapsible Soils (modified after Rogers (1995))

### 2.2.1 Manmade (engineered and non-engineered) collapsible soils

As corroborated by Rogers, Lawton *et al.* (1992) have reported (citing the literature) many cases of significant damages to earthworks related to soil collapse in compacted fills since 1953. Lawton *et al.*, went further to state that there had been a mistaken belief that precludes the possibility of further densification of compacted fills even upon access to water, and that this belief could have been responsible for damages recorded.

From their review, they observed that nearly all kinds of compacted soils are subject to collapse under certain loading and environmental conditions. Whether clean sands or pure clays (including montmorillonite) or soils with significant gravel fractions. Damages caused by collapse in compacted fills included damage to pavements and subgrades placed on highway embankments, cracking and slope failure within fills (piping, seepage losses, and failure in earth dams), damage to foundations and structures placed on fills, and distress or failure of underground utilities (Barden *et al.* (1973); Rogers (1995); Assallay (1998); Houston *et al.* (2001); Alonso *et al.* (2010b); Lawton *et al.* (1992)).

Some case histories of collapse of compacted fills reported in Lawton *et al.* (1992) are: two low earth dams constructed in Western Canada in the late 1930s and early 1940s that failed due to collapse (Peterson and Iverson, 1953). Cracking in three earth dams located in California were found to be due to differential settlement resulting from collapse (Leonards and Narain, 1963). Leonards and Davidson, 1984 cited wetting induced collapse as key factor of the 1976 collapse of the Teton Dam in Idaho. Miranda, 1988 reported collapse of earth dams in the semiarid region of northeast Brazil caused by piping resulting from cracks induced by wetting. Booth, 1977 and Lourens and Cazpla, 1987 have reported a few instances of deformation, cracking, and failure of highway embankments in South Africa resulting from wetting-induced settlement (collapse settlement).

Other wetting-induced collapse cases which Lawton and colleagues were involved as forensic investigators in the early 1980s were in southern California, where a compacted fill placed in a canyon failed resulting in damages and litigations worth over 30 million dollars. Cox 1978 mentioned how important the National House Builders Council of Great Britain considered the problem of collapse settlement of floor slabs placed on fill by discussing it in their 1977 Guidance Notes for House Construction. There were reviews of these failures which reveal that some of them were compacted on the dry side of optimum with low levels of saturation and low dry densities, and with light compactive efforts. Wetting could have occurred from infiltration of water following periods of heavy rainfall, ineffective drainages, regional groundwater build-up, broken water pipes, ponding, flooding, moisture build-up beneath covered areas, irrigation, etc. (Lawton *et al.*, 1989).

Watts and Charles (2015) have also discussed extensively the collapse behaviour of fills, in their book “*building on fill: geotechnical aspects*,” a BRE publication. They recognized generally two forms of fills: engineered and non-engineered fills. According to Watts and Charles (2015), engineered fills are the fills selected, placed and compacted in controlled conditions to achieve specification, so that they exhibit engineering behaviour adequate for their purpose, while non-engineered fills arise as by-products of human activities involving the

disposal of waste materials with no subsequent engineering application in sight. However, both fill types may constitute either natural soils and rocks or several forms of waste material placed over natural ground. Some waste materials, particularly found in the UK include opencast mining backfill, colliery spoil, urban fill, domestic refuse, industrial and chemical wastes, pulverised fuel ash, hydraulic fill, infilled docks, pits and quarries, etc.

Despite that engineered fills may also exhibit some collapse behaviour under certain conditions, Watts and Charles (2015) believe that most of the collapse problems encountered with fill deposits are associated with construction on non-engineered fills. This is particularly due to their variability, differential movements, and the difficulty to characterise them. Watts and colleague identified the following factors generally controlling the engineering behaviour of fills:

- Type of fill material, e.g. colliery spoil, domestic refuse, chemical waste, etc.
- Degree of control during placement, for instance engineered or non-engineered.
- Method of placement, whether the fill was placed in dry or submerged conditions, in thin layers or in high lifts, or transported and hydraulically placed.
- Place of deposition, whether a clay pit, old duck, or opencast mine.
- Age – changes that a fill deposit is subjected to in its environment as time passes, can affect its engineering behaviour.
- Stress history, e.g. stress imposed in the past and present.

Furthermore, Watts and Charles (2015) advice that caution should be taken where development on non-engineered fills is to be carried out, because of the consequent effects of the diverse nature of their constituent materials. For instance, biodegradable wastes will continue to decompose and cause volume changes even after the fill is compacted. There is also the possibility of chemical reactions that may give rise to deleterious materials, e.g. sulfate attack on OPC concrete may cause expansion (Little *et al.* (2005); Little and Nair (2009); Watts and Charles (2015)). The carbonation of portlandite in OPC concretes and other materials present in fills is also a source of concern. Renforth *et al.* (2009) and Washbourne *et al.* (2012) have reported precipitation of secondary (pedogenic)  $\text{CaCO}_3$  contents reaching 19.9 and 40 % in brownfield and demolition sites in the Northeast of England. Watts and Charles (2015) have noted that sulfuric acid can form from the oxidation of iron pyrite ( $\text{FeS}_2$ ) in pyritic shales, and this may cause dissolution of  $\text{CaCO}_3$  present in the fill deposit. Understanding the effect of the dissolution of  $\text{CaCO}_3$  on collapse potential is the main objective of this thesis.

A few examples of buildings founded on fills and damaged by the collapse phenomenon as reported in chapter 5 of Watts and Charles (2015) include: two-storey blocks of eight houses

founded on a 12m deep (clay) opencast mining backfill at Ilkeston; single-storey factory founded on a 18m deep (clay with shale fragments) opencast mining backfill in West Auckland; industrial estate in Brighton founded on a 6m deep chalk fill suffered 110mm settlement; North Tyneside District General Hospital founded on a 20m deep (clay and shale) opencast mining backfill suffered 160mm collapse settlement; Clifford's Tower in York founded on a fill comprising stones, gravel and clay suffered severe cracking; and houses in southern California founded on 30m deep engineered sandy clay fill suffered distress from settlement reaching 0.45m due to increased wetting. Watts and Charles (2015) noted that collapse compression causing these damages occurred several years (5 - 246) post fill placement while suggesting construction specifications necessary to eliminate collapses in engineered fills as well as treatments for non-engineered fill deposits.

### 2.2.2 Natural collapsible soils

Weathering (physical, chemical or both), transportation, and deposition are the natural phenomena that give rise to soil deposits. With the presence of inter-particle bonding an open structure is produced. A classic collapsible soil is the product of a combination of the foregoing processes and particle type and sedimentation mechanisms: resulting in residual, water deposited (quick clay, alluvium), and wind deposited (loess) collapsing soils (Rogers, 1995). They are broadly classified by Fredlund and Gan (1995) into wet or dry collapsible soils, with the dry collapsible ones occurring either as cemented or uncemented.

Residual soils are formed in place from decomposition, disintegration and mechanical alteration of parent rock materials (Rogers (1995); Wesley (2010)). Soil particles produced vary in size from clay to silt, sand, gravel, and large fragments. Residual soils are also characterized by high variation in thickness ranging from few centimetres to several metres depending on regional, environmental and climatic conditions. According to Clemence and Finbarr (1981), the collapsible structure of residual soils develop from the leaching out and loss of soluble and colloidal materials. Good examples of residual collapsible soils are the granitic sands of South Africa whose weathering process was geochemically controlled, manifested by the weathering of feldspar to create a sub-rounded sand having an open structure (Rogers, 1995). Another is the decomposed granites in Rhodesia which were found to have a total collapse of about 7% to 10% (Brink and Kanty, 1961 cited in Assallay *et al.* (1998)).

Water deposited collapsible soils according to Rogers (1995) are classified into: the alluvial deposits and quick clays. The alluvial deposits are usually in the form of alluvial fans, floodplains, and mudflows. However, some (e.g. the calcareous loessial soils of Egypt) are

believed to originate from mixed conditions. They show contributions of marine, wind and rain conditions (El-Sohby *et al.*, 1988).

Clemence and Finbarr (1981) have reported cases of subsidence up to 15% been observed in the San Joaquin Valley of California. Assallay (1998) cited Rollins et al 1994, Holtz and Hilf 1961, Gibbs and Bara 1967, Bull 1964 reporting numerous cases of collapse problems connected with alluvial deposits. That Rollins et al (1994) observed collapsible problems in alluvial fans soils of six case histories, and then concluded from their experience that even coarse-grained soils with high densities and very high SPT blow counts, in a geologically susceptible environment can collapse upon wetting.

Saturated quick clays have been described by Rogers (1995); Terzaghi *et al.* (1996) as soils having metastable structure and are unstable under certain conditions. Quick clay was defined as clay having an undisturbed shear strength at least 30 greater than its remoulded shear strength (Torrance (1983); Geertsema and Torrance (2005)). Rogers (1995) have concluded that quick clay deposits are another set of collapsible soils which are neither unsaturated nor partially saturated. Quick clays consist typically of fine-grained primary mineral particles and are believed to form by the action of glacial grinding and deposited by slow fall sedimentation under shallow marine conditions. The shape of the grains is typical of the blade-shaped and sub-angular like of loess materials created by cold or hot weathering.

Quick clays which are referred as very sensitive clays and defined as type C (2–5 $\mu$ m) particles in the R-size diagram (Figure 2.2) proposed by Jefferson and Smalley (1995) and (1997), have an open structure with short range bonding between particles. They reported their open structure as being sustained by small quantities of carbonate cementation or clay minerals at the intergranular contacts and can collapse when loaded beyond their bearing capacity. The R-size diagram is a model used to assess the possibility of a soil collapsing based on inherent bonding strength and nominal particle size. Further details can be found in Jefferson and Smalley (1995).

According to Assallay (1998), quick clays have moisture contents higher than their Liquid Limit, a sensitivity and plasticity index of 200 and 5 respectively. He also cited Bentley and Smalley, 1984 reporting severe problems for shallow foundations caused by the high shrinking behaviour of quick clays when moisture conditions change. However, the two most commonly studied quick clays, the Canadian and Scandinavian (Bentley *et al.*, 1980) have shown quite different behaviours. For instance, the Canadian quick clay exhibits significant higher strengths than Norwegian quick clay. This is thought to be resulting from the former been geologically

older and developing more carbonate cementation than the latter which have higher clay mineral content and classified as rapid (Rogers, 1995).

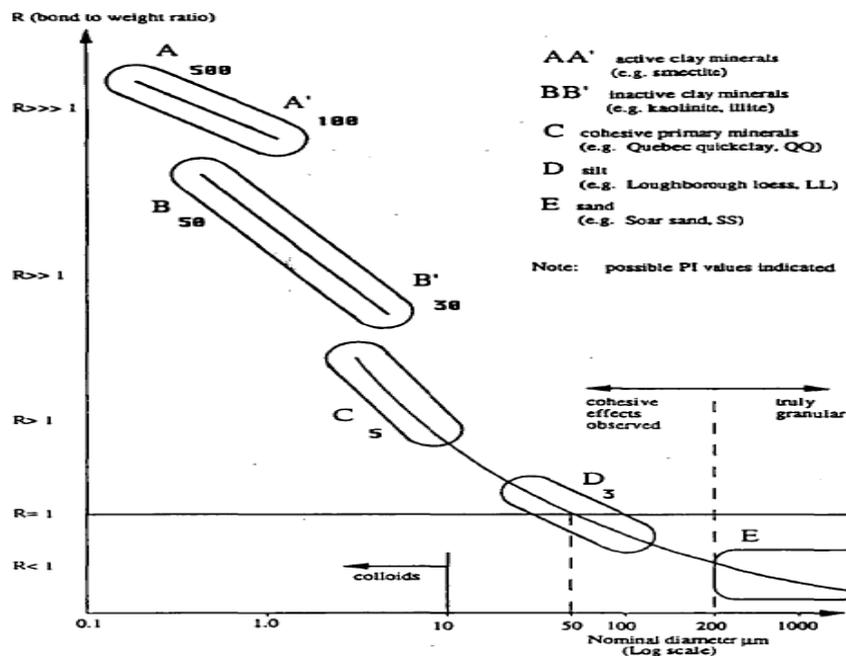


Figure 2.2 The R-size diagram: bond/weight ratio versus nominal diameter of soil particles (after Jefferson and Smalley (1995))

Wind (aeolian) deposited sediments are the predominant type of collapsible soils, especially loess deposits and to lesser extent loose sand deposits (Rogers (1995); Assallay *et al.* (1998)). They are generally unsaturated and are found in arid and semi-arid regions of the world. Collapsible loose sand deposits deposited under desert conditions can be found covering large areas of Southern Africa, and the shifting sand dunes of the Sahara in North Africa. According to Rogers (1995) these sands having an open structure are consisted typically of rounded primary mineral particles (100–200 $\mu\text{m}$ ) supported by short range bonds.

Loess deposits are unsaturated, consisting predominantly of silt-sized particles derived from glacial grinding or cold/hot weathering. The particles are believed to be initially transported/deposited by rivers before picking up by winds, which deposit them at their ultimate places. Glacial grinding produced loess deposits in the North America as reported by Smalley 1966. Loess deposits in the Canary Islands, the Cape Verde Islands and some areas around the Sahara (Northern Nigeria, Libya, and Tunisia) are believed to form from hot weathering processes prevalent in the Sahara (Coude-Gaussen, 1987 & 1991 and Rognon *et al.*, 1996 cited by Assallay *et al.* (1998); Rogers (1995)). Rogers (1995) also reported the Chinese loess and

South American loess from Tibetan mountain ranges and nearby deserts, and the Andes, respectively as good examples of loess deposits produced from cold weathering. Others are the Bulgarian and Romanian loess from the Danube.

The distribution of collapsible soils on the globe is presented as Figure 2.3.

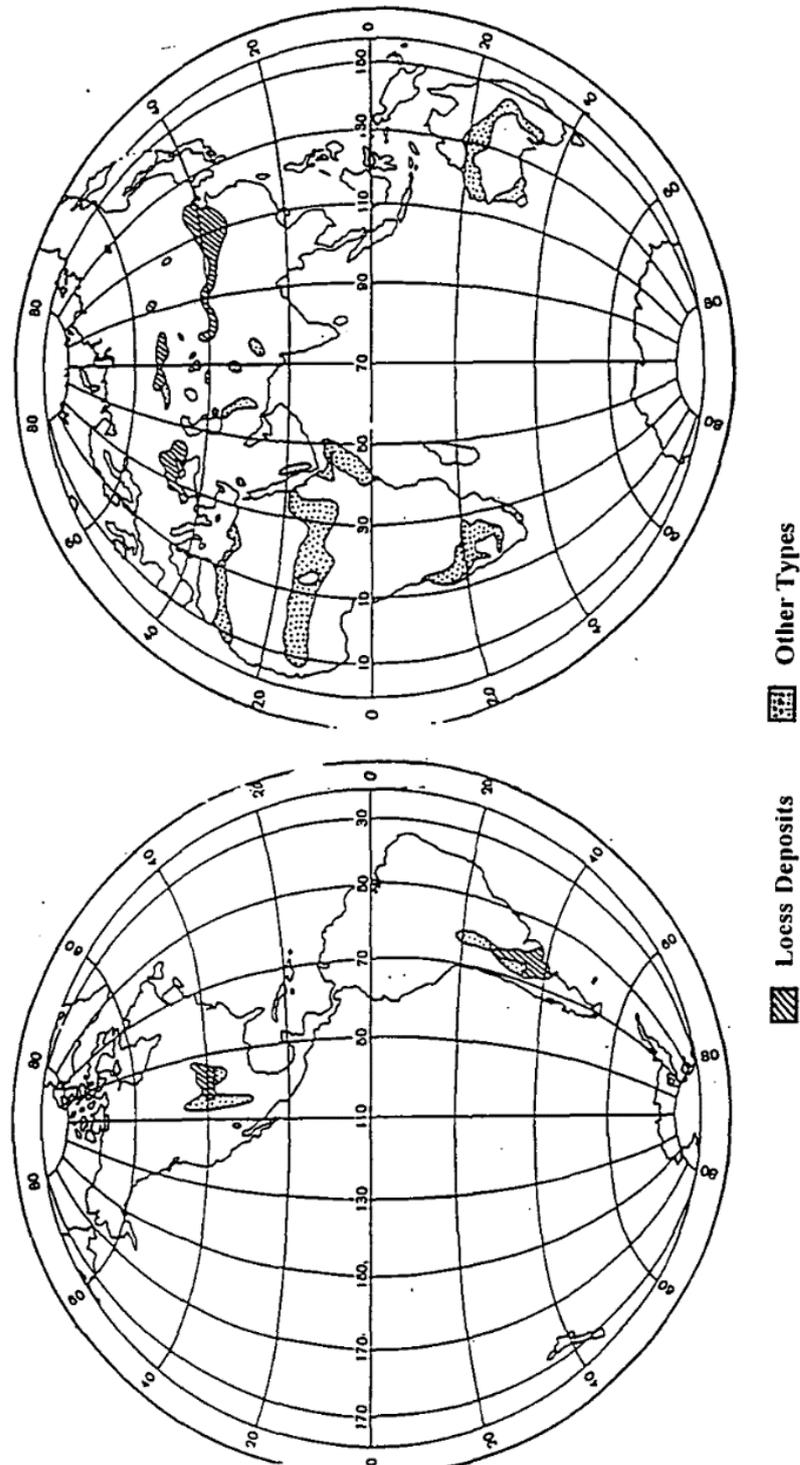


Figure 2.3 Distribution of collapsible soils, Kriger, 1986 (adopted from Assallay (1998))

### 2.3 Understanding Soil Collapse Mechanisms

There is a large body of research aiming to understand the engineering behaviour of both natural and manmade collapsible soils, from a physical (bond alteration) point of view (Feda (1988); Lawton *et al.* (1992); (1995b); Rogers *et al.* (1994); Osipov and Sokolov (1995)) and a mechanical (effective stress) viewpoint (Jennings and Burland (1962); Alonso *et al.* (2010a)).

Despite the many studies, particularly loess soils dominating the literature, the mechanisms of metastability and consequent collapse potential are not fully understood (Derbyshire and Mellors (1988); Bell *et al.* (2003); Milodowski *et al.* (2015)). Consequently, several researchers have described collapsible soils in different ways. Some of these definitions taken from the work of Assallay (1998) and others are presented in Table 2.1.

Table 2.1 Different descriptions of soil collapsibility as in Assallay (1998)

Year	Author	Description of Soil Collapsibility
1970	Dudley	Any unsaturated soil that goes through a radical re-arrangement of particles and great loss of volume upon wetting with or without additional loading.
1973	Handy	A form of under-consolidation related to apparent cohesive strength of unsaturated soil.
1973	Barden <i>et al.</i>	A rapid settlement, which occurs when water easily finds access to partially saturated soils. They went further to note that settlement occurs under loading, and the rate of settlement depended on the rate at which water was available to the soil.
1975	Jennings and Knight	An additional settlement that results from the wetting of a partially saturated soil, normally without any increase in applied pressure.
1977	Booth	Settlement due to an increase in the degree of consolidation.
1981	Clemence and Finbarr	Unsaturated soils which when wetted, whether loaded or not, undergoes rapid particle rearrangement and great volume loss
1989	Rizkallah and Keese	A settlement of soil caused by sudden breakdown of grain-fabric due to wetting, under a constant surcharge.
	Signer <i>et al.</i>	Soils which have such a structure that suffers a high volumetric reduction when soaked, whether subjected to an additional load or not.
	Mackechnie	Reported to the twelfth International Conference on Soil Mechanics and Foundation Engineering that "not all soils with low unit weights are necessarily collapsible, but partial saturation is generally a prerequisite to collapse".
1990	Houston and Houston	When a dry or partially saturated soil contracts upon wetting it is said to be collapsible.

As can be seen from Table 2.1, most definitions identified collapsible soils as partially saturated, but there seems not to be agreement as to whether collapse occurs only with loading or self-weight. Again the principle of effective stress which covers all soil mechanics theory does not seem to be in agreement with the theory of collapse as in the above definitions, because an increase in water content increases pore water pressure and reduces effective stress, thus expected to result in heave and not settlement (Barden *et al.*, 1973).

Clearer explanations to the mechanisms of collapse, and more criticisms to the above definitions were provided by Rogers (1995); Osipov and Sokolov (1995); and Klukanova and Frankovska (1995). Rogers (1995) criticized all the definitions by highlighting three key flaws from a geotechnical perspective:

- (1) that many of the definitions had restricted collapsibility to unsaturated soils collapsed by wetting
- (2) that definitions could not quantify amount of settlement which resulted from collapse of soil structure
- (3) that some of the authors used geotechnical terminologies such as consolidation and under-consolidation, in a conflicting manner.

Following his criticism, Rogers (1995) holistically described soil collapse as change in structure from a metastable form to a stable form, defining a collapsible soil as “*that soil in which constituent parts have an open packing and which forms a metastable state that can collapse to form a closer packed, more stable structure of significantly reduced volume.*”

Assallay (1998) examined the definitions in the literature and made the following conclusions about their general characteristics:

- (1) Collapsible soils have particle sizes ranging from sand, silt, to clay, and angular grains (particularly silts). The size and shape of particles during sedimentation (in the case of natural collapsible soil) or state of compaction (man-made collapsible soil) are responsible for the open structure formed, (Rogers, 1995). The open structure is said to be metastable until collapse is triggered probably by wetting under loading conditions.
- (2) They have high void ratio and low density. This was described by Barden *et al.* 1969 as open potentially unstable structure, which allows the soil readily undergo particle re-arrangement and volume reduction by self-weight and/loading upon wetting (Barden *et al.*, 1973).

- (3) They are highly sensitive. Sensitivity depends on soil mineralogy and type of particle-to-particle bonding agent. For example, quick clays may be more sensitive than sand or silt deposits having calcite as bonding agent.
- (4) Collapsible soils are young geological or recently weathered deposits.
- (5) Collapsible soils possess relatively low inter-particle bond strength. Collapsing rate was also considered by Barden *et al.* (1973) in terms of bonding strength to be immediate, slower and very slow, depending on whether bonding was by capillary suction (Figure 2.4a), clay buttress (Figure 2.4b&c), or chemical cementing (iron oxide, calcium carbonate or some other salts), respectively.
- (6) They are mostly partially saturated, except for saturated quick clay in which collapse is not triggered by wetting but rather triggered by drying resulting in significant shrinkage (Rogers, 1995). Collapse also occurs when internal forces surpass bond shear strength (Jehring 2007 in Langroudi (2014)).

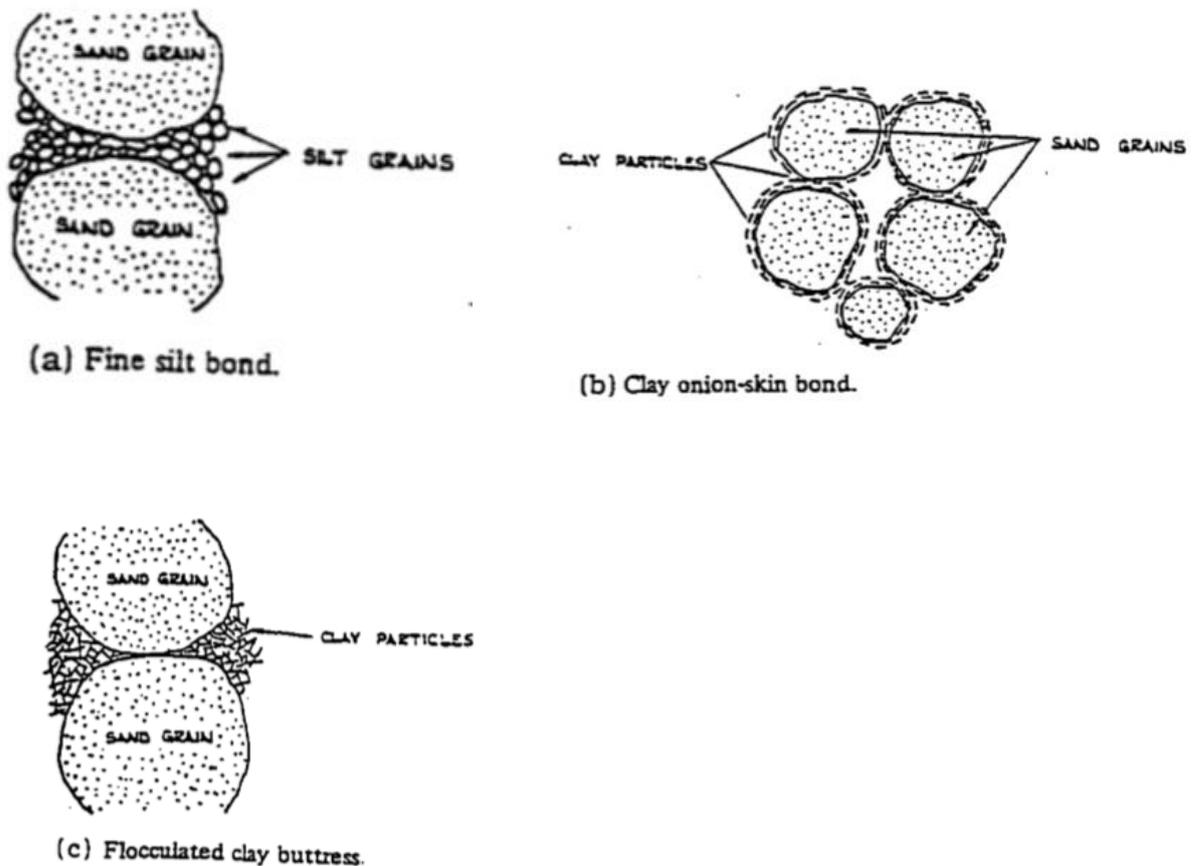


Figure 2.4 Capillary suction bonding and clay bonding in collapsible soils suggested by Barden *et al.* (1973)

### 2.3.1 Micromechanics (or bond alteration) point of view

The influence of internal stress, structural components, and particle arrangements on soil deformation can be described by micromechanics. Discontinuous numerical methods, such as discrete elements are commonly used to formulate relationships in micromechanics but are not often adopted to explain the discrete collapse phenomena in unsaturated soils (Meier *et al.*, 2008).

Mellors (1995) studied the influence of clay component on collapse of the soil structure of loess and described the soil grain arrangement and contact modification on the wetting surface of grains under loading. Mellors argued that prior to wetting, and despite the shear stresses due to the external load, inter-particle bonds remained adequately strong to resist shear failure at points of contact. However, upon wetting, microsheading occurred at the point of contact causing collapse of the soil structure. Mellors noted that wetting with water reduces the shear strength of the bonds such that it becomes less than the shear stresses acting at the point of contact, thus, collapse occurring. Mellors also claim that the translational and rotational movements during the collapse (continuous particles re-arrangement) process are also, controlled by shearing at points of contacts. Mellors (1995) further used an equation introduced by Lamb, 1960 to explain the mechanism of collapse of the loess. This equation (2.1), which modified the effective stress equation, relates the external stress to the internal stresses in a certain soil system.

$$\sigma = \sigma' + u + R - A \dots\dots\dots 2.1$$

where,  $\sigma$  is total external stress,  $\sigma'$  is effective intergranular contact stress,  $u$  is excess pore pressure,  $R$  = interparticle repulsion per unit area of total interparticle contact,  $A$  is interparticle attraction per unit area of total interparticle contact.

On the basis of Mellors' 1995 soil, tested in an air-dry state ( $u = 0$ ) under an external load, Equation 2.1 becomes Equation 2.2

$$\sigma' = \sigma - R + A \dots\dots\dots 2.2$$

Following Equation 2.2, therefore, wetting leads to the rehydration of clay bonds (or connectors) and the expansion of Diffused Double Layer, reduction in attraction forces ( $A$ ), increase in repulsion forces ( $R$ ), and eventual clay bonds' disaggregation. This results in reduced inter-particle shear strength of clayey bonds and consequently shearing occurs at the point of contact (Mellors, 1995). Yun and Santamarina (2005) also recognized the impact of bonding on the behaviour of soil, such as small-strain stiffness, resistance to liquefaction and collapse, noting that increasing degree of cementation affects the drained strength of soil (i.e., increasing the small-strain shear modulus,  $G_0$ ). It is argued that cementation effect depends on the amount and type of cementing material, particle size distribution (the greater the specific surface, the thinner the cement layer around particles), density (intergranular connection increases with density), and the stress-cementation history (i.e. the degree of confinement during cementation) (Clough *et al.* (1981); Feda (1995a); Jarrard *et al.* (2000)). Under triaxial loading, Airey and Fahey (1991) observed inter-particle debonding and skeletal softening. Experimental (Fernandez and Santamarina, 2001) and numerical (Zang and Wong, 1995) observations reveal debonding and softening occurring during unloading, even in isotropic conditions. That soils cemented under confinement expand during unloading and the bond at contact point fails in tension. This is an inherent mechanism in sampling (Tatsuoka and Shibuya, 1992).

An understanding to the collapse mechanism of soils was provided (Figure 2.5) by Feda (1995b), whose micromechanical view identified “debonding”, “grain breakage”, “fabric transition (compression, shear)” and “softening-hardening” as four different mechanisms in collapsible soils. Figure 2.6 also demonstrates the collapse mechanism as described by Rogers (1995). Rogers reported an increase in positive pore water pressure and a decrease in both effective stress and bond shear resistance when wetting takes place. Decrease in bond shear resistance can better explain the collapse phenomenon as increase in positive pore water pressure, according to classic soil mechanics, should lead to increase in volume (heave) rather than collapse.

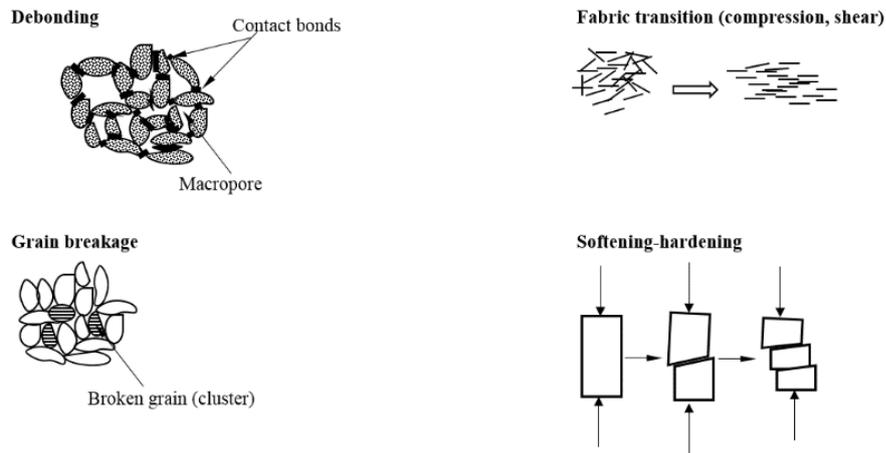


Figure 2.5 Mechanisms of collapse (redrawn after Feda (1995b))

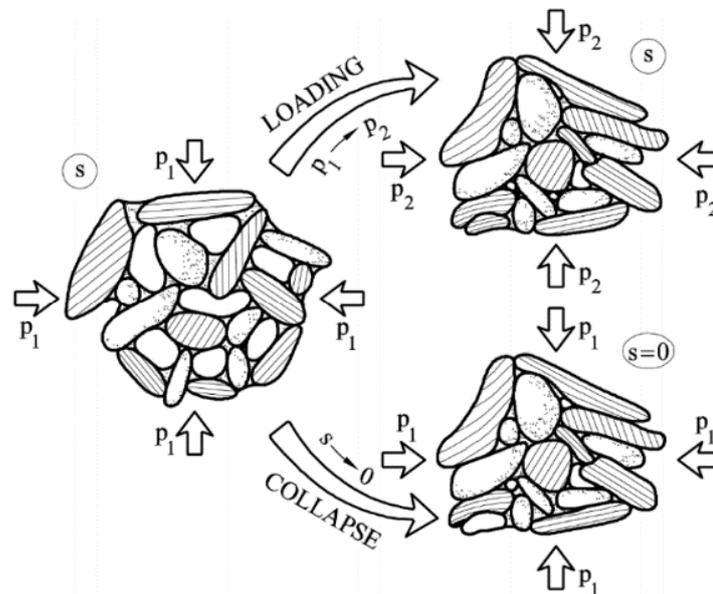


Figure 2.6 Collapse caused by wetting or stressing (after Rogers (1995))

### 2.3.2 The mechanical or effective stress viewpoint

The effective stress concept (Terzaghi, 1936 in Skempton (1960)) is well-established in the mechanics of saturated soils. Terzaghi (1936) developed the concept for a single dominant porosity transmitting single fluid, but later, Bishop (1959) and Matyas and Radhakrishna (1968) developed different forms for pores transmitting two immiscible and miscible fluids, respectively. Efforts have been made by many researchers (e.g., Bishop (1959); Jennings and Burland (1962); Fredlund *et al.* (1993)) to extend the effective stress concept to cover unsaturated soils based on three types of stress variable; attempts to combine pore air pressure, pore water pressure, and total stress within a single effective stress variable. A notable equation (2.3) based on two stress states (net stress and matric suction) (Figure 2.7) and a soil parameter ( $\chi$ ) was proposed by Bishop (1959).



However, earlier studies, e.g., Jennings and Burland (1962) claim that Bishop's equation (2.3) was not capable of adequately relating volume change with effective stress in an unsaturated collapsible soil; since a decrease in suction reduces the effective stress and increases the volume. Consider, for instance, results shown in Figure 2.8 for silt samples loaded under oedometer conditions. The curve with open points representing air-dried silt while the other curve with full points represents identical sample wetted without overburden stress. One of the air-dried samples was wetted under a 400kPa overburden stress, and consequently collapsed onto point A, near the fully saturated line. A second air-dried sample loaded to 400 kPa, wetting took place with a gradual unloading to maintain a nearly constant void ratio. This second sample reached its saturation close to the fully saturated line at point B. In view of the principle of effective stress, as the sample is wetted, resulting in a decrease in suction and effective stress, the samples would have been expected to swell. Conversely, the first sample suffered collapse to point A and the void ratio of the second sample remained unchanged at point B (Burland, 1965).

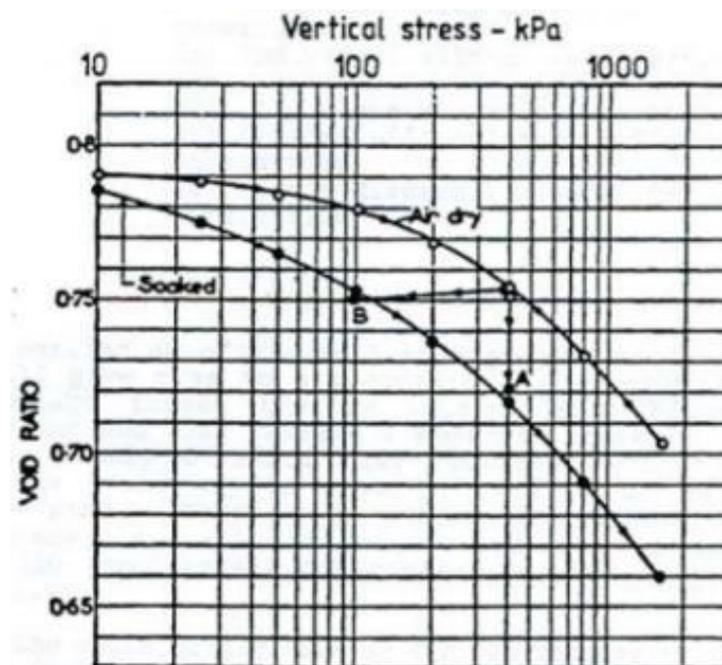


Figure 2.8 Oedometer compression on air-dry and saturated silt showing the effect of soaking (Burland, 1965)

A large body of research (Coussy (2007); Gens (2010)) carried out to develop a suitable function for the soil parameter,  $\chi$  in the Equation 2.3, with  $\chi$  equated to saturation ratio (Houlsby (1997); Borja (2006); Borja and Koliji (2009)), but Tarantino and Tombolato (2005); Pereira and Alonso (2009); Alonso *et al.* (2010a); Vlahinić *et al.* (2011) argue that this choice is for soils containing clay, with the residual saturation ratio greater than zero, thus  $\chi(u_a - u_w) \rightarrow \infty$  when  $(u_a - u_w) \rightarrow \infty$ . To avoid  $\chi(u_a - u_w)$  divergence, the literature contains a suite of methods

proposed, e.g. assuming residual saturation ratio equals zero, idealising water phase as two differentiated residual and free water phases (Fuentes and Triantafyllidis, 2013) or idealising pore phase (air, water, or their combination) as micro- and macro-pore spaces and equating the soil parameter ( $\chi$ ) to the effective saturation ratio (Bagherieh *et al.* (2009); Alonso *et al.* (2010a)). Considering an assumed zero value for the  $\chi$ -parameter along the saturation of micropores (clay fragments of clayey silt soils), it resulted in a constant effective stress equal to  $\sigma - u_a$ , which described the collapse phenomenon. However, it has not been able to describe the early rebound volume change occurring under low external net stresses. It is also not able to describe the effect of seasonal changes in soils. Finally, these formulations have not considered the effect of time on interparticle bond breaking, particularly occurring in chemically bonded unsaturated soils.

Considering the influence of suction on interparticle forces, Burland and Ridley (1996) used the grain column analogy (Figure 2.9) to explain the difficulties posed by the single effective stress in describing unsaturated soil behaviour. They noted that, the impact of a suction acting through the menisci at grains contact points is to provide a stabilising effect, while the influence of a boundary stress is to cause grain slip. The two stress systems give rise to completely different mechanical effects and therefore should not be considered together.

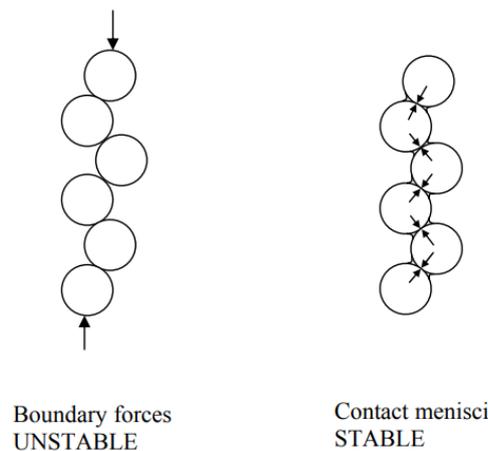


Figure 2.9 Grain column analogy (Burland and Ridley, 1996)

### 2.3.3 Microstructure of collapsible soils

A modelled collapsible structure is shown as Figure 2.10. In spite of the general belief that suction, clay,  $\text{CaCO}_3$  and other salts provide support for the open structure of collapsible soils, some collapsing soils have been reported to gain support by grain mineral-to-mineral contact (see Figure 2.11) where platy grains predominate and clay-size grains are widely scattered (Derbyshire and Mellors, 1988).

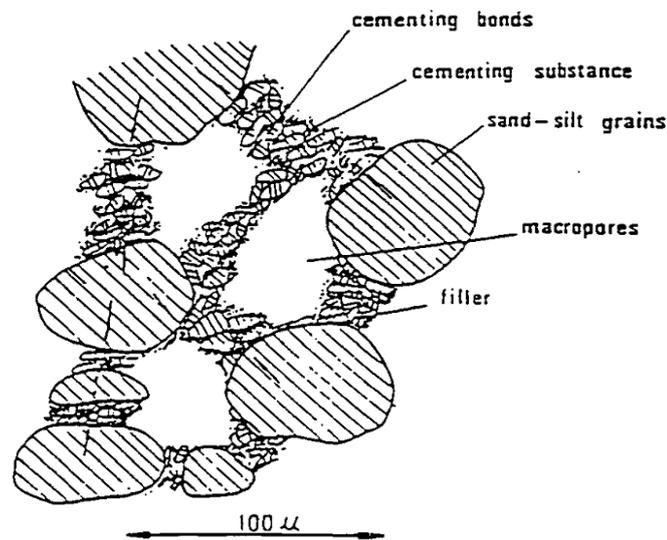


Figure 2.10 Microstructure model of a collapsible loess deposit as suggested by Tan 1988 (after Assallay (1998))

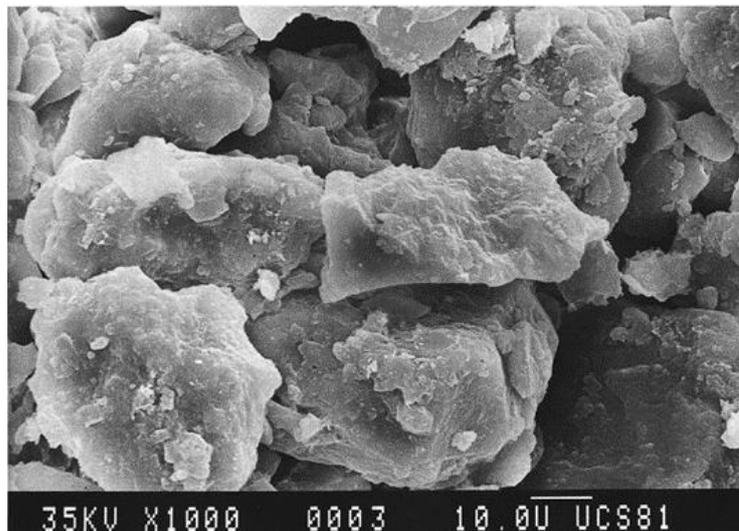


Figure 2.11 Clean edge-to-edge fabric in angular coarse silt Malan loess (Derbyshire and Mellors, 1988)

The understanding of the open flocculated structure of soils compacted dry of optimum has contributed to the understanding of soil collapsibility from a microstructural perspective. Scholars have adopted recent advances in techniques like X-ray diffraction, scanning electron microscope (SEM), and mercury intrusion porosimetry (MIP) to study the microstructure of collapsible soils, and its effect on the mechanical behaviour of such soils and found the techniques useful (Li *et al.*, 2016).

According to Li *et al.* (2016), the literature reports image processing programs developed for extracting from digital images, information relating to soil grains and pores as follows: the Scion (Fang *et al.*, 2013a, b); Videolab (Shi *et al.*, 1995); Mirage (Sha and Chen, 2006; Chen and Sha, 2009a, b); MATLAB (Wang *et al.*, 2011; Mao *et al.*, 2004) and Photoshop (Zhang *et*

al., 2009). These techniques and programs provide strong support for understanding collapse from microstructure information. Commonly used of these techniques are SEM (for acquiring digital images) and MIP (for studying porosity) (Al-Mukhtar et al., 1996; Simms and Yanful, 2001, 2002; Romero and Simms, 2008 cited in Li *et al.* (2016)).

### ***Clay supported structure***

Data from an extensive literature review on the impact of clay content on the collapsibility of loessial soils (Assallay, 1998) has been used to create plots (Langroudi, 2014) to delineate boundaries where clay content influences collapsibility differently under wetted conditions. Langroudi (2014) present 5% to 40% kaolinite clay contents as indicative of possible collapse but most vulnerable between 11% and 24 % with the highest chance of collapse occurring between 8% and 15% regardless of packing state.

Earlier, Handy, 1973 (cited in Bell and Culshaw (2001); Bell *et al.* (2003)) suggested higher collapses with reducing clay content. However, Handy only cited a high probability of collapse for samples having less than 16% clay content, which agrees with Langroudi (2014); Okwedadi *et al.* (2014)). Nevertheless, Handy claims that no collapse should be expected in soils with more than 32% clay content, contradicting the position of Langroudi. El Sohby and Rabbaa (1984)'s data of collapse test of two mixes (sand-clay and silt-clay) as reported by Lawton *et al.* (1992) (Figure 2.12) recorded maximum collapses at clay contents of about 30-40 % and 10-20 %, respectively. Beyond those ranges swelling occurred. Another soil (Ottawa sand-Kaolin mix) studied by Lawton revealed maximum collapse occurring between 12 and 16 % clay content (Figure 2.12). It is obvious that defining collapse solely on clay content can be misleading, as other factors such as type of clay, dry density, bonding strength, etc. can all effect collapse.

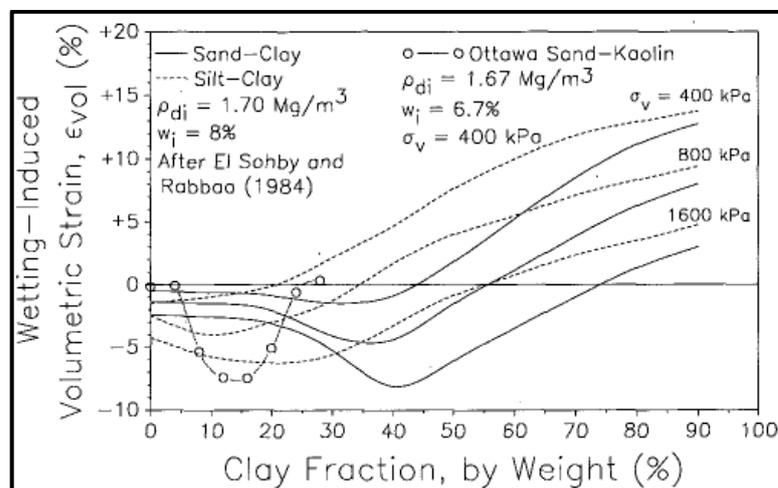


Figure 2.12 Effect of clay content on collapse potential (adopted from Lawton)

The views discussed in Assallay (1998) tended to relate purely -nonactive clays to the collapse structure whereas Langroudi (2014), citing Russell 1944, Handy 1973, Lattman 1973 and Parsons et al., 2009, reported active clay minerals as also contributing to collapse, with special mention of the collapsible loessial deposits in the central areas of North America. Feda (1988) also considered collapse occurring with active clays, but at water contents lower than a certain threshold and overburden stresses higher than their swelling pressures. The collapsible Tucson alluvial clay (majorly montmorillonite) (Barden *et al.*, 1973) is a good example. A few cases of clay-supported microstructure of collapsible soils are shown in Figure 2.13 to Figure 2.15 below:

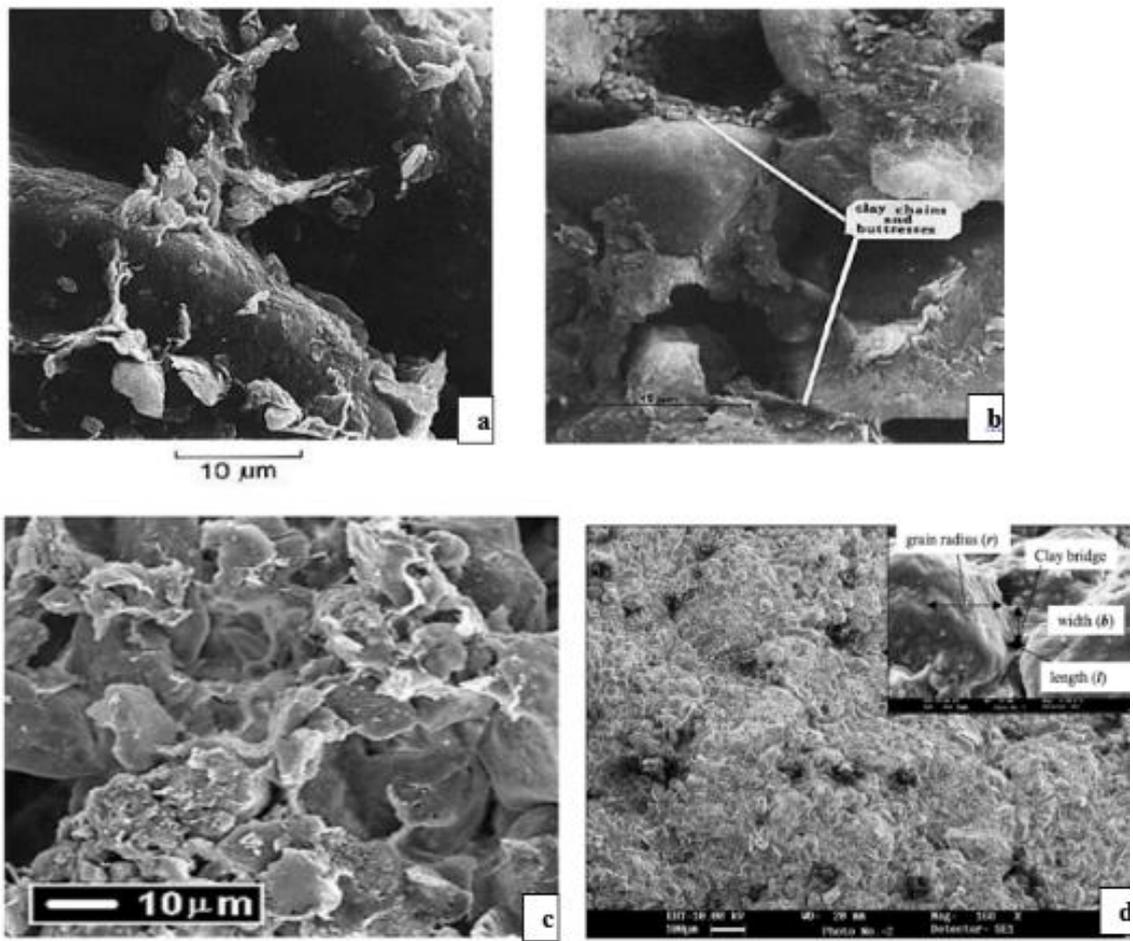


Figure 2.13 Clay connector under scanning electron microscope **a**, UK loess from Kent showing clay bridge (adopted from Derbyshire and Mellors (1988)) **b**, clay buttress in young polish loess in Grabowska Olszweska 1989 **c**, loess at 140km North of Paris, in Delage et al. 2006 (adopted from Langroudi (2014)) **d**, clay bridge in loess (adopted from Jackson et al. (2006)).

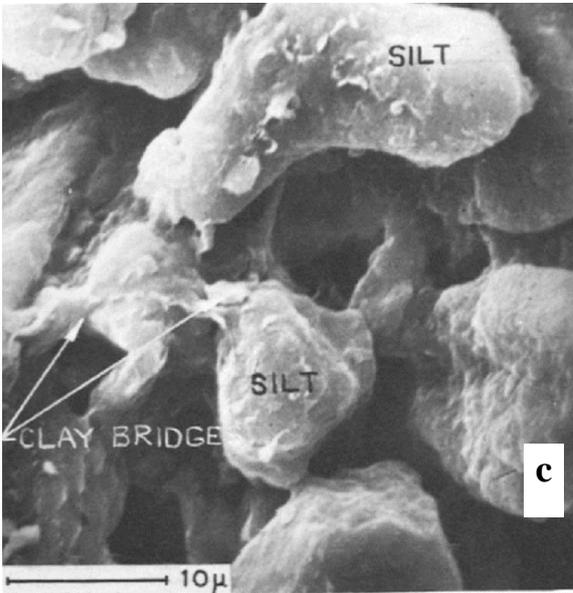
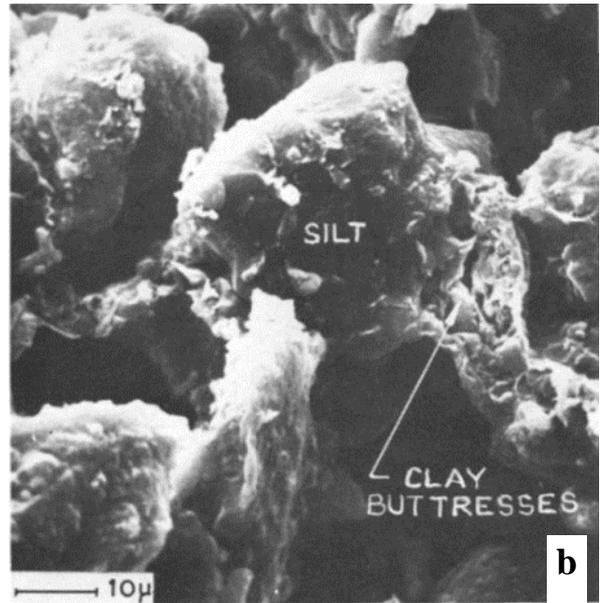
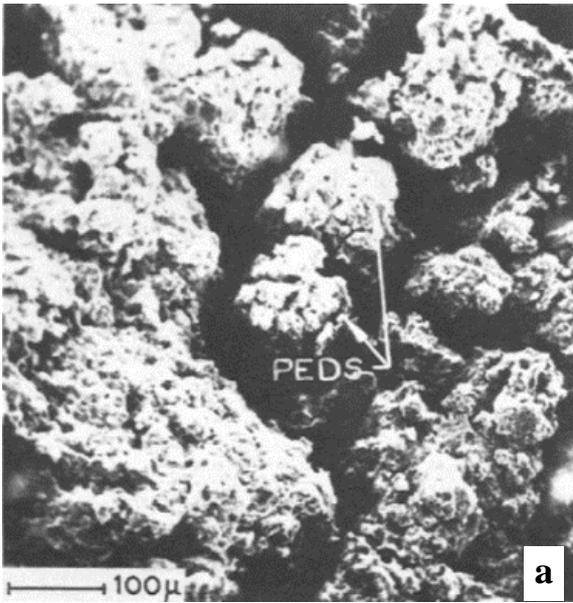
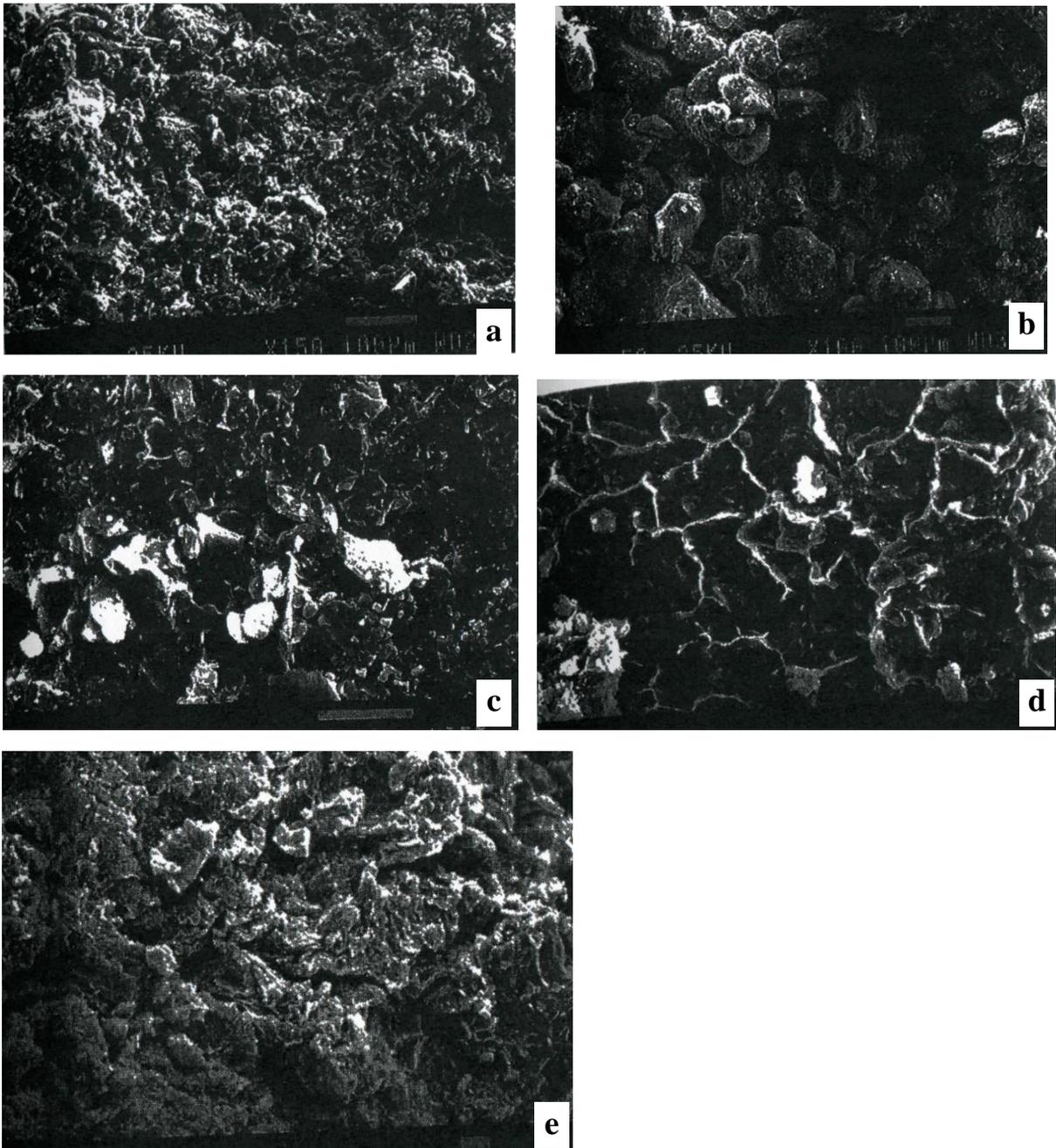


Figure 2.14 micrographs of collapsible soils with clay bonding **a**, bentonite compacted dry of Proctor optimum water content **b**, high clay content of the Tucson alluvial clay **c**, high concentration of clay in Ford brickearth (after Barden et al. (1973))



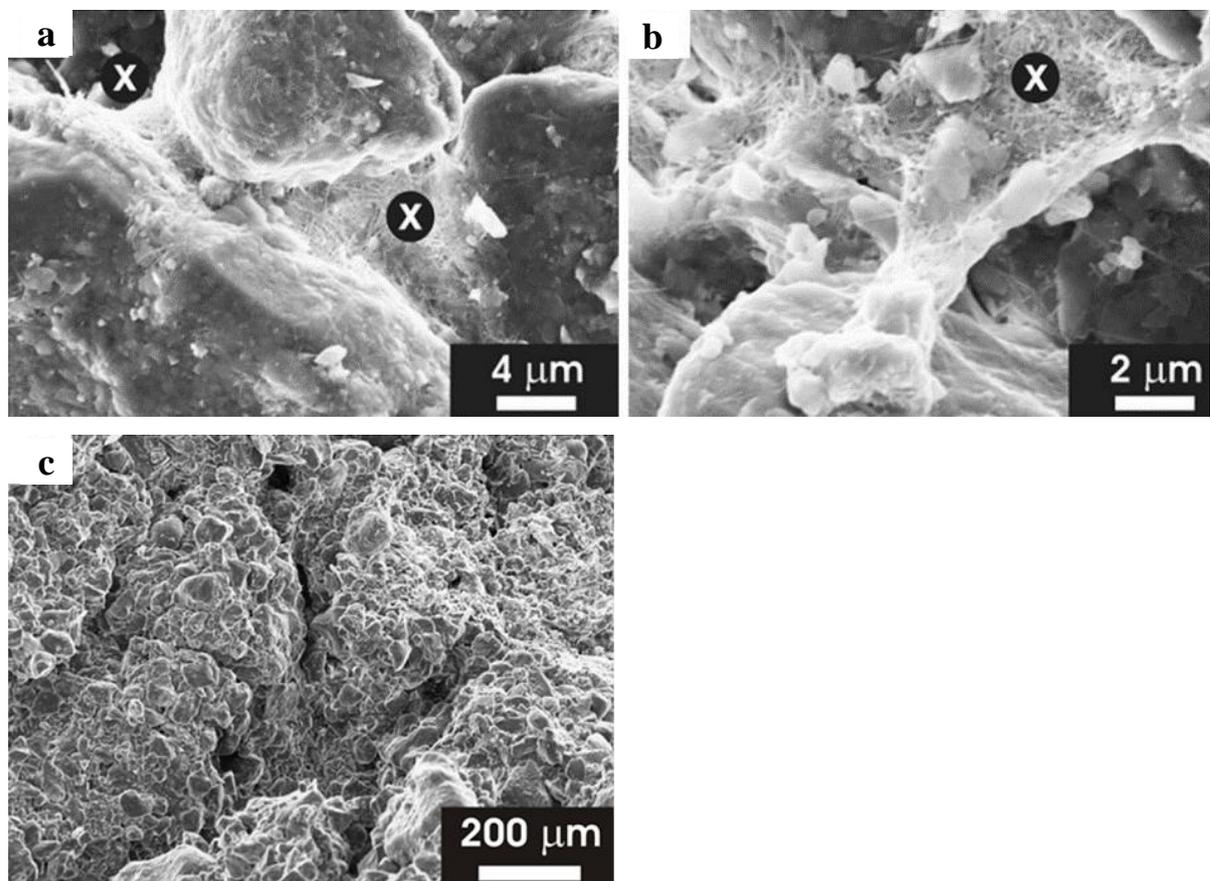
*Figure 2.15 Scanning electron micrographs a, skeletal microstructure of typical loess. Locality Mnesice brick plant near Nove Mesto nad Vahom b, microstructure of loess like sediment from Hajnacka locality. The skeletal microstructure is formed mainly by sand grains c, skeletal-matrix microstructure of clayey loess from Safarikovo locality d, the honey-comb type of microstructure of loess-like sediments from locality Krcava (12m) e, the matrix type of microstructure of loess-like sediment from locality Orechova (8m) (adopted from Klukanova and Frankovska (1995))*

### ***Calcium carbonate supported structure***

The literature has revealed about six structures in which calcium carbonate and other carbonates appear in soils: inter-particle meniscus, nodular bonding, encrusting, reinforcing, scaffolding and lining to rootlet holes (Figure 2.16 to Figure 2.19) (Derbyshire and Mellors (1988); Langroudi (2014) and references therein). Different processes lead to the formation of  $\text{CaCO}_3$

in soils, and each process is associated with a special carbonate crystal shape. For instance, authigenic calcites are nodular shaped while Rhizolithic Calcrete carbonates of high  $\text{Ca}^{++}$  component are needle shaped. Langroudi (2014) has reviewed the various mechanisms of  $\text{CaCO}_3$  formation in soils and reported that the shape of a  $\text{CaCO}_3$  cement affects the soils mechanical response to wetting.

Sometimes, other non-clayey bonds such as free oxides (e.g. haematite, goethite, aluminium and magnesium oxides, colloidal ferric oxides) occur together with carbonate and thereby change certain properties of soil. Free oxides are reportedly able to alter soil plasticity, increase water resistivity, reduce compressibility and increase liquid limit. Boardman *et al.* 2001 report that poly-mineral combinations decrease solubility rate (Langroudi, 2014).



*Figure 2.16 a, SEM photomicrograph showing fine needle calcite (x) forming a meniscus film bridging between adjacent silt grains (2.39 m depth). b, SEM photomicrograph showing a meshwork of fine needle calcite forming a meniscus film (x) bridging between adjacent silt grains and forming a 'scaffold' to support clay particles (2.39 m depth). c, SEM photomicrograph of typical macroporous pelletised brickearth fabric, comprising pellets of compact silt grains (with interstitial clay) in an open-packed arrangement. Calcareous brickearth, 2.39 m, Ospringe Pit 1 (adopted from Milodowski *et al.* (2015)).*

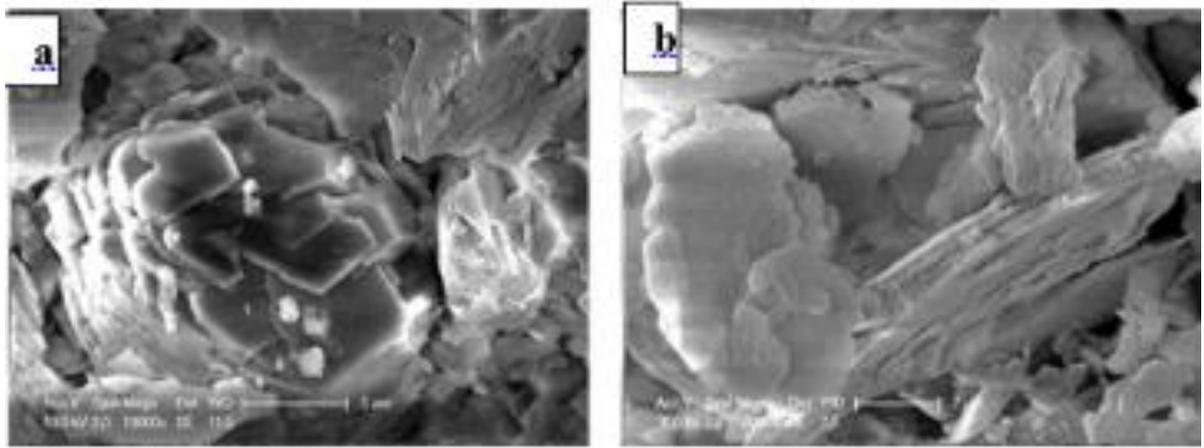


Figure 2.17  $\text{CaCO}_3$  nucleation on kaolinite in electron microscope image **a**, carbonate nodules absorbed by the clay flake surface and overgrowth of carbonate tubular next to the clay laminated aggregate **b**, carbonates bonding with the clay platelets (adopted from Langroudi (2014))



Figure 2.18 Carbonate connectors under scanning electron microscope **a**, authigenic calcite in Melan loess, China (adopted from Derbyshire and Mellors (1988)) **b**&**c**, pedogenic carbonates in Saskatchewan soils, Canada (adopted from Landi et al. (2003)).

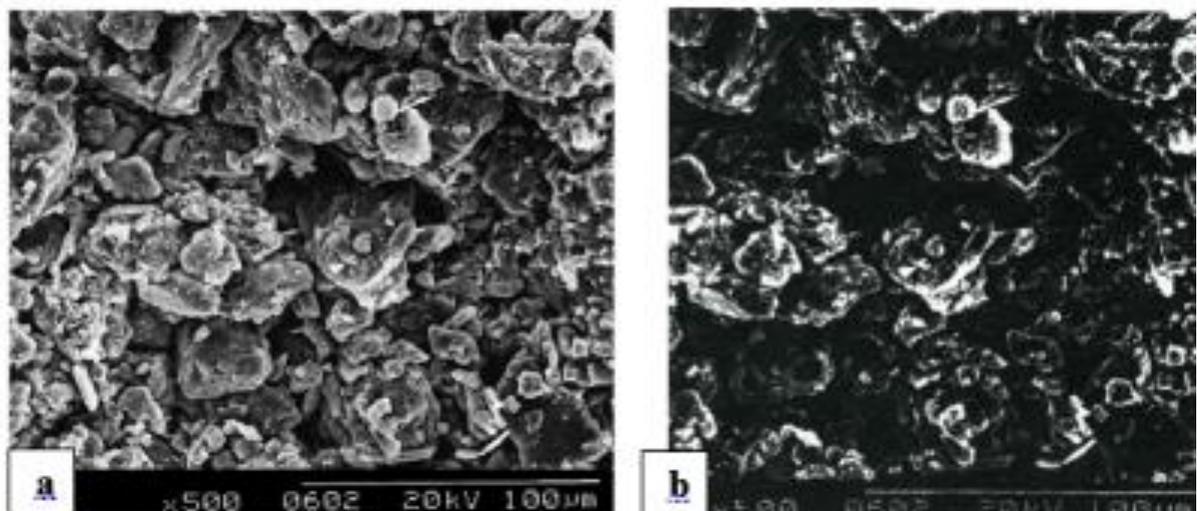


Figure 2.19 SEM of undisturbed Malan loess, (a) vertical face illustrating the characteristic fabric of Upper Pleistocene loess from the Lanzhou region, western Loess Plateau of north China. The fabric consists of a loosely packed quartz silt skeleton, mainly in face-to-face contact, with only minor amounts of clay mineral and some secondary carbonate coatings on some of the larger grains. (b) showing macropores (1.2 void ratio) with abundant carbonate cementation and the absence of supporting clay matrix (after Derbyshire (2001) and (1995), respectively).

The solubility of non-clayey bonds, especially CaCO<sub>3</sub>, has been the subject of many arguments around its role in the collapse phenomenon. There are those who believe that the solubility of CaCO<sub>3</sub> was too slow to demonstrate the sudden volume reduction mechanism, whereas others think CaCO<sub>3</sub> bonding enhances collapsibility.

Osipov and Sokolov (1995) described calcite bonds and other carbonates bonds as low and medium soluble salt bonds, with rates of solubility that range from several hours to several months for low soluble salt dissolution. Mansour *et al.* (2008) also ranked calcium carbonate as the least soluble salt among some common non-clayey bonding elements: calcium chloride to sodium chloride, sodium carbonate, sodium sulfate and calcium carbonate, in descending order of solubility.

In some cases, a particular soil containing CaCO<sub>3</sub> still raises arguments among different investigators. The role of CaCO<sub>3</sub> in the collapsibility of the Pegwell Bay brickearth for example, was questioned by Mellors 1995 while Milodowski *et al.* (2015) emphasized its potential to influence collapse. Milodowski and colleagues based their argument on fabric studies of SEM images that revealed CaCO<sub>3</sub> as the particle-to-particle connector.

The calcareous brickearth of South-East England (2.5m depth, Ospringe Pt. 1) as studied by Milodowski *et al.* (2015) exhibited collapse potential (CP) values averaging 0.5%, 4.2%, and 5.3% for wetting at 100, 200 and 400 kPa, respectively. At a second location (2.5m depth, Ospringe Pt. 2) 100m north of Pt. 1 the results were different. A CP of 3% was recorded for wetting pressures of 100 and 200 kPa while 0.8% was recorded for 400kPa and 0% for 800kPa overburden stress at wetting. The reduction in CP at 400kPa and further down to zero at 800kPa may appear to reveal ‘yielding’ or breaking of intergranular bonds (particularly bonds of clay bridge between silt grains) as loading increased before wetting, apparently reducing void ratios and susceptibility for collapse. Although Pt. 1 and Pt. 2 had similar carbonate contents (10.2 – 12.7 %) other constituents vary as follows: Pt. 1 contained 17.5% clay, 55.5% silt, 26.9% sand, and 0.1% gravel whereas, Pt. 2 had 12.8% clay, 76.1% silt, 9.7% sand and 1.4% gravel. These other factors may well have contributed to the differences in CP. Another potential factor affecting collapse is age of formation; as a time factor relating to the time of formation and the history of alteration and changes undergone (Lin (1995); Mohamed *et al.* (2008); Watts and Charles (2015)).

Numerous researchers have reported significant amounts of CaCO<sub>3</sub> in different collapsible soils, particularly loess. Langroudi (2014) has produced an extensive review of the CaCO<sub>3</sub> contents of more loessial soils; a few examples are presented herein. The literature,

however, is supportive of the impact of CaCO<sub>3</sub> in the collapse behaviour. This notwithstanding, some scholars like Assallay (1998) believe that clay bonds predominantly control collapsibility.

#### 2.3.4 Types of bonding in collapsible soils (loess) and structural bond transformation

Natural loess soils predominate the study of collapsible soils. However, the collapse mechanisms for natural (both loess and others) and artificial collapse soils are similar. It is possible that the proposed (Yang, 1989 in El Howayek *et al.* (2011) and Osipov and Sokolov (1995)) loess bond types (Table 2.2 and Table 2.3 and Figure 2.20) could provide a general understanding for other collapsible soils. Loess bonding can be of various types. The proposal according to Yang 1989 (Table 2.2), recognises two types of bonds: hydro-stable bonding and hydro-labile bonding. Hydro-stable bonding is where the bonding strength does not apparently decrease with water while hydro-labile bonding is easily broken down in contact with water.

Osipov and Sokolov (1995), on the other hand, identified four different bonding elements with varying bonding strength, as summarized in Table 2.3. The weakest being identified as that provided by molecular force. It decreases with increasing water saturation. Bonding by capillary force and ionic-electrostatic force are of similar magnitude, nevertheless, capillary forces only exist at degrees of saturation between 0.35 and 0.80 while ionic-electrostatic force is unstable in the presence of water. The strongest bonding strength can be created by cementation bridges resulting from chemical materials like salts (e.g. CaCO<sub>3</sub>), aluminium and sesquioxides of irons. The water resistance of the bonding forces depends on the types of chemical materials. The transformation of structural bonds in loess under varying degrees of water saturation is presented in Figure 2.20.

Table 2.2 Classification of loess bonding types by Yang 1989 (adapted from El Howayek *et al.* (2011))

<i>Hydro-stable bonding</i>	<i>Hydro-labile bonding</i>
Calcium carbonate	Strongly soluble salts
Hydrous calcium sulfate	Clay cementing agent
Ferrous oxide and ferrous hydroxides	Flocculant
Hydro-stable secondary mica	Non-flocculant
Secondary zeolite	Hydro-labile secondary mica

Table 2.3 classification of structural bonds in loess (adapted from Osipov and Sokolov (1995)).

<i>Forces</i>	<i>Strength of contact between two particles, N</i>	<i>Resistance to water</i>
Molecular	$5 \times 10^{-10}$ to $10^{-8}$	Gradually decrease with increase of water saturation
Capillary	$6 \times 10^{-8}$ to $10^{-7}$	Only exists at degree of saturation between 0.35 – 0.8
Ionic-electrostatic	$5 \times 10^{-8}$ to $4 \times 10^{-7}$	Unstable
Chemical	$5 \times 10^{-7}$ to $5 \times 10^{-6}$	Unstable, stable

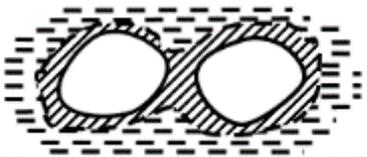
<b>Stage of hydration</b>	<b>Degree of water saturation</b>	<b>Strength of single contact</b>	<b>Scheme of contact</b>
A	<0.15	$\approx 4 \times 10^{-7}$ N	
B	0.15-0.8	$10^{-7}$ - $10^{-8}$ N	
C	>0.8	$10^{-9}$ - $10^{-10}$ N	
 Capillary water		 Adsorbed water	
 Free water		 Solid particle	

Figure 2.20 Structural bonds transformation in loess under water saturation (redrawn after Osipov and Sokolov (1995)).

Osipov and Sokolov (1995), and Klukanova and Frankovska (1995) described two very similar sequences of collapse following wetting. This can be better appreciated if put side by side as in Table 2.4 below. Seven stages were involved according to Osipov and Sokolov, but six stages according to Klukanova and Frankovska.

Table 2.4 reveals that collapse process is both chemically and mechanically driven, depending on soil mineralogy and perhaps the nature of wetting fluid. Nevertheless, over the

years the collapse phenomenon has been exclusively described on the bases of soil mechanics principles; and basic systems (e.g. USCS) for geotechnical characterisation of soils have not considered dissolvable components (e.g.  $\text{CaCO}_3$ ) of soils (Sterianos (1988); Lamas *et al.* (2002)). Thus, almost ignoring the chemical behaviour of the soil.

*Table 2.4 Comparison of collapse sequences according to Osipov and Sokolov (1995), and Klukanova and Frankovska (1995).*

<i>Stage</i>	<i>Osipov and Sokolov (1995)</i>	<i>Klukanova and Frankovska (1995)</i>
1	Formation of hydrated films of adsorbed water and subsequent increase in inter-particle distance	Disintegration of clay coating units, connectors, and aggregates,
2	Destruction of less water-resistant aggregates	Dissolution and migration of carbonates within the pore network,
3	Destruction of most tubular micro-pores (small to medium)	Migration of clay
4	Disappearance of capillary forces	Reduction in carbonate content,
5	Structural collapse	Further compression of soil,
6	Consolidation	Formation of new microfabric of novel basic relations, in which clay aggregates and matrix units replace the coating and connector units.
7	Formation of new contacts	

Considering the arguments around the mechanism of collapse in collapsible soils as related to their inter-particle bonding element, one key aspect not fully considered in the literature is the rate at which collapse may occur. This factor is a function of type and form of bonding element, the chemistry of wetting fluid and the rate of wetting.  $\text{CaCO}_3$  bonding for instance, can exist as three different polymorphs (calcite, aragonite or vaterite) with different solubility. Therefore, considering these as individual bonding elements in relation to the chemistry of wetting fluid will enhance the understanding of their impact on collapsibility. This project aims to provide further understanding to this subject in connection with the role of other key factors of collapsible soils, such as dry density, porosity and clay content.

## 2.4 Predicting Collapse in Soils

Sensitivity of a soil in terms of the rate and magnitude of collapse must be predictable if a geotechnical engineer is to prevent possible damage to infrastructure built with or hosted on

potentially collapsible soils. Various factors are reported to influence the potential of a soil to collapse.

Reznik (2000) identified porosity (void ratio) and moisture content. Ayadat and Hanna (2012) found the following: soil mineralogy and gradation, amount of applied pressures, degree of saturation, density, type of cementing materials, chemistry of pore fluid and the extent of wetting. They further state that the majority of works conducted to investigate the parameters controlling collapse have focused on soil particle size, initial dry density, moisture content (or saturation), and applied pressure. Moisture contents normally falling within those described as partially saturated and unsaturated states except for quick clays. However, soils can also develop secondary porosity when bonding minerals (e.g.  $\text{CaCO}_3$ ) dissolve. This is a potential source of an open structure susceptible to collapse but is often ignored in geotechnical characterization of soils because of the overdependence on mechanical properties.

Several methods of predicting and categorising collapse in soils have been reported by many authors. Apart from reconnaissance studies, these authors have based their methods on three distinct criteria:

- Relationships of fundamental properties like void ratio, degree of saturation, Atterberg Limits, moisture content, and density
- Field tests
- Qualitative and quantitative experimental methods

#### **2.4.1 Reconnaissance**

Reconnaissance studies is useful to gain insight on what to look out for in anticipating collapse. Understanding geological and geomorphological information as well as fill history is necessary, as the assumption for any deposit should be that it is collapsible unless proven otherwise (Jefferson and Rogers (2012); Watts and Charles (2015)). Even all alluvial fans are recommended to be assumed collapsible (Beckwith, 1995 in Jefferson and Rogers (2012)). Geomorphologic information were found to have a strong correlation with collapsibility according to Lin (1995), who also highlighted how age, overburden stress, and degree of saturation and suction affected collapse of loess. Other aspects recommended (Popescu, 1992 in Jefferson and Rogers (2012)) to be considered when assessing soil collapsibility include:



Based on moisture content and Atterberg limits relationship the following equations were proposed. Equation 2.6 by Denisov 1951 and modified by Priklonskij 1952; Equation 2.7 by Gibbs and Bara 1962; Equation 2.8 by Feda 1964 (Alan and Robert (1988); Assallay (1998)).

$$K = \frac{w_0 - PL}{PI} = \frac{w_0 - PL}{LL - PL} \dots\dots\dots 2.6$$

where K is collapsibility index,  $w_0$  is natural water content,  $PL$  is plastic limit,  $LL$  is liquid limit,  $PI$  is plasticity index, and Priklonskij proposed the following K limits collapse categories:

$K < 0$  for highly collapsible soils,  $K > 0.5$  for non-collapsing soils

$K > 1.0$  for swelling soils

$$LI = \frac{w_0 - PL}{LL - PL} \dots\dots\dots 2.7$$

when LI approaches or exceeds a value of unity, collapse may occur

$$K = \left( \frac{w_0}{S_0} \right) - \left( \frac{PL}{PI} \right) \dots\dots\dots 2.8$$

where  $S_0$  is natural degree of saturation.

In 1964, Feda stated that collapse is probable when  $K < 0.85$ , and then in 1966 he employed initial porosity,  $n_0$  to classify collapse reporting soils with  $n_0$  greater than 0.4 as susceptible to collapse (Alan and Robert, 1988).

Based on density and Atterberg limits relationships, the following equations were proposed: Equation 2.9 by Gibbs and Bara 1962; and Equation 2.10 by Anderson 1968 (Assallay (1998)).

$$R = \frac{W_s}{W_L} = \frac{[(\frac{\rho_w}{\rho_d}) - (\frac{1}{G_s})]}{W_L} \dots\dots\dots 2.9$$

where  $R$  is collapse ratio,  $W_s$  is water content at 100% degree of saturation,  $W_L$  is water content at liquid limit,  $\rho_w$  is density of water,  $\rho_d$  is dry density of soil,  $G_s$  is specific gravity of solid particles.

Collapse of soil is imminent on saturation when  $R$  is greater than or equal to unity. A graphical criterion (Figure 2.21) to predict soil collapse was created based on Equation 2.9

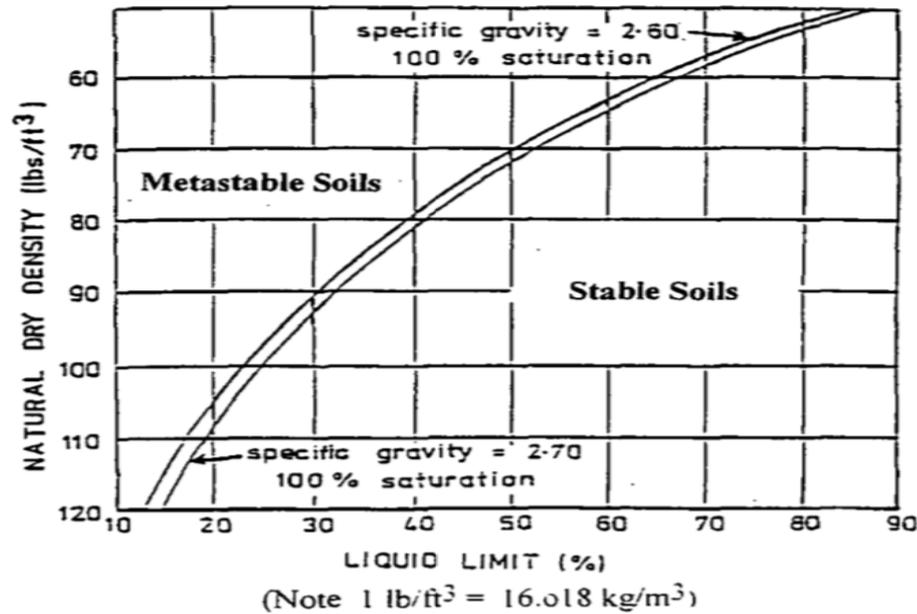


Figure 2.21 Stable and Metastable (collapsible) soil (after Gibbs and Bara 1962) (Assallay, 1998)

Soil dry density has also been used solely to categorize collapse potential. Dry densities less than  $1.28\text{Mg/m}^3$  indicate large settlements to be expected while greater than  $1.44\text{Mg/m}^3$  indicate small settlements to be expected (Clevenger 1958 in Alan and Robert (1988)).

$$R = 5.5 - 3.82\log(LL - PL) - 1.63\log(PL) - 1.24\log(C_u) - 0.918\log P_{10} + 0.465\log\left(\frac{D_{60}}{D_{40}}\right) - 0.45\log\left(\frac{D_{100}}{D_{50}}\right) - 0.303(P_{200}) \dots \dots \dots 2.10$$

where  $C_u$  is uniformity coefficient ( $D_{60}/D_{10}$ )

- $D_{10}$  is particle size corresponding to 10% passing
- $D_{40}$  is particle size corresponding to 40% passing
- $D_{50}$  is particle size corresponding to 50% passing
- $D_{60}$  is particle size corresponding to 60% passing
- $D_{100}$  is particle size corresponding to 100% passing
- $P_{10}$  is fraction passing No. 10 sieve
- $P_{200}$  is fraction passing No. 200 sieve

Equation 2.10 was generated by a computerized step-wise regression programme based on a correlation between collapse ratio (R) and index properties of soil (Anderson 1968, in Assallay (1998)).

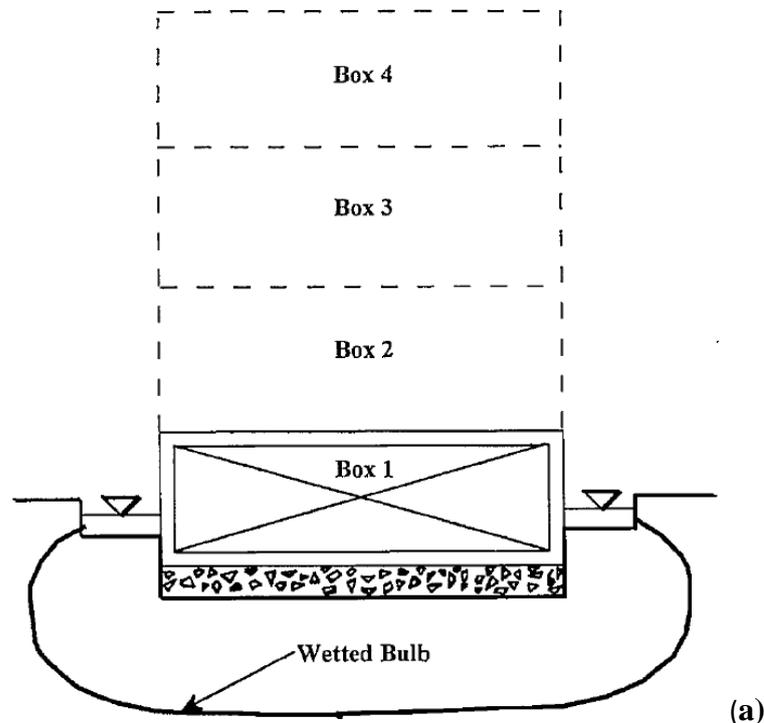
#### 2.4.3 Field predicting methods

Two field collapse-predicting methods were proposed by Jennings and Knight 1957: the “sausage” test and “cylinder” test (Assallay, 1998). According to Assallay, the sausage test involves taking a handful sample from the site and dividing it into two equal parts. One part is wetted and moulded into a ball while the other left dry unmoulded. The volumes of the two are then compared. Collapse is said to be imminent if the volume of the wet ball is less than that of the dry unmoulded part. Accuracy of this test was checked by wetting and moulding the second part, also into a ball to compare the volumes of the two balls. If the same, then the procedures were accurate.

The cylinder test involved sampling a 100mm x 50mm undisturbed sample and dividing it into two equal parts of 50mm x 50mm each. One part is wetted and remoulded by hand into the 50mm x 50mm cylinder while the other remained undisturbed. Collapse was said to be eminent if the height of the wetted remoulded specimen falls below that of the undisturbed (Assallay, 1998). These field methods are only qualitative, and also, do not consider the fact that some bonding agents such as calcite (in some calcareous soils) may not be readily soluble, and collapse may not be predictable with these methods.

There are several field instrumentation methods used to assess collapse. They are generally grouped into in-situ tests such as field vane, standard penetration test (SPT), cone penetration test (CPT), dynamic probing (DP), flat dilatometer (DMT), pressuremeter (PMT) including the Menard pressuremeter (MPM) and the self-boring pressuremeter (SBP); load tests; and geophysical tests (Watts and Charles, 2015). However, the commonly used ones are pressuremeter tests (Schnaid *et al.*, 2004), plate loading tests (Houston *et al.* (1995a); Mahmoud *et al.* (1995)), and geophysical tests (Evans *et al.*, 2004), with each presenting their unique disadvantages. The major disadvantage of in-situ methods is the ground disturbance and point specific nature of assessment (Evans *et al.*, 2004). One vital aspect with any field assessment is the number of tests necessary to satisfactorily characterise the collapse potential (Jefferson *et al.*, 2005). Statistical approaches developed to evaluate the minimum number of tests necessary to adequately characterise a site and its CP, have been discussed by Houston *et al.* (2001).

The conventional plate loading test conducted on or near the ground surface or in shallow trenches (or pits) are commonly used to identify and characterise collapsible soil deposits (Houston *et al.*, 1995a). Although wetting of the soil is done to a considerable depth, the depth is usually not measured. Loads and ground displacement relationships are recorded but no stress-strain relationship is measured (Houston *et al.*, 1995a). A major disadvantage of plate loading test is the non-uniform stress state within the ground region contributing to the displacement, and the attendant difficulty in obtaining stress-strain relationships (Houston *et al.*, 2001). However, recently, the down hole plate tests (Houston *et al.*, 1995b) and the box-plate load tests (Mahmoud *et al.*, 1995) have been developed to determine response to wetting by obtaining wetted stress-strain relationships as presented in Figure 2.22 ((b) shows the effect of partial wetting on actual collapse strain). The procedures to use these techniques are fully described in the respective literatures as well as Houston *et al.* (2001). Some advantages of these tests have been reported to include minimization of sample disturbance, larger soil volume tested and a degree of wetting likely to be comparable to the prototype (Houston *et al.*, 2001). However, there are limitations arising from the amount of overburden that can be applied, and the excavation cost and safety issue that can limit test to shallow depths (Houston *et al.*, 2001).



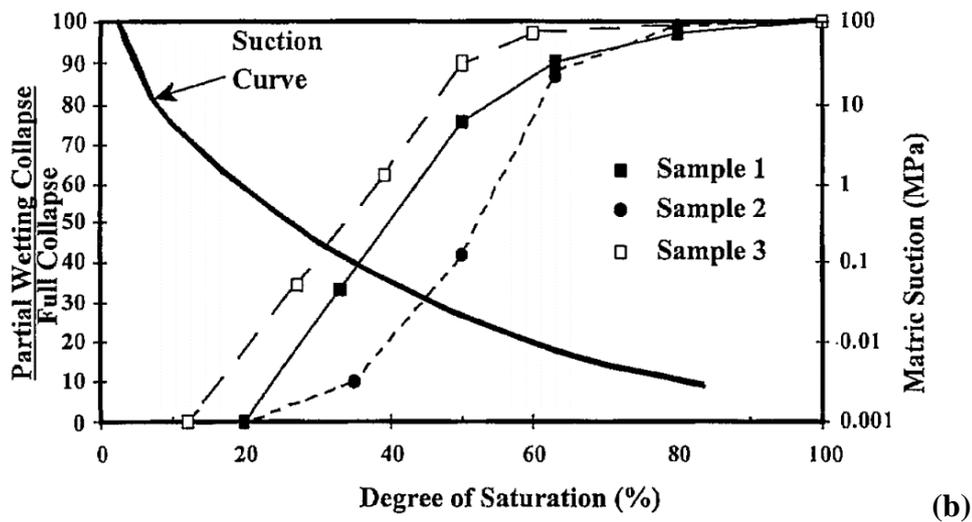


Figure 2.22 (a) Schematic of in-situ collapse testing system using soil boxes on a concrete pad (b) relationship between partial wetting, degree of saturation and matric suction for three silty soils (adopted from Houston *et al.*, (2001))

Geophysical methods such as ground-probing radar and electrical resistivity are occasionally used but, in many circumstances, seismic methods are the most suitable geophysical techniques (Watts and Charles, 2015). Notwithstanding, Evans *et al.* (2004) have demonstrated the usefulness of electrical resistivity (resistivity/moisture content imaging techniques) in measuring moisture content variation in a red clay embankment located in Kenya, and to monitor water movements during a field “hydro-collapse” trial of loess material in Kent. Stiffness changes in the Kent loess material caused by wetting under an applied load, were also monitored by shear-wave transducers installed at depth intervals, to complement the resistivity/moisture imaging (Evans *et al.*, 2004). Additionally, critical properties of collapsible soils such as density and porosity are indirectly obtainable from borehole geophysical logging techniques like resistivity, acoustic, nuclear, and self-potential logs (McDowell *et al.*, 2002). Wyllie *et al.* (1958) proposed an empirical equation (2.11), which has been used for many years to estimate porosity of saturated soil deposits from sonic logs. Nelson (1996) has further described recent procedures to estimate porosity and water content using sonic geophysical logs. Recently, Abatan *et al.* (2016) related acoustic data with estimated porosity data revealing great influence of porosity on  $V_P$  and  $V_S$  with both decreasing with increasing porosity.

$$\frac{1}{V_P} = \frac{n}{V_f} + \frac{(1+n)}{V_m} \dots\dots\dots 2.11$$

where,  $n$  = fractional porosity,  $V_P$  =  $P$ -wave velocity,  $V_f$  = velocity through the pore fluid, and  $V_m$  = velocity through the matrix.

A seismic event produces body-waves and surface waves. While surface-waves travel on the ground surface (mainly on water bodies), body-waves (P (compressional) and S (shear)) travel through the earth's interior, with the wave velocity depending on the mechanical properties of the material it is travelling; the stiffer the material, the faster the wave propagation (Watts and Charles, 2015). However, due to the slow velocities (335 m/s) of near-surface P-waves, they are difficult to measure (Baker *et al.*, 1998), thus, shear wave velocity and damping are of particular interest (Watts and Charles, 2015). Notwithstanding, near-surface waves velocities like P-waves velocity,  $V_P$  and Rayleigh waves velocity,  $V_R$  can be related with shear-waves velocity,  $V_S$  to obtain soil parameters such as dynamic shear modulus,  $G_{max}$  and dynamic constraint modulus,  $D_{max}$  as shown in Equations 2.12 to 2.14 (Charles and Watts (2002); Watts and Charles (2015)).

$$G_{max} = \rho V_S^2 \dots\dots\dots 2.12$$

$$D_{max} = \rho V_P^2 \dots\dots\dots 2.13$$

$$V_S = 1.05V_R \dots\dots\dots 2.14$$

where  $\rho$  is the soil density,

According to Charles and Watts (2002), shear-waves velocity in water equals zero while P-waves velocity in water is approximately 1500 m/s, a considerably higher value than those of most soils and fills. Evans *et al.* (2004) noted that P-wave velocity is governed by undrained bulk modulus. Therefore, for a saturated soil the dynamic constrained modulus will be related to the undrained behaviour of the soil (Charles and Watts, 2002). Shear-wave velocity varies as a function of shear modulus and density (Charles and Watts (2002); McDowell *et al.* (2002)). Therefore, shear-waves can be useful also in saturated conditions (Evans *et al.*, 2004).

Seismic waves are usually generated by either impact (a sledgehammer or weight drop producing a transient pulse), or vibration (producing continuous waves) on the ground surface or in boreholes. These seismic waves are detected by array of geophones fixed either on the ground surface or in boreholes, and a cross-hole tomography is used to measure the waves velocities over a plane section within the ground (Charles and Watts, 2002). Different methods of data analyses are used depending on the source of the seismic wave energy. Spectral analysis

of surface waves (SASW) for impact generated waves, and continuous surface wave system (CSW) for vibratory source waves (Charles and Watts (2002); Watts and Charles (2015)). Examples of some work carried out with these seismic techniques to measure shear stiffness and surface seismic waves can be seen in Matthews *et al.* (1996) and Matthews *et al.* (1997).

According to Charles and Watts (2002) and Watts and Charles (2015), the advantages of geophysical testing may include its non-destructive nature, ability to conduct from ground surface, ability to measure representative values of parameters in a heterogeneous fill, and rapid nature of data acquisition. They also noted limitations to include the necessity to interpret geophysical measurements alongside other forms of ground investigation. Another limitation noted is that, for deformation interpretation, measured dynamic properties need to be related to the static properties required.

#### **2.4.4 Experimental predicting methods**

Houston *et al.* (2001) and Jefferson *et al.* (2005) suggest that the most effective way to assess collapsibility of a soil is by laboratory collapse testing. Assallay (1998) also noted that empirical methods (relationship of properties) are less reliable compared to laboratory approaches, owing that the latter takes soil structure pattern into consideration. Laboratory methods give both qualitative and quantitative results in that amounts of collapse can also be predicted. Two laboratory methods commonly used are standard oedometer and triaxial methods. Both methods take into consideration key conditions such as the dry density, the initial water content, and the applied load, which control densification of most soils under wetting. Both methods also have provision for inundation. However, Houston *et al.* (2001) argue that traditional oedometer tests suffer from effects of specimen disturbance and most times reach saturations not normally encountered in the field. Jefferson *et al.* (2005) therefore, recommend that traditional oedometer tests should at best be considered index tests and a field trial should be conducted for full collapse assessment.

In the triaxial test, inundation is through the lower end of the specimen, while it is complete submersion of specimen in the oedometer (Lutenegger and Hallberg (1988); Assallay (1998)). Normal laboratory procedures are employed: soaked-after-loading is employed in one-dimensional oedometer test and three-dimensional triaxial tests, where specimen is loaded incrementally until equilibrium is reached and then inundated. This is referred to in the oedometer testing method as “single oedometer”. A second procedure involving wetting at loading time have also been adopted in the oedometer method. Figure 2.23 demonstrates these

procedures in predicting collapse. Details of the experimental procedures and other recommendations can be found in Assallay (1998) and Standards for soil testing.

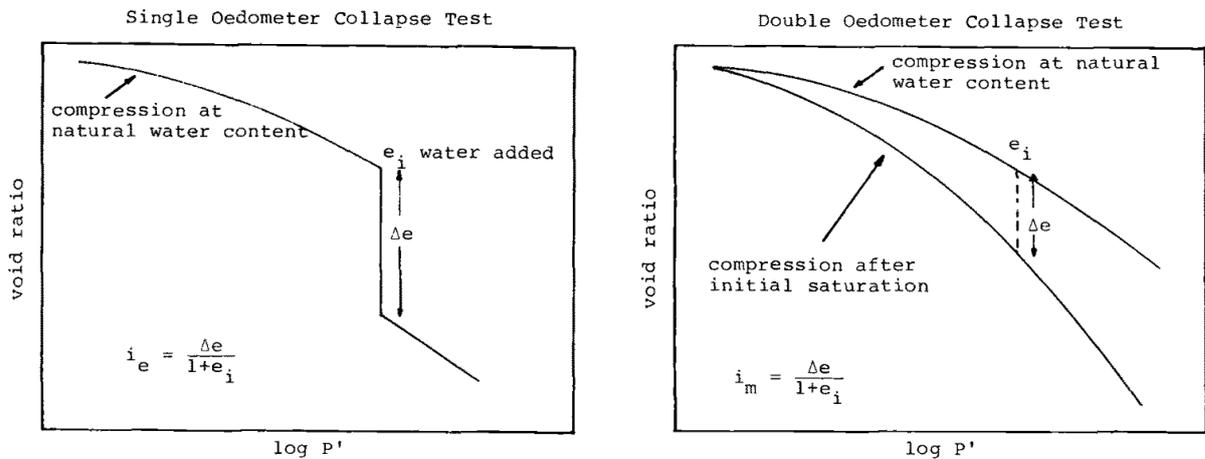


Figure 2.23 Predicting collapse using single and double oedometer procedures (after Lutenegeger and Hallberg (1988))

Incremental vertical stress before inundation up to 300kN/m<sup>2</sup> in one-dimensional single oedometer compression test has been recommended as most meaningful to geotechnical engineers for predicting collapse in soils (Abelev 1948 in Alan and Robert (1988); Lutenegeger and Hallberg (1988)). Their methods involved applying approximately 300kN/m<sup>2</sup> vertical stress at natural moisture content and then inundating the specimen. Lutenegeger and Hallberg (1988) have reported collapse as imminent if volume decreases at constant load, and that the coefficient of collapse, *i*, determined by Equation 2.15 yielding values greater than 0.02 (2%) as indicative of danger with respect to the United States perspective of collapse. However, different regions have adopted different values to delineate collapsible soils. For instance, in Chinese context soils with CP equal to or greater than 1.5% are considered collapsible (Derbyshire *et al.*, 1995) while in the British viewpoint, soils exhibiting CP greater than 1% are regarded as collapsible or metastable soils (Bell and Culshaw, 2001).

$$i = \frac{\Delta e}{1 + e_i} \dots\dots\dots 2.15$$

where Δe = change in void ratio upon wetting, e<sub>i</sub> = void ratio at beginning of wetting

Knight 1963 (cited in Alan and Robert (1988)) and Jenning and Knight 1975 (reported in Mohamed *et al.* (2008) have rather recommended triggering collapse by wetting at 200kPa

incremental stress, and defining CP as in Equation 2.16. However, several workers around the globe have adopted various wetting vertical stresses, in the range of 50 to 600 kN/m<sup>2</sup>.

Therefore, rather than a recommended stress, a stress level close to estimated foundation stresses for proposed projects should be preferred for collapse testing (Alan and Robert, 1988). Again, from these two experimental definitions of collapse potential (Equations 2.15 and 2.16), choice of initial void ratio is a contradictory one. While Equation 2.15 uses void ratio immediately before wetting, Equation 2.16 preferred natural void ratio. This is capable of producing inconsistent results, especially if compression before wetting is large and Equation 2.15 has not taken this into consideration and Equation 2.16 would overestimate collapse as it considers pre-wetting compression as collapse.

$$i_c = \frac{\Delta e_c}{1 + e_o} \dots\dots\dots 2.16$$

where  $\Delta e_c$  = change in void ratio due to wetting,  $e_o$  = natural void ratio

Furthermore, since the oedometer test is performed as a one-dimensional test, collapse can also be defined in terms of percentage change in specimen height as in Equation 2.17 (ASTM, D 5333 - 03).

$$i_c = \frac{\Delta h}{h_o} \times 100 \dots\dots\dots 2.17$$

where  $\Delta h$  = change in specimen height due to wetting,  $h_o$  = specimen height before wetting.

Basma and Tuncer (1992) proposed a collapse-predicting model which reproduces real field and laboratory situations. A multiple regression analysis was performed using experimental data obtained by conducting series of collapse tests. The best-fit equation obtained was Equation 2.18.

$$CP = 48.496 + 0.102C_u - 0.457w_i - 3.533\gamma_d + 2.80 \ln(p_w) \dots\dots\dots 2.18$$

where CP is collapse potential in percent;  $C_u$  is coefficient of uniformity of the soil;  $w_i$  is initial water in percent;  $\gamma_d$  is compaction dry unit weight in kN/m<sup>3</sup>;  $p_w$  is pressure at wetting in kPa. And the correlation coefficient ( $r^2$ ) and the standard error of estimate (SEE) for the equation are 0.94 and 1.94, respectively.

Solutions of Equation 2.18 were presented in a nomographic chart (Figure 2.24) for simplicity in practical applications (Basma and Tuncer, 1992). The purposes of this chart are:

- (1) the straightforward determination of the collapse potential (CP) for different soils under various conditions (dashed line 1);
- (2) to determine the critical initial water content above which no collapse is expected (dashed line 2); and
- (3) to estimate the critical applied pressure below which the soil will not collapse (dashed line 3).

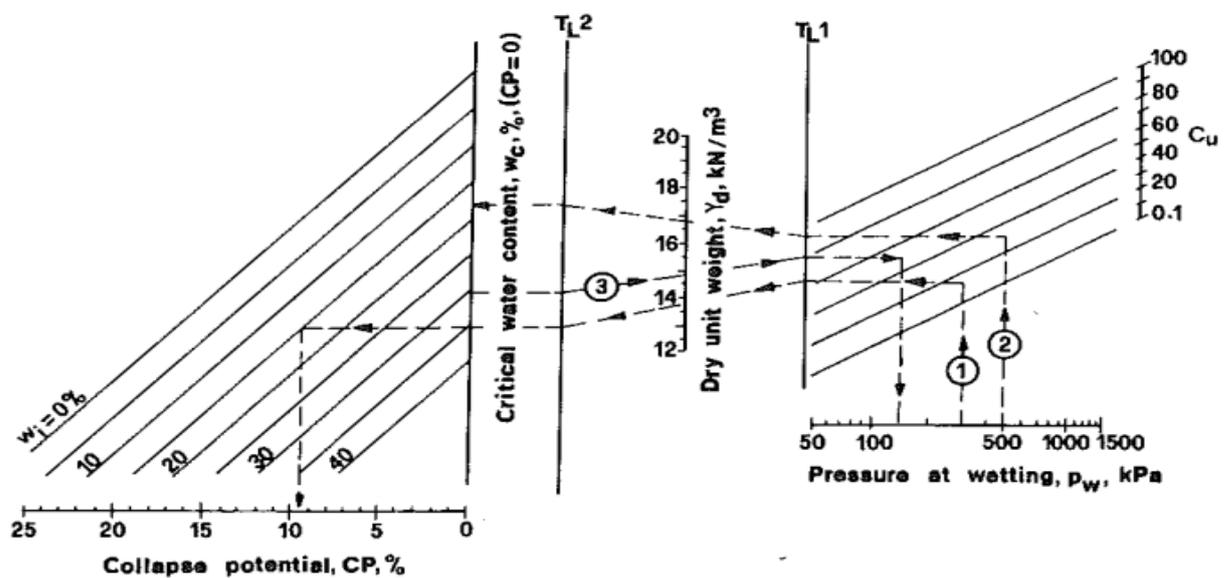


Figure 2.24 Nomograph for determining collapse potential (after Basma and Tuncer (1992))

Ayadat and Hanna (2012) made an extensive comparison of the different collapse predicting methods available in the literature. They used various soils of different depositional histories and some of the soils adopted to model certain of the predicting methods to evaluate other proposed methods. They found many comparative contradictions between most of the proposed methods, and concluded on a generally note that nearly no one criterion would predict the collapsibility of any soil accurately. Delage *et al.* (2008) also compared some of the proposed indirect methods with results of experiments they carried out and found many of them incomparable. Thus, a combination of several techniques is necessary to effectively predict collapse (Jefferson *et al.* (2005); Watts and Charles (2015)).

## 2.5 Formation, Occurrence and Geotechnical Properties of Calcareous Soils

### 2.5.1 Formation and occurrence

In order to put in right and understandable perspective, it is important to identify what calcareous soils are. While some authors, e.g. Fookes (1988) described every soil containing calcium as calcareous, others distinctly describe calcareousness to mean partly or mostly containing calcium carbonate ( $\text{CaCO}_3$ ). In this project, the latter is preferred. Therefore, according to Thompson (2007); Kalra and Maynard, 1991 and Day, 1983 cited in Kishchuk (2000) calcareous soils are defined as any soil containing significant proportion of  $\text{CaCO}_3$  (also referred to as carbonate soils).

To this end, many soils spanning the globe are calcareous in nature. For instance, the commonly studied collapsible soils (loess) generally contain  $\text{CaCO}_3$  contents ranging between 1 – 30 % (Jefferson *et al.* (2005); Li *et al.* (2016)). The Egyptian loessial soils containing 21 – 30 %  $\text{CaCO}_3$  are specifically described as calcareous loess (El-Sohby *et al.*, 1988). Delage *et al.* (2008) also described the main loess deposits of Northern France which contain up to 18%  $\text{CaCO}_3$  as calcareous loess.

For the purpose of this project, calcareous soils have been considered as having two major components, carbonate mineral and detrital mineral components. Carbonate particles occur generally as micritic (< 4 microns) or microsparitic (5–20 microns) crystals in the micromass of calcareous soils (Stoops, 2010).

The formation of calcareous soils considered in this section relies mainly on processes leading to the precipitation of the carbonate component. Calcareous soils have two basic distinct origins. The lithogenic (originating from weathering of parent rock referred to as primary carbonate) or pedogenic (originating from within soil commonly referred to as secondary carbonate). Despite that all carbonate soils have a unifying basic chemistry,  $\text{CO}_3$  component that gives them their identity, their formation processes differ according to Fookes (1988) who grouped these soils based on biological, mechanical and chemical origins:

- The accumulation and subsequent cementation of skeletal carbonate materials, such as shells, coral reefs and related but broken organic material form a rock of primarily biological origin. These are referred to as skeletal or organic carbonates (e.g. bioclastic) (Pettijohn (1975); Semple (1988)). According to Semple, carbonate-rich shells of marine organisms such as planktonic and benthic plants and animals fall to seabed. The shells are reworked and redeposited producing bioclastic soils having reduced grain sizes with high angularity

- Older carbonate materials are broken down, transported and deposited and subsequently cemented forming a rock of mechanical origin
- Physical and chemical conditions such as temperature, pressure and concentration of water bodies, result in precipitation of carbonate material, which either forms a rock or cement of chemical origin. Carbonates of this nature are referred to as evaporitic or non-skeletal or inorganic carbonates (e.g. oolites) (Pettijohn (1975)). They formed through the precipitation of  $\text{CaCO}_3$  from the carbonate saturated or supersaturated warm waters of the equatorial regions (Semple, 1988). This is in agreement with reported evaporitic carbonate (calcium-rich caliche and calcrete) formed in the arid climates (Pettijohn, 1975). Semple noted that the carbonate mineral precipitated coat the detrital particles, and that particles are mostly equidimensional or rounded. However, they also occur as individual particles within pore spaces of host soil (Al-Kaysi, 1983).

It is important to note that the above-described carbonates can either form as pure carbonates or mix with detrital materials. Pettijohn (1975) noted that carbonate accumulations appear in various similar geological environments, and only the associated detrital component and other minerals are used to unravel the depositional environment. Texture was the basic distinguishing property. Based on this he noted five kinds of calcareous accumulations widely distributed in the modern world: shallow-water marine carbonates, deep-sea carbonates, freshwater carbonates, evaporitic carbonates, and eolian carbonates.

The evaporitic carbonate form in regions of low precipitation (semi-arid and arid regions) and are important climatic index. Caliche and calcrete are the common types: they are impure lime-rich deposits, with varying carbonate content ranging between 10% or less and nearly 100% (Netterberg, 1982). They form from the absorption of lime-bearing waters to the surface where, by evaporation, lime-rich caliche or calcrete is formed. Eolian carbonates form from build-up of sand-debris from offshore reefs on beaches and in dunes associated with these beaches. Eolian carbonates or calcareous eolianites are self-cementing and occur in Bermuda and on many of the Bahama Platform islands (Pettijohn, 1975).

Generally, calcareous clays are extensively reported in literature to be widely distributed globally. However, the conditions that favour their formation are believed to be those environmental and climatic ones prevalent in the arid and semi-arid regions of the world. These regions lying approximately within latitudes 30 degrees north and 30 degrees south (Agarwal *et al.*, 1977). However, some calcareous soils have been reported outside these latitudes. For instance, calcareous soils predominate most areas of the Southwestern United States of America and Mexico such as Arizona and New Mexico. They formed from infiltration of surface waters

and precipitation during periods in the past several million years when arid and semiarid climates prevailed in those areas. However, some others have also formed in recent geological time (Beckwith and Hansen, 1982).

In southern Africa, areas where arid and semiarid conditions prevail, such as parts of the Republic of South Africa and Namibia (South West Africa), calcareous soils occur, and have been used for road construction purposes. These calcareous soils are usually dominated by micritic calcite or dolomite, which depending on the stage of development, may occur as individual crystals, silt, and aggregation of sand-sized crystals and porous, carbonate-bonded detrital material (Netterberg, 1982).

In the majority countries of the Near East (or Middle East) Region, extensive occurrence of calcareous soils were reported by Kadry (1973). The Great Konya Basin in the south of Anatolian Plateau in Turkey called the Lacustrine Plain, was reported (De Meester, 1973) to have highly calcareous clayey sediments mapped as Steppe Marl Soils, Marsh Soils and Playa Marl Soils. Calcareous clays have also been reported in Cyprus (Nalbantoglu and Gucbilmez, 2001). In the UK, sections of the brickearth deposits of south Essex and Kent have been described as possessing some characteristics of calcareous silty clays (Milodowski *et al.* (2015); Bell and Culshaw (2001) Jefferson *et al.* (2001)).

In Italy, several clay soils including the popular Pisa Clay (of the Leaning Tower of Pisa), are known to be calcareous in nature. Some of these and their characteristics reported in Jamiolkowski *et al.* (1995) are presented in Table 2.5.

Table 2.5 Characteristics of some Italian calcareous clays (redrawn after Jamiolkowski *et al.* (1995))

Soil	Geological age	Sedimentation	CaCO <sub>3</sub> content (%)	Void ratio, e	Porosity, n (%)
Fucino clay	Pleistocene	Lacustrine	10-30 (*) 50-75 (**)	1.6-3.0	40
Panigaglia clay	Holocene	Marine	12	1.4-1.8	50
Pisa clay	Pleistocene	Estuarine	5-12	0.8-1.8	44
Avezzano clay	Pleistocene	Lacustrine	50-75 (*) 75-100 (**)	1.0-1.8	46
Montalvo di Castro clay	Pleistocene	Marine	14-34	0.6-0.8	40
Garigliano clay	Holocene	Alluvial	18-32	0.9-1.2	58
(*) depth > 25m			(**) depth < 25m		

The distribution of calcareous soils is becoming even wider especially as new findings reveal precipitation of large amounts of secondary  $\text{CaCO}_3$  from brownfields and concrete demolition sites and those used for carbon sequestration. Anthropogenic calcareous soils are developing in urban soils such as in brownfields and concrete demolition sites and in cement- or lime-treated subgrades. Carbon capture has occurred in numerous soils treated with calcium rich substances such as lime, Portland cement or concrete, precipitating significant amounts of  $\text{CaCO}_3$  in those soils. For example, carbonation study carried out in the arid part of Botswana on a road base stabilised with lime confirmed conversion of lime to calcium carbonate to various degrees (Bagoniza *et al.* (1987)).

Many other authors including Netterberg (1984) have also confirmed the transformation of lime-treated soils to calcareous/carbonate soils. Renforth *et al.* (2009) reported secondary (pedogenic)  $\text{CaCO}_3$  contents reaching 19.9%, and 40% by weight of soils in an urban brownfield in Newcastle upon Tyne, and former steelworks site in Durham, respectively, both in NE England. They also estimated huge amounts (~12.7Mt) of carbonates (resulting from construction and demolition wastes) stored in brownfield sites of the UK owing to the country's industrial heritage.  $\text{CaCO}_3$  contents up to 44% by weight of soil have also been reported by Washbourne *et al.* (2012) to precipitate from construction and demolition wastes at the brownfield site, Science Central, located in the urban centre of Newcastle upon Tyne, UK.

### 2.5.2 Geotechnical characterization

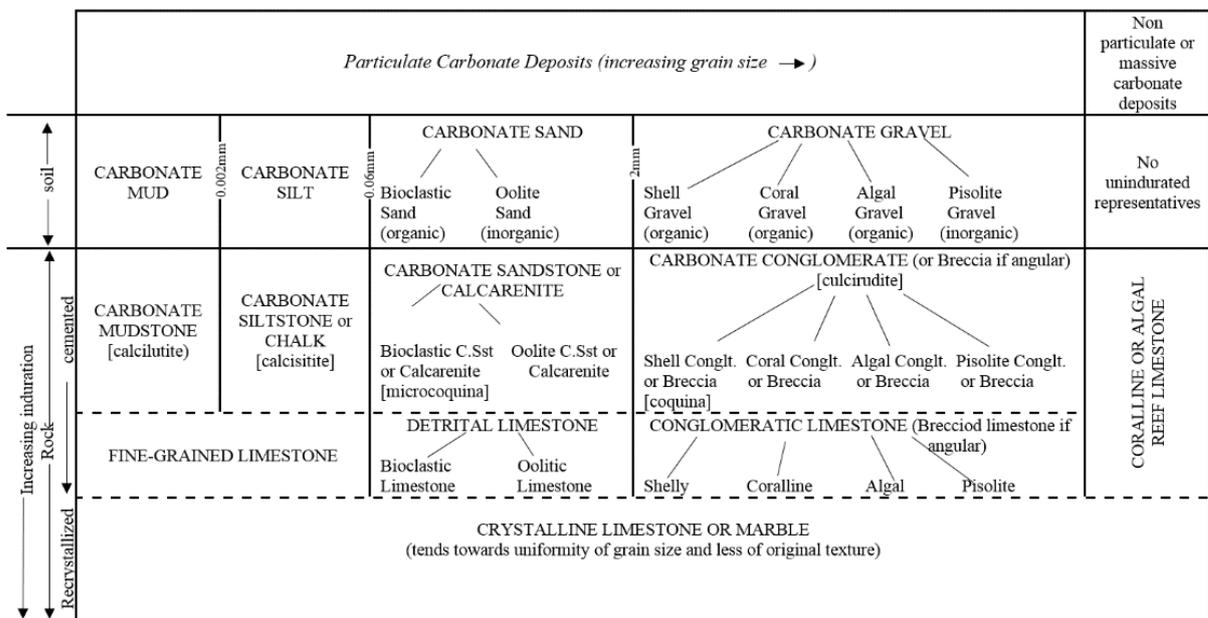
From a geotechnical perspective, characterization of soils is preferred to be based on index properties, which discriminate on mechanical behaviour. For non-calcareous soils, index properties are normally based upon field identification and fairly inexpensive simple laboratory experiments. The index properties used for characterizing such soils fit into two main categories. First is soil grain property, which deals with discrete particle's size, shape, mineralogy and crystal structure while the second category features soil aggregate properties, considering particles microstructure and assemblages.

Before about the last five decades, available systems such as the Unified Soil Classification System (USCS) for instance, developed for soils classification could not be used for carbonate/calcareous soils because they did not consider their distinctive properties. In addition, classification systems and terminology created by geologists for calcareous soils seemed to give little description of the mechanical characteristics of the soils, so were of limited use to geotechnical engineers (Sterianos, 1988). Therefore, this lack of geotechnical standards or systems with precise description and choice of terminology for calcareous sediments often

resulted in misrepresentation. It led to wide discrepancies in description of calcareous sediments by different engineers, where the same sample could be described by one engineer as limestone whilst another describing it as chalk or marl or even clay (Fookes and Higginbottom, 1975).

Another concern was that calcareous/carbonate soils have varying engineering properties from location to location, and even on the same sediment column, especially with the biogenic carbonate deposits. A simple example is a Steppe Marl soil described as calcareous silty-clay studied by De Meester (1973) posing much difficulty in interpreting common grain-size distribution. These variations have posed a lot of limitations in describing their mechanical characteristics using any single method, and has led to the development of various experimental models that describe the observed characteristics of some types of these soils (Rashidian and Hassanlourad, 2014).

Numerous researchers have investigated the mechanical characteristics of carbonate soils in the last few decades. Various schemes proposed for their engineering characterization (e.g. Fookes and Higginbottom (1975) (Figure 2.25); Clark and Walker (1977); Fookes (1988); Semple (1988); Renfrey et al. 1988 cited in Carter *et al.* (1999)) have based on a combination of a number of parameters such as amount of carbonate, structure, consistency or cementation, particle size, void ratio, strength and compressibility.



[established alternative names are in square brackets]

(a)

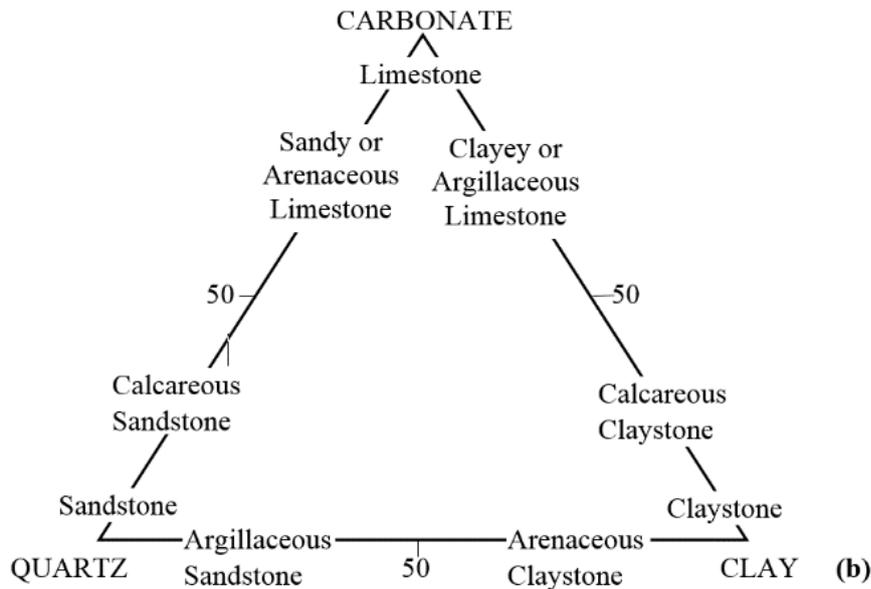


Figure 2.25 Proposed classification for engineering purposes (a), pure carbonate sediments (b), impure carbonate, sandstone and clay sedimentary rocks (adapted from Fookes and Higginbottom (1975))

The Fookes and Higginbottom (1975) engineering characterization system was later modified by Clark and Walker (1977) and further modified by Beringen *et al.* (1982) as presented in Figure 2.26 and Figure 2.27, respectively.

CARBONATE CLASSIFICATION								
Degree of Induration	Additional Descriptive Terms Based on Origin of Constituent Particles				Total Carbonate Content, % (constituent particles plus matrix)			
	Not Discernible	Bioclastic (organic)	Oolitic (inorganic)	Shell (organic)		Coral (organic)	Algal (organic)	Pisolitic (inorganic)
	Increasing Grain Size of Particle Deposit →							
	0.002mm	0.074mm	4.76mm	76mm				
(Soils) Non-Indurated	Carbonate clay	Carbonate Silt (1)	Carbonate Sand (1)	Mixed Carbonate and Non-carbonate Gravel (2)	50%			
	Calcareous clay (3)	Calcareous Silt (1)	Calcareous Sand (1)		12%			
(Rock) Moderately Indurated	Clay	Silt	Silica Sand	Gravel	88%			
	Limestone	Limestone	Limestone	Limestone Conglomerate or Breccia	50%			
	Argillaceous Limestone	Fine-grained Limestone	Argillaceous Limestone					
	Calcareous Claystone	Calcareous Siltstone	Calcareous Sandstone	Calcareous Conglomerate or Breccia	12%			
	Claystone	Siltstone	Sandstone	Conglomerate or Breccia				

Terms Related to Carbonate Classification	Degree of Cementation
Algal – Composed of the remains of calcareous-secreting algae.	(a) Well cemented – cannot be manually broken but grains can be dislodged.
Arenaceous – Having a notable proportion of detrital quartz sand or silt.	(b) Weakly (or lightly) cemented – can be manually broken down without difficulty; individual grains can be dislodged.
Argillaceous – Having a notable proportion of clay.	(c) Moderately cemented - intermediate
Authigenic – Formed in place by chemical or biochemical action.	<b>Notes:</b>
Bioclastic – Consisting of fragmental remains of organisms.	(1) Non carbonate constituents are likely to be siliceous apart from local concentration of minerals such as feldspar and mixed heavy minerals.
Coral – Calcareous skeleton of a coral or group of corals.	(2) In description, the rough proportions of Carbonate and non-carbonate constituents should be quoted and details of both the particle minerals and matrix minerals should be included.
Detrital – Derived of pre-existing rock fragments.	(3) Calcareous is suggested as a general term to indicate the presence of unidentified carbonate.
Oolitic – Made up of Ooliths (0.25 to 2mm, spherical particle, usually carbonate).	When mineral identification is possible, calcareous referring to calcite or alternative adjectives such as dolomite, or aragonite, etc.
Pisolitic – Made up of pisoliths (2 to 10mm round particles usually carbonate).	
Shell – The generally hard rigid covering of an animal, commonly calcareous.	
Siliceous – Containing abundant quartz or silica, generally cryptocrystalline.	

Figure 2.26 classification of carbonate sediments (adopted from Clark and Walker (1977))

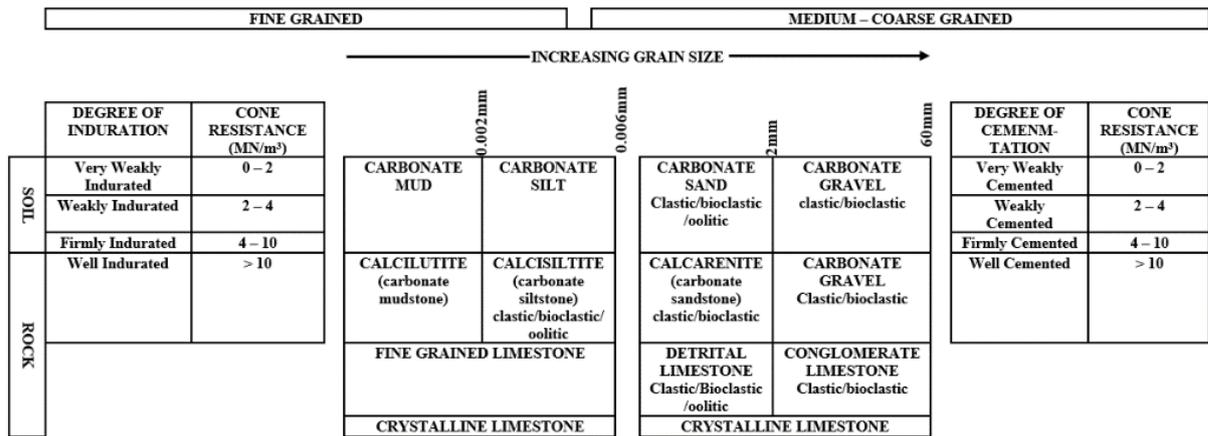


Figure 2.27 classification of carbonate sediments (90-100% carbonate) (adopted from Beringen *et al.* (1982))

Figure 2.26 and Figure 2.27 were based on particle size, carbonate content, degree of cementation or induration, and geological origin of carbonate. A little emphasis was also made on bedding and lamination, colour, and composition of non-carbonate material.

- Particle size - They adapted the words, "clay or mud", "silt", "sand" and "gravel" to indicate particle size, as for non-carbonate sediments
- Carbonate content – Beringen *et al.* (1982)'s system (Figure 2.27) relate to soils containing 90 to 100 % CaCO<sub>3</sub> but for lower contents they proposed changing the prefix (carbonate) as follows:

<u>CaCO<sub>3</sub> content (%)</u>	<u>Prefix</u>
50 to < 90	siliceous carbonate
10 to < 50	calcareous silica
0 to < 10	silica

- Degree of cementation or induration – the hardening of sediments is referred induration. It happens through processes of diagenesis such as cementation, pressure, heat, etc. Although, induration used to be a term applied to carbonate clay cementation not distinguishable from natural apparent cohesion while cementation was used for sands or silts. However, the degree of cementation has been adopted for all soils and measured on the basis of unconfined compressive strength (Clark and Walker, 1977) and cone resistance (Beringen *et al.*, 1982)

- Geological origin of carbonate component – the following terms were adopted: clastic - particles of inorganic origin; bioclastic - particles of organic origin (e.g., fossils and fossil fragments); oolitic - spherical particles coated with precipitated CaCO<sub>3</sub>; pelltitic - elliptical particles coated with precipitated CaCO<sub>3</sub>; and reefoidal - particles consisting of old reef fragments (Fookes and Higginbottom, 1975).

Datta *et al.* (1982) applauded the foregoing systems for covering the entire spectrum of carbonate soils and rocks. More so, they heavily criticised those schemes following a study conducted, stating that “*in covering the width, they seem to lack the depth necessary for serving as an indicator of engineering behaviour*”. They pointed out the following as limitations to the schemes:

- Susceptibility to crushing was not considered in the schemes. As observed from their investigation Datta *et al* noted that two different sands which would both be described as "bioclastic carbonate sands" in Figure 2.26, exhibited significantly different engineering behaviours on the account of their difference in particle crushing
- The terminology used do not give indication of the soils engineering behaviour. For instance, the terms "clayey carbonate silt/mud" and "calcareous silt/clay" used to describe two soils studied by Datta *et al* (Figure 2.28) did not specify that the former soil is uncemented and behaves similar to a normally consolidated soil while the latter is cemented and demonstrates apparent overconsolidation
- The classification schemes have neither specifically stated the significance of nor attempted to include the typical classification parameters like gradation and plasticity (e.g. consistency limits as in the USCS)
- The usefulness of carbonate content as an index property was also noted to be limited. The amount of carbonate material present does not solely control the engineering behaviour, but its nature often plays a significant or dominant role. For calcareous soils, the degree of cementation and susceptibility to either collapse or exhibit other forms of behaviours can also be a function of the type of CaCO<sub>3</sub> polymorph present. Soils composed of aragonite, the unstable form of CaCO<sub>3</sub>, will behave differently from those composed of calcite.

Datta *et al.* (1982) believed that it was too early to propose a geotechnical characterization system for carbonate soils because of the following difficulties. Instead, only a system of

description incorporating cementation, grain size distribution, plasticity, nature of both carbonate and non-carbonate components, may be formulated. Thus Figure 2.28. The difficulties they recognised included (a) lack of method to readily identify and quantitatively express the degree or consistency of cementation (b) lack of method to simply quantitatively assess the susceptibility to crushing of carbonate grains - visual or microscopic studies merely give qualitative indication of crushability and (c) lack of clear knowledge of the influence of carbonate material on different non-carbonate components of carbonate soils.

Description of	Remarks
1. Cementation (a) No cementation (b) Weak cementation (c) Strong cementation (i) Uniform (ii) Partial	The soil has a soft rock-like appearance. Unconfined compressive strength should be indicated. The soil contains cemented aggregates – this should be noted.
2. Grain Size Distribution (GSD) and Plasticity (a) Grain size distribution  (b) Plasticity	For strongly cemented soil, GSD is not very relevant; for uniform cementation, size of constituent particles should be indicated; for partial cementation, GSD of soil after removing aggregates should be indicated and size and proportion of aggregates noted separately. For fine-grained soils in which intra-particle voids cause error in GSD and Atterberg Limits, field classification procedures may be used for providing the relevant information in a qualitative sense.
3. Nature of Carbonate Component (a) Carbonate content  (b) Particle size of carbonate material  (c) Particle characteristics and origin  (d) Mineralogy (e) Geologic name	Soils having more than 30% carbonate content should be termed carbonate soils.  The carbonate content in the sand and in the silty-clay fractions should be determined separately and indicated. Microscopic studies mentioned below will also give information about particle size. Microscopic studies – optical microscope for sands and scanning electron microscope for fine-grained soils should be conducted. Presence of thin-walled material and intra-particle voids should be highlighted. X-ray diffraction analysis should be performed. If possible, to identify, the geologic name may be indicated.
4. Nature of Non-carbonate Component  (a) Particle size  (b) Particle characteristics (c) Mineralogy	Information on non-carbonate material is determined by dissolving the carbonate material in HCl, separating the remaining soil, and conducting the following tests on it. Grain size distribution analysis. As for carbonate soils Microscopic studies. X-ray diffraction analysis.

Figure 2.28 Proposed system of description (adopted from Datta et al. (1982))

Carter et al. (1999) identified and discussed under the following four sections some common properties unifying calcareous soils despite challenges and differences posed at relatively short distances:

- (1) Discrete particles, which are majorly composed of bioclastic materials, are feeble and remarkably angular. However, wide range of particle shapes and complex structural formations have been noted in Brandes, 2011 (Rashidian and Hassanlourad, 2014).

- (2) They are extremely compressible. Compressibility resulting from their large intra- and inter-granular porosity, their grain irregularity and brittleness.
- (3) Calcareous soils have variable grades of cementation. Semple (1988) recognized physiochemical environment, sediment stability, and sediment porosity as facies-controlled factors, which may be responsible for the spatial variation in cementation.
- (4) Calcareous soils textural and structural features such as grain type and size distribution, and level of cementation, which influence their mechanical properties (e.g. shear strength, compressibility and permeability) can significantly contrast over small distances. A behaviour that pose problems of interpretation of site investigation results and prediction of foundation behaviour.

Calcareous soils may appear indurated (e.g. limestone) at one end as weak rock (good engineering material) (Semple (1988); Carter *et al.* (1999)), and at the other end as very compressible (uncemented sands, silts and clays) (problematic) (Carter *et al.*, 1999). Again, Carter *et al.* (1999) noted that particle-to-particle bonding which results from precipitation and growth of CaCO<sub>3</sub> crystals is usually highly irregular. This irregularity brings about variations at very short distances, in strength, density, permeability and degree of cementation.

Many of the foregoing unifying behaviours of calcareous soils fall into the band for soils collapsibility. In Table 2.6 below characteristics of natural calcareous/carbonate soils with relevant references, are compared with characteristics of natural collapsible soils as reported in the literature (e.g. Assallay (1998); Rogers (1995); Barden *et al.* (1973)) to see to what extent they conform. The degree to which calcareous soils meet such characteristics are represented in three categories: strong agreement is indicated by “+ +”, agreed with “+” and no agreement with “- -”

Table 2.6 Degree to which calcareous soils meet characteristics of collapsible soils

S/N	Characteristic features of collapsible soils	Degree to which calcareous soils meet character	Reference
1	High porosity & high initial void ratio	++	Semple (1988); Rogers (1995); Assallay (1998); Carter <i>et al.</i> (1999).
2	Low density & high compressibility	++	Semple (1988); Carter <i>et al.</i> (1999).
3	Low inter-particle bond strength	+	Rogers (1995); Carter <i>et al.</i> (1999); Barden <i>et al.</i> (1973).
4	Maximum in the clay, silt, and sand particle size	++	Stoops (2010); Grine and Glendinning (2007); Kadry (1973); Al-Kaysi (1983).
5	Geologically young	++	Pettijohn (1975); Rogers (1995).
6	Predominantly primary minerals	+	Pettijohn (1975); Semple (1988).
7	Collapse with increasing saturation	+	Jackson 1956 cited in Al-Kaysi (1983).
8	High sensitivity	++	Agarwal <i>et al.</i> (1977); Barden <i>et al.</i> (1973).
9	Collapse yields a considerably reduced volume	++	El-Sohby <i>et al.</i> (1988).

Note:

#3– inter-particle bond strength may dependent upon factors such as: type and amount of CaCO<sub>3</sub> present, degree and pattern of cementation.

#6– calcareous soils are formed by both primary and secondary minerals and either may predominate.

#7– in calcareous soils collapse will depend on both mechanical and chemical properties of the soil components as well as nature of wetting fluid. Depending on the pH of wetting fluid, carbonate component may be soluble or insoluble.

Some naturally occurring calcareous soils may not meet all features often mentioned as characteristics for collapsible soils. Most of them however, meet the most essential characters of collapsible soils. Furthermore, with atmospheric pollution and increasing acid-rain precipitations, more dissolutions are imminent of carbonates in natural or engineered calcareous soils when allowed access to these acidic waters. This will transform them to a metastable state. The susceptibility of carbonate soils to become metastable under mentioned condition is of geotechnical consequence, and therefore should be considered collapsible soils.

### 2.5.3 Effect of CaCO<sub>3</sub> content on geotechnical properties

Some variables may influence the shear strength, compressibility and collapsibility of a soil consisting both non-carbonate and carbonate components. These variables may include: whether or not a carbonate component has a cementing impact on either itself, the non-carbonate component or both; the relative proportions of non-carbonate and carbonate components in the soil; the nature and composition of both the carbonate component (e.g. particle size, skeletal or non-skeletal particles, etc.) and the non-carbonate component (e.g. clay or sand, sensitivity of clay fraction, etc.). Nature, composition and degree of cementation in these soils are largely controlled by geological and sedimentary history, regional and environmental conditions. Therefore, a terrestrial clay for instance, could differently be affected by carbonate content compared to a marine clay. Some examples are presented under the following headings:

#### *Shear strength*

Demars *et al.* (1976) and Agarwal *et al.* (1977) stated sure favourable impact of carbonate content on the geotechnical parameters of calcareous clays.

Table 2.7 show results of consolidated undrained triaxial tests performed on calcareous clays containing between 10 to 90 % CaCO<sub>3</sub> (by Demars *et al.* (1976)). The strain at which maximum pore-water pressure parameter A occurred was selected as the failure criterion. This occurred commonly between 3 and 4 % strain. As demonstrated in the Table 2.7, an increase in CaCO<sub>3</sub> content was observed to increase effective frictional angle, with a corresponding decrease in pore-water pressure parameter A at failure, with a change from a cohesive behaviour to a granular at 40 % CaCO<sub>3</sub> content. They suggested the significance of particle shape and packing on strength behaviour was demonstrated by the results.

Table 2.7 Consolidation undrained triaxial test results (adapted from Demars *et al.* (1976))

CaCO <sub>3</sub> (%)	<i>c'</i> (kPa)	$\phi'$ (degrees)	<i>A<sub>f</sub></i>
0 – 25	0	27.7	0.7
25 – 40	0	29.4	0.55
40 – 60	0.69	31.0	0.4
> 60	0.69	31.3	0.25

Failure strain,  $\epsilon_f$ , taken at maximum pore-pressure parameter-A, *A<sub>f</sub>* – typically  $\epsilon_f = 3 - 4$  %

The effect of CaCO<sub>3</sub> content was studied by Agarwal *et al.* (1977) on Indian offshore calcareous clays, silts and sands collected from three sites. The study employed the direct shear behaviour and found that an increase in CaCO<sub>3</sub> content increased shear strength of the clay samples. Also, that CaCO<sub>3</sub> content had significantly influenced the stress-strain behaviour of the sands, especially at CaCO<sub>3</sub> contents higher than 45% and higher normal stress. For the high CaCO<sub>3</sub> sands, the behaviour and pattern of stress-strain curves seemed to indicate cementation effect and likely breakage of aggregated composition. A detrimental effect on the strength of the soil became obvious at greater than 45% CaCO<sub>3</sub> content.

The relationship between CaCO<sub>3</sub> content, bulk density and shear strength was investigated on deep-sea calcareous sediments. The shear strength was noted to depend mainly on whether a carbonate framework or the non-carbonate matrix dominated the soil, and on the physical nature of the carbonate matrix (Lee, 1982). Based on relations between bulk density and CaCO<sub>3</sub> content Lee developed a model that was quantitatively examined with triaxial shear strength, vane shear strength, CaCO<sub>3</sub> content and density data from different studies of deep-sea calcareous sediments. The model described three different types of behaviour: matrix-dominant, transition and carbonate framework dominant, with the following trends emerging: vane shear strength was not dependent on CaCO<sub>3</sub> content in the matrix dominant zone but increased with increasing CaCO<sub>3</sub> content in the carbonate framework dominant zone. However, it could not be established, whether the cause for increase was cementation or the granular nature of carbonate particles. The character of the non-carbonate matrix mainly determined the three behavioural types described by the model.

### ***Compressibility***

Bryant *et al.* (1974) performed over 120 consolidation tests on calcareous marine clay samples from the Gulf of Mexico and noted a general decreasing trend of compressive index (Cc) with increasing CaCO<sub>3</sub> content, and a clear difference of Cc between normally consolidated and overconsolidated samples (see Figure 2.29). They maintained that the consolidation results were similar for non-carbonate samples and those for carbonate silty clays. However, the compressed carbonate samples did not reach comparable low final void ratios as the non-carbonate samples.

Nevertheless, McCarel and Beard (1984) argue that decreasing compressibility with increasing CaCO<sub>3</sub> content was only valid with some fine-grained carbonate sediments because carbonate sands are already believed to be more compressible than silica sands.



### ***Collapsibility***

Apart from calcareous loessial soils, only a few other natural calcareous soils have been investigated in terms of wetting induced collapsibility. This, however, does not preclude other natural calcareous soils exhibiting this phenomenon.

In Southwestern USA and Mexico calcareous soils predominate most areas of Arizona and New Mexico. Gile et al 1965 was cited in Demars and Chaney (1982) to have grouped these calcareous soils into five different stages based on their collapsibility. The Stage 1 calcareous soils are of Holocene age and described as collapsible. They have weak cementation with filaments and grain coatings. Stages 2 and 3 are Pleistocene in age with moderate and strong cementation, respectively. Stage 2 are thus, moderately weakened whereas Stage 3 are only slightly affected by moisture increase. Stages 4 and 5 are Pleistocene or older in age. Stage 4 are very strongly cemented with properties akin to soft rock and are not considerably affected by moisture increase. Stage 5 calcareous soils fall into the category of moderate hard rocks where rock mechanics approaches are recommended for their investigation and analysis.

#### **2.5.4 Geochemical characteristics**

Calcareous soils have been defined in general terms on the basis of  $\text{CaCO}_3$  content (Kalra and Maynard 1991, and Day 1983 cited in Kishchuk (2000); Thompson (2007)), and at some points some referred to it as marl (e.g. Pettijohn (1975)).  $\text{CaCO}_3$  (especially pedogenic) may occur in soil as either carbonate particles precipitates in pore spaces of parent material, coating of grains, or inter-granular cementing agent (Al-Kaysi (1983); Landi *et al.* (2003)). Hunt, 1972, and Brown *et al.*, 1978 cited in Kishchuk (2000) have reported  $\text{CaCO}_3$  (calcite and aragonite), dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) and magnesite ( $\text{MgCO}_3$ ) as the calcium and magnesium carbonate minerals found in calcareous soils, but noted that these minerals occur mostly as calcite (see also Doner and Pratt, 1969 cited in Al-Kaysi (1983)) and to a lesser degree as dolomite. Mitchell and Soga (2005) also noted calcite as a common soil mineral.

Assuming all carbonate minerals in calcareous soils as calcite can be misleading though, because aragonite (an unstable  $\text{CaCO}_3$ ) behaves very differently from calcite. In some regions aragonite has been reported to occupy large areas. For example, it occurs above 60% on the North-West Shelf of Australia (Carter *et al.*, 1999). In terms of crystal structure aragonite is described as an orthorhombic-shaped (Carter *et al.*, 1999) crystalline polymorph of  $\text{CaCO}_3$  belonging to the holosymmetric class of the orthorhombic system whereas calcite forms rhombohedral crystals (Bragg, 1924), also described as tridiagonal crystal polymorph of  $\text{CaCO}_3$ . The aragonite grains were further described to have elongated and needle-like shape whose

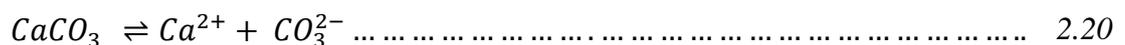
microstructural arrangement is responsible for the high void volume (Mao and Fahey 1999, in Carter *et al.* (1999)).

It is understandable that calcareous soils may consist of  $\text{CaCO}_3$  and any other soil material. Most soil minerals, except some clays, are inert to  $\text{CaCO}_3$ . In any case, the major problem posed by  $\text{CaCO}_3$  in soils is its dissolution and collapsing effects. On the other hand, calcareous clays may either collapse (when  $\text{CaCO}_3$  is dissolved out) or swell (when clay absorbs excess water) depending on the clay mineral present.

Note that most previous works on characterization of calcareous soils for engineering constructions have based almost entirely on mechanical properties (Fookes and Higginbottom (1975); Clifford Teme (1991); Esu *et al.* (1994)), and neglecting their chemical interaction with the environment, which is a vital property to understanding their long-term effectiveness. Corroborating the dissolution nature of  $\text{CaCO}_3$ , Al-Kaysi (1983)'s study of calcareous soils of Iraq reported both calcite and aragonite as dissolvable to aqueous solution and re-precipitating with different thermodynamic equilibrium constant values of  $10^{-8.35}$  for the former and  $10^{-8.22}$  for the later. Jackson 1956 was cited by Al-Kaysi (1983) as quoting pH 5 and less for the complete decalcification of carbonate soils. This simply implies that aqueous solutions with pH 5 or less can dissolve  $\text{CaCO}_3$  content of calcareous soils. Acid rains with less than pH 5 are common in some parts of the globe and with increasing atmospheric pollution in many urban cities (Huang *et al.*, 2019), there may be concerns about the safety of earthworks hosted or constructed with carbonate soils. Earlier, Lamas *et al.* (2002) and Mansour *et al.* (2008) have reported collapse of calcareous (marly) soil due to dissolution of  $\text{CaCO}_3$  after inundated with distilled water. The rate of dissolution however, would depend on the type of  $\text{CaCO}_3$  present in the marl.

***Solubility of calcium carbonates***

Salts of both weak acids and bases dissolve in aqueous solutions at differing rates depending on the pH of the solvent. A good example of pH dependence solubility is  $\text{CaCO}_3$ . This can be explained as in Equations 2.20 and 2.21:

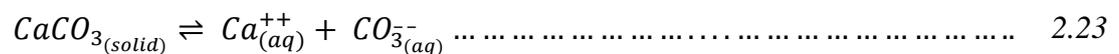
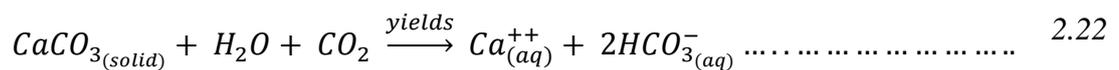


of which some of the carbonate hydrolyses to produce  $\text{HCO}_3^-$ :



From Equation 2.20, the concentration of  $CO_3^{2-}$  is decreased by the hydrolysis, and the solubility equilibrium is pulled to the right making  $CaCO_3$  more soluble.

Al-Kaysi (1983) has reported other factors responsible for the solubility of  $CaCO_3$  such as thermodynamic equilibrium constant, level of  $CO_2$  saturation in solvent, and grain size. Equation 2.21 was presented by Al-Kaysi to represent gradual removal of soil carbonate by solution in a leaching environment. He cited Abedi and Talibudeen 1974 who compared carbonate contents at surface and depths of Iraqi calcareous soil profiles and reported that leaching of carbonates from surface and reprecipitation of same at greater depths was responsible for the higher carbonate contents experienced with increasing depths of profiles. That, composition, occurrence, and distribution of  $CaCO_3$  were directly affected by their solubility in aqueous solutions as determined by their thermodynamic equilibrium constant (see Equations 2.22 and 2.23).



The thermodynamic equilibrium constant from Equation 2.22 has a value of  $10^{-8.35}$  and  $10^{-8.22}$  when the solid phase is calcite and aragonite, respectively, showing the higher solubility of aragonite (Krauskopf, 1967 cited in Al-Kaysi (1983)).

Quoting the works of Krauskopf 1967 and Vilenskii 1957, Al-Kaysi reported the degree of  $CO_2$  saturation in dissolving solvent as a controlling factor in the solubility of  $CaCO_3$  minerals and gave a 0.01 – 0.05 g/dm<sup>3</sup> range depending on the degree of  $CO_2$  saturation for the solubility of calcite in water at 25<sup>0</sup>C. The crystal size of carbonate grains were also identified experimentally by Suarez 1977 and Fairbridge 1972 as another factor responsible for differing solubility of  $CaCO_3$  resulting from surface energy contributions: smaller grains possess higher surface energy than larger grains, and thus more soluble (Al-Kaysi, 1983). This effect was studied on two samples of calcite with surface areas of 13.5m<sup>2</sup>/g and 0.8m<sup>2</sup>/g which demonstrated the effect of surface area as the former with a higher surface area showed higher solubility (Al-Kaysi, 1983). Table 2.8 shows results of effects of grain size on the thermodynamic equilibrium constant for calcite, reported by various researchers and compiled by Chave and Schmalz, 1966 (Al-Kaysi, 1983).

Table 2.8 Effect of grain size on thermodynamic equilibrium constant for calcite at 25°C (after Chave and Scmalz 1966 (Al-Kaysi, 1983))

Average particle size diameter (mm)	Thermodynamic equilibrium constant $K_{25}^0$
10	$10^{-8.41}$
$1 \times 10^{-3}$	$10^{-8.40}$
$2 \times 10^{-4}$	$10^{-8.34}$
$1 \times 10^{-4}$	$10^{-8.33}$
$1 \times 10^{-5}$	$10^{-7.70}$

Al-Kaysi (1983) presented findings by various investigators relating to the factors that may be responsible for  $\text{CaCO}_3$  solubility. Some of which included Olsen and Watanabe, 1959 who reported that pure calcite dissolved less compared to calcite occurring within a clay soil. According to them, solubility increased as soil/distilled water ratio also increased. However, it is unclear whether this study considered changes to the characteristics of the dissolving solvent which can affect the solute's solubility. Marion and Babcock, 1977 have also shown that  $\text{Mg}^{++}$  can interfere with  $\text{CaCO}_3$  precipitation and can increase its solubility. This is in agreement with the general claim that calcite stability decreases as its Mg content increases as demonstrated by the solubility values estimated by Winland, 1969. Also, Suarez, 1977 and Hassett and Jurinak, 1971 have established that there is an increasing instability of calcites with large Mg substitution (>10%) as compared to lower Mg calcites.

## 2.6 Simulation of Collapsible Soils

### 2.6.1 Non-calcareous collapsible soils

Geotechnical engineers have often considered the collapse of soils as exclusively a mechanical phenomenon and almost deliberately ignoring the chemical breakdown of bonding material. As such many researchers have produced artificial collapsible soils in the laboratory principally by compaction producing targeted mechanical properties. Some of which include Lawton *et al.* (1989); Basma and Tuncer (1992); Assallay (1998); Medero *et al.* (2005); Jefferson and Ahmad (2007) and references therein. Their most interested variables being void ratio, dry density and saturation. These factors were established either by compacting soil to predetermined dry densities or by varying compaction moisture contents. In these artificial collapsing soils, the particles' bonding element is probably suction or clay bridging. However, in many natural

collapsible soils, crystalized  $\text{CaCO}_3$  and other salts can also act as particles bonding substances which on dissolution would result in soil volume reduction.

### 2.6.2 Calcareous collapsible soils

The study of the collapse phenomenon on artificially cemented carbonate soils seems scarce in the geotechnical literature. Only a few reported cases are available. Hu et al., 2000, 2004 and Jiang et al., 2012 were cited in Li *et al.* (2016) to have created artificial structural calcareous loess by mixing different proportions of quicklime with natural loess and then adding water and carbon dioxide to produce  $\text{CaCO}_3$  bonding. Nevertheless, no experimental data or procedures for their study were presented.

However, there are methods reported in literature used to simulate natural carbonate cementation for different geotechnical and non-geotechnical studies. The knowledge of these may be relevant to produce collapsible calcareous soils needed for the present study.

## 2.7 Simulation of Calcareous Soils for other Geotechnical Studies

There are reports (e.g., Allman and Poulos (1988)) about difficulties in recovering undisturbed cemented calcareous samples from field. This, coupled with the non-uniformity of cementation in naturally occurring calcareous soils, compelled the option of studying laboratory controlled artificially cemented samples to gain insight on the impact of cementation on different geotechnical properties, especially strength related properties. However, Poulos et al, 1982 cited in Allman and Poulos (1988) suggested that methods to induce artificial cementation should closely simulate natural cementation processes, and satisfying the following criteria:

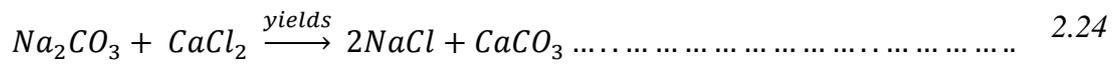
- (1) the artificial cementing material should mimic the natural cement as closely as possible
- (2) the process should be realistic enough to produce sufficient cementation within a practical timeframe;
- (3) the procedure should be reasonable to avoid ridiculously complex and expensive equipment.

Nevertheless, the literature seems to be agog with attempts (e.g., Clough *et al.* (1981); Allman and Poulos (1988) and references therein) to artificially produce cementation in calcareous soils, but with most, if not all of these attempts only satisfying criteria (2) and (3) above at the detriment of criterion (1). The criteria above can be affected by the amount of cemented sample needed for such study and practical constraints connected with meeting criteria (1) and (2) concurrently. Another drawback found in the literature is the use of non-

carbonate or impure carbonate substances to cement or produce calcareous soils. A few examples are discussed below in this section.

The artificial cementation of carbonate soils with Portland cement also featured in the literature, e.g., Allman and Poulos (1988) and references therein. Sterianos (1988) also reported Ertec Western Inc., 1983 to have studied artificially produced carbonate samples where they cemented shell fragments with Portland cement Type I. The authors referred to here, used between 0.5 and 4 percent cement by weight to mix with dry sand-size calcareous sediments and then added water.

Reacting calcium chloride (CaCl<sub>2</sub>) with sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) to precipitate CaCO<sub>3</sub> according to Akili and Torrance (1981); Ni and Ratner (2008) has also been tried. Akili and Torrance permeated CaCl<sub>2</sub> solution through a dry soil mixed with powdered Na<sub>2</sub>CO<sub>3</sub> precipitating CaCO<sub>3</sub> through cation exchange reaction. Sodium ions in Na<sub>2</sub>CO<sub>3</sub> were exchanged with calcium ions from the CaCl<sub>2</sub> solution. As shown in the Equation 2.24 below, sodium chloride, an undesired salt will also precipitate from this reaction.



A special branded procedure known as the calcite in-situ precipitation system (CIPS) process, which reportedly replicated natural processes of calcite precipitation was developed by the Commonwealth Scientific and Industrial Organization (CSIRO) (Kucharski *et al.*, 1996). The process involved flushing the proprietary CIPS solution through soil, leading to precipitation and coating of the soil particles with calcite and developing bonding at the particles contacts (Kucharski *et al.* (1996); Ismail *et al.* (2000)). This product, however, is not commercially available.

Cailleau, 1982 developed artificial CaCO<sub>3</sub> cementation method where a supersaturated CaCO<sub>3</sub> solution was used. This method was later modified by Molenaar and Venmans (1993). The modified process involved continuously circulating the supersaturated solution through a porous soil deposit over a period of time while CO<sub>2</sub> was simultaneously passed through the supersaturated solution to maintain the CaCO<sub>3</sub> concentration. The supersaturated solution aimed to precipitate CaCO<sub>3</sub>, nucleating soil particles at favourable sites. Even though the process was successful, it seemed overly complicated and Molenaar and Venmans (1993) noted that it failed to produce repeatable samples as their UCS results scatter demonstrated. Again, in

principle, the reaction of CaCO<sub>3</sub> solution with CO<sub>2</sub> (as in Equation 2.25) would rather not yield CaCO<sub>3</sub> precipitate but produce dissociated calcium ions and bicarbonates, which will rely on certain conditions to recombine. This position was supported by the works of Poulos et al., 1982 (Sterianos, 1988) and McKown and Ladd (1982).

Poulos et al., 1982 tried to produce artificial cementation of Bass Strait sands adopting the following different procedures:

- Uncemented carbonate sand samples were allowed standing under a stress of 248 kPa in an oedometer, in the presence of a CaCO<sub>3</sub> supersaturated solution for a period of ten weeks.
- Two of the above samples were further subjected to an increased stress up to 310 kPa and allowed standing for a further six to eight weeks.
- Some samples were allowed to stand under similar stress conditions as above, in the presence of a lime rich solution.
- Other samples were mixed with 6% Portland cement.

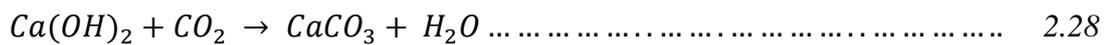
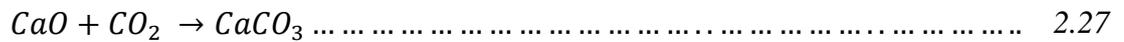
The experiments revealed that in a laboratory environment, cementation was not developing in a static CaCO<sub>3</sub> solution condition. It was observed that signs of cementation were only obvious in samples mixed with Portland cement. Even in circulatory conditions, McKown and Ladd (1982) demonstrated that circulation was not sufficient in itself to ensure precipitation of large amounts of CaCO<sub>3</sub>. McKown and Ladd (1982) leached samples of natural clays with a supersaturated CaCO<sub>3</sub> solution under hydraulic gradients up to 9600. The leaching was done for a period of about four months with approximately 35 void volumes of solution passed through samples. Index properties, chemical, and x-ray diffraction analyses performed before and after the leaching periods indicated no substantial changes occurring. It became obvious from these experiments, that: simply treating a sample with a supersaturated CaCO<sub>3</sub> solution may not yield substantial CaCO<sub>3</sub> precipitate and its cementation; most, if not all artificially cemented samples adopted in the studies of their geotechnical engineering properties did not adequately simulate natural cementation (McKown and Ladd, 1982).





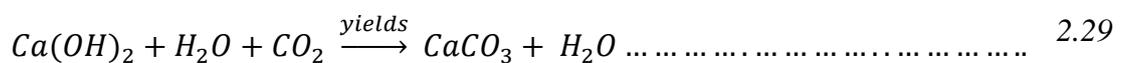
Different research interests ranging from capturing of flue CO<sub>2</sub> to production of CaCO<sub>3</sub> or soil carbonation have adopted this chemical reaction.

In geotechnical engineering, the use of lime (both quick and hydrated forms) to treat soils in earthworks engineering is a common practice (Glendinning and Rogers (1996); Rogers *et al.* (2000); Boardman *et al.* (2001)). Such earthworks exposed to atmospheric CO<sub>2</sub> are usually transformed or carbonated following Equations 2.27 and 2.28 (Moorehead (1986); Bagoniza *et al.* (1987); Vance *et al.* (2015)). For instance, Bagoniza *et al.* (1987) reported carbonation of pavement soils in parts of Africa from the reaction of lime and CO<sub>2</sub>. Langroudi (2014) also showed that adding lime slurry to soil and then passing CO<sub>2</sub> through can reduce the systems pH to about 7.5 triggering instantaneous precipitation of CaCO<sub>3</sub>.



A few research works done outside geotechnical engineering, on the carbonation of lime, especially hydrated lime exist in literature (Moorehead (1986); Shih *et al.* (1999); Beruto and Botter (2000); Van Balen (2005); Materic and Smedley (2011); Materic *et al.* (2014); Vance *et al.* (2015); Despotou *et al.* (2016)) and are discussed below.

Hydrated lime (Ca(OH)<sub>2</sub>) was known to react faster with CO<sub>2</sub> than would quicklime (CaO). This may be due to the presence of hydroxyl ions. However, from preliminary studies made in this project, it was revealed that hydrated lime in its dry state would also react extremely slowly with CO<sub>2</sub>. Some moisture at a certain range was necessary to catalyse the reaction. Some very recent studies conducted have also confirmed the role of moisture as a catalyst but reported moisture in the form of relative humidity (RH) or water vapour. Equations 2.27 and 2.28 therefore, modified to incorporate moisture as in Equation 2.29. Reports suggesting increase in Ca(OH)<sub>2</sub> conversion to CaCO<sub>3</sub> with increase in moisture (Shih *et al.* (1999); Beruto and Botter (2000); Materic and Smedley (2011); Materic *et al.* (2014); Vance *et al.* (2015); Despotou *et al.* (2016)).



Though no relationship was established between RH and soil moisture content, Van Balen (2005) has on the contrary noted that, due to the low diffusion of CO<sub>2</sub> in water (nearly 10000 times lower than in air), high moisture contents in this kind of carbonation system will act as barrier preventing CO<sub>2</sub> from reaching potential reaction sites. This claim may be enlightening as to why two identical samples with different moisture contents reacted at the preliminary stage of this study showed varying results (see Figure 3.5 on page 105). However, density also influence the results.

Shih *et al.* (1999) reported insignificant reaction in terms of Ca(OH)<sub>2</sub> conversions, occurring at RHs below 8%, above which conversion increased with RH up to 70% tested. Shih and colleagues further noted that temperatures within range (60–90 °C) tested did not affect Ca(OH)<sub>2</sub> conversion. In Materic *et al.* (2014) a near total conversion was achieved at RH of 88% at ambient temperature within 12 hr but a very slow reaction was observed at very low RH, achieving only 12% conversion at 0.5% RH in 48 hrs. The reaction, under high temperatures (200–425 °C) isothermal conditions have also been reported as alternative route for catalysing rapid carbonation of Ca(OH)<sub>2</sub>. This suggests activity of a direct CO<sub>2</sub>–solid Ca(OH)<sub>2</sub> carbonation mechanism (Equation 2.28) (Materic *et al.*, 2014) through a self–catalysing effect as water is released from Ca(OH)<sub>2</sub> at those temperatures (Nikulshina *et al.*, 2007).

Furthermore, ambient temperatures (15–30 °C) are agreed by many authors to play an insignificant role while moisture which dissolves lime at water–adsorbed surface appearing to be the controlling mechanism for the reaction (Shih *et al.* (1999); Beruto and Botter (2000); Van Balen (2005)). Vance *et al.* (2015) added that even temperatures in the range of as low as 8°C and high as 42°C, and pressures in the range of 60–100 bars only influence this reaction very slightly. Despotou *et al.* (2016) also confirmed ambient pressures to have little or no effect on the reaction but that at ambient conditions (temperature and pressure), thickness determines the time taken to completely carbonate a lime sample.

A summary of the reaction mechanisms of lime and CO<sub>2</sub> in a compact according to Moorehead (1986) include:

- Dissolution of lime in compact moisture forming Ca<sup>++</sup> and OH<sup>-</sup> ions.
- Reduction of pH and formation of H<sup>+</sup>, HCO<sub>3</sub><sup>-</sup> and CO<sub>3</sub><sup>-</sup> ions by dissolving CO<sub>2</sub> in compact capillary water.
- Interaction between those ions to produce CaCO<sub>3</sub> which precipitates in greater volume compared to the initial lime.

- Micro-crystalline or amorphous calcium carbonate are usually precipitated as the water evaporates from the reaction sites. Evaporation is due to reaction heat.
- The reaction continues until full conversion is achieved unless compact water content is reduced by reaction heat below sufficiency to drive further reaction.

## 2.9 Collapse Mitigating Measures: Available Techniques

A wide range of collapsible soil improvement techniques exist. However, not too many have been implemented at field scale. An overview of available treatment processes is presented in the following sections based on six key categories, mechanical or physical, hydraulic, chemical, biological, electro-kinetics, and soil gassing. Even though most have been targeted at improving collapsible loess soils, their processes and potentials can serve as guide to treat other problematic soils.

### 2.9.1 Mechanical or physical processes

These processes usually target at densification. Most of these processes are usually carried out pre-construction while a few can apply both pre- and post- construction. Common examples of these techniques are the injection of clay suspension to reduce porosity in granular loess (loess-like sand and sandy loess) (Jefferson *et al.*, 2005), and modification of the grading curve of collapsible soil by introducing sub-rounded grains to both decrease plasticity and coefficient of volume compressibility ( $m_v$ ) (Langroudi, 2014).

Some other mechanical processes include: preloading and the use of vertical drains, dynamic compaction, vibrocompaction, hydro-blasting compaction, compaction by ponding, compaction by moistening and explosions, shallow compaction, compaction grouting and the installation of densification piles or columns (lime, cement, lime-fly ash, stone), water treatment, vacuum isotropic consolidation, use of mixed material and re-compression, vibroflotation (Jefferson *et al.* (2005); Essler (2012); Langroudi (2014)).

These methods have some kind of limitations in their suitability under certain instances, e.g., because of the low permeability of high clay loess soils, treatment by vibroflotation and densification piles are impracticable (Watts and Charles, 2015). For soils such as collapsible calcareous clays low permeability will also pose challenges for some of the treatment methods. Again, densification by compaction for instance, may break/crush carbonate bonds and increase their solubility.

### 2.9.2 Hydraulic processes

Hydraulic methods aim at the control of groundwater movement or its extraction from dangerous zones. Different techniques based on the principles of gravity, electrical gradients and increased stresses are used (Chris DF, 2012). These may include the application of sump pumps, well points, ejector wells, deep wells, vertical drains, electro-osmosis and barrier walls (William (2012); Chris DF (2012)). The soil's permeability which is a factor of porosity, density and natural water content, controls the effectiveness of any of these methods (Gibbs and Holland, 1960). For instance, due to the cracks resulting from shrinking or rhizoliths (root-like macro-pore spaces) occurring in young dried clayey silt loess or reworked calcareous loess, these soils could exhibit high degrees of permeability compared to dense and heavily reworked loess deposits, which exhibit far less degrees of permeability. Again, water outflow may eliminate 2 to 20  $\mu\text{m}$  quartz filling particles and therefore, reduce the lateral support of void spaces, dissolve the chemical binders and drive clays into the residual macro-pore spaces. These can considerably decrease the collapsibility (Langroudi, 2014).

### 2.9.3 Chemical processes

Chemical processes concern the use of chemical substances or principles to alter the chemistry of soils towards a better performance. They can be applied pre-construction as well as post-construction. Commonly known techniques used here can be classified into chemical stabilization and others.

Chemical stabilization includes grouting and deep mixing of soils with calcium-based reactants (lime and cement), pozzolans (fly ash, ground granulated blast slag) or reactants such as sodium silicates (discussed in section 2.10). Quicklime has been successfully used to desiccate and stabilize water-saturated loess in the former Soviet Union by compacting into boreholes (Abelev, 1983 in Jefferson *et al.* (2005)).

Others include electro-kinetics and soil gassing (see sections 2.9.5 and 2.9.6, respectively). The environmental damages caused by some of these chemicals have raised concerns about their continuous usage. Before the 21<sup>st</sup> century, performance of agglomerated materials and cost were the major requirements for soil improvement exercises, but today, while performance is still a critical aspect of soil improvement planning, environmentally friendly materials that are still reasonably priced are most demanded. Therefore, attention is being diverted to materials such as sodium silicate, which has been investigated to treat calcareous silty-clays in the present study.

#### 2.9.4 Biological processes

Biological processes involve the inducement of microbial to precipitate cementing material into soil. The technique usually involves introducing a calcium source (e.g. calcium chloride) and a urea source bacteria (*Sporosarcina Pasteurii*) into a suitable soil environment where the bacteria survives and precipitates calcite cementation into soil pores of granular medium. Severally works have reported the potential of this process and the principles are detailed in Van Paassen (2009); Harkes *et al.* (2010) and Martinez *et al.* (2013). Van Paassen (2009) cited series of works since 1995 on the microbial induced calcite cement.

The idea of calcite cementation to treat collapsibility is debatable as there are arguments for and against the beneficial impact of calcite in collapsible soils. However, the present study investigated this and concludes that calcite cementation can be both beneficial as well as promote collapse under certain conditions.

#### 2.9.5 Electro-kinetics (EK)

Electro-Kinetics (EK) is a recent innovation that involves using its phenomena in functions such as electro-osmosis, electrolysis, ion migration, electrophoresis, etc. to enhance chemical, biological and physical changes (e.g. hydraulic conductivity) in a soft ground for quicker improvement (Jones *et al.* (2006); (2008); Ou *et al.* (2009); Chien *et al.* (2014)). This method is applied in reinforcement (chemical/biological) and consolidation types of improvement as a measure to speed up the hydraulic flow in fine-grained soils. Electro-Kinetics has been successfully used to strengthen soft clays through water draining and consolidation (Rogers *et al.*, 2002). Jones *et al.* (2008) noted that Electro-osmotic consolidation is far faster than preloading method. That flow rate created under electric field can be greater than that under hydraulic gradient by up to 100 to 10000 times. It has also been used to mitigate swelling in expansive clays through ion migration, cation exchange and reduction of plasticity (Jones *et al.*, 2008). Successes have also been recorded in chemical improvement methods using electro-osmotic technique (Chien *et al.*, 2014).

Ozkan *et al.* (1999) revealed shear strength increases of 500 – 600 % for a study involving electrochemical treatment of kaolinite by injecting phosphate and aluminium ions. The process however, showed inhomogeneous distribution of strength over specimens, with shear strength increase restricted to regions close to the cathode or anode. By using the same method Alshawabkeh and Sheahan, 2003 achieved a 160% increase in shear strength near the cathode after injecting phosphoric acid over 14 days to stabilize a marine illitic soil.

Asavadorndeja and Glawe (2005) applied anode depolarization method and were able to increase the improvement region of a soft clay treated. Strength increases up to 170 % just after treatment and up to 570 % after 7 days curing were recorded. Otsuki et al., 2007 applied the electrochemical method to inject anolyte solutions of  $\text{MgSO}_4$ ,  $\text{Mg}(\text{CH}_3\text{COO})_2$ ,  $\text{Mg}(\text{NO}_3)_2$ ,  $\text{ZnSO}_4$ , and  $\text{AgNO}_3$  and catholyte solutions of  $\text{NaOH}$  and  $\text{Na}_2\text{CO}_3$  into kaolin soil. Shear strength increases up to 300 kPa were recorded but the improvement region was also restricted to near the cathode. Chien et al. 2009, during osmosis tests injected a  $\text{CaCl}_2$  solution into a soil over a 7-day period and reported average undrained shear strength increase of 400 to 500 %. Abdullah and Al-Abadi, 2010 demonstrated that cationic–electrokinetic processes adopting the  $\text{K}^+$  and  $\text{Ca}^{2+}$  ions as stabilizing agents can be effectively used for transferring cationic-stabilizing agents systematically and homogeneously into soils.

Electro-kinetics could be a useful strategy to treat calcareous silty-clays but this was not considered in the present study.

#### 2.9.6 Soil gassing

Ground improvement by soil gassing is usually applied in the following areas:

- Ground freezing – liquid nitrogen injected into the ground freezes and stabilizes the ground. Nitrogen gas is released to the atmosphere.
- Ground water level in tunnels is balanced by flushing in compressed air.
- Dissolution and reprecipitation of carbonates – this involve a chemical reaction between  $\text{CO}_2$  and soil elements (e.g., calcic plagioclase anorthite,  $\text{Ca}(\text{OH})_2$ ,  $\text{CaCO}_3$ , etc.) to form secondary carbonates. Another method under this is the carbonation bubbling stabilization where an original soil is enriched with alkaline  $\text{Ca}(\text{OH})_2$  and flushing the soil with  $\text{CO}_2$ . This reaction reduces the alkaline pH 14 to about neutral triggering  $\text{CaCO}_3$  nucleation (Molenaar and Venmans, 1993).

Soil gassing has also been adopted in some instances to enhance other stabilization method. In silicatization of carbonate and humus soils, Sokolovich and Ibragimov (1971) for instance, reported a further 30 – 50% strength increase for a  $\text{CO}_2$  preactivated soil compared to a non-activated. They noted that the preactivation enhanced a more uniform distribution of the injected silicate solution in the ground because carbonic acid formed during the preactivation can actively absorb the silicate solution.

### 2.9.7 Nanotechnology

This technology deals with the use of nanoscale ( $\leq 100$  nm) materials. Recent studies have been conducted to examine its prospective use in geotechnical engineering, targeting both to understand the impact of inherent nano soil particles on the soil behaviour and the applicability of commercially available nanomaterials in ground improvement (Gallagher and Mitchell (2002); Huang and Wang (2016); Iranpour and haddad (2016); Ghasabkolaei *et al.* (2017)). Some of the nanomaterials employed in soil treatment studies included colloidal- and nano-silica, nano-clay, nano-copper, carbon nanotube (CNT), and nano-alumina. The potential of some of these nanomaterials to improve soil properties such as strength, collapse potential, hydraulic conductivity, and liquefaction has been reported.

Iranpour and haddad (2016) studied the impact of different nanomaterials on the collapsibility of Iranian soils and presented the collapse results in Figure 2.30 for two different soils. Iranpour and colleague concluded that only were appropriate amounts of nanomaterials are used could desired improvement be achieved and quickly added that collapse was also dependent on moisture content and dry unit weight.

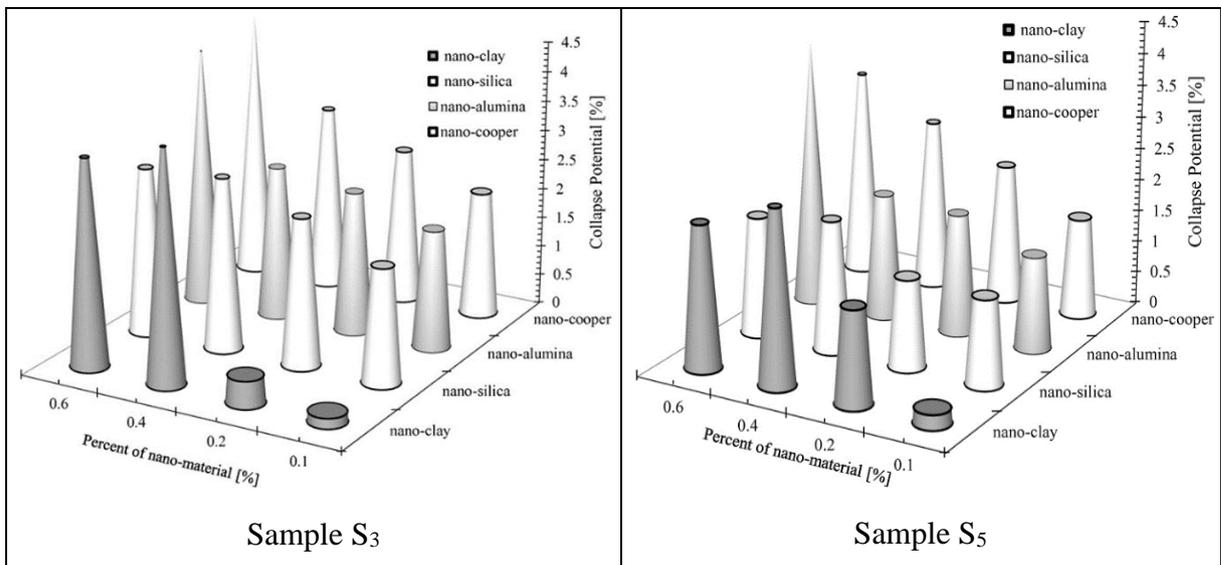


Figure 2.30 Comparing the impact of different nanomaterials on the collapse potential of Iranian soils (adopted from Iranpour and haddad (2016)).

Persoff *et al.* (1996) and Yonekora and Miwa, 1993 cited in Gallagher and Mitchell (2002) have grouted sands with colloidal silica and reported compressive strength increase with increasing colloidal silica concentration and increasing weight percent. Persoff and colleagues found that Monterey sands grouted with different concentrations of colloidal silica resulted in

a linear relationship between UCS and concentration. Towhata and Kabashima, 2001 in Ghasabkolaei *et al.* (2017) noted that liquefiable sand specimens at 40% relative density treated with a 4.5% weight colloidal silica displayed liquefaction resistance and deformation behaviour akin to those of the untreated dense sand with 75% or greater relative density. Gallagher and Mitchell (2002) also investigated loose sand with about 5% weight colloidal silica and noted that the risk of liquefaction of the sand was considerably mitigated. They further revealed that strain levels were also decreased (i.e., stiffening) with increasing weight percent of colloidal silica but higher than 10% weight was seen as uneconomical relative to others. They further investigated the UCS of the sand grouted with 5% and 15% concentrations of colloidal silica and noted a 32 kPa and 110 kPa, respectively.

Hydraulic conductivity of sands have been reported to linearly reduce with increasing colloidal silica (Persoff *et al.* (1996)) and nanoclay (Kananizadeh et al 2011 in Ghasabkolaei *et al.* (2017)). Nanoclays have also been found to improve both soil strength and susceptibility to collapse. The nanoclays act as filler reducing pore voids and modifying the microstructure of the soil (Ghasabkolaei *et al.* (2017)).

According to Huang and Wang (2016), nanomaterials have overwhelming advantages over traditionally used grouts in terms of environmental effect and price/performance ratio.

## 2.10 Sodium Silicate: A Binder

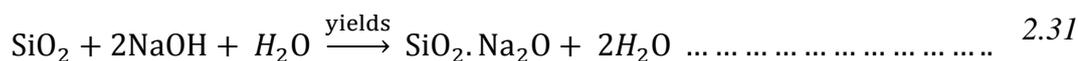
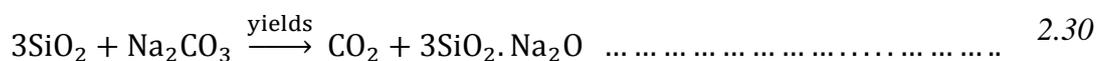
### 2.10.1 Brief history

Sodium silicate, an alkali silicate, is an inorganic high-molecular chemical compound which comprises silica ( $\text{SiO}_2$ ) and sodium oxide ( $\text{Na}_2\text{O}$ ). They are a group of chemicals having a wide range of chemical and physical properties (described in detail in section 2.10.3).  $\text{Na}_2\text{SiO}_3$  was described as one of the oldest and most friendly manufactured chemicals, dating from the late 1810s (McDonald and Thompson (2006)). However, Iler (1979) argued that commercial alkali silicate production began only in 1855 in America and Europe.

Sodium silicates industrial application spans a wide range including manufacturing of silica gel, refractories, foundry, wood processing, protective coatings, rust inhibitors, detergents, deflocculants, catalyst bases, cleaning compounds, bleaching agents, adhesives, and cements (Hurley and Thornburn (1972); Sharafudeen *et al.* (2017)). Iler (1979) noted that Johann Nepomuk von Fuchs proposed in 1850 after a systematic investigation, that alkali silicates could be used as adhesives, cements, and fireproof paints. However, for the purpose of this study they will only be reviewed as binders (adhesives and cements).

### 2.10.2 Manufacture of sodium silicates

The raw materials for manufacturing Na<sub>2</sub>SiO<sub>3</sub> are usually derived from the abundant natural silica, sodium salts and water. Its production is simply by fusing natural silica (sand) and a sodium salt (e.g. Na<sub>2</sub>CO<sub>3</sub> or NaOH) (see Equations 2.30 and 2.31) in large open-hearth furnaces at temperatures described as red heat or ranging from 1100 and above 1300<sup>0</sup>C (Hurley and Thornburn (1972); Vail, 1952 in Iler (1979); McDonald and Thompson (2006); Stephens *et al.* (2003); Sharafudeen *et al.* (2017)). The simple process of manufacture was the reason for their early development (McDonald and Thompson, 2006). The process yields a molten glass that is allowed to either solidify and thereafter ground into various particle sizes or is dissolved in steam under pressure to produce solutions at different concentrations.



Additionally, recent advances in the manufacture of Na<sub>2</sub>SiO<sub>3</sub> indicate that, silica can also be extracted from various kinds of agricultural wastes. Some of these include bagasse ash, rice husk ash and rice hull ash, through different process, e.g. acid leaching, burning rice hull ash at high temperature, or gasification of rice hull (Chakraverty and Kaleemullah (1991); Kamath and Proctor (1998); Kalapathy *et al.* (2000); Mukunda *et al.* (2004); Shelke *et al.* (2010); Hariharan and Sivakumar (2013); Sharafudeen *et al.* (2017)).

### 2.10.3 Properties of sodium silicate

Sodium silicates also known as water glass, are available commercially as lumps or glass form, partially hydrated and hydrated powders, and concentrated aqueous solutions. They are produced in different molecular ratios, SiO<sub>2</sub>:Na<sub>2</sub>O (or weight ratio, since both SiO<sub>2</sub> and Na<sub>2</sub>O molecules weigh similar), different water contents and particle sizes depending on their proposed application. The key variables in the solutions revolve round the molar ratio and the concentration of solids (Iler (1979); Weldes and Lange (1969)). Even though alkali and silica may theoretically be combined in all proportions, there are no commercially available products exceeding a silica to alkali ratio of about 4:1 by weight because fused silicates above this ratio possess very low solubility. See typical commercial Na<sub>2</sub>SiO<sub>3</sub> composition in Tables A1-1 &

A1-2 in Appendix A1. Figure 2.31 is a ternary system of Na<sub>2</sub>O-SiO<sub>2</sub>-H<sub>2</sub>O covering commercial and non-commercial Na<sub>2</sub>SiO<sub>3</sub> composition.

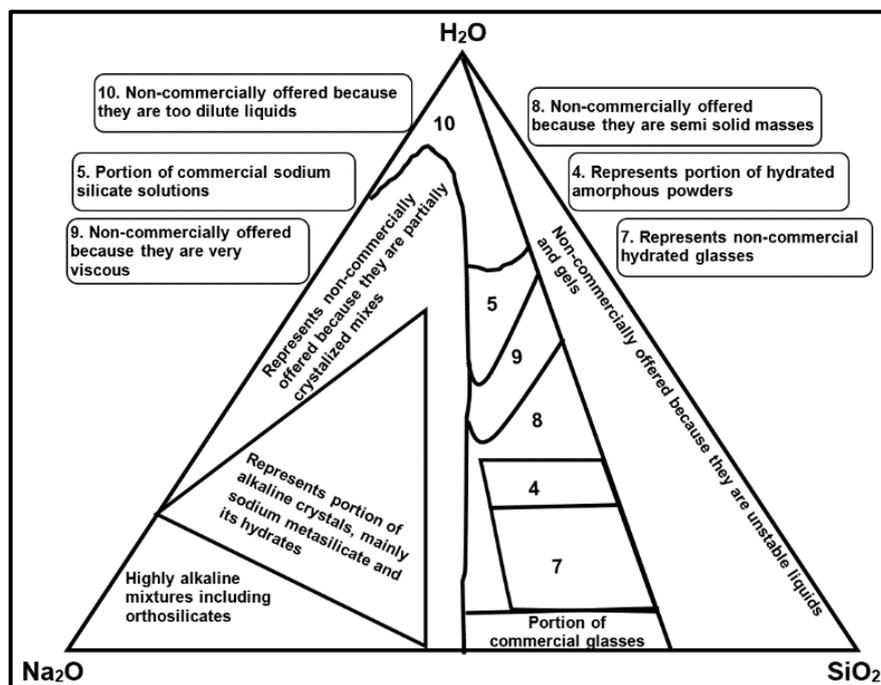


Figure 2.31 A ternary of commercial and non-commercial sodium silicate composition (redrawn after Weldes and Lange (1969)).

### Physical properties

Sodium silicate solutions are colourless, clear and odourless. Hurley and Thornburn (1972) have described the physical nature of the soluble silicates as ranging widely from readily (or highly) soluble crystals to lumps of transparent glass that are lightly soluble even in boiling water and from marginally sticky liquids of the consistency of maple syrup to very heavily viscous materials that can hardly flow. Roggendorf *et al.* (2001) described concentrated Na<sub>2</sub>SiO<sub>3</sub> solutions structurally, as dispersions of colloidal amorphous silicates in aqueous solution, with sizes of colloids ranging between 30 and 100 nm. Sizes of colloids between 1 and 5 nm have also been extracted by tetrahydrofuran and light scattering (Böschel *et al.*, 2003). Na<sub>2</sub>SiO<sub>3</sub> are silicate polymers with a range of different SiO<sub>2</sub> and Na<sub>2</sub>O polymers in existence. The degree of polymerization depends on the proportion of oxygen shared between [SiO<sub>4</sub>]<sup>2-</sup> tetrahedra, and this determines the SiO<sub>2</sub>:Na<sub>2</sub>O ratio, which controls the properties and functional activity of any Na<sub>2</sub>SiO<sub>3</sub> (Lee and Stebbins, 2009).

Commercially produced ratios can be between 1.6 - 3.9 (Iler, 1979), 1.5 – 3.2 (McDonald and Thompson, 2006) while ratios of 2.0 and 3.3 are standard compositions (Roggendorf *et al.*, 2001). Ratios of 1.6 – 3.8 are currently produced by Ingessil S.r.l Silicate Industry, supplier of

the Na<sub>2</sub>SiO<sub>3</sub> solution used for this project. The ratio represents an average of various molecular weight silicate species that control the bonding properties of the solution. Notwithstanding that high “3.2” ratio silicates is mostly commonly used in agglomeration, all Na<sub>2</sub>SiO<sub>3</sub> ratios may be ideal for application depending on factors such as: type of material being agglomerated, type of bonding needed, and set-up time needed. For applications where pumping is required, viscosity becomes an important property to be considered.

However, viscosity tends to increase with increasing siliceous concentration and decreases with increasing alkalinity, it shows a distinct minimum at a SiO<sub>2</sub>:Na<sub>2</sub>O ratio of 2:1, and tends to rapidly increase with either an upward or downward change from this ratio (Hurley and Thornburn, 1972). Therefore, workable viscosity required may dictate the weight ratio of aqueous solutions, since low enough viscosity may be needed for ease of pumping (Weldes and Lange (1969); Iler (1979)). But this may pose other challenges as dominant silicate species in solution determines the rate of gelation, precipitation, dehydration, water resistance, etc. (McDonald and Thompson, 2006).

### ***Chemical properties***

The chemistry of soluble silicates remains complicated despite decades of investigations (Iler (1979); Nordström *et al.* (2011)). The chemical behaviour of silicates is also controlled majorly by their SiO<sub>2</sub>:Na<sub>2</sub>O molecular ratio, solid concentration (for liquid silicates) (Lee and Stebbins, 2009) and pH (Iler (1979); Nordström *et al.* (2011)). Concentration of solids influences expansion coefficient whereas, molecular ratio determines alkalinity and specific gravity. Available silicates are in the pH range of 11 to 13, and the lower the SiO<sub>2</sub> to Na<sub>2</sub>O ratio, the higher the alkalinity and specific gravity. Hurley and Thornburn (1972) reported silicates with alkalinity greater than that of SiO<sub>2</sub>:Na<sub>2</sub>O ratio of 1.6 as definite crystalline salts with fixed composition and particular chemical formula. The following examples (Table 2.9) are in the order of decreasing alkalinity:

*Table 2.9 silicates with greater alkalinity than SiO<sub>2</sub>:Na<sub>2</sub>O = 1.6 (after Hurley and Thornburn (1972))*

<i>SiO<sub>2</sub>:Na<sub>2</sub>O</i>	<i>Name</i>	<i>Formula</i>
1:2	Sodium orthosilicate	Na <sub>2</sub> SiO <sub>4</sub>
2:3	Sodium sesquisilicate	Na <sub>6</sub> SiO <sub>7</sub>
1:1	Sodium metasilicate	Na <sub>2</sub> SiO <sub>3</sub>
2:1	Sodium disilicate	Na <sub>2</sub> Si <sub>2</sub> O <sub>5</sub>

Those silicates with alkalinity less than that of metasilicate form colloidal solutions, which are soluble as well as chemically unstable. These unstable materials can form silica gel when mixed with any other inorganic material. However, the gel properties may vary greatly depending on factors such as type of salts, acids or bases available in the reaction,  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio, concentration of solution, and temperature. The traditionally used silicate for treating building materials is a solution of  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio of about 3.22 with a specific gravity of 1.394 and density of 41B at 20°C (Hurley and Thornburn, 1972). As discussed earlier, the reaction of acids and bases with  $\text{Na}_2\text{SiO}_3$  produces a silicate gel. Mostly a complex metal hydroxide silica gel whose structure is not understood (Hurley and Thornburn, 1972). Little or no pattern is shown under X-rays, and is thus, regarded amorphous. Its specific surface is quite high, 600m<sup>2</sup>/g, of the order of magnitude of montmorillonite. Silica gel shrinks (syneresis), becomes opalescent, and cracks on aging; it dissolves readily in caustic (Hurley and Thornburn, 1972).

According to Iler (1979) and McDonald and Thompson (2006),  $\text{Na}_2\text{SiO}_3$  can undergo four distinctive chemical reactions which are: hydration/dehydration, polymerization and gelation, precipitation, and surface charge modification. They note that these reactions result in film bonding, matrix bonding, and/or chemical bonding of agglomerated material; and that best performances of silicate-based formulations sometimes are achieved from more than one bond mechanism.

*Hydration/Dehydration:* Aqueous silicate becomes stickier and more viscous as water is gradually removed from it (McDonald and Thompson, 2006). However, the viscosity can be influenced by various factors, such as  $\text{Na}_2\text{O}$  weight percent, temperature, density, and  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio, as presented in Figure 2.32 and Figure 2.33 (PQ Corporation 2004 online publication (*accessed 14/05/2019*)). Weldes and Lange (1969) had also reported a sharp break in viscosity–concentration curves of more siliceous  $\text{Na}_2\text{SiO}_3$  solutions having high water content. They said beyond the break, a relatively small amount of water removed rapidly increases viscosity making the aqueous silicate a glassy film. High (3.2) ratio aqueous  $\text{Na}_2\text{SiO}_3$  are best suitable for acting as film binders. According to McDonald and Thompson (2006), High 3.2 ratio silicates are usually low in alkalinity, and as such, they dry faster because of their low affinity for water. In addition, that when dried the bond is less prone to water absorption, but complete water resistance can only be achieved with some degree of either heat or chemical setting. Achieving sufficient green bond strength has been identified as a common difficulty when using silicates as film binders, but a number of things can be done on the processing side to achieve earlier and more expected green bond strength. Some of these as recommended by Iler (1979) may include, either of the following: (1) incorporating relatively small quantities of

other quicker setting film forming bonding agents such as dextrin, glycerin, starch or molasses; (2) temperature control of the agglomerated material, moisture control of the agglomerated material, lay-up time to correspond with tack development or proper mixing. Another is the incorporation of calcium source material (Pietsch 1978, in McDonald and Thompson (2006)).

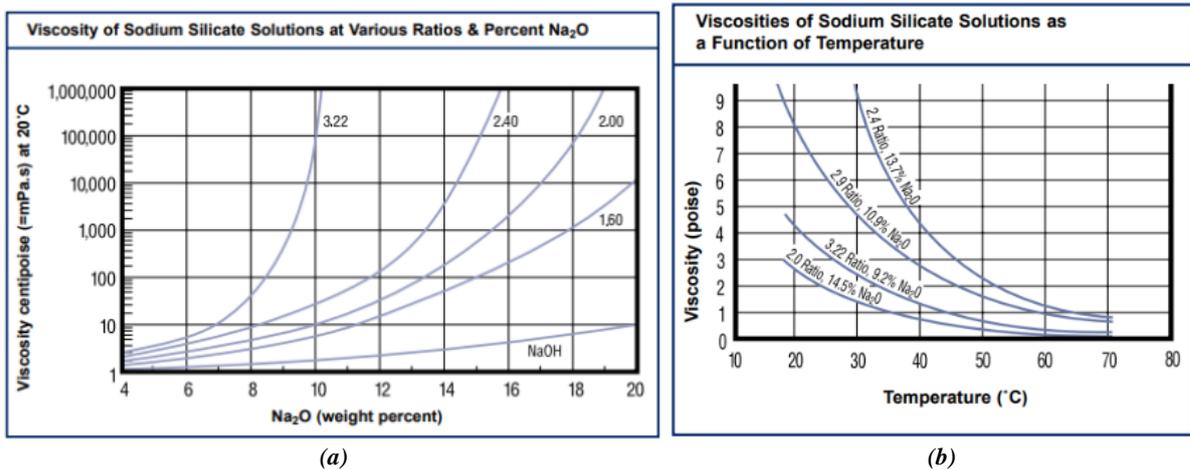


Figure 2.32 Viscosities of  $\text{Na}_2\text{SiO}_3$  solutions (a) at various weight ratios and  $\text{Na}_2\text{O}$  percent (b) as a function of temperature (adopted from PQ Corporation 2004 online publication (accessed 14/5/2019))

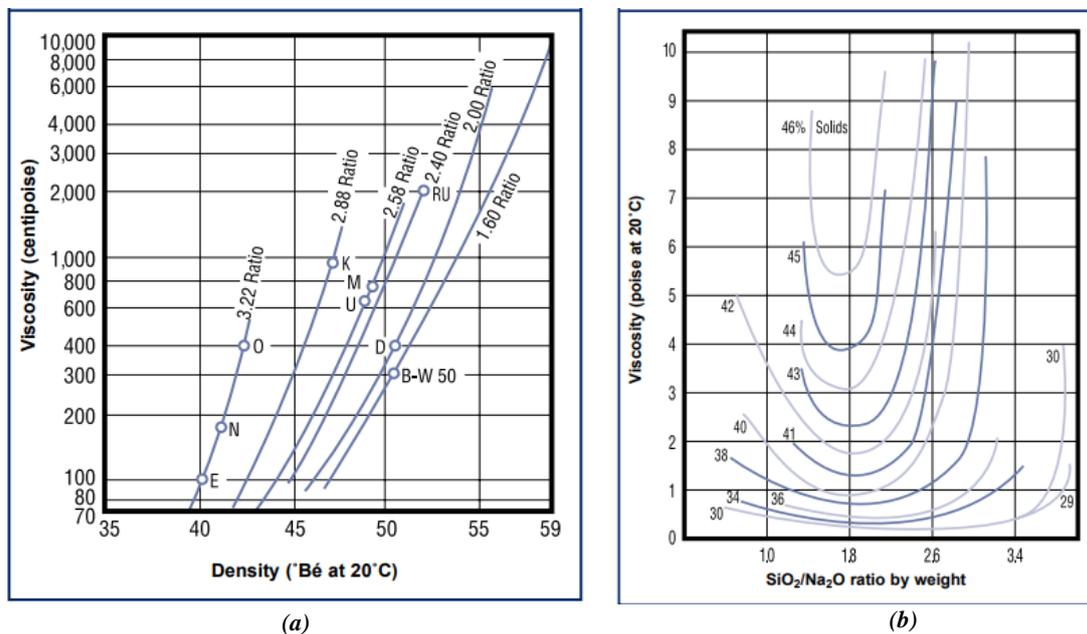


Figure 2.33 Viscosities of  $\text{Na}_2\text{SiO}_3$  solutions (a) as a function of density (b) as a function of weight ratio at constant solids (adopted from PQ Corporation 2004 online publication (accessed 14/5/2019))

*Polymerization/Gelation:* Iler (1979) and McDonald and Thompson (2006) have reported this reaction occurring rapidly at pH levels lower than 10.7, where crosslinking of silicate species is triggered, and polymers formed. However, Weldes and Lange (1969) identified

increased viscosity as the trigger for monomers of the  $\text{Na}_2\text{SiO}_3$  species crosslinking to form polymers/gel. It should be noted that increasing viscosity is making solution more siliceous, and therefore, crosslinking is almost entirely with silica monomers. This process was demonstrated by Iler (1979), noting that polymerization occurs in distinct ways at two pH boundaries (see Figure 2.34):

- (1) pH below 7 down to 2: Within this range, monomer polymerization is slow enough to allow the early progressive stages to be followed leading to the formation of three-dimensional condensed silica polymers. Changes may occur in hours, especially at pH 2. The polymer units here have no charge. Particles are very small and cease to grow after reaching a diameter of 2–3 nm, and aggregation occurs just as polymeric particles form and aggregation can also occur at low silica concentration.
- (2) pH above 7: Here aggregation may not occur as particle continues growing to a larger diameter. Changes are very rapid, occurring in few minutes or seconds. Aggregation and gelling may only occur in the presence of a significant amount of an electrolyte.

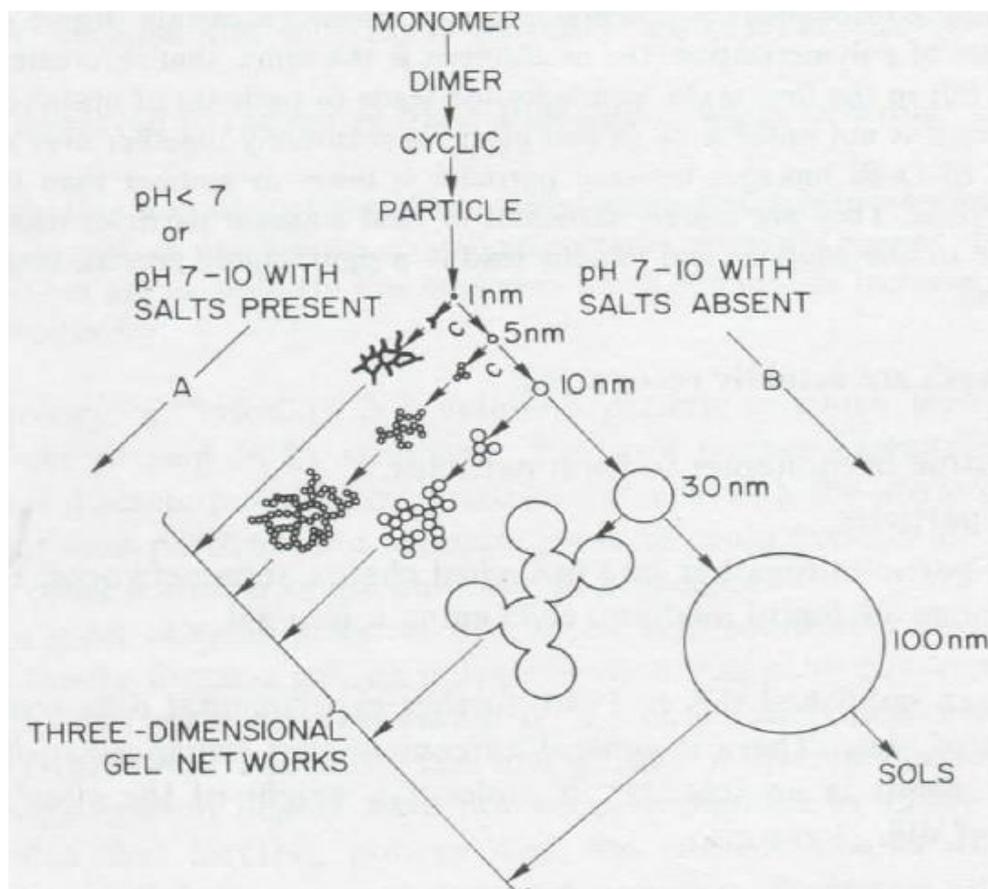


Figure 2.34 polymerization and gelling of silica monomers in (A) alkaline solution, and (B) in acid solution (adopted from Iler (1979))

Silicate polymerization produces lower strength compared to dehydration but produces higher levels of water resistance for treated materials (McDonald and Thompson, 2006). It was noted that agglomeration by polymerization/gelation is better achieved where agglomerated material is acidic or is exposed to high CO<sub>2</sub> environment, and that setting can be enhanced by materials such as acidic salts, organic acids, esters of acidic acid or carbonates ((Iler, 1979); McDonald and Thompson (2006); Lee and Stebbins (2009)). The addition of polymerizing aid (or hardeners) to silicate treatments is relatively common in grouting and foundry industries, with low alkaline 3.2 ratio silicate further recommended as best suitable for polymerization/gelation reaction since it neutralizes faster (McDonald and Thompson, 2006).

*Precipitation:* According to McDonald and Thompson (2006), when an agglomerated material containing multivalent metal ions (e.g. Ca<sup>+2</sup>, Mg<sup>+2</sup>, Zn<sup>+2</sup>, Cu<sup>+2</sup>, Fe<sup>+3</sup>, etc.) reacts with aqueous silicates, a corresponding insoluble metal silicate is precipitated almost instantly. The presence of significantly high amounts of cations on the surface of the agglomerated material allows the silicate to act as a chemical binder. Iler (1979) reported precipitates of gel and excess Ca(OH)<sub>2</sub> from a silicate of 3.3 ratio and suggest that 3.2–3.3 ratios are most suited for chemical bonding since it is the negative siliceous portion of the silicate that reacts with the cations.

#### 2.10.4 Sodium silicate - soil reaction mechanism

The exact reaction between Na<sub>2</sub>SiO<sub>3</sub> and soil is not well understood because only a little is known of the silicate gel formation process. Some authors such as Laws and Page in 1946 have without a detailed investigation of the nature of the reaction mechanism, declared that Na<sub>2</sub>SiO<sub>3</sub> reacted with several common clay minerals present in soil. Again, Ruff and Davidson in 1961 stated that because the reaction of Na<sub>2</sub>SiO<sub>3</sub> with calcium salts in water solution forms insoluble gelatinous calcium silicate (CaSiO<sub>3</sub>), and because hydrate CaSiO<sub>3</sub> is well understood as a cementing agent, it could be used in soil stabilization (Hurley and Thornburn, 1972). Consequently, the Na<sub>2</sub>SiO<sub>3</sub>–soil reaction merely occurs between calcium ions (Ca<sup>2+</sup>) within soil and the silicate ions (SiO<sub>3</sub><sup>2-</sup>) in the Na<sub>2</sub>SiO<sub>3</sub>. Thus, calcium-based substances, such as lime, CaCl<sub>2</sub>, etc. have been introduced to soils in order to react with Na<sub>2</sub>SiO<sub>3</sub> for stabilization purposes (Ou *et al.* (2009); Maaitah (2012); Chien *et al.* (2014)). However, silicates can also result from solubilisation of soil's hydrated silica as the alkalinity of the system is raised by the Na<sub>2</sub>SiO<sub>3</sub> solution, thus, enhancing the CaSiO<sub>3</sub>-hydrate levels produced (Hurley and Thornburn, 1972). Furthermore, gel-like sodium-aluminosilicate-hydrates have been identified as the

cement produced from clays treated with a special  $\text{Na}_2\text{SiO}_3$  brand (TX-85) (Latifi *et al.* (2014); Pakir *et al.* (2015)).

In spite of much study carried out on the exact nature of the bonding action of these  $\text{CaSiO}_3$ -hydrates, no adequate explanation exists (Hurley and Thornburn, 1972). Iler (1979) considers the silicate to be in the form of polymeric chains of silicon-oxygen connections with sodium spread along the chain of the oxygen. A polyvalent ion ( $\text{Ca}^{++}$  or  $\text{Mg}^{++}$ ) added, results in  $\text{Na}^+$  been replaced by the divalent ion permitting bonding together of 2 chains to create an insoluble gel with a cross-linked structure.

Laws and Page (1946) recognized the purpose of silicate addition to suitable soils to be to decrease their swelling and plasticity index, to provide water-stable aggregation and resistance to crushing. According to Murray, 1952 the  $\text{CaSiO}_3$  precipitates as a continuous matrix with a strong and rigid ionic polar bond formed between stabilizer and soil (Hurley and Thornburn, 1972). This understanding informed Wooltorton (1955)'s claim that anionic exchange was the process involved, leading to the development of insoluble silicate sheaths surrounding the soil grains, with a cementation effect.

From these ideas, Hurley and Thornburn (1972) cited the 1959 M.I.T Cambridge soil stabilization research report, indicating key variables to be looked out for: (i) the amount of polymeric silica, responsible for the volume of gel; (ii) the rate of occurrence of sodium atoms along the chain, which dictates the amount of possible cross-linking; and (iii) the concentration of the precipitant, which controls the degree of cross-linking.

Laws and Page (1946) on the other hand had state that, though low active clays like kaolinite may react satisfactorily with  $\text{Na}_2\text{SiO}_3$ , the addition of silicate to highly active clays like montmorillonite, weakens their stability. Mainfort 1945; 1955; and the Massachusetts Institute of Technology 1959; 1960 were cited to have confirmed  $\text{Na}_2\text{SiO}_3$  as not reacting adequately with many fine-grained soils (Hurley and Thornburn, 1972).

When a  $\text{Na}_2\text{SiO}_3$ -treated soil is exposed to basic groundwater or to air, the life of the stabilized soil may become limited. Processes that may result in the destruction of silica gel in the voids of a soil include shrinkage due to either syneresis (i.e. spontaneous expulsion of moisture) or desiccation, and erosion or dissolution by groundwater (Hurley and Thornburn (1972) and references therein). On the contrary, McDonald and Thompson (2006) recently found that air drying or heating is necessary for the setting of silicate bonds, and where a water-resistant bonding is needed either heating at very high temperatures is required to form a glassy bond or by chemical setting.

All varieties of silica gels are known to suffer syneresis, and there are laboratory reports indicating the loss of moisture ranging between 20% and 60% by mass within a period of two months. Depending on the concentration of the original silicate solution and reacting agents, there is the possibility for some gels to shrink to about 25% of their initial volume. In a personal communication, Terzaghi in 1947 (cited in Hurley and Thornburn (1972)) reported a loss of cohesion and a reversed permeability of a single-step sand-gel due to desiccation. Although the loss of moisture in the  $\text{Na}_2\text{SiO}_3$ -treated soils studied in this project produced minor cracking, the strength of specimens under compression was rather observed to increase with drying (see Figure 5.8 in Chapter 5). A position in agreement with Dogra and Uppal, 1958 (Hurley and Thornburn, 1972). Dogra and Uppal (1958) found that a  $\text{Na}_2\text{SiO}_3$ -treated sand-clay cured under room ambience for long periods increased compressive strength.

#### 2.10.5 Geotechnical application of sodium silicate

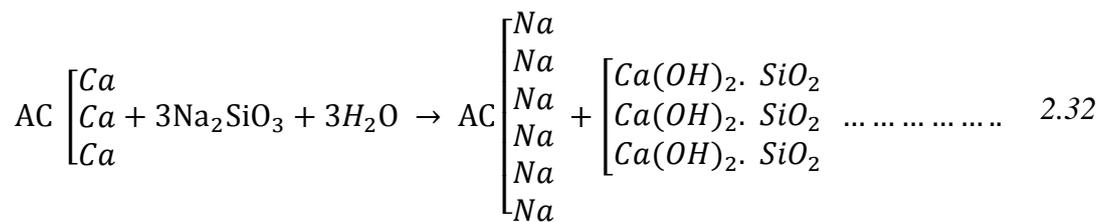
It is over a century since  $\text{Na}_2\text{SiO}_3$  has been applied in improving building materials. In the early 19<sup>th</sup> century, it was proposed by a pioneer in the development of commercial soluble silicates, that silicates have the potential for hardening artificial stones, as well as recognizing their reaction with calcium compounds. During that period, the manufacture of artificial stones from the mixing of silicate solutions with solution of calcium compounds became common as several patents were issued (Hurley and Thornburn, 1972).

Sodium silicate has been used as dust palliative since the early 20<sup>th</sup> century. Cementing agents such as calcium silicates and aluminium silicates were precipitated into voids of road stones by reacting  $\text{Na}_2\text{SiO}_3$  solution with calcium and aluminium salts, respectively. This brought about the development of macadam roads, where a slurry of finely ground limestone and  $\text{Na}_2\text{SiO}_3$  were used to form cementitious matrix to hold crushed stones in place (Hurley and Thornburn, 1972). Also, a French engineer, Albert Francois in about 1915 found that  $\text{Na}_2\text{SiO}_3$  was effective in aiding deeper penetration of cement grouts because of its slipperiness. The slippery silicate gel coats granular grains and aid movement of succeeding grouts (Hurley and Thornburn, 1972).

Despite the fact that many polyvalent metals are known to precipitate cement element with  $\text{Na}_2\text{SiO}_3$  (McDonald and Thompson, 2006), its effectiveness on soils appears to be more promising in the presence of calcium ions, precipitating an insoluble  $\text{CaSiO}_3$  gel. This fills soil pores, provides imperviousness and increases strength. This method has been widely adopted in foundation works to control seepage. Riedel 1952 however, noted that it is not capable to

solidify soil within three feet of the ground surface. Injecting  $\text{Na}_2\text{SiO}_3$  with other compounds like sodium bicarbonate may as well produce a bonding gel but is not insoluble, deteriorates with time and also has lower strength than  $\text{CaSiO}_3$  gel (Hurley and Thornburn, 1972).

The use of  $\text{Na}_2\text{SiO}_3$  in geotechnical engineering practice became very popular for treating collapsible loessial soils in the mid-20<sup>th</sup> century after Askalonov 1949 developed a technique coined “Silicatization,” which was a method of injecting silicate solution into collapsing loess soils to reduce collapse and prevent water permeation (Sokolovich, 1965). The method which based on Equation 2.32 (Sokolovich, 1965) was successful in increasing UCS, imperviousness, and non-slumping qualities of a treated loess. Silicatization which was developed at the Institute of Foundation Beds, Russia has most of its applications and study on loess and carbonate sands in the former Soviet Union (Sokolovich (1965); Askalonov (1966); Sokolovich and Ibragimov (1971); Sokolovich (1976); Beketov *et al.* (1967); Sokolovich and Gubkin (1970); Rzhantsyn (1967); Sokolovich (1971)).



From the preceding discussions,  $\text{Na}_2\text{SiO}_3$  appeared to be applied mainly in granular soils by two different processes: a 2-step and single-step injection methods. The 2-step injection involves injecting a  $\text{Na}_2\text{SiO}_3$  solution followed by a second solution or vice versa while the single-step involves one-time injection of a mixture of a  $\text{Na}_2\text{SiO}_3$  solution and an acid or base. In all injection processes, the solution is nearly the same, containing colloidal silicic acid and free sodium hydroxide. The introduction of acids or salts into the silicate solution causes formation of a gel. However, the processes each present advantages over the other. For instance, in the 2-step process the zones and degree of mixing by the separate solutions cannot be determined. Mainfort in 1952, raised concerns that in a 2-step process there is the likelihood of the second chemical pushing the first forward of it, preventing satisfactory mixing or at least mixing in the required proportions. However, recently, 2-step electroosmotic method has been successfully used to treat soft clays both in laboratory and field scale tests by the injection of a  $\text{CaCl}_2$  solution followed by a  $\text{Na}_2\text{SiO}_3$  solution (Ou *et al.* (2009); Chien *et al.* (2014)). Chien *et al.* (2014) reported an electroosmotic system referred to as suitable operation process (SOP).

Nevertheless, in this process, after injection of the  $\text{CaCl}_2$  solution, careful monitoring of current and timing was needed before subsequently injecting  $\text{Na}_2\text{SiO}_3$ , in order to achieve desired result. Through the SOP implemented in the field, Chien *et al.* (2014) reported increases in cone resistance and undrained strength of 130% and 125%, respectively.

The single-step injection process on the other hand, presents various uncertainties. Some of these may include, selecting the correct proportions of the mix components to ensure that gel formation occurs at an appropriate time. Setting should not occur during pumping but at a reasonable period after pumping has ended. This may be challenging except where  $\text{Na}_2\text{SiO}_3$  was used solely. McDonald and Thompson (2006) stated that reaction of  $\text{Na}_2\text{SiO}_3$  with any metal or acidic compound forms an instantaneous setting, and their addition should be after  $\text{Na}_2\text{SiO}_3$  treatment. That, setting time can also be controlled by the nature of soil being grouted. Setting times as short as 15 minutes may be appropriate for a highly pervious soil with rapid flow of groundwater while a longer time may be necessary for moderately permeable sand, to allow penetration of the solution to the desired distance from the point of injection. However, lime-sodium silicate mixes were used to reduce plasticity, improve swelling potential and CBR of an expansive clay in Karak, Jordan (Maaitah, 2012).

Environmental factors generally may also affect the setting time of the silica gel and therefore, the success rate of treatment. Terzaghi, 1947 noted that solution injected into saturated sand will become diluted, and other conditions being the same, dilution can delay setting time by 2 to 20 times of those for undiluted material. On the contrary, he stated that setting time could be shortened up to 30% when solution is injected into dry sand, and that the presence of considerable salt contents (even  $\text{CaCO}_3$ ) in soils or groundwater, substantially reduces setting time. Further evidence revealed that setting may be completely prevented where organic matter is present.

Terzaghi 1947 and Riedel 1952 (Hurley and Thornburn, 1972) presented laboratory compressive test results of impregnated sands that indicate a better performance of the 2-step process over the single-step. Terzaghi, while comparing the strength of an acid gel and that of an ultrabasic gel formed from the single-step method, noted that the acid gel exhibited about 5 to 8 times higher strengths than the ultrabasic gel. The acid gel-sand exhibited strengths between approximately 172 to 193 kPa at periods ranging up to 5 days. These values according to Riedel, were merely a fraction for specimens treated with the 2-step injection process, which showed strength ranging between approximately 689 to 2413 kPa, few minutes post treatment.

Recent publications in field of study generally appear to suggest uses of  $\text{Na}_2\text{SiO}_3$  solutions to be predominant in two areas. The first one is its use as alkaline activator for pozzolans such as PFA, GGBS, etc. (Sugama *et al.* (2005); Bernal *et al.* (2011); Phoo-ngernkham *et al.* (2015); Davari Algoo *et al.* (2019)) while the second area is its use along with calcium-based solutions (Ou *et al.* (2009); Maaitah (2012); Chien *et al.* (2014)). These are applied through various techniques to treat a range of soils including soft clays.

#### ***Sole application of sodium silicate solution or with an additive***

Even though the sticky nature of  $\text{Na}_2\text{SiO}_3$  solution is capable of agglomerating soil particles, it may not provide strengths required for geotechnical applications. Consequently, most reported research indicates using the solution with an additive. This notwithstanding, Sherwood in 1961 had stated that  $\text{Na}_2\text{SiO}_3$  could be used solely; however, it is suitable for only stabilizing sandy soils in a moderate climate. He argued further that treating soils with silicates combined with other chemicals will yield variety of results. Sherwood, however lamented the near absence of field data to corroborate the promising results acquired in the laboratory (Hurley and Thornburn (1972) and references therein).

Nevertheless, the possibility of  $\text{Na}_2\text{SiO}_3$  solely improving clays has recently been argued by Moayedi *et al.* (2011) who noted that  $\text{Na}_2\text{SiO}_3$  can only be effective with an additive. Moayedi and colleagues argue that, the negative silicate ions will typically attach to the positively charge broken edges of clay particles, and therefore causing the entire clay particles to become negatively charged. That, this will result in the particles repelling one another, thus, the clay structure becomes weak and dispersed.

More recently, however, a few studies have also revealed the potential of  $\text{Na}_2\text{SiO}_3$  solely improving the engineering properties of clays (Latifi *et al.* (2014); Pakir *et al.* (2015)). Both Latifi *et al.* (2014) and Pakir *et al.* (2015) reported a sodium silicate solution specially branded “TX-85” to have increased UCS of a lateritic soil and an expansive marine clay, respectively. The results show that UCS increases with curing time, and with increasing amount of the solution up to certain thresholds. Above those thresholds, UCS values began to plunge. The plunge was probably due to the increase in the positive surcharge and the subsequent repulsion of soil particles inside the mixture (Latifi *et al.*, 2014) or increased moisture content (Pakir *et al.*, 2015). However, these results may not be relied upon because the composition of the specially branded solution (“TX-85”) was not made known.

## 2.11 Chapter Summary

### 2.11.1 Prerequisite for collapse

There appears to be some consensus on what features make a soil metastable or collapsible whether natural or anthropogenic. Foremost of these features include an open structure, unsaturated state, low dry density, high void ratio and porosity, and high enough initial bonding. Almost all kinds of soils can exhibit these features given the right conditions of placement or ageing, but loess or loessial soils appear to be the commonest forms of collapsible soils dominating the literature. Thus, available knowledge on the collapse mechanisms of loessial soils is vital for the study of other collapsible soils. However, in general, the mechanism of metastability and consequent collapse potential is yet to be fully understood. While the main prerequisite of collapse occurring in any soil is the formation of an open metastable structure, some arguments exist around whether collapse can occur merely by wetting without loading, whether the degree of saturation controls collapse, whether bonding element type and pH of wetting fluid determine the magnitude of collapse.

### 2.11.2 The mechanism and prediction of collapse

There is a large body of knowledge attempting to describe the mechanism of collapse base on either the principle of effective stress or bond shear resistance (bond weakening and/or softening). However, the latter appears to more adequately describe this phenomenon because the former defines an increase in water content to result in volume increase rather than volume decrease. Notwithstanding the modifications made over the years to fit the principle of effective stress in describing unsaturated collapsible soils, it is still not adequate to explain key elements of the collapse phenomenon, e.g., the destruction of chemical bonding.

Several techniques for predicting and classifying collapse have been proposed. They are grouped into indirect correlations, laboratory, geophysical, and field loading methods. Although laboratory methods are seen to be best for describing these soils, it is argued that no single testing method is adequate to fully describe collapse in any soil. Notwithstanding the importance of standardized testing methods, there is the need for collapse testing to be site specific and anticipatory. For instance, standardized wetting fluid in laboratory testing may not mirror all sites pore fluid. There is the need to investigate the varying impact of different bonding materials on collapse relative to wetting fluid type.

A major drawback of many of the collapse predicting techniques (particular, with the indirect correlations) is the fact that they base mostly on a single soil parameter, e.g. dry density, clay content, degree of saturation, porosity, etc. Researchers have criticised most of them as

been misleading, e.g., a low dense soil having an open structure with strong bonding may not show significant collapse upon wetting, except a high enough stress breaks bonds before or during wetting. This explains why many of the classification methods at best only suit those soils they were derived. There is also the need to understand how different soil parameters influence collapse interdependently.

### 2.11.3 Inter-particle bonding material and collapse potential

The open structure of collapsible soils is argued to form from the support of bonding existing at particle contacts, which provides an initial high strength and stiffness. Either or a combination of, suction, clay, calcium carbonate, or other salts provide this bonding. The presence of significant amounts of  $\text{CaCO}_3$  in these soils, however, defines them as calcareous soils.

Fundamentally, collapse is said to occur where the bonding element at the particle contacts is weakened or destroyed upon wetting with or without stressing. However, bonding materials can either be stable or unstable depending on the wetting fluid type and degree of stressing. The bonding material type and wetting fluid type are either unwittingly or wittingly ignored in the defining of collapsible soils; and this is potentially one reason the mechanism of collapse remains poorly understood. While soil suction, for instance, is commonly believed to reduce with increased wetting, with a consequent collapse effect on such soils, the impact of some bond materials on collapse due to wetting is still been debated.

The impact of  $\text{CaCO}_3$  bond on collapse has largely been argued. Some scholars believe that its rate of dissolution (in ordinary water commonly used in laboratory testing) is too slow to demonstrate the “sudden collapse upon wetting phenomenon” while others are of the opinion that since  $\text{CaCO}_3$  is a common bond material in natural collapsible soils, the collapse effect is a result of such bonds been weakened/softened upon wetting. Nevertheless, the literature is yet to quantify the impact of  $\text{CaCO}_3$  on collapse. Again, though calcite is seen as the commonest polymorph of  $\text{CaCO}_3$  occurring in soils, it is important to take a closer look at the impact of all different polymorphs (calcite, aragonite and vaterite) on the collapse phenomenon in relation to different wetting fluids. This issue forms the main objective of this study and has been investigated further, thus our concentration on collapsible calcareous clay soils.

The role of clay bonding on collapse appears to be adequately described. Nevertheless, describing collapse potential on the bases of clay content remains debated. Considering that all soil types can be collapsible under certain conditions, and that a pure clay soil can also be collapsible, then claims that certain amounts of clay make a soil either collapsible or

noncollapsible cannot be generalized. A further understanding on this is also provided as part of the main objectives of the present study.

#### 2.11.4 Collapse mitigating measures

Collapse mitigating measures have been well reported. These measures may be grouped broadly into mechanical, physical, chemical, and biological methods, which have been applied “successfully” in some ways. Surprisingly, the effectiveness of most of these measures to mitigate collapse has merely been inferred from their effectiveness to improve strength, neglecting the impact of mineral dissolution upon wetting. Again, some of the methods can only apply preconstruction while others like the chemical grouting have the advantage of applying at both pre- and post- construction stages. However, some commonly used chemicals are also known to be harmful to the environment or produce deleterious substances in certain conditions. Thus, the use of safer and effective chemicals such as sodium silicate has become a target for researchers.

Because of the environmental harmless nature of  $\text{Na}_2\text{SiO}_3$  solution, it has been used as a stabilisation chemical mainly to treat granular soils but in most cases, it was applied with additives to enhance gelation. The key properties of  $\text{Na}_2\text{SiO}_3$  solution controlling its effectiveness are  $\text{Na}_2\text{O}/\text{SiO}_2$  molar ratio and concentration. The effectiveness of  $\text{Na}_2\text{SiO}_3$  when adopted without an additive to treat soils is still argued whereas, the impact of different curing methods on silicized samples is almost not reported.

Again, the use of  $\text{Na}_2\text{SiO}_3$  as a sole chemical for soil treatment appears to be lacking attention in recent researches. Rather, most recent works have shown interest in its use with different additives, or merely as an alkaline activator of pozzolans. Therefore, much direct knowledge of  $\text{Na}_2\text{SiO}_3$  behaviour with soil is absent. Calcium-based chemicals are typically utilized (as additives) with  $\text{Na}_2\text{SiO}_3$  and one major challenge with the use of  $\text{Na}_2\text{SiO}_3$  solution is its high viscosity or the efficiency of a solution with lowered viscosity. Consequently, this research investigated the potential of a well formulated  $\text{Na}_2\text{SiO}_3$  solution to solely stabilise calcium-based soils (calcareous silty-clays).

## 2.12 Research Questions and Gaps in the Literature

Despite the large body of knowledge on soil collapse, this review of the literature shows that the available knowledge is still insufficient to fully understand the phenomenon of collapse. Pertinent research questions in key areas remain unanswered:

1. Notwithstanding the wide discussions on the impact of calcium carbonate bonding in collapsible soils, there appear to be little or no simplified laboratory techniques available in the geotechnical theme, for effectively producing carbonate samples for collapsibility studies. A rigorous process has been followed to develop a suitable technique effectively producing laboratory calcareous samples satisfying objectives (2) and (3) of this study.
2. What is the impact of a chemical bonding element (e.g., calcium carbonate) on the collapse phenomenon? Notwithstanding a few researches available in this area, there is no clear evidence of how different amounts of  $\text{CaCO}_3$  content may control the rate and magnitude of collapse. This question need addressing through laboratory investigation to satisfy objective (4), and part of (5).
3. Many of the available collapse classification systems only based on one criterion, e.g., clay content, dry density, suction and degree of saturation, porosity, etc. The question that needs addressing here is the global validity of those classification systems and whether there is no dependence between different criteria? Assessing the role of some of these criteria and establishing relationships between them is also necessary to satisfying objective (5).
4. Under what wetting conditions and stress states are certain chemical bonding (e.g.,  $\text{CaCO}_3$ ) of engineering consequence, and worthy of consideration in geotechnical characterization of soils? The impact of mineral dissolution on soil behaviour is typically not considered by available soil classification systems (e.g., USCS). This is perhaps why wetting in collapse testing has mostly been performed with ordinary water, not considering the effect of other wetting fluids with differing pH as may be the case in field situation. Addressing this question will meet objective (6).
5. How is soil strength related to its collapse potential? Despite the debate about the strength and stiffness of unsaturated collapsible soils, a correlation between these properties (particularly for chemically bonded soils) appears to be absent in the literature. This question need addressing through laboratory investigation to satisfy objective (7).
6. Do calcareous silty-clay soils have the potential to be used as foundation or fill materials in the face of  $\text{CaCO}_3$  dissolution? Alternatively, can they be effectively treated with a sodium silicate solution for geotechnical application even in acidic environment? What strategy of application, type and amount of solution can better enhance strength and mitigate collapse in calcareous silty-clay soils? What is the impact of curing method on a silicatized soil? These are questions not fully answered in the literature. The potential of a well-selected  $\text{Na}_2\text{SiO}_3$  solution has been investigated as part of objective (8).

## Chapter 3 Methodology

### 3.1 Overview

The problem of collapse has been associated with almost all kinds of soils. The microfabric criteria for collapse to occur have also been reasonably established. However, the influence of different soil parameters on the collapse phenomenon under varying testing conditions remain debatable. This thesis was set out to investigate mainly two aspects of soil collapsibility. Firstly, the impact of soil parameters such as calcite bonding, clay content, etc. on the collapse phenomenon (including rate of collapse) in calcareous silty-clay samples as related with wetting fluid type. The other aspect is the mitigation of collapse – the potential of a well-selected sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) solution to improve these samples. Also, compressive strength testing of both untreated and treated samples was carried out to evaluate the relationship between UCS and collapsibility.

This chapter, however, only presents the laboratory methodological framework adopted to produce precipitated  $\text{CaCO}_3$ , selection of soil materials, design and manufacture of artificial calcareous silty-clay samples for the main testing programme, and a description of the geotechnical testing methods adopted. The chapter is broken into three broad stages as shown in the Figure 3.1. Details of each of the stages are described under sections 3.3 to 3.5.

### 3.2 Summary of each Stage Objectives

- 1) Stage 1 consist two levels of preliminary studies. First is to review the literature to identify potential techniques for precipitating  $\text{CaCO}_3$  in a clay soil, then evaluate and identify both advantages and disadvantages of each technique with emphasis on which is most promising to produce pure  $\text{CaCO}_3$  samples. Secondly is identifying the most promising technique and conducting advance study on it; assessing factors capable of influencing the process of  $\text{CaCO}_3$  precipitation (desired contents in view) as well as production of consistent samples and developing a suitable setup for the process.
- 2) Stage 2 is the production of main tests samples that poses collapsible features. This involves selection of soil materials, sample design (including sample physical properties) and their rationale.
- 3) Stage 3 involves determining the geotechnical properties of samples, such as collapse potential, rate of collapse and unconfined compressive strength, in a manner sufficient to evaluate relationships that satisfy objectives set out in section 1.2.

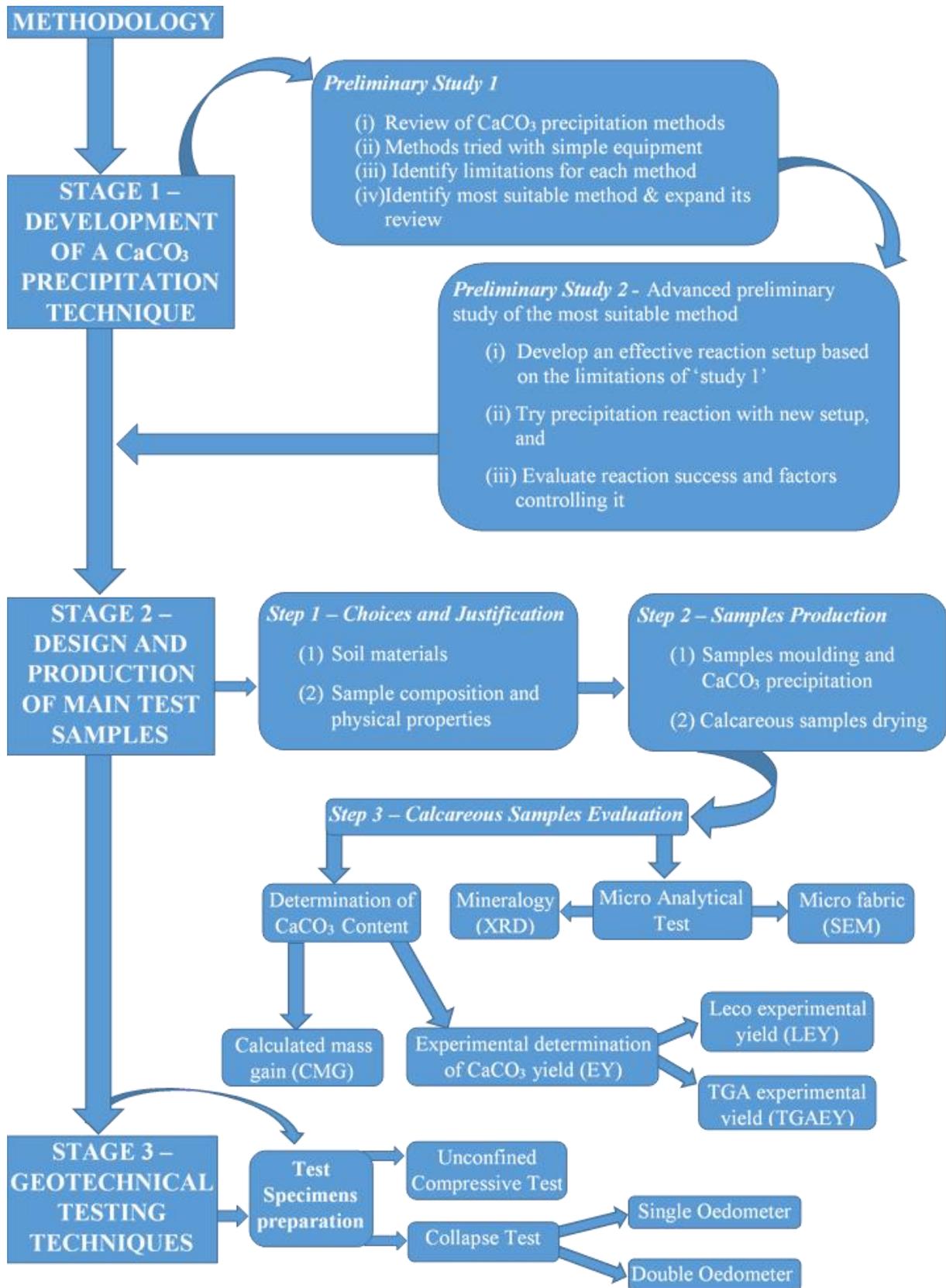


Figure 3.1 Stages of the laboratory methodological framework of the study

### 3.3 Stage 1 – Development of a CaCO<sub>3</sub> Precipitation Technique

#### 3.3.1 Preliminary study 1

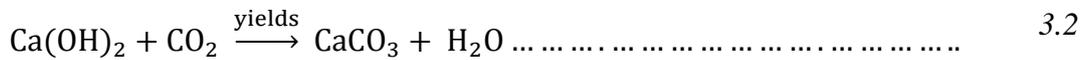
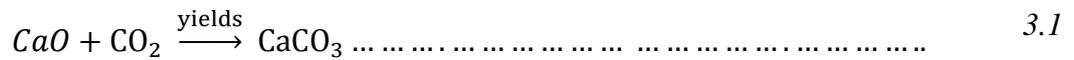
In nature, both primary and pedogenic CaCO<sub>3</sub> in soils develop from precipitations from either CaCO<sub>3</sub>-rich shell of organisms (organic carbonate) or from chemically saturated solutions (inorganic carbonate) under certain physicochemical processes in both marine and terrestrial environments. Carbonates may precipitate either purely or in the presence of detrital materials, producing different carbonate soils with varying amounts of CaCO<sub>3</sub> or levels of calcareousness. When these soils undergo diagenetic processes and recrystallization, carbonate/calcareous soils and rocks with different degrees of induration or cementation are created (Pettijohn (1975); Fookes and Higginbottom (1975); (1988); Semple (1988); Carter *et al.* (1999)).

The geotechnical engineering literature seems to contain only a little information pertaining to effective methods of artificially manufacturing calcareous cementation in soils. For instance, it was observed by Allman and Poulos (1988) that modelling natural process of cementation was impracticable. However, there are some out-of-discipline reports about carbonation, from which knowledge can be adapted. Some of these involved reacting a calcium-rich material with another rich in carbonate. In order to mimic the natural processes of inorganic CaCO<sub>3</sub> synthesis or precipitation in soils in laboratory conditions, three chemical reaction routes (Equations 2.24, 2.26 and 2.27 described in sections 2.7 and 2.8) involving calcium-rich (Ca(OH)<sub>2</sub>, CaCl<sub>2</sub>) and carbonate-rich (Na<sub>2</sub>CO<sub>3</sub>, CO<sub>2</sub>) compounds were tried at a pilot scale to ascertain which could most effectively produce calcareous samples. Amongst those, the reaction route involving calcium oxide (CaO) and carbon dioxide (CO<sub>2</sub>) was adjudged most appropriate because others were observed to precipitate additional unwanted components, such as NaCl and NaOH, capable of influencing the behaviour of samples. Section 3.3.2 briefly describes this process and its adjudged merits over others. However, health and safety concerns related to long-hours exposure to CaO and others (detailed in section 3.3.2) informed sourcing its hydrated form (Ca(OH)<sub>2</sub>); hence Equation 3.4 above.

#### 3.3.2 Our understanding about lime-carbon dioxide reaction

Lime carbonation by the capture of carbon dioxide is a well-established process (Bagoniza *et al.* (1987); Beruto and Botter (2000); Despotou *et al.* (2016)). Both forms of lime, calcium oxide (quicklime) and calcium hydroxide (hydrated lime) can absorb atmospheric carbon dioxide to precipitate CaCO<sub>3</sub>. A preliminary study was initiated where availability of materials informed an initial reaction of quicklime (CaO) and carbon dioxide (CO<sub>2</sub>) following Equations 3.1 and 3.2, using a reaction system and procedures presented in Appendix A3. The aim was to

assess both efficiency of the reaction system as well as the effectiveness of the reactants (CaO and CO<sub>2</sub>) to precipitate relatively predictable amounts of CaCO<sub>3</sub>.



Unfortunately, the following drawbacks were identified from the above soil-CaO-CO<sub>2</sub> reaction as well as reaction setup:

- Even though this reaction is known to be near instantaneous in nature, the main concern was whether the reaction apparatus effectively distributed permeating CO<sub>2</sub> gas to reach all possible reaction sites within the soil-lime system. On the other hand, it was also possible that CO<sub>2</sub> escaping from the outlet pipe could have only passed through weak zones developed within the sample.
- The longer the permeation time allowed, the more CO<sub>2</sub> wasted assuming vents were created in the sample. This results to poor cost effectiveness.
- As indicated in Equation 3.2, carbonation of quicklime first goes through the slaking process to form hydrated lime before proceeding to precipitate CaCO<sub>3</sub>. The slaking process requires large volumes of water, and this could impede the carbonation reaction, which is diffusion controlled. Therefore, if quicklime was to be used in this process, complete slaking should first be achieved before proceeding to carbonation. Evaluation of this was complex and undesirable for the scope of this study.
- Apart from the health and safety challenges (e.g. respiratory and skin irritation, and eye damage) identified with the usage of CaO, the heat generation from the exothermic reaction during slaking causes significant loss of water through evaporation and this made it difficult to estimate the amount of water required to prepare samples. Calculation of the moisture content was necessary to determine the initial dry density of the samples.
- The increased temperature from the exothermic reaction would possibly reduce CO<sub>2</sub> density making it easier to permeate through (possible weak zones) the soil-lime system to escape. In addition, CO<sub>2</sub> becomes even less diffusive thus delaying the carbonation process.

### 3.3.3 Preliminary study 2

To counter the above drawbacks, a new reaction setup, which allows for an effective and non-destructive production of samples and specimens was developed in-house (Figure 3.2). An industrial slaked lime, calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) was preferred over quicklime ( $\text{CaO}$ ).

Therefore, a further preliminary investigation was conducted, reacting the industrial  $\text{Ca}(\text{OH})_2$  and  $\text{CO}_2$  within the innovative reaction setup shown as Figure 3.2. The following probable influencing factors on the success of carbonation were considered at this stage: reaction period, initial dry density and moisture content of sample.

### 3.3.4 The innovative reaction setup developed in-house

The reaction apparatus as shown as Figure 3.2 was developed from a pressure cell. The cell was modified to include the following features:

- A gas inlet and an outlet. The inlet allows for  $\text{CO}_2$  admittance into cell loaded with the soil-lime compact while the outlet allowed escape of free air from the cell at the early reaction stage.
- Staking rack. This was used to hold several soil-lime mix prepared either into oedometer test specimens or as large samples. It increases the capacity of the cell to hold a greater number of specimens for each reaction cycle.
- Pressure gauges. One fitted to the cell was for closely monitoring both cell pressure and  $\text{CO}_2$  capture. The second one was a pressure transducer connected to a computer to monitor rate of  $\text{CO}_2$  capture in real-time. This was only employed at the preliminary stage to understand at what time  $\text{CO}_2$  capture ceased or became insignificant.
- Thermometer. This was used to measure temperature rise during the carbonation process. Because the thermometer could only read temperatures on the outer pressure cell, the readings were not considered reasonably accurate. If not for time constrain the setup could have been modified to fit thermometer inside cell.

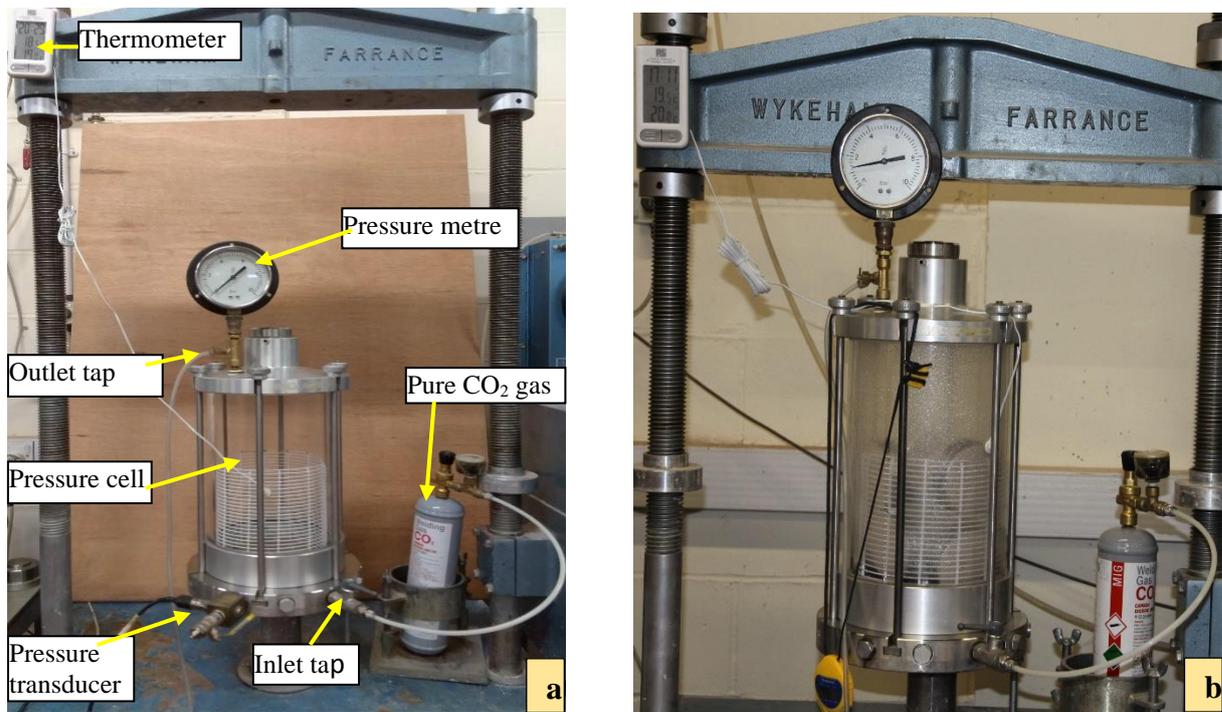


Figure 3.2 Reaction apparatus for sample carbonation *a*, empty setup *b*, stacked with oedometer specimens

The setup was calibrated by admitting gas into cell up to a cell pressure of about 3 bars, leaving all valves closed, and allowing it to stand for up to 48 hours. The pressure gauge was observed to deflect only slightly after this period. To remove free air from pressure cell, lime slurry bubbling was adopted. The free air was released (bubbled) into a lime slurry and the slurry becomes milky and thickens when CO<sub>2</sub> starts bubbling into it. This was observed to begin within the first minute. The escape of CO<sub>2</sub> gas was believed to follow that of free air in the cell because, air is lighter and should float on CO<sub>2</sub> gas. The reason for this test was to maintain the high concentration of the CO<sub>2</sub> in the reaction cell.

Key advantages of the above setup include:

- The apparatus could confine and distribute gas within cell to virtually all possible reaction fronts in the soil-lime system. Therefore, even carbonation can be achieved.
- Since the pressure distribution is uniform on all surfaces of samples (rather than being fed from one end of the sample), there is a reduced tendency for the gas to cause mechanical damage to the samples (i.e. creation of weak zones or vents).
- Wastage of CO<sub>2</sub> is minimized because only an approximately enough is admitted into the cell while its outlet valve remained closed. The pressure dial gauge deflection indicates CO<sub>2</sub> capture and becomes almost stationary when capturing is nearly complete. A rapid drop in pressure is noticed during the early periods of CO<sub>2</sub>

admittance, which gradually slows down. This informs how much more gas should be allowed in the cell enough for a probable complete carbonation.

### ***Samples preparation and carbonation experiment***

The commercial English China clay (Kaolin) was mixed in dry state with 10% hydrated lime (by dry mass of kaolin), that is, 9.09% by total mass of kaolin-lime mix. This mix was used to prepare several samples with relation to two compactive variables, moisture content and dry density. Different amounts of water were used to mix with proportions of the kaolin-lime mix and kneaded for several minutes until smooth consistency was observed before storing in sealable plastic bags for about 24 hours. At the end of storing, wet mixes were statically compacted (by volume control) into cylindrical samples at predetermined dry densities ranging between 1.00 and 1.61 Mg/m<sup>3</sup>.

Compacted cylindrical specimens (of the size of a standard compressive test specimen) were placed in the reaction cell (without rack) and setup assembled as shown in Figure 3.3.



*Figure 3.3 Cylindrical specimens undergoing carbonation*

While the outlet valve remained closed, CO<sub>2</sub> gas was admitted into the reaction cell through the inlet valve to cell pressures maintained between 2 and 3 bars for the initial few minutes when the rate of CO<sub>2</sub> capture was still high. After capture rate was observed to significantly reduce, the inlet valve was then closed, and reaction left to continue for different periods up to a maximum of 48 hours. However, the amount of CO<sub>2</sub> (in terms of cell pressure)

was monitored at intervals to see if there was need to admit additional CO<sub>2</sub> into the cell. Each time further significant pressure drops occurred, more CO<sub>2</sub> was admitted.

At the end of carbonation experiments, simple qualitative and quantitative methods outlined in the following section were employed to evaluate the success level of the reaction.

### ***Methods for the confirmation of calcium carbonate precipitate***

Before proceeding to carry out a quantitative test to determine the amount of CaCO<sub>3</sub> precipitate post-carbonation and drying, two qualitative methods ((1) and (2) below) were adopted to confirm the presence of CaCO<sub>3</sub> in the samples. Leco experimental technique ((3) below) was used for quantitative analysis. However, at later stages in the project thermogravimetric analysis and x-ray diffraction were also adopted.

- (1) *Acid–base colour indicator*: phenolphthalein turns colourless in near neutral (pH < 8.2) or acidic solutions. At pH approximately 8.2 it begins to show light pinkish colour and progressively deeper colour with increasing pH up to about 9.8 where deep pink emerges. Phenolphthalein solution has been used by many to detect carbonation of lime–treated soils and lime–mortars by spraying it on their fresh broken surface. Where surface indicated deep pink, it shows untransformed portlandite (pH ~12.4) implying no carbonation took place whereas light pink to colourless indicated different carbonation degrees (Despotou *et al.* (2016); Bagoniza *et al.* (1987)). CaCO<sub>3</sub> has a pH between 8.0 and 8.5 (Bagoniza *et al.*, 1987). The initial soil-Ca(OH)<sub>2</sub> mix before reacting with CO<sub>2</sub> was sprayed with phenolphthalein, and it indicated very bright/deep pink-red colour. Just after reaction, samples were halved, and phenolphthalein solution sprayed on the inside surface. Non-carbonated portions of the sample showed bright pinkish-red colour while converted portions maintained original sample colour (see Figure 3.4 for example).
- (2) *Calculated mass gain (CMG)*. The dry mass of sample before and after carbonation where determined. Their difference represented yield. This was compared to the expected theoretical yield (i.e., the change in mass). The theoretical change in mass expected was determined as follows: since the molar masses of Ca(OH)<sub>2</sub> and CaCO<sub>3</sub> are 74.09 and 100.09 grams, respectively, in principle 1 gram of Ca(OH)<sub>2</sub> completely carbonated (reacted with CO<sub>2</sub>) was expected to be converted to CaCO<sub>3</sub> in the ratio of 1:1.35, representing a 35% increase in mass. This method reveals in semi-quantitative terms the amount of CaCO<sub>3</sub> produced.
- (3) *Experimental determination of CaCO<sub>3</sub> content*. The Leco equipment (see details in Appendix A5) was employed to determine CaCO<sub>3</sub> content in terms of percentages, in

each of the carbonated samples. Test specimens were obtained from the core of randomly selected samples, and test performed in accordance with BSI7755 (1995), Section 3.8; ISO 10694, 1995; Soil Quality, Part 3 (Chemical Methods), determination of organic carbon and total carbon after dry combustion. Total carbon (TC) and organic carbon (OC) were determined and then inorganic carbon deduced by difference.

**Results of preliminary carbonation studies**

The influence of three main variables (as discussed in section 3.3.3) on carbonation success were investigated. These are: carbonation period, initial moisture content and dry density. Figure 3.4 to Figure 3.6 show the relationship between these variables and success level of the reaction process in terms of percent conversion (compared to the theoretically expected) of lime to calcium carbonate.

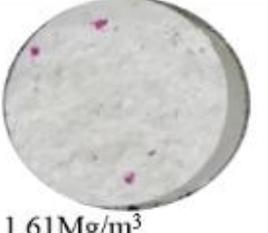
<i>Moisture condition before carbonation</i>	<i>Colour after spraying phenolphthalein solution after carbonation</i>		
Specimens prepared and sprayed additional water up to 35 - 40% moisture content before carbonation	 1.61Mg/m <sup>3</sup>	 1.31Mg/m <sup>3</sup>	 1.01Mg/m <sup>3</sup>
Specimens prepared with 22.08% moisture content before carbonation	 1.61Mg/m <sup>3</sup>	 1.31Mg/m <sup>3</sup>	 1.01Mg/m <sup>3</sup>
Specimens prepared with 8.7% moisture content before carbonation	 1.61Mg/m <sup>3</sup>	 1.31Mg/m <sup>3</sup>	 1.01Mg/m <sup>3</sup>
Specimens prepared and oven dried to 0% moisture content (allowed to cool) before carbonation	 1.61Mg/m <sup>3</sup>	 1.31Mg/m <sup>3</sup>	 1.01Mg/m <sup>3</sup>

Figure 3.4 Colour observation by spraying phenolphthalein solution after carbonation

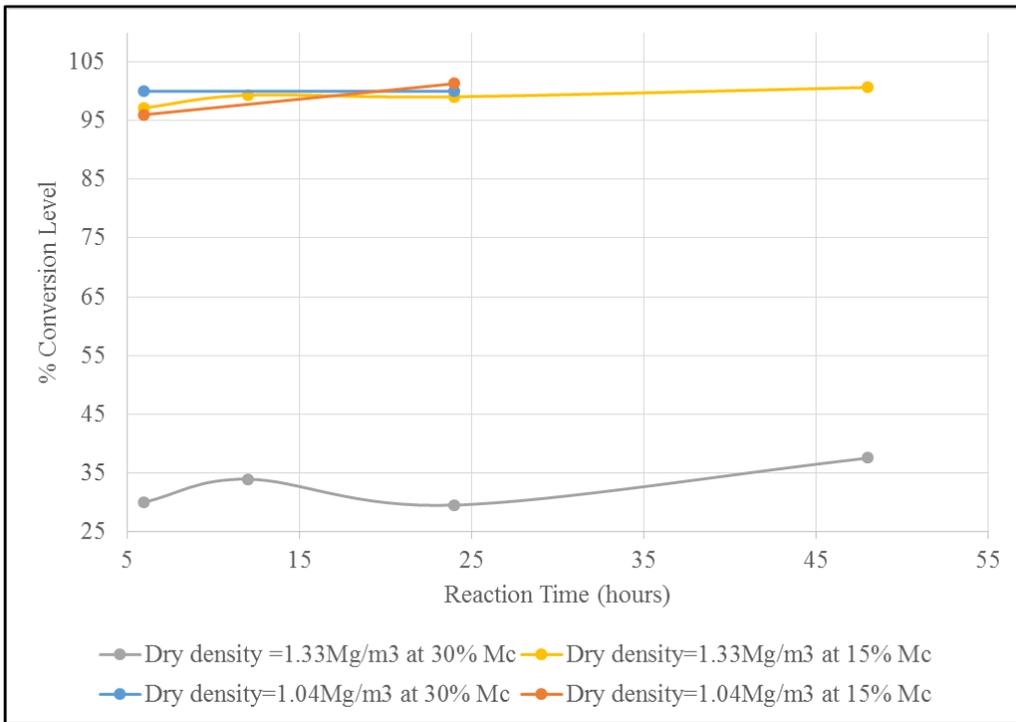
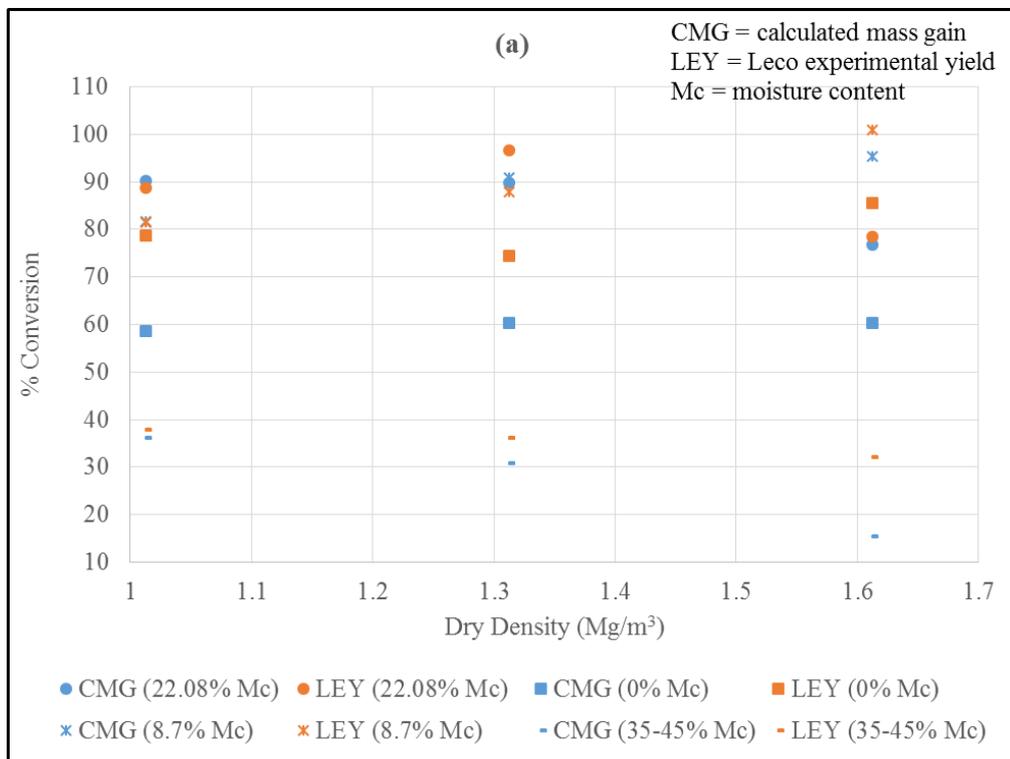


Figure 3.5 Varying carbonation (reaction) periods and conversion level of a clay-lime system prepared differently



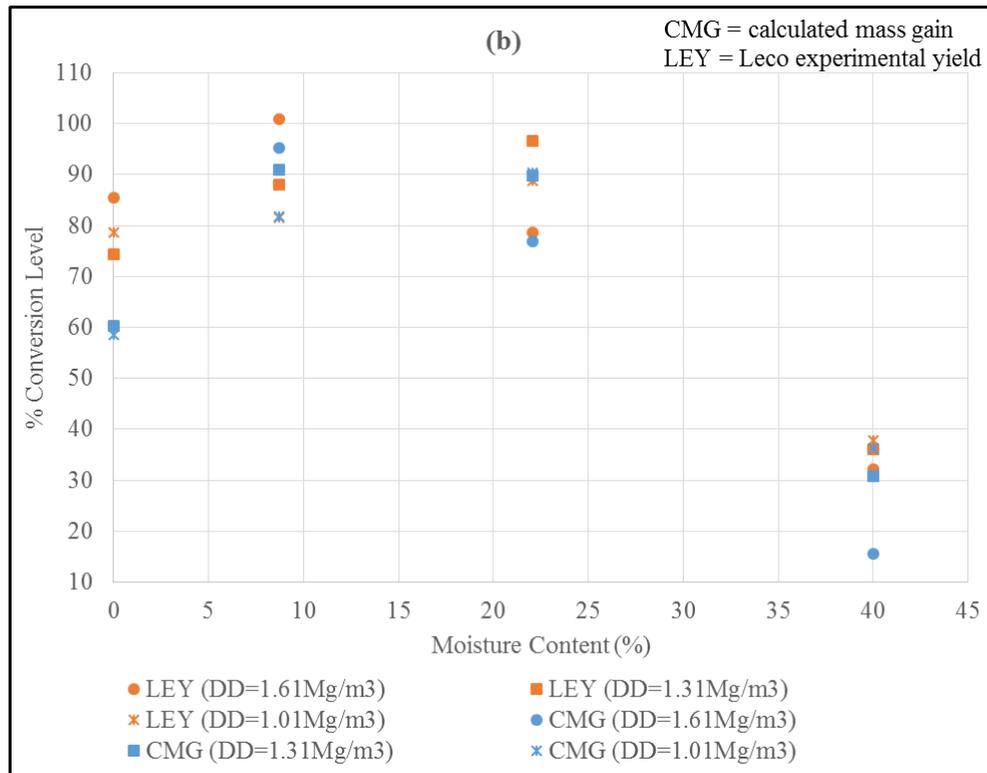


Figure 3.6 Carbonation success determined by the experimental (Leco) yield and calculated mass gain (a) the effect of initial dry density (b) effect of initial moisture content, before carbonation.

### ***Preliminary conclusions about the carbonation method***

From the results presented in the foregoing section, the following conclusions are drawn:

- The durations of reaction, at least those adopted here (6, 12, 24 and 48 hours) did not significantly influence the degree of lime-to-calcium carbonate conversion. This agrees with existing knowledge that lime-CO<sub>2</sub> reaction is instantaneous and the possible delay in this reaction would be reduced accessibility of CO<sub>2</sub> to lime particles.
- Initial reaction moisture content is a major controlling factor for the CO<sub>2</sub> carbonation of lime. Moisture content at a certain threshold (depending on density of sample) appears to favour the reaction. Above or below that level carbonation was strongly affected, although to a lesser effect below the threshold.
- Observations reveal that at dry state, the reaction was delayed and then later progresses slowly. This appear to indicate that the carbonation reaction might require some form of moisture to proceed. It is possible that the CO<sub>2</sub> gas confined within the reaction cell will start condensing after a prolonged period, thus providing moist conditions that subsequently stimulate and gradually progresses the process.
- Though initial dry density appeared to influence the carbonation reaction, it does not in itself affect the process. As the results indicate, samples with similar densities behaved

differently following their moisture contents, confirming that the rate and degree of lime-to-calcium carbonate conversion through CO<sub>2</sub> carbonation was mainly diffusion controlled. That is, by the level of CO<sub>2</sub> accessibility to lime fronts. High moisture content in both dense and low dense compacts resulted in low degrees of lime conversion. This is because CO<sub>2</sub> diffusion in water is very low and access to reaction fronts is rather impeded.

- For effective carbonation, both high and low-density compacts must contain moisture contents low enough to permit gas access to reaction fronts, and dry carbonation of compacts may not be desired.
- Following the understanding provided and successes demonstrated above, this method appeared to be valid for manufacturing the ‘main’ calcareous samples required for the present thesis.

### 3.4 Stage 2 – Design and Production of Main Test Samples

#### 3.4.1 Material choices and justification

Calcite is a common soil mineral (Mitchell and Soga, 2005) that develops and cement particles enabling soil to maintain a loose structure (Coop and Airey, 2003 in Mitchell and Soga (2005)). Even though calcareous clays abound in many parts of the globe including the UK (see section 2.5.1 for details), artificial samples were preferred in this study, to allow both the inherent variations in natural calcareous soils to be avoided and target properties such as, degree of calcareousness, dry density, etc. to be accurately controlled (Allman and Poulos, 1988).

Commercial kaolin and an alluvial quartz-silt material were used to form primary soils (silty-clay) before the precipitating CaCO<sub>3</sub>. The choice of these materials was informed by:

- 1) Their physicochemical nature. Both kaolin clay and quartz-silt are stable and chemically inert, which will mean they will not interfere with the chemical reaction required to precipitate CaCO<sub>3</sub>.
- 2) These form the composition of commonly studied (natural and engineered) collapsible soils, e.g., alluvial silts are typical of loessial soil (Delage *et al.*, 2008). Common collapsible soils have grain sizes ranging between clay ( $\leq 2 \mu\text{m}$ ) and silt ( $2 \mu\text{m} < 63 \mu\text{m}$ ) and sand in some cases (Barden *et al.* (1973); Rogers (1995); Assallay (1998); Derbyshire and Mellors (1988); Mansour *et al.* (2008)).

Even though some of the key criteria for collapse, e.g., low dry density, high void ratio and porosity have been deliberately exaggerated ( $\sim 1 \pm 0.1 \text{ Mg/m}^3$ , 1.1 – 1.8, 52 – 65, respectively) in the studied samples to better understand the cementing impact of CaCO<sub>3</sub> on the collapse

phenomenon, values are however, comparable to soils studied by authors such as Hull (1988); Allman and Poulos (1988); Ismail *et al.* (2002b). Furthermore, normally consolidated clay have void ratio,  $e$  and porosity,  $n$  of 1.73 – 3.86 and ~60%, respectively (Kokusho *et al.*, 1982), and natural sediments have  $e = 1.0 – 6.0$  and  $n = 50\%$  (Shibuya *et al.*, 1998). Sterianos (1988) reported void ratio values between 1 and 2 as typical of natural carbonate sediments. These values compare to those of the present samples. Also see Table 2.5 in section 2.5.1 and Bui *et al.* (2010) for more global void ratios and porosities.

Following researchers, e.g., Lawton *et al.* (1989); Basma and Tuncer (1992); Assallay (1998); Medero *et al.* (2005); Jefferson and Ahmad (2007), who produced (non-chemically cemented) artificial collapsible soils mainly by compaction (either by volume control or varying compaction moisture contents) to achieve predetermined dry densities, void ratio and saturation, this study has adopted the same methods in conjunction with the  $\text{CaCO}_3$  precipitation method examined in section 3.3.3, to produce samples. Proportions of  $\text{CaCO}_3$  precipitated are similar to those occurring in many natural clays (see Table 2.5 for example) and some loessial soils (see El-Sohby *et al.* (1988); Jefferson *et al.* (2005); Delage *et al.* (2008); Li *et al.* (2016)).

### 3.4.2 Kaolin clay

The clay used in this project was a brand of English-China clay (Polwhite E grade) supplied by *Imerys Performance Minerals Ltd*, UK. Its properties as reported by the supplier are presented in Table 3.1 and Figure 3.7. Kaolin clay, a hydrated aluminium silicate mineral (kaolinite) is generally preferred in many geotechnical laboratory experiments because of its unique characteristics such as chemical inertness and insolubility.

*Table 3.1 Properties of Polwhite E English China clay as supplied by Imerys Performance Minerals ltd, UK*

<i>Property</i>	<i>Value</i>
Appearance	Powder
Colour	White/off-white
Solubility	Insoluble in water
pH	5
SiO <sub>2</sub> content	50%
Al <sub>2</sub> O <sub>3</sub> content	35%
Particle density	2.6
Surface area (BET)	8 m <sup>2</sup> /g
Particle size	See Figure 3.7
Moisture content	1.5%

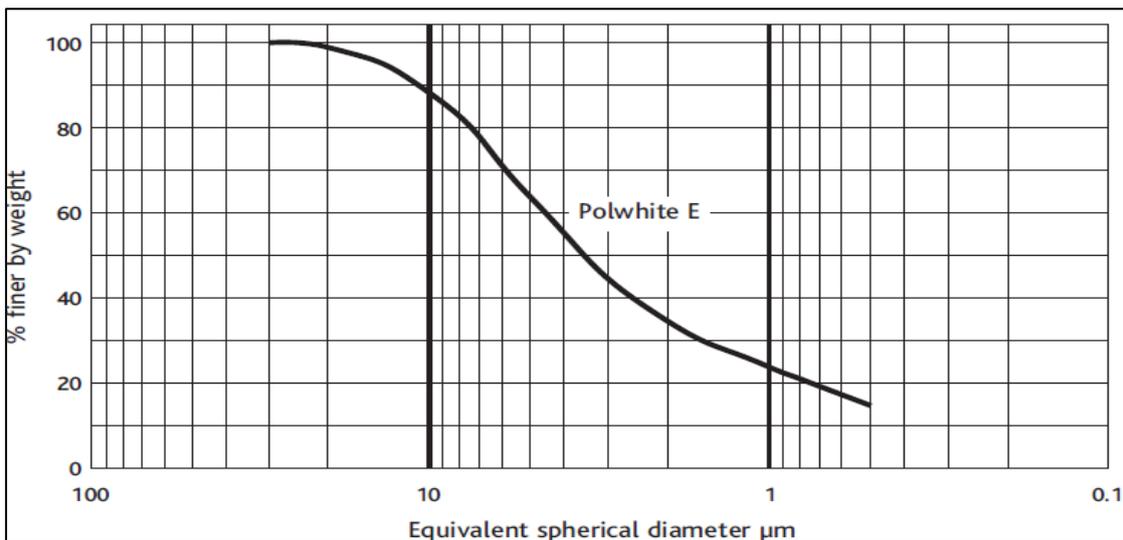


Figure 3.7 Particle size distribution of Kaolin (data by supplier)

### 3.4.3 Quartz silt

Due to a lack of availability of appropriate commercially sourced silt material, the quartz-silt for this project was extracted from a natural alluvial material. The Lanton alluvium from Lanton River, Northumberland, UK was observed to contain mainly silt and fine sand. The alluvium was sieved to obtain the sub-63 μm portion. However, it was originally concluded that the sub-63 μm soil would only be usable if it was free of carbonate content and its plasticity index classed as low because a stable silt material was desired for this study.

Wet and dry sieving methods (according to BS1377 (1990b)) were examined on the Lanton alluvium for the purpose of recognizing which could be more viable for the extraction exercise. The following steps were followed:

#### *Wet Sieving*

- The wet alluvium was washed into a bucket through a 63 μm mesh and allowed to settle until water above becomes clear. The clear water was decanted and soil air dried. The dry soil was then weighed to know mass of sub-63 μm obtained. Lumps and aggregates that resulted were crushed, then soil goes through a round of dry sieving process again.

#### *Dry Sieving*

- The wet alluvium was air-dried, then lumps and aggregates were crushed using mortar and pestle. The crushed soil was sieved through the 63 μm mesh with the aid of a mechanical shaker, then the sub-63 μm soil was weighed to know amount retrieved.

Even though wet sieving produced more of the sub-63  $\mu\text{m}$  portion, it was much more labour intensive than dry sieving which produced enough. So, it was more efficient to dry sieve only.

The index properties of the extracted sub-63  $\mu\text{m}$  were determined in accordance with the relevant section of the BS1377 (1990b) to further classify it following classification systems such as the unified soil classification system (USCS), AASHTO, and ISC. An attempt also, was made to remove the sub-3  $\mu\text{m}$  portion of the sub-63  $\mu\text{m}$  soil to obtain a near pure silt portion. This was done by means of dispersing soil in a cylindrical bucket, allowing an appropriate period of time, based on Stokes' Law of sedimentation (Equation 3.3) for  $\geq 3 \mu\text{m}$  particles to settle and then decanting clay in suspension.

$$V = \frac{H}{T} = \frac{D^2 g(\rho_s - \rho_L)}{18\eta} \dots\dots\dots 3.3$$

Where

$V$  = velocity of falling particle (m/s),  $H$  = height (m) of particle fall in time  $T$  (s),

$D$  = effective diameter of falling particle (mm),  $g$  = acceleration due to gravity ( $\text{m/s}^2$ )

$\rho_s$  = specific gravity of falling particle,  $\rho_L$  = specific gravity of the liquid

$\eta$  = viscosity of the liquid (poise)

The Atterberg limits for the  $\geq 3 \mu\text{m}$  portion were also determined in accordance with BS1377 (1990b). Their index properties as well as classification by three distinct methods are presented in both Figure 3.8 (PSD) and

Table 3.2. Figure 3.8 reveals ~10% clay size content. A fizzing test to confirm the absence or otherwise of carbonates using dilute (1%) hydrochloric acid (HCl) was also carried out and the soil was observed to be free of carbonates. These outcomes informed the acceptability of the extracted silty soil for this project and the clay content in the sub-63  $\mu\text{m}$  extract was considered in the main sample design.

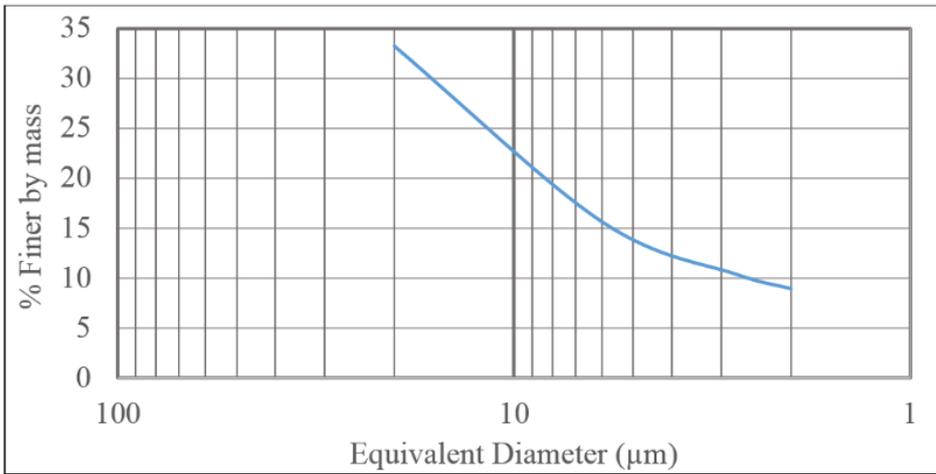


Figure 3.8 Particle size distribution of the Sub 63 microns of the Lanton Alluvium

Table 3.2 Index properties of the sub-63 µm of the Lanton Alluvium

Index Property	Sub-63 µm portion	3 µm to 63 µm portion
<i>Atterberg limits</i>		
Liquid Limit (%)	34	33
Plastic Limit (%)	23	26
Plasticity Index (%)	11	7
<i>Presence of carbonates (by dilute HCl spraying)</i>	No fizzing	No fizzing
<i>Classifications</i>		
USCS	ML, inorganic silts of low plasticity.	ML, inorganic silts of low plasticity
AASHTO	A-4 Silty soil	A-4 Silty soil
ISC	CL, inorganic clays of low plasticity	Between ML & CL

### 3.4.4 Hydrated lime (Ca(OH)<sub>2</sub>)

The cementation by precipitated calcium carbonate was performed by a carbonation reaction involving Ca(OH)<sub>2</sub> (Table 3.3) supplied by *Lafarge Tarmac, UK* and a pure CO<sub>2</sub> gas supplied by *SIP Industrial, Italy*. This reaction, carried out in the innovative setup, converted Ca(OH)<sub>2</sub> into CaCO<sub>3</sub> bonds following Equation 3.4.

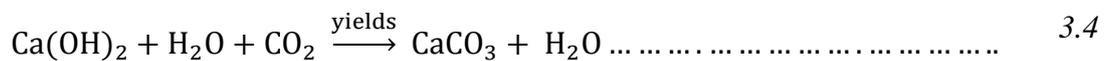


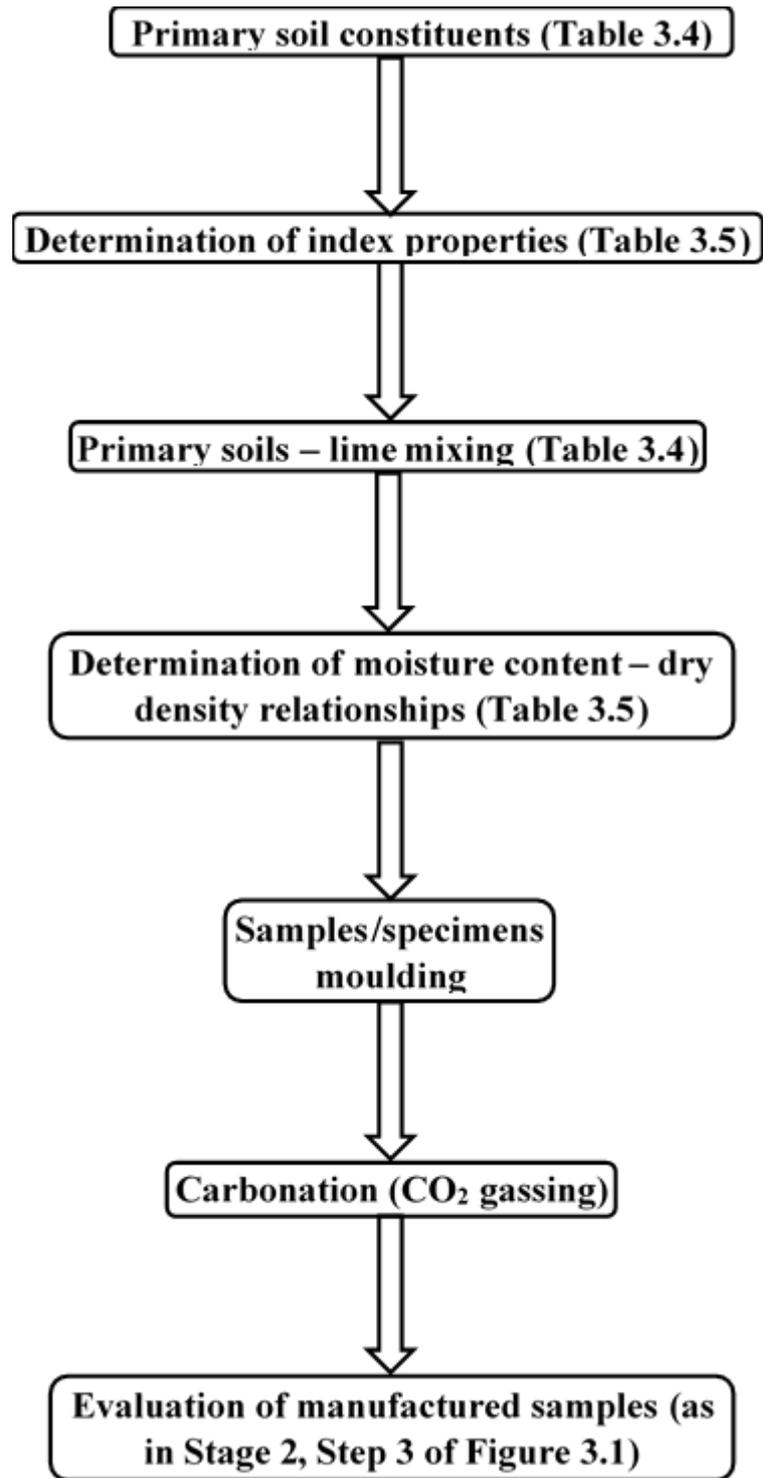
Table 3.3 Properties of the hydrated lime as presented by Lafarge Tarmac, 2016.

<i>Property</i>	<i>Value</i>
<i>Chemical properties</i>	
Calcium Hydroxide – $Ca(OH)_2$	97.0 % (minimum 95.0)
Magnesium Hydroxide – $Mg(OH)_2$	0.50 % (maximum 1.4)
Carbon Dioxide – $CO_2$	0.70 % (maximum 1.00)
Silica – $SiO_2$	0.70 % (maximum 1.00)
Alumina – $Al_2O_3$	0.10 % (maximum 0.20)
Iron Oxide – $Fe_2O_3$	0.06 % (maximum 0.10)
Sulphur – $S$	0.01 % (maximum 0.025)
Arsenic – $As$	0.3 ppm (maximum 1.0)
Fluorine – $F$	65 ppm (maximum 110)
Lead – $Pb$	1.3 ppm (maximum 5.0)
Manganese – $Mn$	175 ppm (maximum 250)
Loss on Ignition	24.80 %
<i>Physical properties</i>	
Moisture – $H_2O$	0.25 % (maximum 0.75)
Form	Fine white / off-white powder
Surface Area (SSA)	1400 – 1900 m <sup>2</sup> /kg
Bulk Density	450 – 520 kg/m <sup>3</sup>
Mean particle size (D50)	7 – 9 μm

### 3.4.5 Sample constitution and production procedures

As noted in section 3.4.1, samples were simply constituted with material proportions not mimicking any particular pre-existing sample. The procedures summarized as Figure 3.9 and succeeding details (including Table 3.4 and

Table 3.5) below were followed.



*Figure 3.9 Summary of main samples production procedures*

Table 3.4 designed soil samples composition: degree of calcareousness defined according to Day's 1983 classification of calcareous soils, cited in Kishchuk (2000)

Composition						
Sample	Before carbonation				Theoretical expected after carbonation	
	% silt (quartz)	% clay (kaolin)	Clay/silt ratio	% Ca(OH) <sub>2</sub>	% CaCO <sub>3</sub>	Calcareousness
A0	50	50	1	0	0	Non
B0	35	65	1.9	0	0	Non
C0	20	80	4	0	0	Non
A1	45	45	1	9	~ 12 %	Moderately
B1	31	59	1.9	9	~ 12 %	Moderately
C1	18	72	4	9	~ 12 %	Moderately
A2	40	40	1	20	~ 25 %	Strongly
B2	28	52	1.9	20	~ 25 %	Strongly
C2	16	64	4	20	~ 25 %	Strongly
A3	33	33	1	33	~ 40%	Very Strongly
B3	23	43	1.9	33	~ 40%	Very Strongly
C3	13	53	4	33	~ 40%	Very Strongly

Table 3.5 Index properties of Primary Soils with varying proportions of lime (uncarbonated)

Property	Primary Soil A – 50% QS & 50% KC				Primary Soil B – 35% QS & 65% KC				Primary Soil C – 20% QS & 80% KC			
	0% Ca(OH) <sub>2</sub>	9% Ca(OH) <sub>2</sub>	20% Ca(OH) <sub>2</sub>	33% Ca(OH) <sub>2</sub>	0% Ca(OH) <sub>2</sub>	9% Ca(OH) <sub>2</sub>	20% Ca(OH) <sub>2</sub>	33% Ca(OH) <sub>2</sub>	0% Ca(OH) <sub>2</sub>	9% Ca(OH) <sub>2</sub>	20% Ca(OH) <sub>2</sub>	33% Ca(OH) <sub>2</sub>
Specific Gravity, Gs (Calculated)*	2.65	2.61	2.57	2.51	2.64	2.58	2.56	2.50	2.62	2.56	2.54	2.49
OMC (%)	17	24	25	27	22	25	28	29	24	28	29	33
MDD (Mg/m <sup>3</sup> )	1.55	1.48	1.45	1.38	1.48	1.45	1.39	1.34	1.53	1.43	1.39	1.29
Atterberg Limits	LL (%)	42	-	-	46	-	-	-	52	-	-	-
	PL (%)	32	-	-	34	-	-	-	34	-	-	-
	PI	10	-	-	12	-	-	-	18	-	-	-

Notes: Maximum dry density (MDD) and Optimum Moisture content determined using standard Proctor (2.5 kg rammer); QS = quartz silt, KC = kaolin clay  
Gs calculated by weighted average of constituent materials from values reported by their producers: Polwhite E China Clay – 2.6; Hydrated lime – 2.24; and silt assumed 2.7; Calcium carbonate – 2.71 adopted from Moorehead (1986).

### *Details of procedures*

- (1) Mix designs presented in Table 3.4 were chosen for this project. Three soils with clay/silt proportions of 50/50, 65/35, 80/20 were first created and referred to as primary soils *A*, *B*, and *C*, respectively and their Atterberg limits determined (
- (2) Table 3.5). Each of these primary soils were then mixed with 0, 10, 25, and 50 %  $\text{Ca(OH)}_2$  (by mass of primary soil) in a dry state for several minutes until a uniform distribution was obtained. This formed twelve samples with constituent proportions as shown in the Table 3.4. The samples were labelled *A0*, *A1*, *A2* and *A3*; *B0*, *B1*, *B2* and *B3*; *C0*, *C1*, *C2* and *C3*. Where *0*, *1*, *2* and *3* represent the 0%, 9%, 20% and 33%  $\text{Ca(OH)}_2$ , respectively. 0%  $\text{Ca(OH)}_2$  represented non-calcareous sample while the other values correspond to estimates expected to precipitate  $\text{CaCO}_3$  contents to produce *moderately*, *strongly* and *very-strongly* calcareous samples, respectively. Apart from these main samples, others (named *D1*, *D2* & *D3*) have also been constituted for certain checks as the project progressed
- (3) Primary soil- $\text{Ca(OH)}_2$  mixes were tested to obtain their optimum moisture contents (OMC) and maximum dry densities (MDD) (
- (4) Table 3.5 and Figure 3.10) in accordance with relevant sections of BS1377 (1990a). After this, the nine samples containing lime were each prepared to two different moisture contents ( $100\pm 2\%$  of OMC and  $50\pm 2\%$  of OMC) for compaction to vary density related properties. The soil- $\text{Ca(OH)}_2$  systems were mixed with the appropriate amount of water by thoroughly kneading by hand to avoid local wetting and the formation of lumps. Kneading continued for several minutes until samples were observed to be smoothly consistent. Then stored in air-tight sealable plastic bags for a period not less than 48 hours under room temperature for moisture homogenization. Compaction using the standard Proctor (lightweight rammer, 2.5kg), and static methods were then adopted to obtain various densities.
- (5) (i) Samples compacted with the standard Proctor method (Appendix A2), having a height and diameter of 115 and 105 mm respectively, were extruded from compaction mould (by means of an hydraulic jack) and divided into approximately four equal parts (to reduce thickness for effective  $\text{CO}_2$  penetration) within a short space of time. Cut samples were covered with plastic bags to avoid moisture loss. Samples prepared with water contents higher than approximately 15% were allowed to air dry until moisture contents fell to or below that level (based on preliminary results) before proceeding to

the carbonation stage. On the other hand, those samples mixed with water contents lower than this were taken for carbonation immediately after compaction and cutting.

(ii) For statically compacted specimens, only samples mixed with  $50\pm 2\%$  of OMC were used to prepare specimens to predetermined dry densities. Here, the volume-controlled compaction method was adopted in order to easily obtain the initial density required. The oedometric and compression test (CT) specimens were prepared using respective split moulds (Appendix A4). These moulds, with known volumes, were assembled with their bases and appropriate amounts of prepared soil (to achieve predetermined dry densities) were carefully and gradually emptied into the moulds, ensuring even distribution. The top pistons were then placed on top of the soil and then statically compacted by the aid of a dead load until the gap between the mould and top piston closed completely. Where a dead load could not fully compact, a loading machine was employed. At full closure of the top piston, compacted specimens were left a few minutes before removing the load and disassembling the mould to avoid possible rebound of the soil. The oedometric specimens were compacted into an oedometer ring assembled into the compaction mould while CT specimens were compacted directly into the moulds. Both specimens were retrieved by splitting the moulds. However, in some cases CT specimens were better retrieved by extrusion using a hydraulic jack. Moulded specimens were then weighed and transferred into the carbonation cell.

- (6) The next stage was carbonation of the compacted samples/specimens following the procedures noted in section 3.3.3. The preliminary study indicated that any time between 6 to 48 hours enough for the carbonation required. Thus, 24 hours was chosen for convenience.
- (7) Wet carbonated samples/specimens were weighed immediately upon removal from the carbonation cell and then oven dried (temperatures between 45 and 50 °C) to obtain dry or unsaturated conditions.
- (8) The success of the reaction process, in terms of degree of lime conversion was also evaluated following methods (2) and (3) stated in section 3.3.3 in addition to thermogravimetric analysis and X-ray diffraction (XRD). Scanning electron microscopic (SEM) images were also obtained to examine the microstructure of the manufactured specimens in comparison with collapsible soils in the literature. Specimens used here were oven dried at standard temperatures (between 110 to 115 °C), which revealed moisture contents ranging from 0.12% to 0.45% retained after the first

oven (45 – 50 °C) drying. This would likely cause some form of discrepancies in the “calculated mass gain” evaluation method.

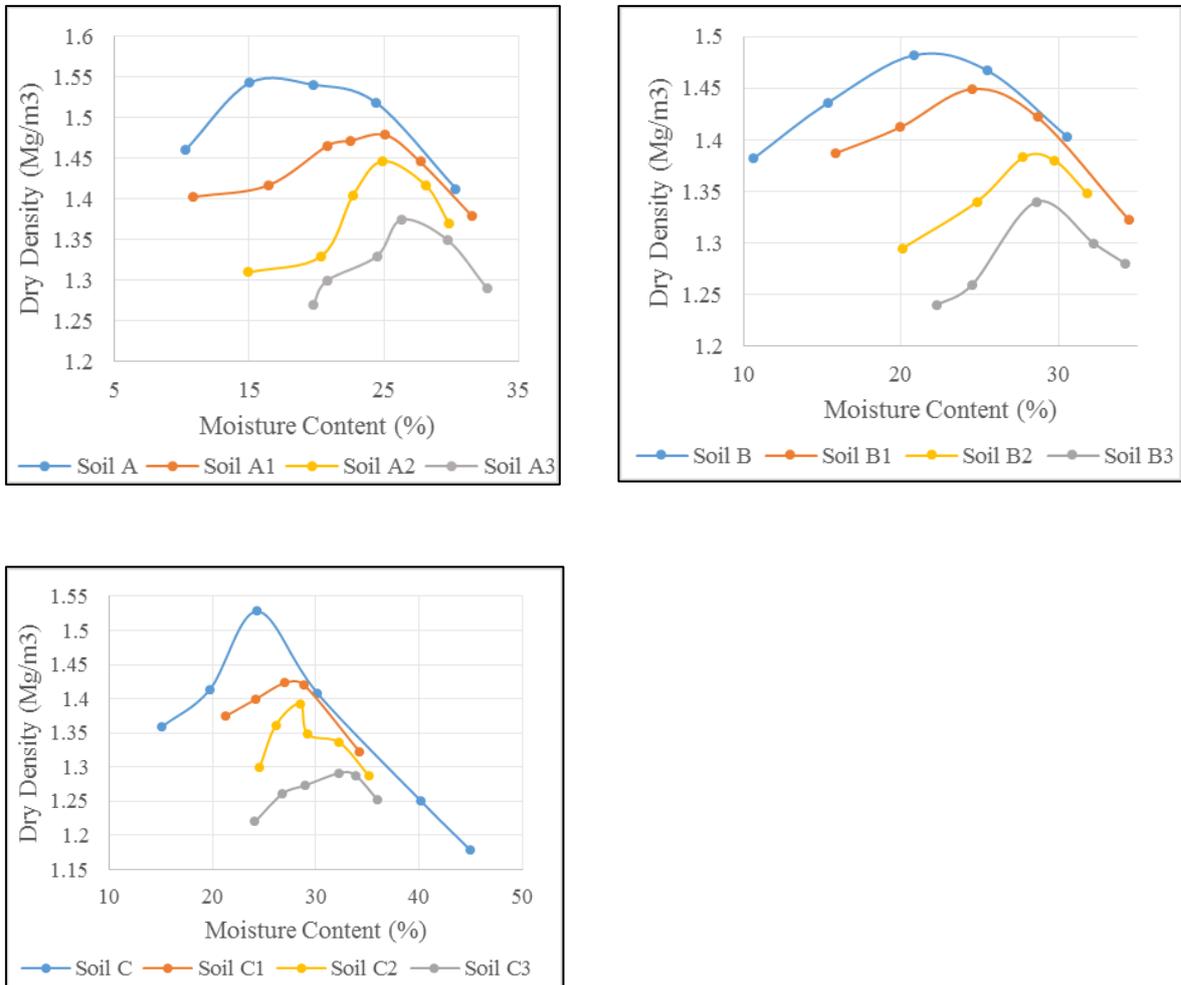


Figure 3.10 Moisture content – Dry density relationship of primary soils with varying lime contents

**Determination of amount of CaCO<sub>3</sub> precipitate post-carbonation and evaluation of degree of success - the CMG, LEY and TGA EY**

The procedures for the calculated mass gain (CMG) and Leco experimental yield (LEY) methods have been outlined in section 3.3.3. Another experimental technique used to quantify CaCO<sub>3</sub> precipitate was the thermogravimetric analysis (TGA) whose results are herein referred thermogravimetric experimental yield (TGA EY). Summary of signatures of the TGA are presented as Figure 3.11 and Figure 3.12 for *sample series A* and *C*, respectively. The experiment was a simultaneous thermal analysis (STA) of thermogravimetry and diffraction scanning calorimetry (TG/DSC). Further details of the signatures and experimental principles and procedures are presented in Appendix A6.

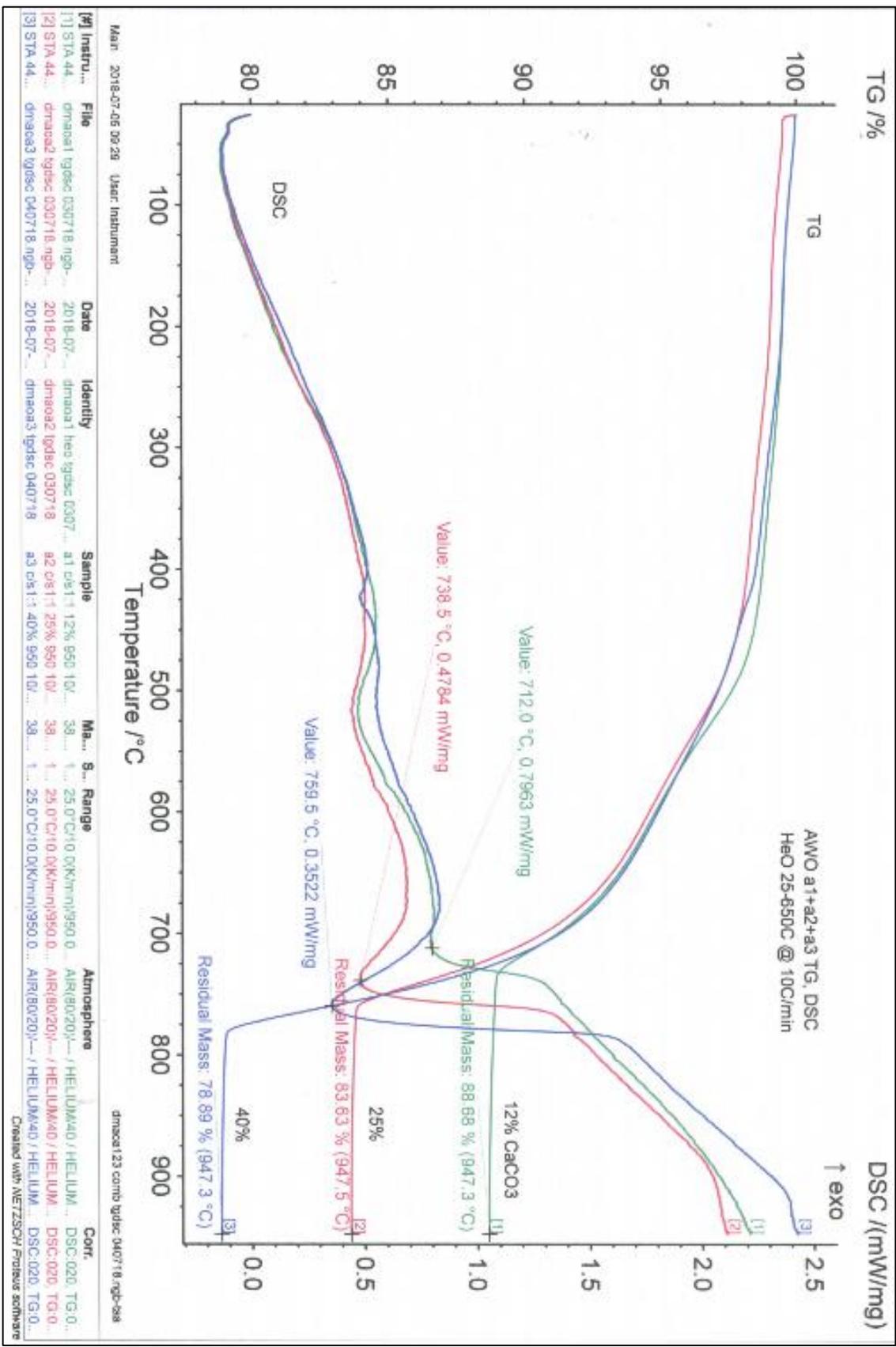


Figure 3.11 Thermogravimetric and differential scanning calorimetric analysis of samples A1, A2, and A3



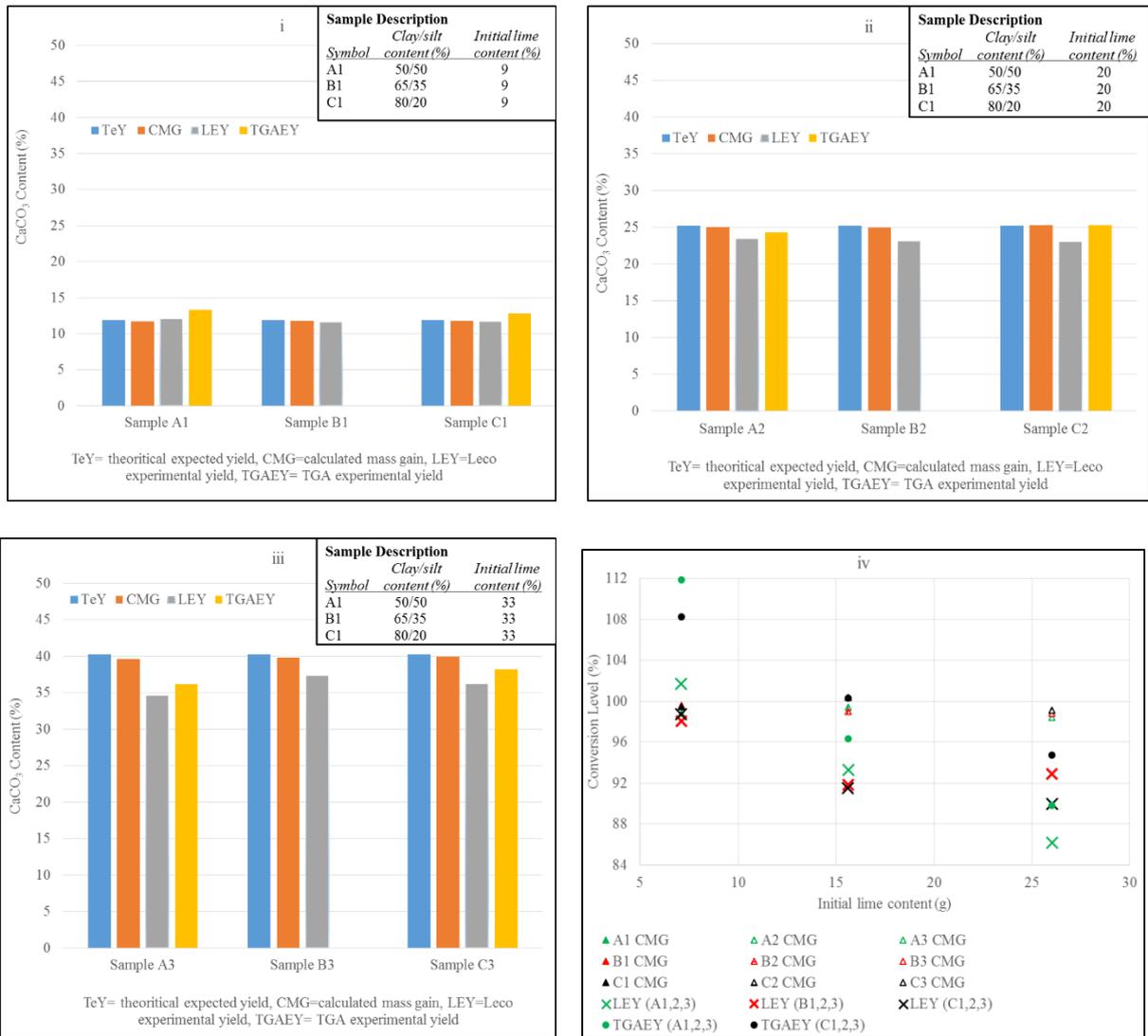


Figure 3.13 Carbonation success level as related to the theoretical expected  $\text{CaCO}_3$  yield (i) moderately calcareous (ii) strongly calcareous (iii) very strongly calcareous samples (iv) percent lime-to-calcium carbonate conversion levels

The CMG results plotted in the Figure 3.13 represent average of 8 specimens for each sample and revealed carbonation successes in the ranges shown in Table 3.6. The experimental yields (EYs) on the other hand, were determined in each case with a representative specimen chosen at random. A summary of approximate degrees of carbonation from both experimental methods are also shown in the Table 3.6.

Table 3.6 Summary of the degree of lime-to-carbonation conversion as evaluated by the different methods

Degree of Lime-to-Calcium Carbonate Conversion (%)			
Sample	Calculated mass gain (CMG)	Experimental yield (EY)	
		Leco yield (LEY)	TGA yield (TGA EY)
A1	99±1.5	101.7	111.9
A2	99±0.3	93.3	96.4
A3	98±0.3	86.2	89.8
B1	100±0.7	98.1	-
B2	99±0.7	91.8	-
B3	99±0.3	92.9	-
C1	99±0.9	98.7	108.2
C2	100±0.5	91.5	100.3
C3	99±0.2	89.9	94.7

Results of the lime-to-carbonate conversion levels evaluated by the three different methods show close comparisons between methods in many cases but differ quite significantly in a few others possibly due to systematic error. However, on a general note, adequate carbonation of approximately 90% were achieved. As can be seen in all plots in Figure 3.13, CMG appear to match closer with the TeY than the experimental methods. Nevertheless, in the samples with low initial lime contents (9 and 20 %), CMGs are comparable to the experimental yields (EYs) with variations not more than 2% CaCO<sub>3</sub> content. However, in samples with high initial lime contents (33%), variations reaching about 5% are observed.

Apart from experimental errors, these variations between methods could happen due to several factors. For instance, loss of particles during handling and incomplete drying would, to some extent invalidate results determined by CMG. On the other hand, metal oxides other than calcium oxides inherent in samples constituent materials could react with CO<sub>2</sub> to form carbonates heavier than CaCO<sub>3</sub>, presenting mass gains different from that resulting from lime

conversion. Consequently, more credence has been accorded the EY data (experimentally obtained) in the following discussion.

As would be expected, more  $\text{Ca(OH)}_2$  reacted produced more  $\text{CaCO}_3$ . This is not surprising since in all reaction cases sufficient pure carbon dioxide was available to be absorbed by all available lime particles. However, carbonation (or conversion) levels achieved were rather of the contrary. Lower degrees of conversion with increasing initial lime contents was almost a general trend but more evident in the EY results.  $\text{Ca(OH)}_2$ -to- $\text{CaCO}_3$  conversion levels recorded for the nine samples range between 86 and 112 % as indicated in Figure 3.13iv. While it remains unclear why conversion levels may exceed 100% (possibly, due to systematic error), there are possible explanations as to why lesser conversion levels could have been achieved, especially those with higher initial lime contents. Perhaps, yields in excess of 100% of expected are the result of carbonate precipitates other than  $\text{CaCO}_3$ , which the experimental techniques could not distinguish.

Early carbonation can result into  $\text{CO}_2$  flow restrictions. Since hydrated lime (particle density 2.24g/m, molar volume 33.01) increases in volume by ~ 11.8% when carbonated (Moorehead, 1986), it is understandable that higher lime content in a compacted sample will bring greater volume of expansions filling the internal pore spaces. Thus,  $\text{CO}_2$  accessibility to other potential reaction fronts could be limited bringing about slower carbonation rates at later stages of the reaction process. Despite that this trend was noticed only with the EY data, another reason for this occurrence can be explained from the perspective of energy yield. Because a mole of  $\text{Ca(OH)}_2$  reacted with  $\text{CO}_2$  yields about 74 KJ heat energy (Moorehead, 1986), the carbonation reaction can be impacted by heat energy in the following ways: (1) increased temperature reduces  $\text{CO}_2$  density, potentially reducing its diffusion in water; (2) high temperatures accelerate moisture loss and can cause moisture starvation needed to proceed the carbonation reaction. It has been observed in the present study, that heat generated during the carbonation process increased with increasing amount of lime reacted. From this understanding, it is possible that higher heat energies generated in higher lime compacts would have caused evaporation of both the catalysing moisture as well as those generated during the process, leaving some lime particles in the compact devoid of moisture needed to progress the reaction.

An interesting situation regarding moisture was observed during the carbonation period. Surprisingly, samples with higher lime contents having higher initial moisture contents before carbonation, retained lower moistures after carbonation as shown in Figure 3.14. Since water is one of the yields from this reaction, it was expected that moisture generation would increase with increasing lime content. This appeared so but only for the water that condensed on the

reaction cell. It may imply, therefore that the excess heat generated resulted in higher evaporations leaving samples dryer.

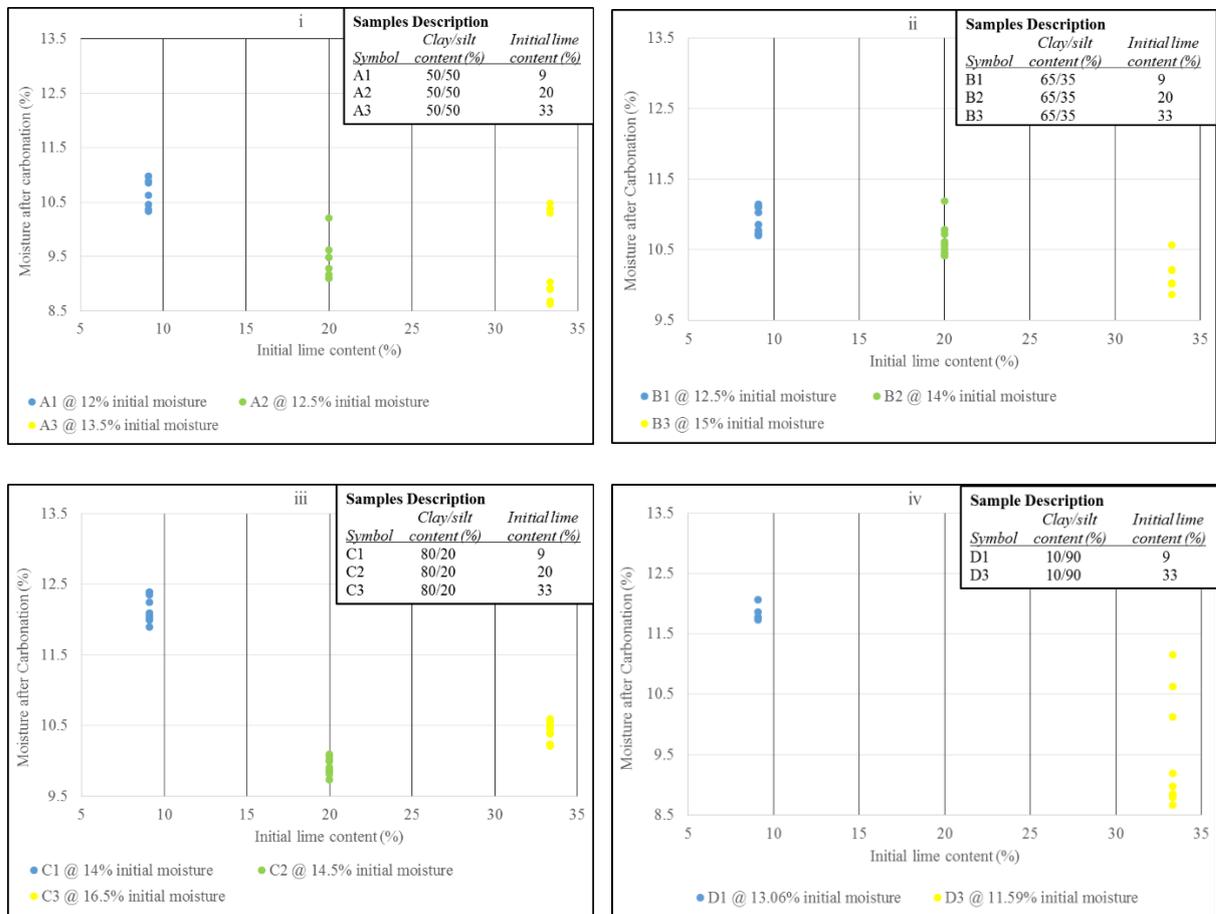


Figure 3.14 Relationship between amount of lime carbonated and moisture retained

Again, the higher heat energies, according to Moorehead (1986) could have reduced the CO<sub>2</sub> solubility in water, delaying the reaction. However, this will be insignificant for temperatures up to 90<sup>0</sup>C (Shih *et al.* (1999); Beruto and Botter (2000); Van Balen (2005); Vance *et al.* (2015)). As outlined in section 2.8, CO<sub>2</sub> solubility is critical to produce CO<sub>3</sub><sup>-</sup> ions, which will react with dissolved Ca<sup>++</sup> to form CaCO<sub>3</sub>.

Van Balen (2005) also considered surface passivation of lime particles at early carbonation as a possible condition that could prevent CO<sub>2</sub> from reaching particles cores, resulting in reduced conversion levels. Conversely, Vance *et al.* (2015) are of the opinion that early carbonation and surface volume expansion should result in surface exfoliation of the early formed CaCO<sub>3</sub>, and coupled with the nonuniform morphological structure and irregular growth of calcite, more vents should develop allowing for greater CO<sub>2</sub> penetration.

The research carried out by Moorehead (1986) also highlighted more factors which may be responsible for low carbonation rates with increasing lime content in a compacted soil. These present results agree with most of Moorehead's arguments except that he stated that carbonation levels could only reach a maximum of 90%. However, he used quite a simple compacted material; that is, pure glass particles and lime. Again, Moorehead's samples carbonated for a maximum of 60 minutes while 24 hours was adopted here. While lime carbonation reaction may be considered instantaneous, 60 minutes may be insufficient time because the carbonation process of a lime sample progresses from surface to core (see Figure 3.15) and therefore, the rate and duration to achieve complete carbonation will depend on the physical conditions of the sample.

It is hoped that in future, advanced chemical analysis of carbonated samples could identify reasons for excessive mass gain as found in this study. Nonetheless, it is worth noting (from the material data sheet) that the hydrated lime carbonated herein consisted of about 3 – 5 % other metal compounds and oxides, such as magnesium hydroxide ( $Mg(OH)_2$ ), iron oxide ( $Fe_2O_3$ ), alumina ( $Al_2O_3$ ), Lead (Pb), and Manganese (Mn), which can react with  $CO_2$  to possibly form other minor carbonates heavier than  $CaCO_3$ .

As evident in Figure 3.13, in general, results of both assessment methods do not differ considerably with the theoretical one, which is an indication of nearly pure carbonate samples.

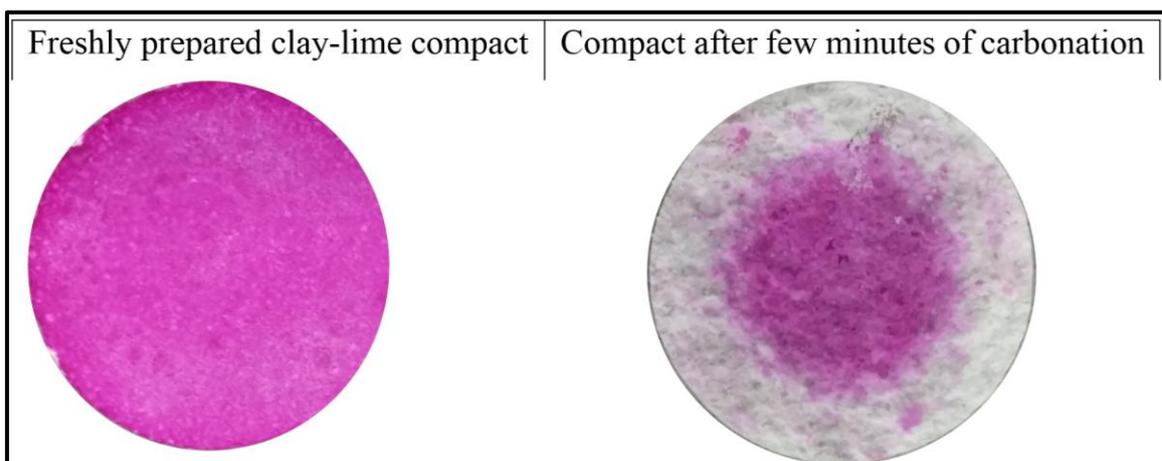


Figure 3.15 carbonation progressing from compact surface towards core

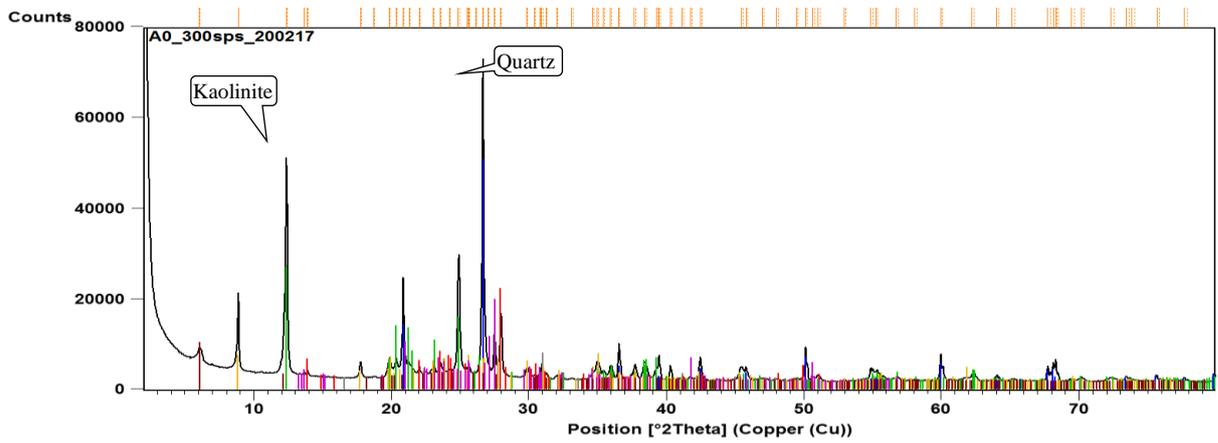
#### 3.4.6 X-Ray diffraction (XRD) analysis

XRD analyses were performed on both non-carbonated and carbonated samples to identify the crystalline phases and physical characteristics of their components. Identifying the main phase

of the CaCO<sub>3</sub> precipitated from the carbonation process was crucial. Crystalline CaCO<sub>3</sub> is known to exist in three polymorphs (calcite, aragonite or vaterite) with varying chemical stabilities. Calcite being the most stable and vaterite the least stable. Mineral phase or crystal structure controls the pattern of X-ray diffraction obtainable, and almost all crystalline solid materials possess a unique powder X-ray diffraction form in terms of the peak intensities and the positions of the observed reflections (Weller, 1994), which aid their identification when compared to X-ray databases. Usually, their X-ray spectra show strongest diffraction peaks at their unique angles (degrees, 2 theta). Stronger diffraction peaks also indicate dominance of a mineral in a sample. Some diffraction peaks defined in literature (Table 3.7) are adapted to evaluate those obtained in this present study (Figure 3.16 and Figure 3.17).

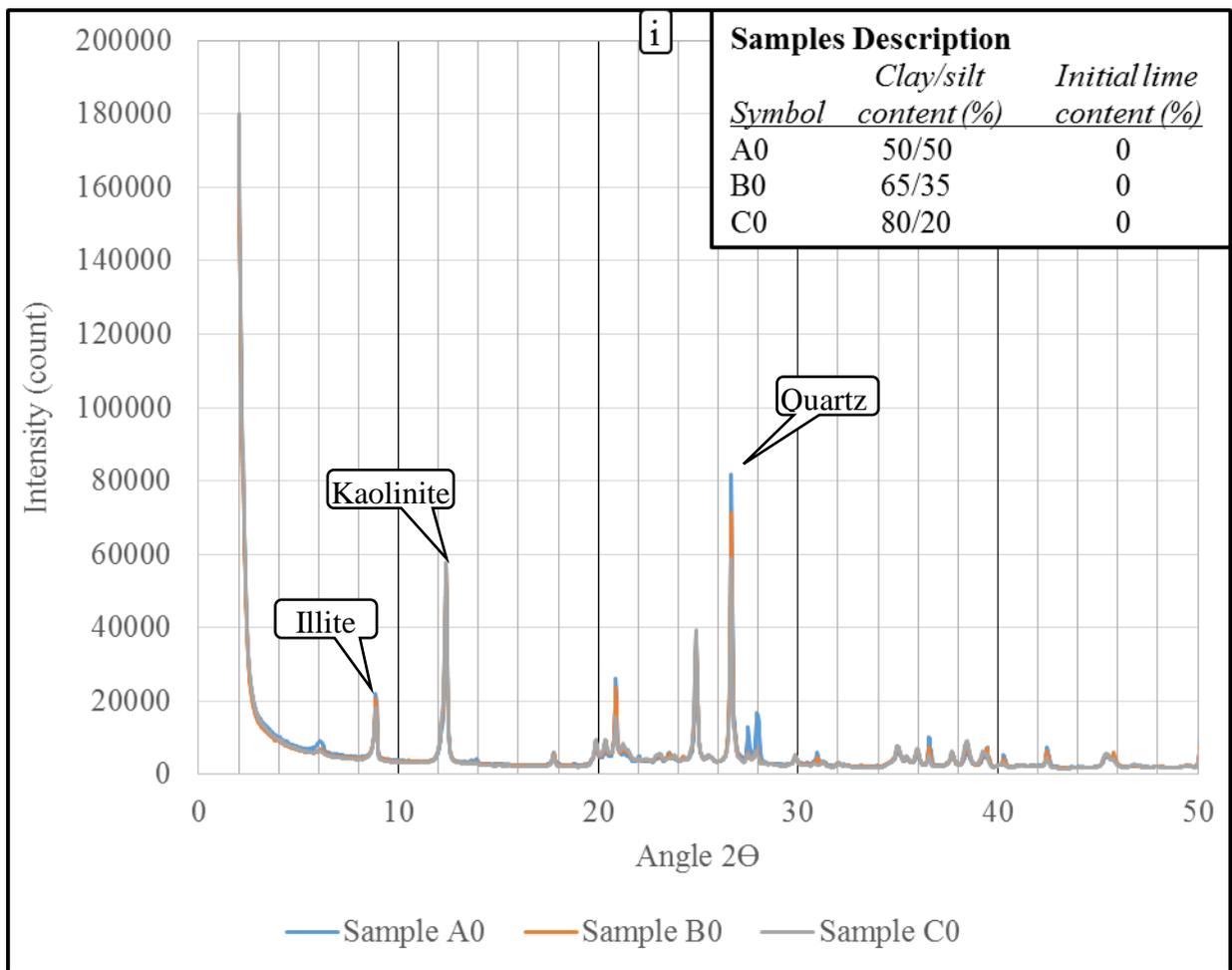
Table 3.7 X-Ray diffraction angles of minerals as adapted from the literature

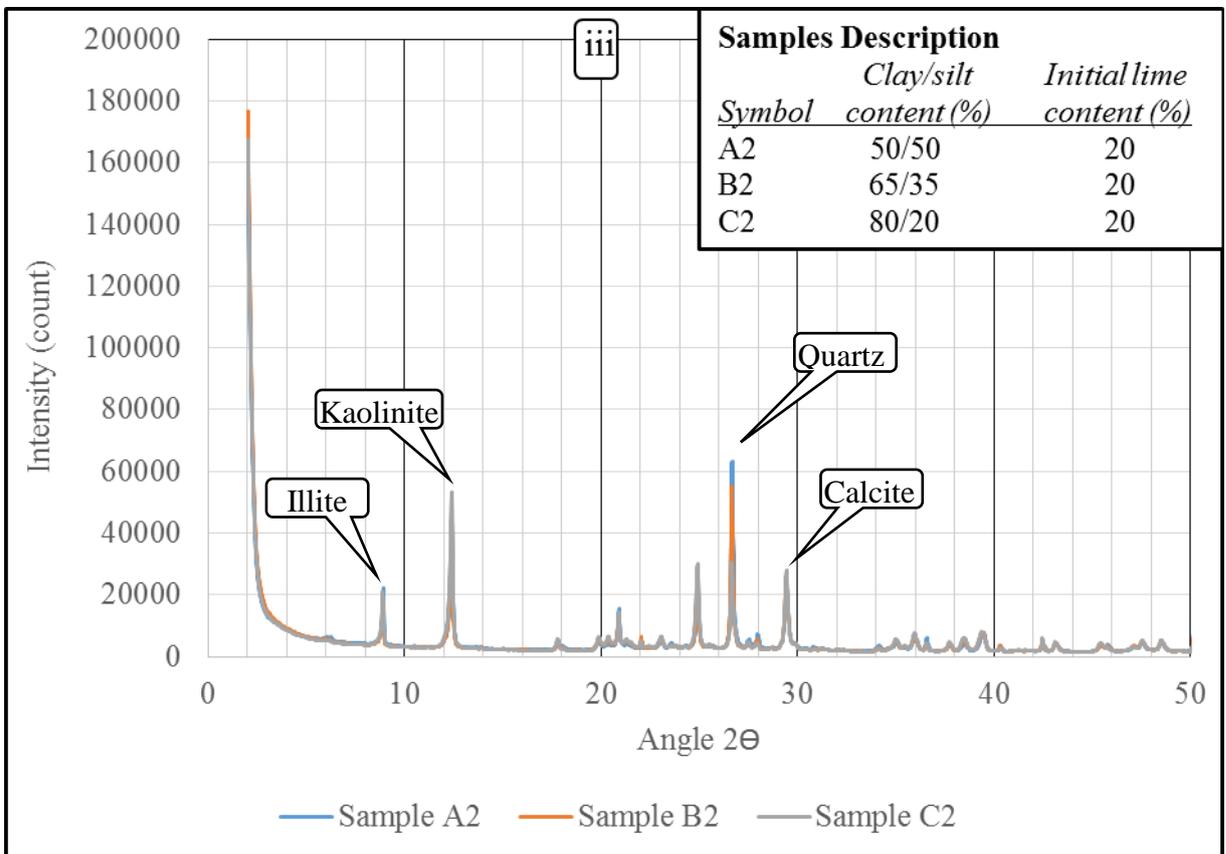
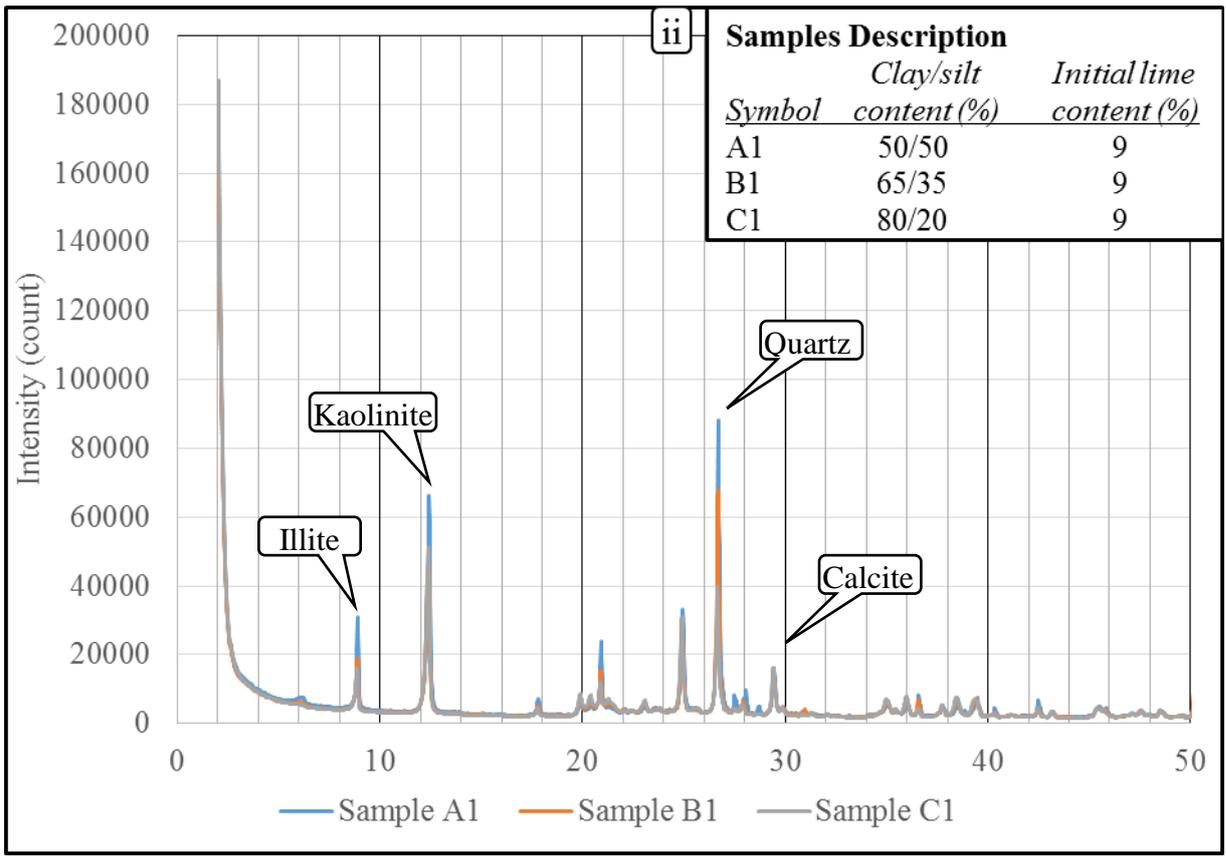
<i>Mineral</i>	<i>Angle at strongest diffraction peak (degree 2 theta)</i>	<i>Reference</i>
Lime (Ca(OH) <sub>2</sub> )	17.5 <sup>0</sup>	James and Subba Rao (1986); Rodriguez - Navarro <i>et al.</i> (1998).
Calcite	29.5 <sup>0</sup>	Derbyshire and Mellors (1988); Al-Mukhtar <i>et al.</i> (2010); (2012); Bontognali <i>et al.</i> (2010); (2012); Wang <i>et al.</i> (2013).
Aragonite	26.25 <sup>0</sup>	Wang <i>et al.</i> (2013); Wang <i>et al.</i> (2017).
Vaterite	32.73 <sup>0</sup>	Kjellin (2003).
Dolomite	31 <sup>0</sup>	Bontognali <i>et al.</i> (2010); (2012).
Quartz	26.5 <sup>0</sup>	Derbyshire and Mellors (1988); Al-Mukhtar <i>et al.</i> (2010); (2012); Bontognali <i>et al.</i> (2010); (2012).
Kaolinite	12.38 <sup>0</sup>	Al-Mukhtar <i>et al.</i> (2010); (2012); Mutaz and Dafalla (2014).
Illite	8.75-8.93 <sup>0</sup>	Al-Mukhtar <i>et al.</i> (2010); (2012); Mutaz and Dafalla (2014).
Smectite	5.91-7.37 <sup>0</sup>	Al-Mukhtar <i>et al.</i> (2010); (2012); Mutaz and Dafalla (2014).
Halite	27.5 <sup>0</sup>	Bontognali <i>et al.</i> (2010); (2012).
Anorthite (feldspar)	28 <sup>0</sup>	Derbyshire and Mellors (1988); Bontognali <i>et al.</i> (2010); (2012).



Peak List
Kaolinite 11TA1RG; Aluminum Hydroxide Silicate; Al <sub>2</sub> ( Si <sub>2</sub> O <sub>5</sub> ) ( O H ) <sub>4</sub> ; 01-078-1996
Dolomite; Calcium Magnesium Carbonate; Ca Mg ( C O <sub>3</sub> ) <sub>2</sub> ; 01-075-1655
Albite, ordered; Sodium Aluminum Silicate; Na Al Si <sub>3</sub> O <sub>8</sub> ; 00-009-0466
Quartz low, syn; Silicon Oxide; Si O <sub>2</sub> ; 01-089-1961
Muscovite-2M1; Potassium Aluminum Silicate Hydroxide; K Al <sub>2</sub> ( Si <sub>3</sub> Al ) O <sub>10</sub> ( O H ) <sub>2</sub> ; 01-076-0637
Microcline, intermediate; Potassium Aluminum Silicate; K Al Si <sub>3</sub> O <sub>8</sub> ; 00-019-0932
Clinchrysothile-2Mc1; Magnesium Silicate Hydroxide; Mg <sub>3</sub> ( Si <sub>2</sub> O <sub>5</sub> ) ( O H ) <sub>4</sub> ; 01-073-9904

Figure 3.16 Typical mineral composition of initial samples without lime/carbonate treatment, identified by X-ray diffraction analysis





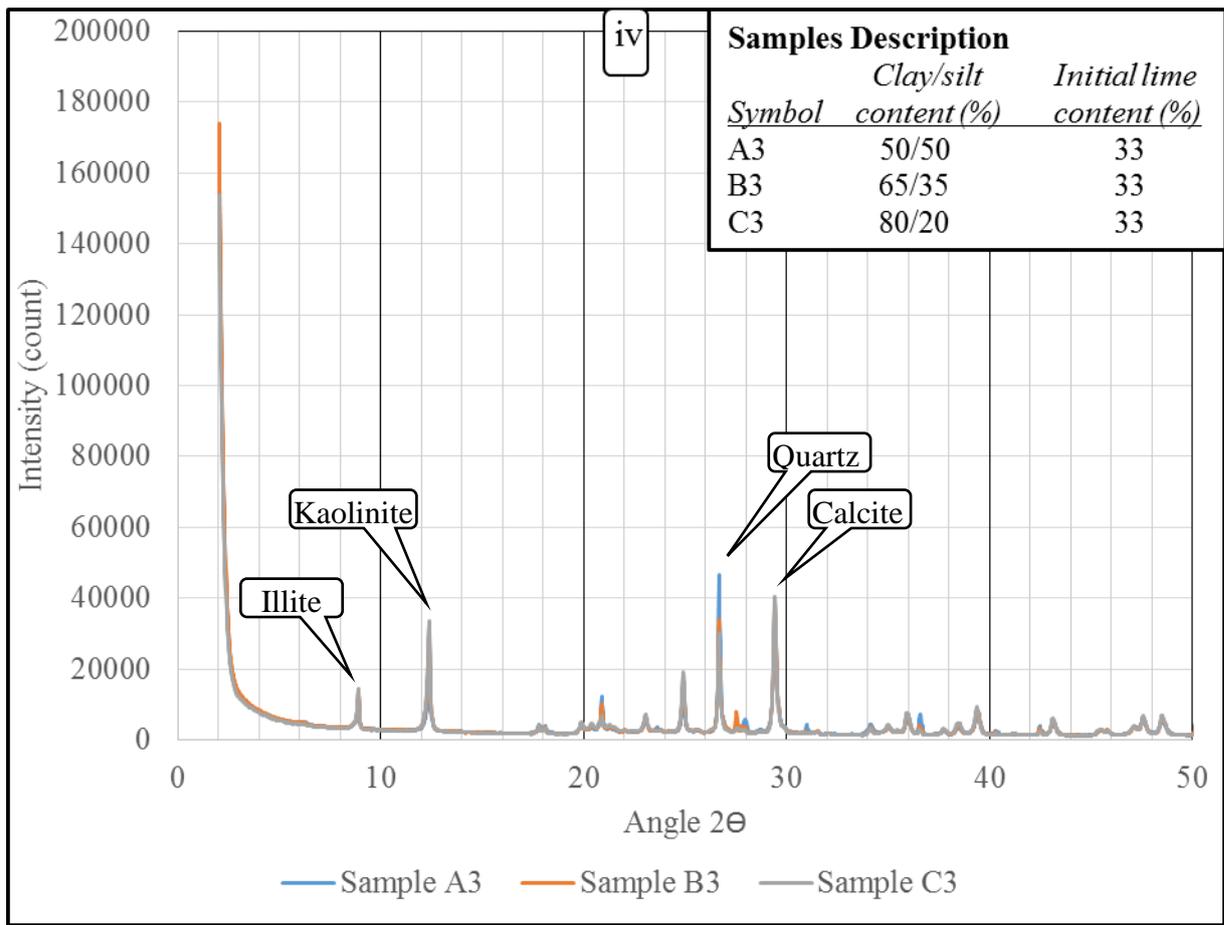


Figure 3.17 X-Ray diffraction analysis of manufactured samples (i) initially constituted primary soils (ii) moderately calcareous (iii) strongly calcareous and (iv) very strongly calcareous samples

### ***XRD spectra obtained from this present study***

A powder X-Ray diffractometer, PANalytical X'pert Pro Multipurpose Diffractometer (MPD), powered by a Philips PW3040/60 X-ray generator fitted with an X'Celerator detector was used to examine samples. The X'Celerator is an ultra-fast X-ray detector, which uses RTMS (Real Time Multiple Strip) technology. It operates as an array of a hundred channels which can simultaneously count X-rays diffracted from a specimen over the range of 2 theta angles specified during a scan. The detector is thus, able to produce high quality diffraction data in a significantly shorter period compared to older style diffractometers.

Diffraction data were acquired by exposing powder specimens to Cu-K $\alpha$  X-ray radiation, having a typical wavelength ( $\lambda$ ) of 1.5418 Å. X-rays were generated from a Cu anode supplied with 40 kV and a current of 40 mA. The data were acquired over a range of 2-80 $^{\circ}$  (2 theta) with a step size of 0.0334 $^{\circ}$  (2 theta) and nominal time per step of 300 seconds using the scanning X'Celerator detector. Fixed divergence and anti-scatter slits of  $\frac{1}{4}^{\circ} + \frac{1}{4}^{\circ}$  were employed together with a beam mask of 10mm and all scans were performed in 'continuous' mode. Phase

identification was performed by means of the X'Pert accompanying software, PANalytical High Score Plus in conjunction with the ICDD Powder Diffraction File 2 Database (2004), ICDD Powder Diffraction File 4 - Minerals (2018), the American Mineralogist Crystal Structure Database (2010) and the Crystallography Open Database (2016. [www.crystallography.net](http://www.crystallography.net)).

Figure 3.16 and Figure 3.17 present spectra of the powder X-Ray diffraction acquired for samples studied herein. All samples were constituted with the same mineral materials. Figure 3.16 shows the typical initial mineral constituents of samples before lime or calcium carbonate treatment. Samples are only different from one another in proportions of these minerals. Predominantly, the exercise was to identify and quantify the calcium carbonate phase precipitated from the carbonation process, since other key components of the samples were fairly well known from initial constituent proportions.

All the X-ray diffraction spectra of both initially constituted soils and manufactured samples show good consistency; an indication of the repeatability of the production method. By comparing diffraction angles identified in Table 3.7, with Figure 3.16 and Figure 3.17, it becomes clear that primary soils, i.e. samples before addition of lime, were composed mainly of quartz ( $26.5^{\circ} 2\theta$ ) and kaolinite ( $12.38^{\circ} 2\theta$ ) minerals with minor others. Interestingly, all the different manufactured samples revealed that, after lime addition and subsequent carbonation, only calcite ( $29.5^{\circ} 2\theta$ ) was the new mineral introduced. Calcite is the most stable form of  $\text{CaCO}_3$  (Mitchell and Soga, 2005). Its solubility in ordinary water is extremely low, and therefore, in such conditions calcite cementation effect on soil collapsibility may just be mechanically controlled rather than a bond cement dissolution.

#### 3.4.7 Micro-fabric analysis

Scanning electron microscope (SEM), X-ray Computed Tomography (XRCT) and other advanced image processing technologies provide new opportunities for examining the internal structure of soils and rocks. Previous studies have related microstructural properties of materials with their macroscopic behaviour (references in Liu *et al.* (2011)). Therefore, an attempt has been made here to relate the microstructure of samples (obtained by SEM) and their mechanical behaviour. However, due to poor images much of what could be studied was more of a micro-fabric.

SEM images were obtained using a TESCAN VEGA LMU scanning electron microscope. Prior to scanning, specimens were oven dried and mounted on an aluminium stub with Achesons Silver Dag and then dried overnight, followed by gold coating (standard, 15nm),

using a Polaron SEM Coating Unit. SEM images obtained and their microporosity analyses are shown in Figure 3.19.

Microporosity identification and quantification was carried out by digital image processing using PCAS (Particles (Pores) and Cracks Analysis System). PCAS is a professional software developed according to image processing technologies by Dr Chu Liu of the School of Earth Sciences and Engineering, Nanjing University, to automatically identify multiscale pores and cracks. It has been employed by several researchers (Liu *et al.* (2011); Liu *et al.* (2013) and references therein) in the quantitative analysis of SEM images and photographs of rock and soil cracks, micropores and mineral particles. The software is almost automatic, requiring just two thresholds to be (1) gray-level threshold (80 chosen here) and (2) minimum pore area (50 pixel chosen here). The image is then, automatically processed in the software and returns all geometric parameters of the pores and the statistical parameters of the pore system.

After scanning, further chemical examination was performed on specimens, using an Energy Dispersive X-ray analyser (EDX) attached to the electron microscope. An area on the specimen, as indicated by the squares on images in Figure 3.20 was mapped and Table 3.8 presents the elemental composition and quantification of the various manufactured samples.

Samples constituent materials (kaolinite, quartz-silt and carbonated lime) were initially scanned individually (Figure 3.18) to form a guide for the interpretation of micrographs of the main samples shown in Figure 3.19 and Figure 3.20.

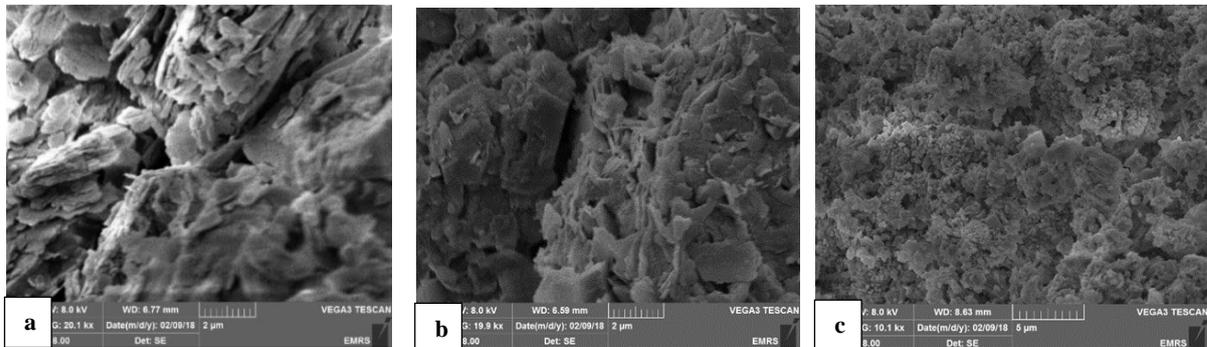
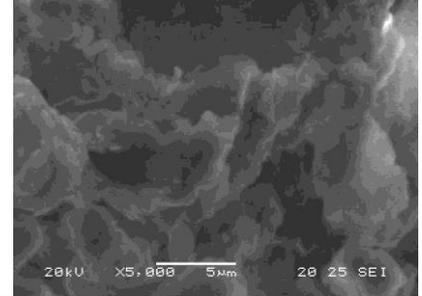
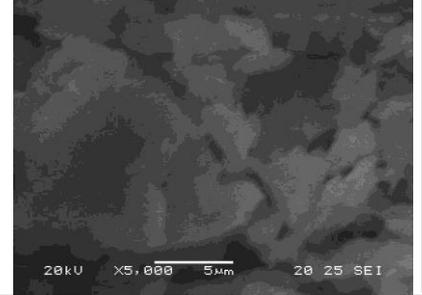
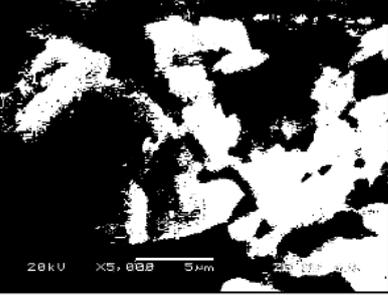
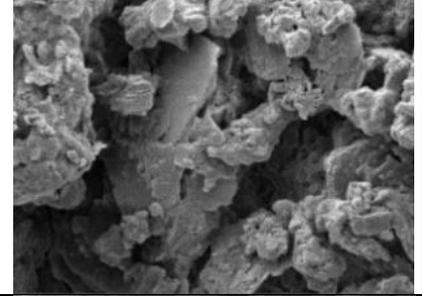
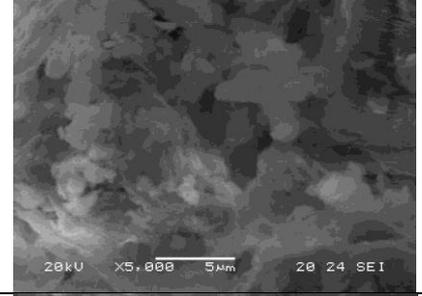
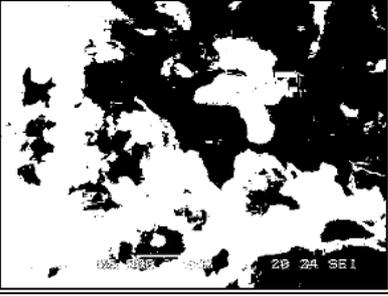
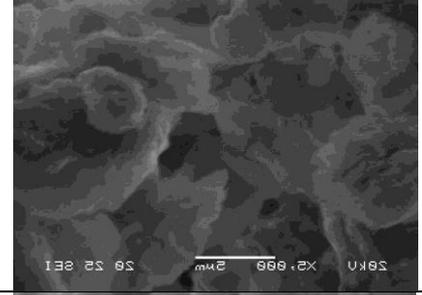
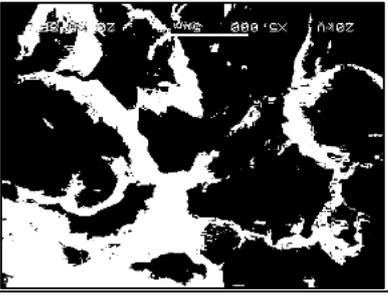
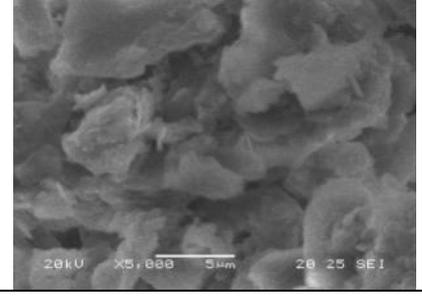
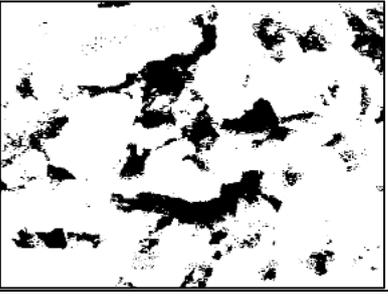
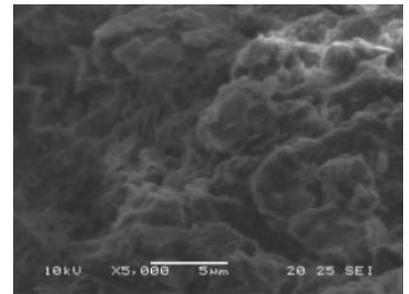
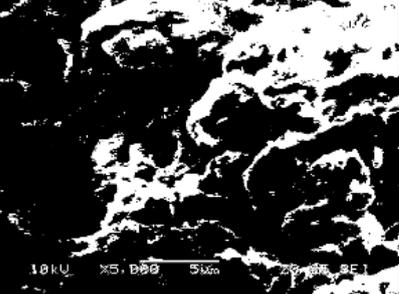
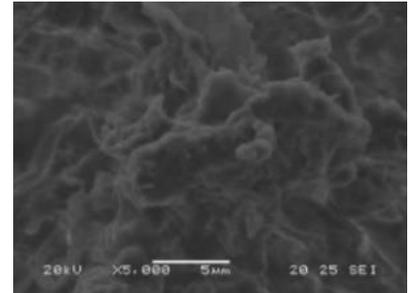
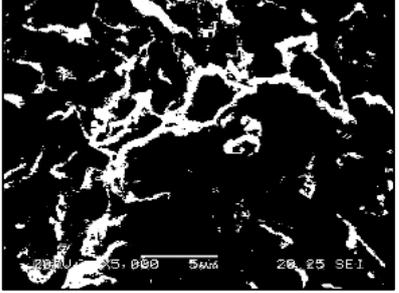
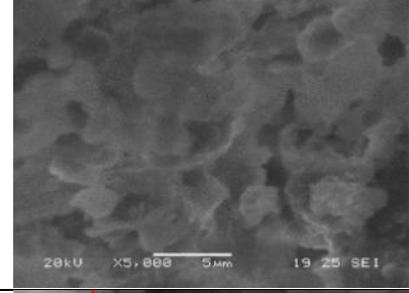
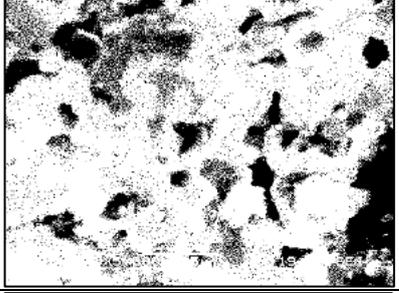
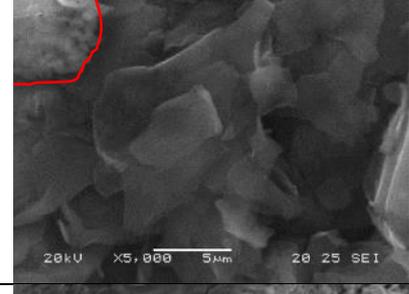
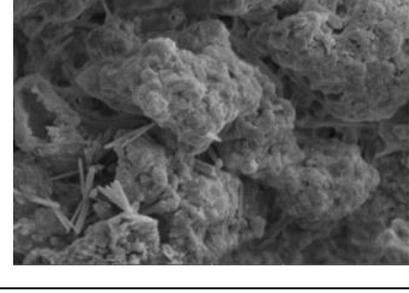


Figure 3.18 SEM images of the individual components of manufactured samples (a) Kaolinite (Polwhite E) – shows clay flakes in a parallel arrangement (b) Lantan alluvium (sub 63  $\mu\text{m}$ ) – image show a few clay flakes surrounding angular silt-size quartz (c) carbonated hydrated lime – calcium carbonate globules are formed covering entire surface

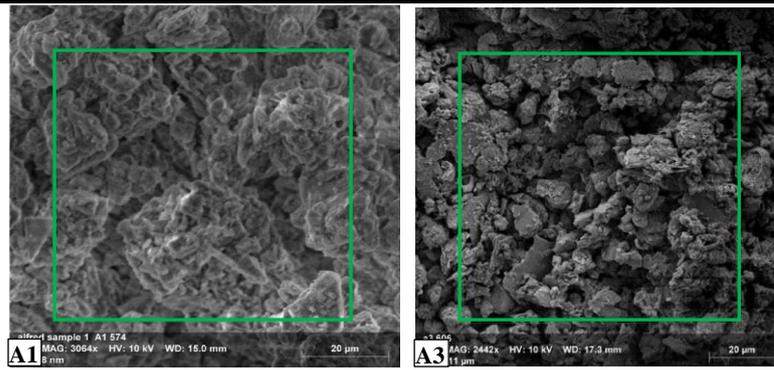
Original SEM Image	PCAS Analysis	Remarks
		<p><b>A1 (50/50 clay/silt, ~12% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 41.62% An open structure of silt grains and clay clods supported by CaCO<sub>3</sub></p>
		<p><b>A2 (50/50 clay/silt, ~25% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 65.88% An open structure of silt grains and clay clods supported by CaCO<sub>3</sub></p>
		<p><b>A3 (50/50 clay/silt, ~40% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 43.07% The structure is comparable to FIGURE 2.12a&amp;b. A loosely packed silt fabric interconnected by abundant CaCO<sub>3</sub>-clay matrix</p>
		<p><b>B1 (65/35 clay/silt, ~12% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 45.5% An open structure of silt grains and clay clods supported by CaCO<sub>3</sub></p>
		<p><b>B2 (65/35 clay/silt, ~25% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 68.45% An open structure of silt grains and clay clods supported by CaCO<sub>3</sub></p>
		<p><b>B3 (65/35 clay/silt, ~40% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 16.25% CaCO<sub>3</sub> and clay mix connecting few silt grains as well as clay clods.</p>

*a*

Original SEM Image	PCAS Analysis	Remarks
		<p><b>C1 (80/20 clay/silt, ~12% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 74.09% Clay and CaCO<sub>3</sub> overwhelmingly surrounded the minor silt grains. Structure comparable to Figure 2.18a</p>
		<p><b>C2 (80/20 clay/silt, ~25% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 86.16% CaCO<sub>3</sub> bonding with the clay platelet overwhelmingly surrounded minor silt grains. Structure comparable to Figure 2.18a.</p>
		<p><b>C3 (80/20 clay/silt, ~40% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 28.8% Clay and CaCO<sub>3</sub> overwhelmingly surrounded the few silt grains. An open clay structure supported by CaCO<sub>3</sub> bonding.</p>
		<p><b>D1 (10/90 clay/silt, ~12% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 83.89% CaCO<sub>3</sub> globules visible at top left corner but more calcite could be embedded within silt grains. The structure can be compared to the Malan loess shown in Figure 2.11</p>
		<p><b>D3 (10/90 clay/silt, ~40% CaCO<sub>3</sub>) –</b></p> <p>PCAS porosity = 52.67% D3-Needle shaped CaCO<sub>3</sub> outgrowth from the CaCO<sub>3</sub> globules, coating and bridging silt particles. Structure comparable to Figure 2.16c, Calcareous brickearth, 2.39 m, Ospringe Pit 1.</p>

*b*

Figure 3.19 *a* and *b* - SEM micrographs of manufactured samples and their microporosity analysis using PCAS. Black areas represent pores in the PCAS images

*SEM image with EDX mapping**Remarks*

**A1 & A3 (50/50 clay/silt, with ~12% & ~40% CaCO<sub>3</sub>, respectively). Green square showing EDX mapped area -** Silt grains coated with clay-carbonate system. Carbonate globules are also visible, especially in A3. The A1 structure is comparable to Figure 2.16c, calcareous brickearth, 2.39 m, Ospringe Pit 1 from Milodowski et al. (2015) while A3 structure is comparable to Figure 2.19a, Melan loess with a  $9.55 \pm 1.97$  % collapsibility

Figure 3.20 SEM micrographs showing EDX mapping

Table 3.8 Chemical composition of manufacture samples obtained by EDX mapping

Sample	% Weight of Elements											
	C	O	Na	Mg	Al	Si	K	Ca	Ti	Fe	Sn	Cl
A1	14.00	47.58	-	0.21	11.35	16.66	1.34	7.11	-	1.53	-	-
A2	8.90	46.67	-	0.15	11.29	12.73	1.54	17.15	-	0.63	0.63	-
A3	41.25	46.39	0.11	1.01	2.85	4.1	0.24	3.96	-	-	-	0.10
B1	8.96	50.47	-	-	13.68	19.29	2.43	4.2	-	0.97	-	-
B2	9.4	50.17	-	0.18	12.60	14.97	1.35	10.38	-	0.53	0.43	-
B3	94.57	2.51	-	-	0.29	0.43	-	2.21	-	-	-	-
C1	4.95	50.68	0.11	0.23	13.91	19.52	2.13	7.51	0.16	0.8	-	-
C2	15.67	48.52	-	-	12.69	14.94	0.65	7.26	-	0.26	-	-
C3	20.51	43.58	-	-	5.78	6.53	0.45	23.14	-	-	-	-

### **Concluding remark**

As noted earlier, samples manufactured in this study did not mimic a particular soil but rather merely simulate in general terms, an open metastable structure. Despite that, observations of the SEM micrographs reveal similarities in microfabric appearance with some SEM images of natural collapsible soils reported in the literature. This proves the validity of the procedures adopted here.

Regardless of the identical mineralogy of samples, the SEM micrographs show different microfabric from one sample to another. This may be because of differences in proportions of constituent materials, suggesting that the microfabric was basically controlled by the mix design.

However, nearly all samples exhibited an open structure (i.e. large porosity), with intergranular pore sizes reducing with increasing clay and carbonate content. Conversely, the PCAS image processing revealed an increase in pore number (porosity) with increasing clay content.

The image processing results depended mainly on discrimination of pores and particles as black and white spots, respectively. This was enhanced by threshold adjustments. To be able to compare processed image results logically, general threshold values were chosen. However, a major drawback was that of the quality of the original SEM images. For instance, in some cases, soil particles appeared dark on the original SEM image and those were also identified as pores in the PCAS analysis. Notwithstanding these issues, observations by eye revealed that intergranular pore sizes in all of the micrographs are visible measuring in the range of macroporosity (i.e. pores wider than  $0.05\mu\text{m}$  (Reedy *et al.*, 2014)).

Whereas other experimental data confirmed the precipitation of calcite in the main samples (A, B, and C series) of this project, it was difficult to physically recognise  $\text{CaCO}_3$  in the SEM images, in terms of identifying  $\text{CaCO}_3$  shapes. However, carbonate globules were suspected in the sample A series. It was concluded that, perhaps because of the high quantity of clay mineral in the samples, it was possible that clay could have overshadowed the systems making carbonate shapes opaque.

Consequently, a silty soil with only about 10% clay was carbonated following identical procedures as with the main samples. In a subsequent SEM analysis, therefore calcium carbonate shapes (globules and needle-like) became visible in samples with high silt content (that is, samples A and D series) being more conspicuous in the high carbonate ones (A3 and D3). The calcium carbonate can be seen to coat silt particles in sample D3, and carbonate forming contacts between coated grains while samples of the A series (especially Figure 3.20) appeared to have both carbonate and clay occurring together as the silt grains coating. Large silt grains appear clean in sample D1. No grain coating was however, observed. Rather, silt particles are seen to contact each other in a random manner with what appears to be menisci of calcite and accumulation of globules at the top left corner.

### **3.5 Stage 3 – Geotechnical Testing Techniques**

Two main geotechnical testing methods have been adopted to study the impact of calcite cementation, clay content, etc. The oedometer testing investigated their collapsibility while unconfined compressive testing was employed to study the strength and stiffness properties of samples. Both methods tested samples before and after silicate treatment.

However, for every laboratory study, there are limitations, and the concern is usually raised about the accuracy and reliability of data obtained and conclusions reached. Some of the limitations identified in this laboratory study are described in section 3.6, whereas, accuracy and reliability of the data obtained were ensured as follows:

1. Relevant sections of Standards, such as the British Standards 1377(1990) and the *Manual of soil laboratory testing* by K. H. Head, were followed in both the test specimen preparation and testing procedures. Care was taken to ensure all specimens were tested under identical conditions, except that room ambience varied each day.
2. Even though calibration of the testing equipment had been carried out in routine maintenance by the lab technicians, calibration confirmation tests were carried out before the main testing programme of this study.
3. For each data point, not less than three identical specimens were tested, and the mean value (of CP or UCS) represented data points. However, as acceptance criteria, it was specified that the individual values of at least three identical specimens should not differ by more than 10% of the mean value.

### 3.5.1 Tests specimen preparation

All compressive test specimens were tested as manufactured whereas, oedometric test specimens were prepared into two different groups. The first group were those tested as manufactured while the second group were those cored/trimmed from larger manufactured samples. These were carbonated samples larger than the oedometer rings. Specimens were prepared from them by gradually and carefully shaving sample to fit into the oedometer ring as can be seen in Figure A4-1 (Appendix A4). All test specimens were cylindrical in shape. Their physical properties such as volume, dry density, void ratio and porosity were determined following Equations 3.5 to 3.12.

$$V = \pi r^2 * h \dots\dots\dots 3.5$$

$$\rho_d = \frac{\text{dry mass}}{V} (Mg/m^3) \dots\dots\dots 3.6$$

$$e = \frac{\rho_s}{\rho_d} \dots\dots\dots 3.7$$

$$n = \frac{e}{1 + e} * 100 (\%) \dots\dots\dots 3.8$$

where  $V$  = volume,  $r$  = radius,  $h$  = height,  $\rho_d$  = dry density,  $e$  = void ratio,  $\rho_s$  = particle density, and  $n$  = porosity.

### 3.5.2 Collapse testing apparatus and procedures

The one-dimensional oedometer method for determining collapse potential (CP) was adopted. The conventional oedometer apparatus used, its components and data acquisition system are shown below as Figure 3.21.

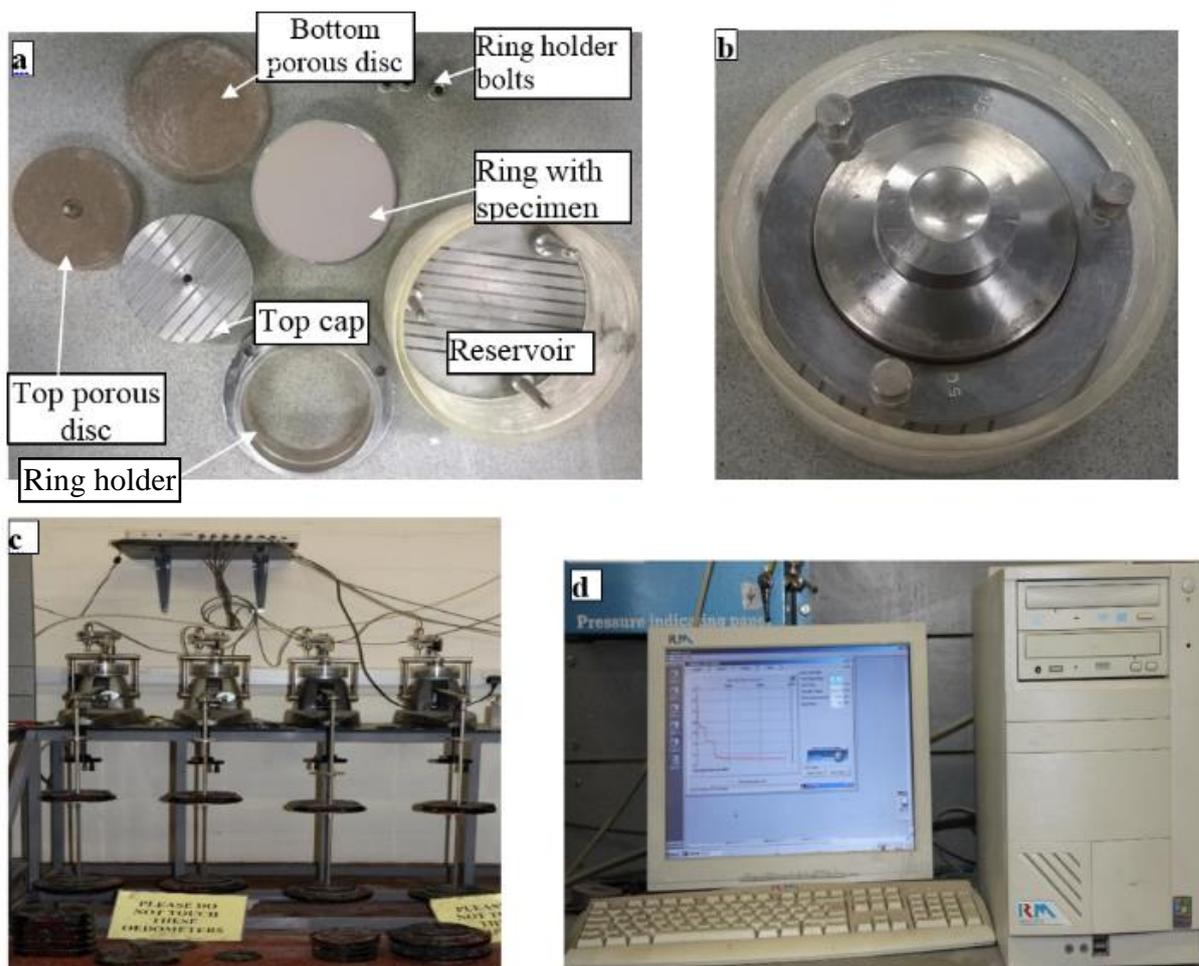


Figure 3.21 conventional oedometer equipment **a**, specimen and pot components **b**, assembled pot with specimen **c**, setup of test running **d**, data acquisition system

### *Calibration of apparatus*

Calibration of both the conventional oedometer apparatus and transducers for measuring the vertical compression was previously carried out during routine laboratory maintenance by the technical staff. The elastic deformation characteristics were determined in accordance with method described in BS1377 (1990).

### *Testing procedures*

Two testing processes referred to as the “single oedometer” and “double oedometer” were adopted. In both, specimens prepared into oedometer rings were assembled into the oedometer cell. Two air-dried porous discs were placed, one at the base and the other on top of specimen. Dried porous discs were preferred to avoid altering moisture contents of specimens, as well as preventing further dilution of wetting fluid (acidic solution in particular). The assembled oedometer cell with specimen was then placed under the oedometer loading frame. A displacement transducer attached to the equipment and connected to a data acquisition system, was lowered onto the top hanger of the loading frame lever arm.

GDS data acquisition software was used to collect displacement data at intervals of 10 seconds. However, only total deformation at the end of each stage was desirable (Alan and Robert, 1988). Data collection by the software was started nearly simultaneously with loading. Because of the low compressibility of the calcareous specimens tested, loading was started at a 100kPa. Incremental loadings of 100kPa up to 500kPa was applied to all testing while unloading in reverse order was applied in a few cases to investigate any possible rebound or swelling of the specimen. A few specimens were also loaded to beyond 500kPa to a maximum of 860 kPa.

Collapse was triggered by wetting specimens with either distilled water or a 5% acetic acid solution at different overburden stresses. Against the traditionally used distilled water, an acidic solution was adopted for two reasons. Firstly, as an anticipated pore fluid in environments where acidic precipitations may be common, and secondly, the effect of dissolution of the particle bonding mineral ( $\text{CaCO}_3$ ) on the collapse phenomenon may not be seen if only distilled water was used.

In order to avoid air entrapment in the centre of specimens during wetting, care was taken to ensure that wetting was done in one-dimension. That is, wetting gradually from bottom to top according to the recommendation of Alan and Robert (1988). CP was defined in terms of

the percentage decrease in height or void ratio of specimens resulting from wetting, as shown in Equation 3.9 (ASTM, D 5333 - 03) and Equation 3.10 (Alan and Robert, 1988).

$$CP = \frac{\Delta H}{H_0} \times 100 \dots\dots\dots 3.9$$

where  $CP$  = collapse potential,  $\Delta H$  = change in specimen height resulting from wetting,  $H_0$  = specimen height before wetting

$$CP = \frac{\Delta e}{1 + e_1} \times 100 \dots\dots\dots 3.10$$

where  $\Delta e$  = change in specimen void ratio resulting from wetting,  $e_1$  = specimen void ratio before wetting

A system traditionally adopted by many authors for classifying the degree or severity of collapse was proposed in 1975 by Jennings and Knight. Table 3.9 was modified by Bell and Culshaw (2001) in line with British perspective of collapse, and was adapted here to classify sample collapsibility. There, soils exhibiting CP less than 1% are not regarded as collapsible or metastable. However, this value may vary from one region to another. For example, in a Chinese context, soils with CP less than 1.5% are considered non-collapsible (Derbyshire *et al.*, 1995) while Lutenegger and Hallberg (1988) said that, in the United States, collapse is only regarded as significant if CP exceeds 2%.

The potential of a silicate solution to improve both collapse and strength properties of samples was investigated (see Chapter 5). Treatment focused on samples exhibiting degrees of severity within “trouble” and “very severe trouble” as described in the Table 3.9.

*Table 3.9 Collapse percentage as an indication of severity (from Jennings and Knight, 1975 modified after Bell and Culshaw (2001))*

Collapse (%)	Severity of Problem	Abbreviation
0–1	No trouble	NT
1–5	Moderate trouble	MT
5–10	Trouble	T
10–20	Severe trouble	ST
> 20	Very severe trouble	VST

### ***Single oedometer test (SOT)***

In the SOT, specimens are loaded incrementally in their 'as prepared dry state' up to a predetermined stress level, and allowed to equilibrate, then wetted progressively until completely inundated to induce collapse. Wetting was performed at different overburden stresses but 300 kPa was the foremost stress used. Though the literature appear to favour 200kPa as a general wetting stress, 300kPa (roughly equivalent to the self-weight of a 15m high embankment) was chosen here because of the high dry stiffness of samples caused by CaCO<sub>3</sub> content (Langroudi (2014) recognizes high dry stiffness as common with calcareous soils). See also section 2.4.4 for argument on wetting stress. Each stage of loading increment before wetting was maintained until compression was completed or no more than 0.05mm/h compression is recorded (Alan and Robert, 1988) while after inundation loading was maintained for 24 hours before further increments. While collapse was defined to be the settlement occurring within 10 minutes of wetting (Fookes and Best, 1969), longer periods were necessary to allow for sufficient bond dissolution in accordance with ASTM (D 5333 - 03). At the end of the 24-hour soaking, further increments to reach 500kPa were carried out before decrements in the reverse order down to 200kPa.

### ***Double oedometer test (DOT)***

The DOT involved testing two nominally identical specimens, one as in the SOT and the second differing as follows. The specimen placed under the oedometer loading frame was inundated simultaneously with the initial 100kPa loading. This is here referred to as '*presoaked*'. Incremental loading and unloading procedures are identical to the SOT.

The DOT is used to estimate the amount of deformation or collapse that would occur if a soil sample was wetted at different stages of its stress history, a process well presented by Fredlund and Gan (1995); Rogers (1995); Alan and Robert (1988). The curves of overburden stresses against either axial deformations or void ratio change for the both presoaked and single oedometer tests are compared. The amount of deformation at each stress level on the presoaked curve relative to identical stress level on the single oedometer curve represent an estimate of the degree of collapse that could occur if the soil was wetted at such stress level during its loading history (Alan and Robert, 1988).

### ***Merits, demerits and sources of error of the testing procedures***

1. The testing procedure is not cumbersome. However, a lot of time can be spent monitoring rate of deformation where further load increments are desired.
2. Where absolute care is not given, the process of manually loading and unloading specimens during testing can cause irregularities in the data retrieved.
3. In some cases, specimens are manufactured directly in oedometer rings and therefore disturbance can be very minimal while in other cases where samples are shaved to fit into rings, the structure of specimen surfaces can be altered. Sample disturbance could lead to either a reduction or an increase in the estimated CP.
4. The oedometer pot is relatively small and can only hold a limited amount of fluid, which in certain conditions may not be enough to satisfactorily dissolve the particle bonding element. This may lead to underestimated collapses compared to field situations.
5. The acidic solution adopted in this research could corrode oedometer machine.
6. A possible reaction of the acidic solution with the oedometer components can change the chemistry of wetting fluid, thus altering the expected reaction between the soil and the wetting solution.
7. Though the principle of the collapse phenomenon is based on the elimination of inter-particle bonding and consequent densification, yet the ingress of fluid to the specimen will initially generate excess pore pressures, which are subsequently dissipated before densification. It may be interesting to monitor these pressures during testing but the conventional oedometer system does not possess the means.
8. The degree of wetting that occurs in the laboratory is higher than that which will likely occur in the field. This effect is conservative, because partial wetting leads to only partial collapse.

### **3.5.3 Unconfined compressive strength testing equipment and protocol**

#### ***Testing system***

A loading machine, “Instron 5585H series floor model testing systems” shown in Figure 3.22 was used for the compression testing. The machine has a 250kN loading capacity designed to carry out tension, compression, bending and component testing. The loading frame is internally fitted with a force transducer connected to a computer for automatic data (force/displacement) logging. It has a 1256 mm by 575 mm test area with a 0.001 – 500 mm/min speed range and is compatible with Bluehill<sup>®</sup> 2 data retrieving software.

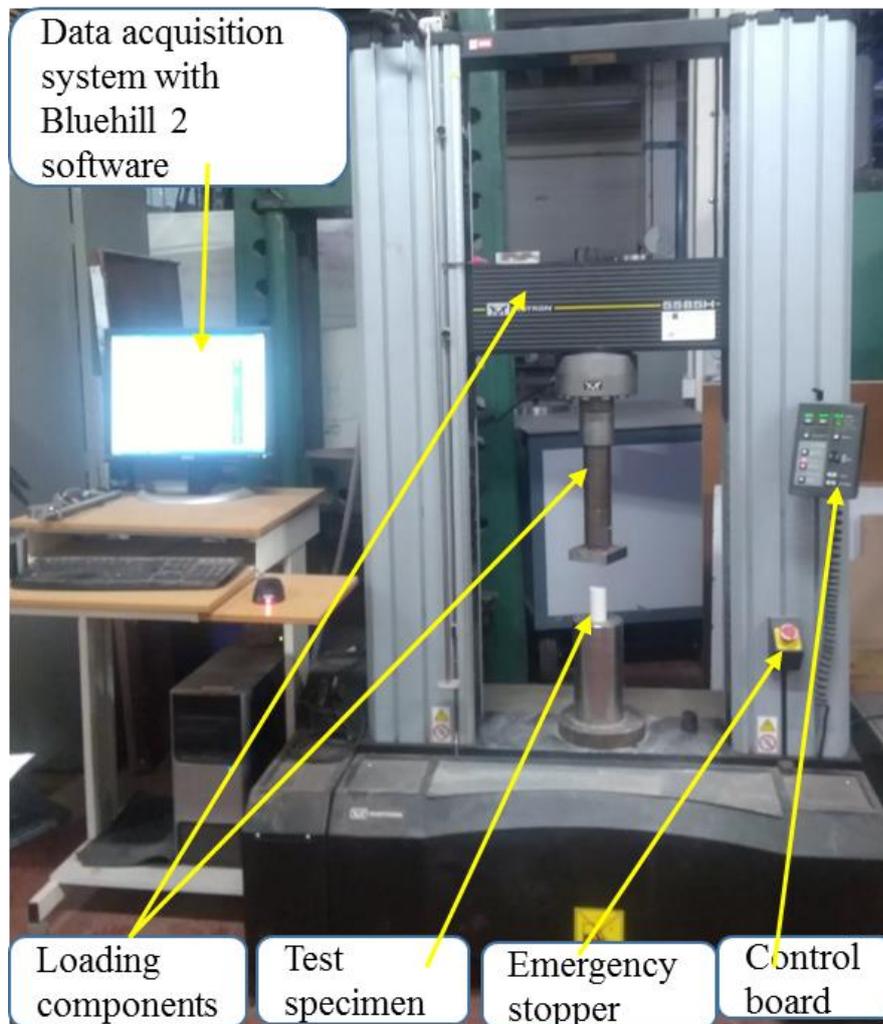


Figure 3.22 Instron 5585H series floor model testing systems showing major components (Newcastle University Geotechnical Labs)

### ***Testing protocol***

This test was performed in accordance with BS1377 (1990c) using the load frame method. Cylindrical specimens of height and diameter, approximately 76 and 38 mm, respectively manufactured as described in section 3.4.5 were uniaxially compressed to failure under the loading machine in Figure 3.22. Each sample was tested in triplicate and where stress-strain curves vary significantly from one another further specimens were tested to find closely related ones, from which mean behaviour was determined. Test specimens were placed on the lower platen of the loading machine and were capped with a smooth flat metal cap. The data logger (Bluehill 2 software) was then launched and programmed to log force/deformation data. Before loading proper, the loading rod was lowered until slightly in contact with the specimen's top cap. Displacements resulting from lowering of the loading rod recorded on the data logger were reset before testing was started. A loading rate of 0.2mm per minute was used for all specimens.

At the end of testing, i.e., after failure, load was removed, and the mode of failure examined. In the case of sodium silicate treated specimens, moisture content was thereafter determined.

UCS was calculated in accordance with BS1377 (1990c) following Equations 3.11 to 3.14 whereas, Young's modulus (stiffness) was estimated from the gradient of the linear (elastic) portion of the stress-axial strain curve as in Figure 3.23.

$$\text{Strain, } \varepsilon = \frac{\Delta L}{L_0} \dots\dots\dots 3.11$$

$$\text{Corrected area, } A = \frac{A_0}{1 - \varepsilon} \text{ (mm)} \dots\dots\dots 3.12$$

$$\text{Axial stress, } \sigma_1 = \frac{1000P}{A} \text{ (kPa)} \dots\dots\dots 3.13$$

$$\text{Unconfined compressive strength, } q_u = \sigma_1 \text{ at failure (MPa)} \dots\dots\dots 3.14$$

where  $\Delta L$  = Change in length,  $L_0$  = initial length,  $A_0$  = initial area,

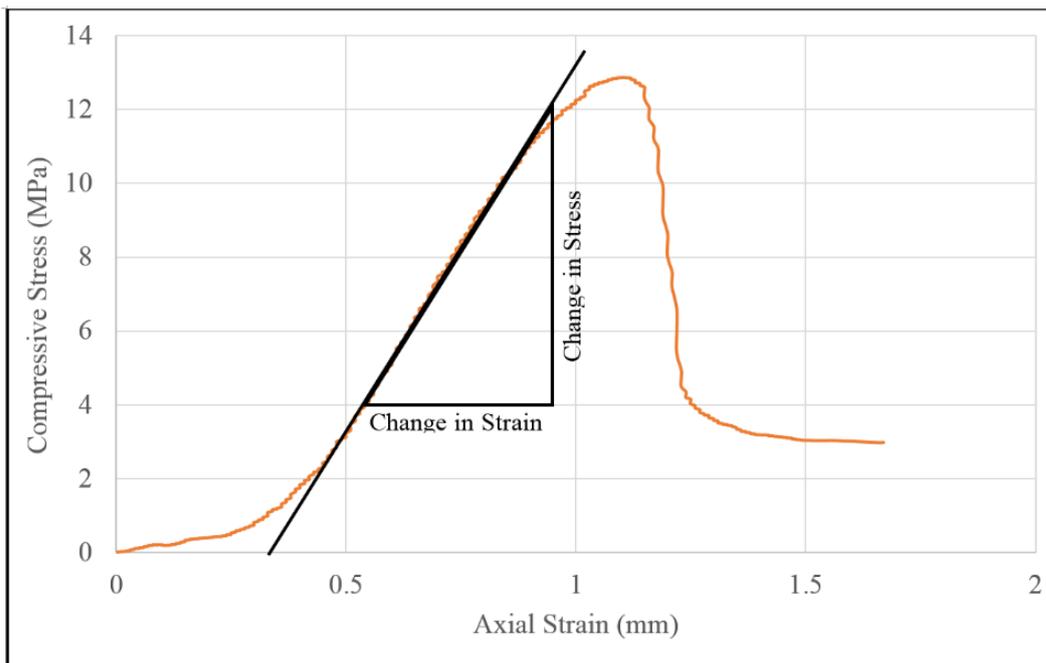


Figure 3.23 Estimation of Young's Modulus of elasticity (stiffness)

### ***Merits, demerits and sources of error of the testing method***

1. The testing procedures are quite easy to follow
2. Testing time is relatively short and as such several specimens could be tested in a day
3. Because of lack of lateral confining pressure, UCS testing is usually limited to cohesive types of samples. However, it has been used here to test relatively loose samples (uncemented non-calcareous). Notwithstanding the care taken in their handling there were tendencies for errors.
4. Without confinement, the shear strength parameters obtainable from this testing are limited.

## **3.6 General Limitations in the Laboratory Works**

A few limitations to the experimental works that might have affected the quality of data obtained and conclusions reached in this research are discussed under the following headings:

### **3.6.1 Scope of work**

Restricting the testing programme to unconfined compressive strength and collapse potential has not provided the opportunity to understand, for instance, the ease of water infiltration in both untreated and treated samples. Permeability is an important factor that influences the rate of collapse and could have also provided an understanding as to whether the sodium silicate treatment (in chapter 5) produced an impervious layer on treated samples.

### **3.6.2 Limited number of test samples**

Due to time and budget constraints the number of test samples were limited. This practically reduced the data points, preventing a more detailed trend. Thus, interpretations are done with some level of uncertainty. In some cases, error bars also indicated large discrepancy. Testing of more samples could have increased the level of confidence, and more data points would have increased the understanding of the impact of the various parameters considered in this study, e.g., influence of clay and CaCO<sub>3</sub> contents on both the CP and UCS.

### **3.6.3 Experimental methodology and conditions**

The testing techniques used did not allow the acquisition of enough data to explore other sample parameters needed to aid a more robust interpretation of the results. For instance, the effective parameters of samples could not be obtained under the unconfined compressive testing. Again, suction and degree of saturation are key factors controlling collapse potential and strength, but these factors were assumed absent in the test samples since they were oven-dried (45 - 50°C) before testing.

Additionally, the uncontrolled ambience of the laboratory. Batches of samples have been prepared (manufactured, treated, cured and tested) on different days under varying room temperature and humidity in the ranges  $18\pm 3$  °C and 25 – 35 %, respectively. Although the effect of these variations was not measured, they are believed to impact on the physical properties of the soil samples. For instance, Bulut and Leong (2008) report that a small change in the relative humidity (0.999656 and 0.999063) in soil air phase leads to a large change in suction (47.199 and 128.6 kPa, respectively). That is, a 0.06% decrease in humidity causing about 172% increase in suction. However, the relationship between suction and shear strength was shown to be nonlinear, and in some cases change in suction did not significantly affect shear strength (Alonso *et al.*, 2010a). Although in the present study temperature and humidity variations were not directly related to the mechanical properties of samples, it is assumed that such changes contributed to the variations in measured properties such as strength and stiffness. These variations were significant in some cases. For instance, variations generally ranging between 5 to 30 kPa for UCS and 0.01 to 0.2 MPa for stiffness data were measured.

#### **3.6.4 Quality of SEM images**

The poor quality of SEM images used to study the microstructure of test samples led to a limited understanding of the relationship between samples' microstructure and parameters tested. Good quality images were necessary to study the general microstructure of samples and in particular, the type of clay-bridge fabric and meniscus cement (including shape and mode of CaCO<sub>3</sub> occurrence: whether as inter-particle meniscus, nodular bonding, encrusting, reinforcing, or scaffolding as recognized in section 2.3.3), as they affect collapse behaviour (Langroudi, 2014).

### **3.7 Conclusion on Study Approach and Methodology**

To be able to achieve comprehensible and practical results satisfying the aims and objectives of this study, laboratory procedures were implemented. These procedures were developed following background knowledge in three themes:

1. formation of carbonate bond in potentially collapsible soil samples
2. geotechnical behaviour (UCS and CP) of cemented unsaturated collapsible soils
3. ground improvement of fine-grained soils

Following rigorous investigations on each of these themes, a high-quality data set was generated, and presented. The present chapter deals largely with theme (1) while themes (2) and (3) form the two following chapters.

## Chapter 4 **Mechanical Behaviour of Untreated Samples**

### 4.1 **Introduction**

This chapter presents results and discussions characterizing the mechanical behaviour of the artificial calcareous silty-clay samples analogous to natural marls, comparing them with non-calcareous samples of similar physical properties. These samples prepared to exhibit features of collapsible soils have been targeted to study mainly the role of calcite and clay contents on collapse potential (CP) and rate of collapse under different wetting conditions. The single and double oedometer techniques (described in section 3.5.2) were employed here using distilled water and a low pH (acidic) solution as wetting fluids. This study furthers the understanding of the mechanism of collapse as related to the already established knowledge on bond weakening/softening caused by pore fluid. Unconfined compressive test (UCT) (detailed in section 3.5.3) was adopted to study the relationship between strength and CP as strength is usually believed to characterise collapse potential. Generally, in ground improvement of collapsible soils, efforts have generally been targeted at increasing strength (e.g., Jefferson *et al.* (2005); Iranpour and haddad (2016)). The results presented under the various discussion sections are in one standard deviation of mean values.

### 4.2 **Unconfined Compressive Strength (UCS) Testing**

#### 4.2.1 **Introduction**

Under UCS testing, stress-strain behaviour defines the deformational characteristics of a cohesive soil specimen subjected to axial loading. These characteristics include compressive strength (the minimum stress causing shear failure) and stiffness, which is the resistance to deformation defining a brittle or ductile behaviour.

The present study aimed to investigate a collapse mitigating strategy for calcareous silty-clay soils using a sodium silicate solution. Thus, since inter-particle bonding strength is a criterion for assessing soil collapsibility and a vital property usually considered in the context of ground improvement, it was imperative that strength characteristics of samples before and after treatment are investigated to evaluate the potential of the improvement programme. UCS and stiffness of untreated non-calcareous and calcareous specimens have been examined and presented in the following sections while treated (or silicized) samples are dealt with in

chapter 5. A relationship between strength characteristics and collapse behaviour was also intended to be established between comparable specimens.

Oven dried cylindrical specimens of height and diameter approximately 76 and 38 mm, respectively were created as described in section 3.4.5. In uniaxial and triaxial testing, specimens with length-to-diameter ratio of about 2:1 are recommended to ensure that effects of radial shear stresses between soil specimen and machine platens (top cap and base-pedestal) are not significant at the middle of specimen (Clayton *et al.* (1995); Head (1998) p 197). This is because, ratios less than 2 will affect the results as shear stresses at the ends of the specimen in contact with platens will constrain the failure planes. However, BS1377 (1990c) recommends that a length variation from 8 % under-size to 12 % over-size will not considerably affect the results.

Compressive test (CT) specimens with comparable physical properties to the tested oedometer specimens were desired to be produced and tested here in order to relate mechanical behaviours acquired from both tests, especially as concerning impact of inherent bonding. However, there were difficulties in moulding CT specimens with dry densities identical to their oedometric counterparts because of differences in the internal volume of available specimen moulds. For instance, the same amount of soil-mix required to mould an oedometric specimen of a certain low dry density was insufficient to mould a standard compressive test specimen of comparable dry density. The minimum initial dry density before carbonation that it was possible to prepare CT specimens to was about 1.1 Mg/m<sup>3</sup> as against approximately 0.9 Mg/m<sup>3</sup> for oedometric specimens. After carbonation the CT specimens had the following estimated physical properties shown in Table 4.1.

*Table 4.1 Physical properties of cylindrical compressive test specimens*

<i>Specimen ID</i>	<i>Clay/Silt ratio</i>	<i>CaCO<sub>3</sub> content (%)</i>	<i>Dry density (Mg/m<sup>3</sup>)</i>	<i>Void ratio, e</i>	<i>Porosity, n (%)</i>
A0	1	0	1.11	1.39	58.11
A1	1	~12	1.11	1.38	57.92
A2	1	~25	1.14	1.29	56.33
A3	1	~40	1.18	1.14	53.35
B0	1.9	0	1.10	1.39	58.25
B1	1.9	~12	1.10	1.37	57.80
B2	1.9	~25	1.15	1.26	55.83
B3	1.9	~40	1.18	1.14	53.26
C0	4	0	1.10	1.38	58.01
C1	4	~12	1.11	1.35	57.39
C2	4	~25	1.15	1.28	56.23
C3	4	~40	1.19	1.11	52.53

#### 4.2.2 Stress-strain relationship of uncemented non-calcareous samples

In order to understand the impact of  $\text{CaCO}_3$  cementation on strength development in the samples, non-calcareous specimens with similar dry densities as to the calcareous ones were initially prepared and tested using the load frame method according to BS1377 (1990c). The samples labelled A0, B0, and C0 are described in Table 4.1 above. Prepared to a low dry density and oven dried to attain an unsaturated state, these specimens could only be transferred to the loading frame with extra care employed as they were extremely friable and perhaps only held together by their inter-particle attraction and some sort of frictional interlock. It is common knowledge that fine soils obtain their shear strength from adhesive forces. However, in these low-density samples, which can be comparable to dry normally consolidated clays, adhesion may be nearly negligible. Nevertheless, under drained conditions normally consolidated clays exhibit some frictional behaviour (Smith (2014), p 121). The stress-strain relationship and failure modes of these non-calcareous samples are presented in Figure 4.1 and Figure 4.2, respectively.

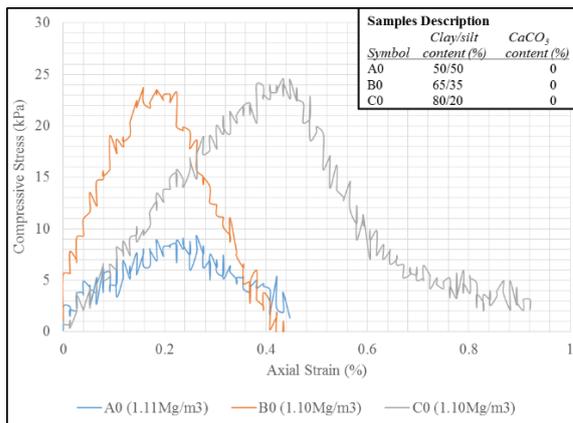


Figure 4.1 Stress-strain curves of non-calcareous specimens under unconfined compressive stress

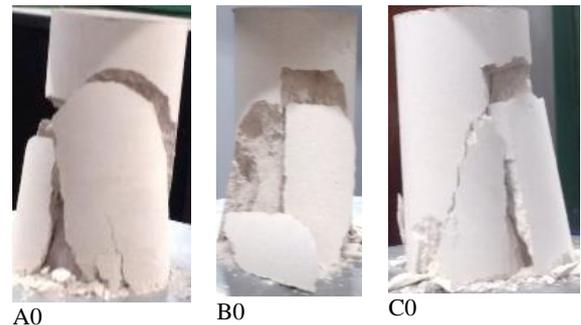


Figure 4.2 Failure modes of non-calcareous specimens under unconfined compressive stress

Ordinarily, these non-cemented samples possibly described as unsaturated cohesionless silty-clay samples, were not suitable for testing under the UCS system because of their lack of substantial stability (Cernica, 1995). However, with great care it was possible to achieve minimal handling disturbances. Their lack of apparent cohesion and low dry density state were mainly responsible for the low compressive strengths obtained, which ranged between 9 to 25  $\text{kN/m}^2$  with higher clay content producing higher UCS. These values, which are assumed to be obtained under drained conditions (dry specimens) are still well below the upper limit ( $40\text{kN/m}^2$ )

of undrained shear strength known for normally consolidated soft clays (Smith, 2014). That is, the samples are weaker than normally consolidated clays.

Typically, in frictional conditions, samples with lower clay/silt ratio would have been expected to yield higher shear strengths than higher clay/silt ratio. Nonetheless, frictional interlock of silt particles was unexpected due of the low density and relatively high clay contents. The silt particles have been widely dispersed within the clay so could not interact with each other. Again, the idea by Lawton *et al.* (1992), that clay soil-water mixing creates macropeds which can improve strength may be applied here. Because higher clay contents will normally produce more macropeds, strength could be expected to be greater with higher clay contents in certain clay/silt-systems.

Even though the failure patterns (Figure 4.2) tend to demonstrate brittle behaviours, the irregular or corrugated surfaces of curves (Figure 4.1) makes it difficult to define a definite peak stress, which can be interpreted as being the result of multiple microscale alternations between stiffening and softening. The specimens appeared friable during handling and as such compressing at a relatively slow rate of 0.2 mm/min could allow for localised micro straining and hardening until maximum shear stresses were mobilized. At this point deep longitudinal cracks developed (Figure 4.2) initiating a wedge deformation in most of the specimens with some also failing via the formation of a cone at the specimen base.

#### **4.2.3 Stress-strain relationship of cemented calcareous samples**

The stress-strain behaviour of the nine calcite-cemented (calcareous) samples shown in Table 4.1 alongside the non-calcareous ones (A0, B0 and C0), have been investigated. These oven dried specimens were designed to simulate young unsaturated sediments having calcite cementation in order to study the impact of the degree of calcareousness on the strength of fine-grained soils.

The stress-strain relationships and pictures showing their failure modes are presented in Figure 4.3, Figure 4.5 & Figure 4.7 and Figure 4.4, Figure 4.6 & Figure 4.8, respectively. In each Figure, samples presented are of a similar degree of calcareousness, but differentiated by clay/silt ratio (indicated by different line colours in the stress-strain curves). Specimens were prepared to approximately the same initial dry density before carbonation ( $1.0 \text{ Mg/m}^3$ ). However, after carbonation dry densities differ between  $1.10 - 1.19 \text{ Mg/m}^3$ , increasing in order of increasing  $\text{CaCO}_3$  contents.

Some authors, such as Nagaraj and Srinivasa (1994) and Nagaraj and Miura (2001) have recognized a combination of two key factors to control the load bearing capacity of cemented soft clays. These factors included the strength of soil skeleton and the strength of cementing bonds. Although it may be observed from the results presented in Table 4.2 that specimens with higher degree of calcareousness (and consequential higher dry density) produced higher UCS and stiffness values, the impact appears to result more from the calcite-cementation and clay/silt ratio rather than dry density. For instance, each group of different samples (Figure 4.3, Figure 4.5 and Figure 4.7) had similar dry densities, yet there are significant differences in their UCS values. Some of those with nominally higher dry density yielded lower UCS values.

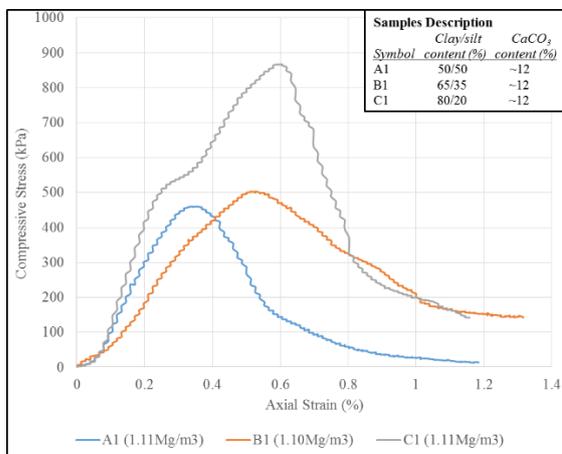


Figure 4.3 Stress-strain curves of “moderately calcareous” specimens under unconfined compressive stress

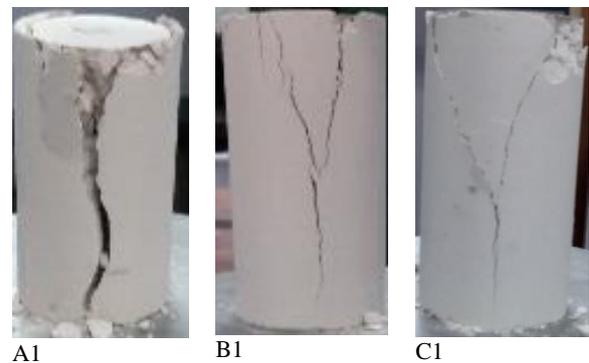


Figure 4.4 Failure modes of “moderately calcareous” specimens under unconfined compressive stress

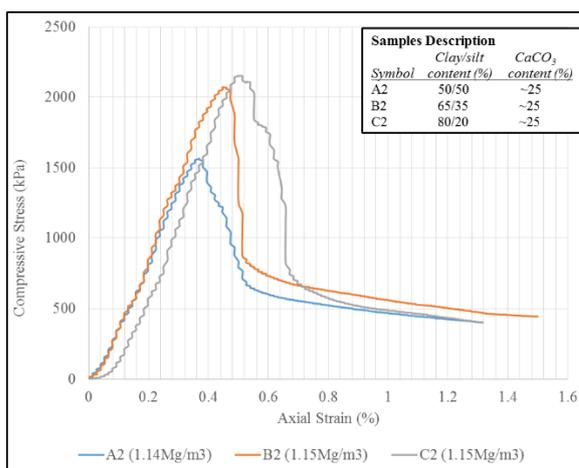


Figure 4.5 Stress-strain curves of “strongly calcareous” specimens under unconfined compressive stress

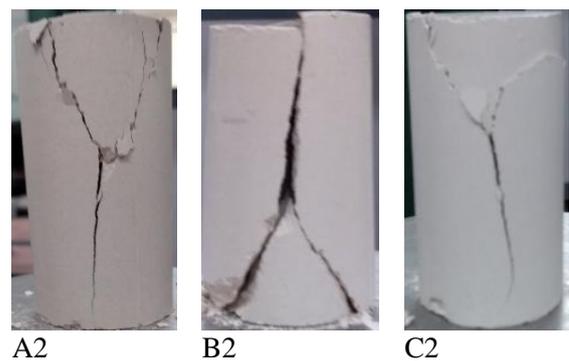


Figure 4.6 Failure modes of “strongly calcareous” specimens under unconfined compressive stress

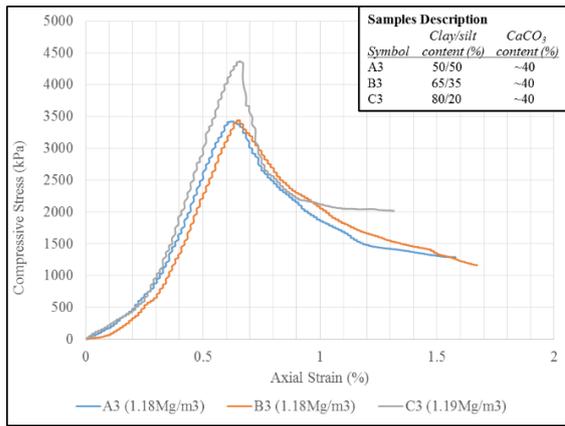


Figure 4.7 Stress-strain curves of “very strongly calcareous” specimens under unconfined compressive stress

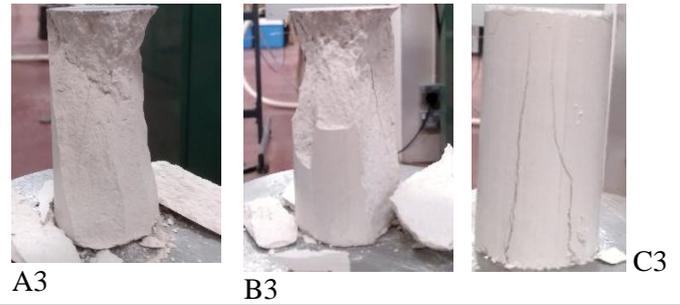


Figure 4.8 Failure modes of “very strongly calcareous” specimens under unconfined compressive stress

Table 4.2 Mechanical properties of the calcareous silty-clay samples under UCT

Sample	Peak UCS (kPa)	Strain at peak stress (%)	Stiffness (MPa)
A1	460	0.35	2.07
A2	1575	0.37	4.69
A3	3422	0.62	8.93
B1	550	0.53	1.25
B2	2071	0.45	5.00
B3	3437	0.66	8.33
C1	867	0.60	2.81
C2	2155	0.50	6.25
C3	4369	0.66	10.42

Expectedly, calcite-cementation resulted in strength increase, with increasing calcite content producing higher UCS values. Table 4.2 shows UCS values for *moderately*, *strongly*, and *very strongly* calcareous specimens. It appears that the proportion of strength development can be related to the amount of CaCO<sub>3</sub> increase. For instance, about a hundred percentage increase of calcite content (from the ~12 to ~25 %) produced about 150 – 200 % increase in UCS. A further sixty percent increase in calcite content produced strength increases in the range of about 60 – 100 %. This means that the proportion of strength increase is a function of the proportion of cement increase. However, this cannot be endless. Similar results have been reported, where carbonate contents at certain levels drastically changed the engineering behavioural trends of carbonate soils (Demars *et al.* (1976); Agarwal *et al.* (1977)).

Because of the significant differences in the chemical and engineering properties of silt and clay, the ratio of clay/silt in fine-grained soils usually influences the effectiveness of cementation, thus controlling the resultant strength properties of the cemented soil. Based on

the superior engineering properties of silt compared to clays, Teerawattanasuk and Voottipruex (2014) recognised a reducing clay/silt ratio to favour higher strength development in cemented soft clays. On the contrary, results presented here have suggested otherwise.

At each degree of calcareousness, UCS values were higher with greater clay/silt ratios. This may perhaps, be the effect of inherent void ratios and porosity values, although void ratio and porosity are only marginally lower with increasing clay/silt ratios. Nonetheless, it can also be noted that the prominence of the impact of clay/silt ratio was minimal with the *very strongly* calcareous (~40% CaCO<sub>3</sub> content) samples, especially between samples A3 and B3. This is in agreement with the claim that at greater than 30% carbonate content in calcareous sands, the influence of quartz on the strength of the sands was eradicated (Agarwal *et al.*, 1977). This indicates that at high CaCO<sub>3</sub> content in soil, the behaviour of the soil becomes mostly controlled by CaCO<sub>3</sub>.

Notwithstanding the above, nearly all samples tested exhibited brittle behaviour with notable peak stresses, and an increase in stiffness, but with higher strains at failure as CaCO<sub>3</sub> content was increased (Table 4.2). Failure patterns are also comparable in all samples tested. Shear failure was suspected to occur as a result of sudden bond breakage as no form of bulging was observed. This is like what is normally seen with shearing of stiff rocks. In *moderately* and *strongly* calcareous samples a conical failure occurred producing two shear planes near the end cap, joining together at the centre of the specimen and propagating across the specimen causing complete rupture into three pieces as loading was allowed to continue after failure. However, in the *very strongly* calcareous samples, conical failures were associated with longitudinal splitting. Several shearing surfaces developed in the form of large cracks parallel to the longitudinal axis of specimen and resulting in spalling and breaking away of large slabs in these *very strongly* calcareous samples.

### **4.3 Discussion on Parameters Affecting UCS and Stiffness**

#### **4.3.1 Effect of CaCO<sub>3</sub> content on USC and stiffness, and density related properties**

Even though Figure 4.9 illustrates a nonlinear relationship, UCS and stiffness increase with increasing calcite content, irrespective of soil type. Nevertheless, UCS curves indicate a lower order of increase than stiffness with each calcite increment. These lower responses of strength to increasing calcite content may imply that brittleness or ease of cracking of calcite-cemented silty-clays under compressive stress could be unwittingly enhanced where an attempt was made to improve strength by cement increase. Thus, caution is required in such ground improvement.

Figure 4.10 shows a direct linear relationship existing between UCS and stiffness in all samples tested.

Earlier reports (e.g., Demars *et al.* (1976); Agarwal *et al.* (1977)) noted that increased carbonate content was beneficial to strength parameters (e.g. effective frictional angle, and bonding) of soils. See Table 2.7 on page 61. This was believed to be the result of an increased chemical bonding with increasing carbonate content. However, Agarwal *et al.* (1977) identified a detrimental effect of CaCO<sub>3</sub> contents above a certain amount (unnamed). They stated that beyond that level, the stress-strain behaviour of a calcareous soil was exclusively controlled by carbonate cementation, which caused breakage of aggregated composition. Similarly, Demars *et al.* (1976) noted a change of behaviour of calcareous clays from cohesive to granular behaviour at a threshold of about 40% carbonate content, but without identifying the cause. Iorliam (2019) has also reported an increase in UCS with increase in CaCO<sub>3</sub> content of a kaolin clay but noted a diminishing effect just above about 8% CaCO<sub>3</sub> content. The reason for this diminishing effect was also not made clear.

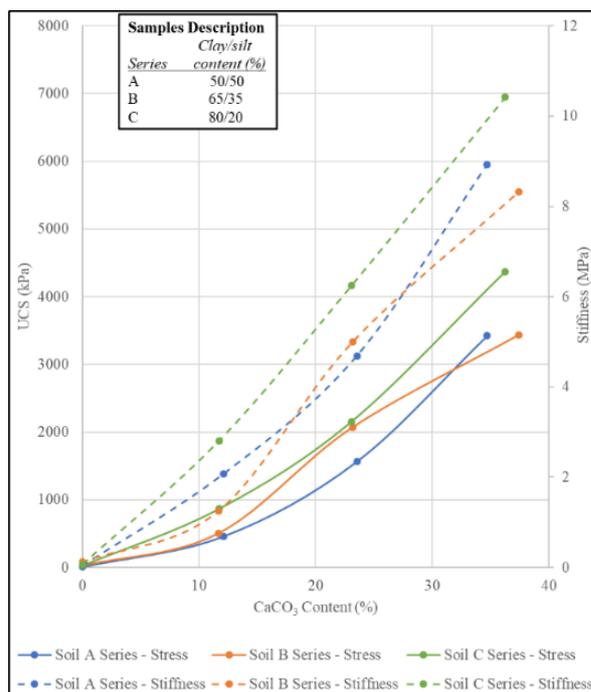


Figure 4.9 The impact of calcite cementation on UCS and stiffness of low density silty-clays

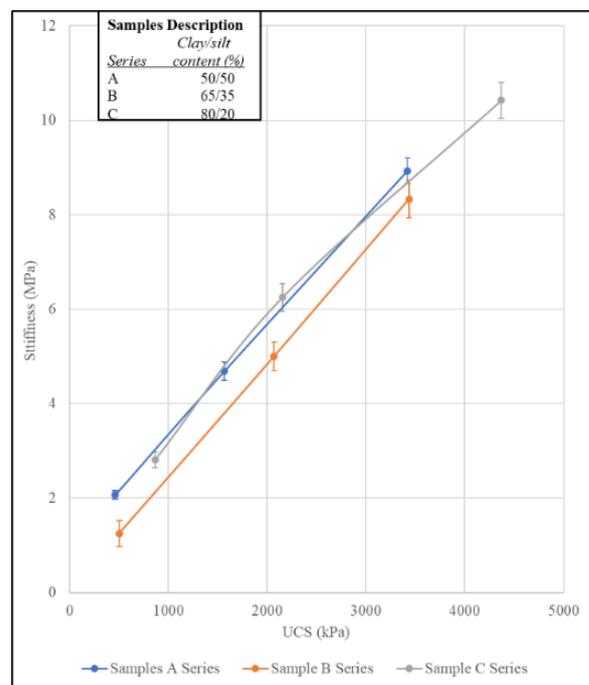


Figure 4.10 Relationship between UCS and Stiffness

Even though specimens tested in the present study barely contained CaCO<sub>3</sub> content greater than 40%, Figure 4.9 above show an obvious indication that though each carbonate increment resulted in an increased UCS and stiffness but the proportion of increase was lower each time CaCO<sub>3</sub> was increased. For instance, strength improvement in the range of 2023 –

4839 % from a zero to an ~12% CaCO<sub>3</sub> content, about 149 – 311 % from ~12% to ~ 25% CaCO<sub>3</sub> content and between 66 – 119 % improvement from ~25% to ~ 40% CaCO<sub>3</sub> content. This behaviour appears to agree with Agarwal *et al.* (1977). However, considering the low dry densities (~ 1.1 – 1.12 Mg/m<sup>3</sup>) of the present samples, it is difficult to assume the source of such influence. However, perhaps due to carbonation, micro-cracking could have become more prevalent with increasing quantity of carbonated lime (Moorehead (1986); Bagoniza *et al.* (1987); Vance *et al.* (2015)). Conversely, the condition reported for the natural calcareous soils by Demars *et al.* (1976) and Agarwal *et al.* (1977) did not involve precipitated carbonate.

Although there is scarcity of recent data in the literature relating to the impact of precipitated CaCO<sub>3</sub> on the strength of clayey soils required to compare with the present data, many researchers (e.g., Whiffin *et al.* (2007); Park *et al.* (2014); Montoya and DeJong (2015)) have investigated the potential of both biologically and chemically precipitated CaCO<sub>3</sub> to improve sand deposits. There is a general believe that CaCO<sub>3</sub> precipitates enhance the mechanical properties of such sands, and just like most conventional soil improvement chemicals, increasing CaCO<sub>3</sub> content produces higher compressive strength in the sand deposits. Two examples from the literature showing trends comparable to the present results are given here as Figure 4.11 (drawn from Park *et al.* (2014) data) and Figure 4.12 (Whiffin *et al.*, 2007). The Source 2 CaCO<sub>3</sub> shown in Figure 4.11 was precipitate from Ca(OH)<sub>2</sub>, similar to the one used in the present study.

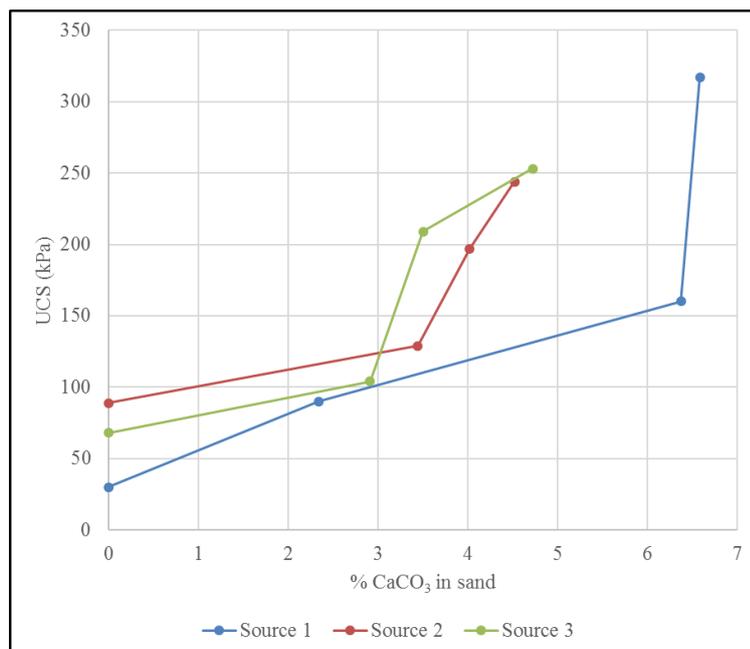


Figure 4.11 Effect of CaCO<sub>3</sub> content (chemically precipitated from 3 different sources) on the UCS of sand (plotted from Park *et al.* (2014) data).

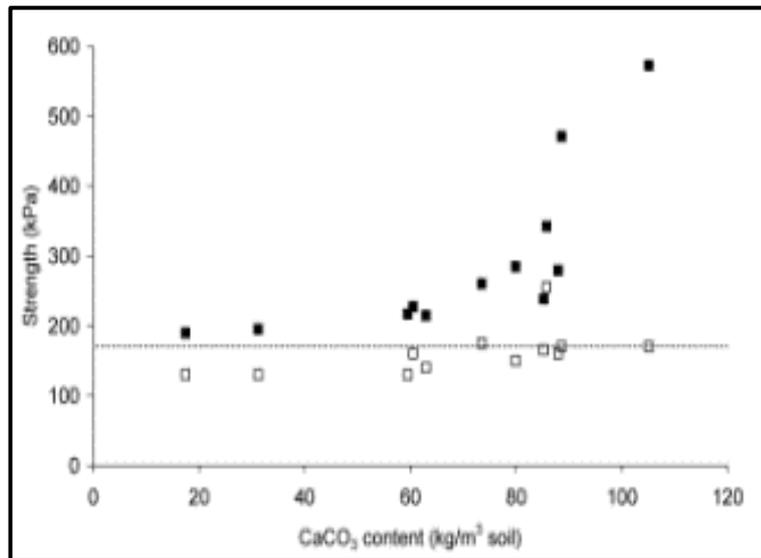


Figure 4.12 Effect of microbially precipitate  $\text{CaCO}_3$  content on confined compressive strength (■) and residual strength (□) of sand at 50kPa confining pressure (adopted from Whiffin *et al.* (2007))

Clay treatments with Portland cement or lime are also commonly known to increase strength and stiffness with increasing content (Croft (1967); Teerawattanasuk and Voottipruex (2014); Estabragh *et al.* (2013)). While it is a common strategy in ground improvement to increase cement material content with the aim of achieving a more packed soil with better strength properties, in the present samples (though containing differing cement content) packing (dry density) was controlled and only marginally vary with a standard deviation of 0.019. Figure 4.13 shows that although dry density may affect UCS, the variations recorded here have not in themselves influenced the compressive strength. Park *et al.* (2014)'s findings appear to agree with this.

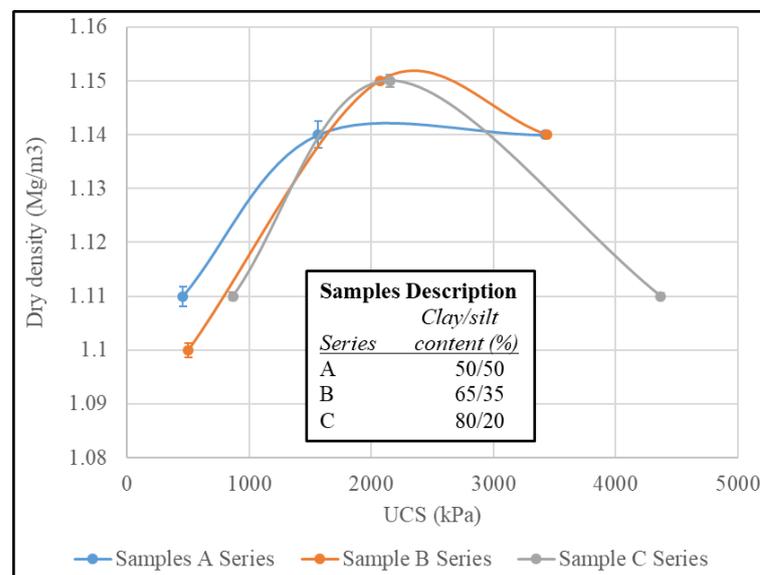


Figure 4.13 Dry density relationship with compressive strength

However, increased calcite content significantly reduced void ratio with a maximum difference of 0.23 and produced a stronger cementing bond as demonstrated by UCS in both Figure 4.9 above and Figure 4.14 below. The reduced void ratio with greater carbonate content as presented in Figure 4.15 is perhaps a consequence of volume of lime expansion following carbonation; as a result, those specimens with higher degree of calcareousness resulted in a less open structure and stronger cementation. Obviously, these factors are necessary to improve strength and stability.

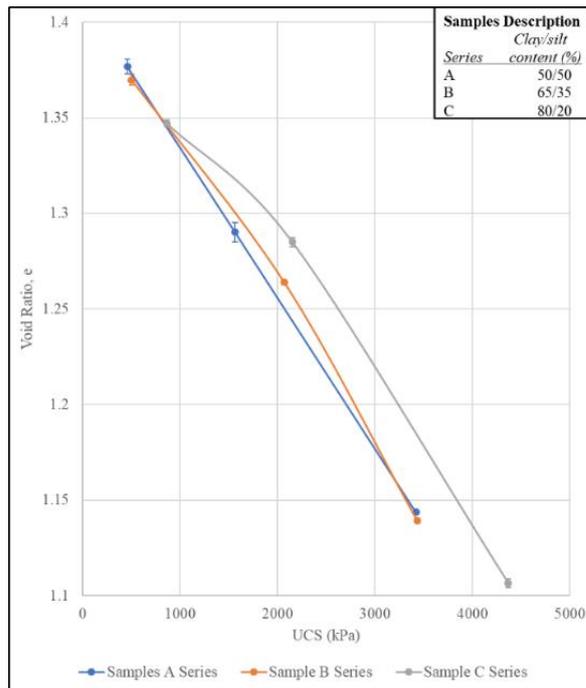


Figure 4.14 Void ratio relationship with compressive strength

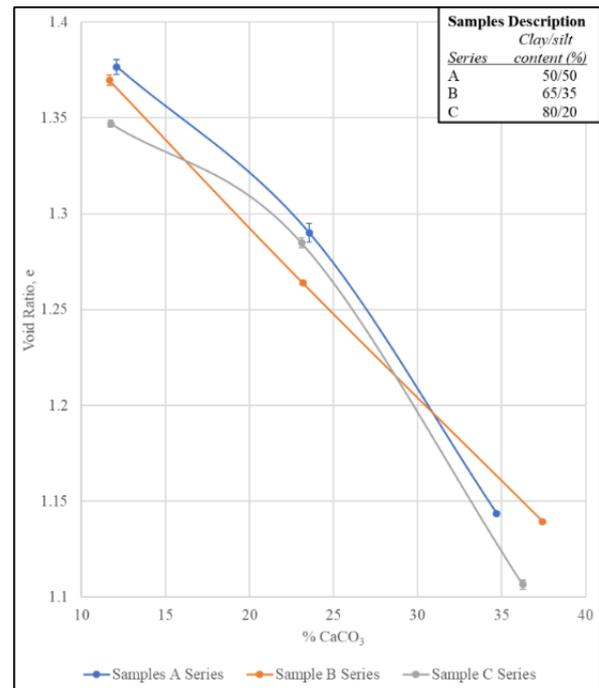


Figure 4.15 Void ratio relationship with carbonate content

Furthermore, strain at peak stress (i.e., at failure) have not been largely influenced by the changes in calcite content as illustrated in Figure 4.16. Arguably, a change in CaCO<sub>3</sub> content changes strain at failure. However, only sample A series show a clear trend (Figure 4.16) where strain at failure increased with CaCO<sub>3</sub> increase. An indication that increased degree of calcareousness could bring about some form of ductility, but this does not appear to reduce strength (Figure 4.17).

However, the stress-strain curves of all the calcareous samples (Figure 4.3 to Figure 4.8) present response associated to a brittle failure with defined peaks and no macroscopic cracking occurring before peak stress. However, it is established that rupturing at peak stress of a brittle material under compression is a consequence of the coalescence of microcracks (Amann *et al.*, 2011). Fedá (1995b) also argue that inter-particle bonds start breaking before the peak strength of a cemented soil. Non-calcareous samples on the other hand, having no cementation showed

a zigzag response (see stress-strain curves in Figure 4.1) indicating alternations of stress-dependent stiffening and softening typical of cohesionless soil behaviour under shear (Yun and Santamarina (2005)).

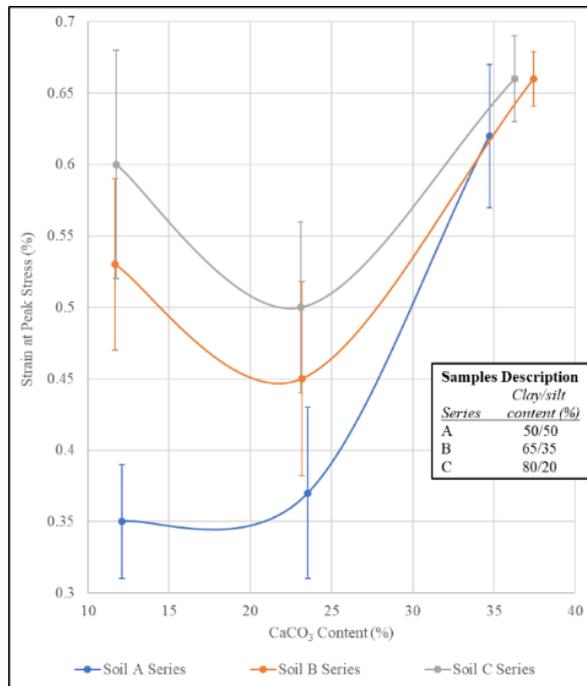


Figure 4.16 The impact of calcite cementation on strain at failure of low density silty-clays

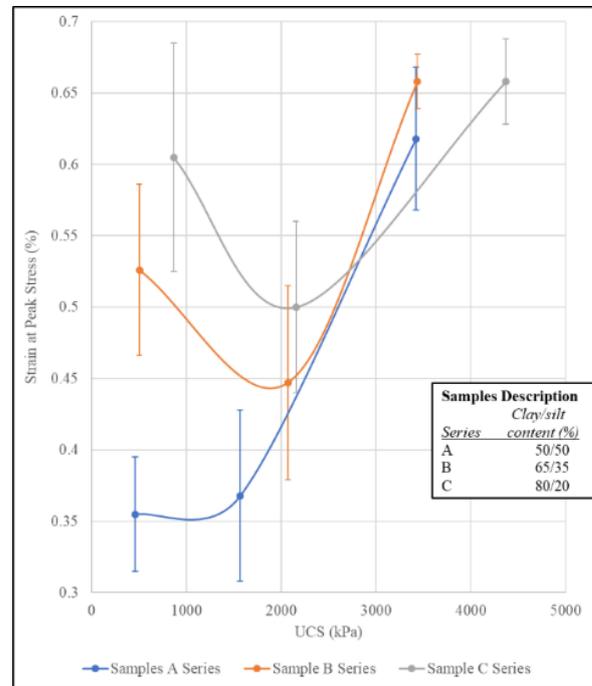


Figure 4.17 Relationship between strain and compressive strength

#### 4.3.2 Effect of clay content on UCS and stiffness

Clays and silts are both fine-grained soils but with differing engineering and chemical properties. Clays and silts usually occur together in nature in different proportions and commonly classified as soft clays (Teerawattanasuk and Voottipruex, 2014). Because of their varying properties, the mechanical behaviour of silty-clays can be related to the ratio of clay to silt. Typically, silts have greater enhanced engineering behaviours than clays.

The impact of clay content on the UCS and stiffness of specimens reported here as Figure 4.18 and Figure 4.19, respectively have demonstrated improvement of these properties with an increasing clay content. Although UCS values are greater with higher clay contents irrespective of samples degree of calcareousness, stiffness does not appear to follow a clear pattern. This impact of clay content on UCS seems to be in agreement with Ismail *et al.* (2002a), which claim that, the finer a soil is, the better the performance of a calcite cementation. However, the present non-calcareous samples have showed similar trend. This may be explained based on the effect of aggregation (Croft (1967); Lawton *et al.* (1992)) but it contradicts claims by some other researchers who argue that generally cementation produces higher strength with reducing

clay/silt ratio (Currin et al., 1976 cited in Sariosseiri and Muhunthan (2009); Teerawattanasuk and Voottipruex (2014)). Teerawattanasuk and Voottipruex (2014) for example, presented UCS results (see Figure 4.20 for comparison) of cemented fine-grained soils that indicated better performance (i.e., higher strengths) with increasing silt content, noting this as a consequence of superior engineering properties of silt compared to clays. Though bentonite constituted Teerawattanasuk and Voottipruex (2014)'s soil (bentonite-kaolin-silt) as against the present soil (kaolin-silt), they have agreed kaolin has better engineering properties than bentonite.

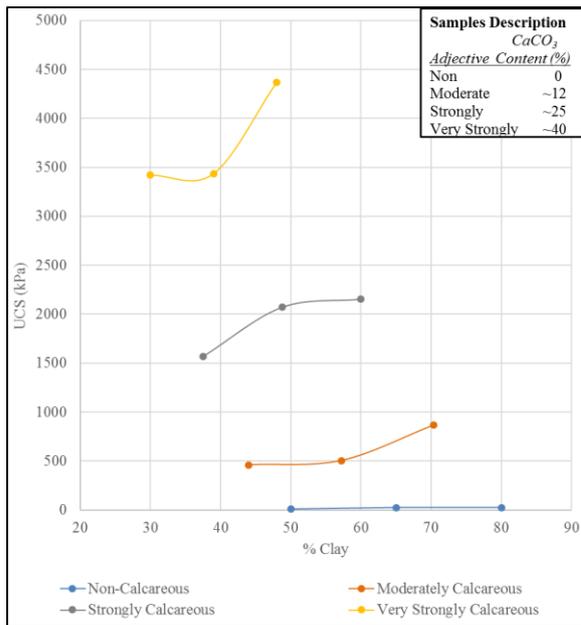


Figure 4.18 Effect of clay content on the UCS of calcite cemented silty-clays.

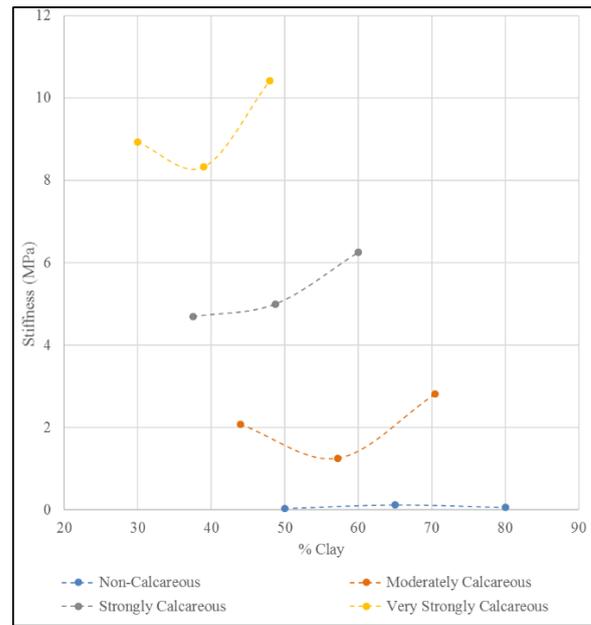


Figure 4.19 Effect of clay content on the stiffness of calcite cemented silty-clays.

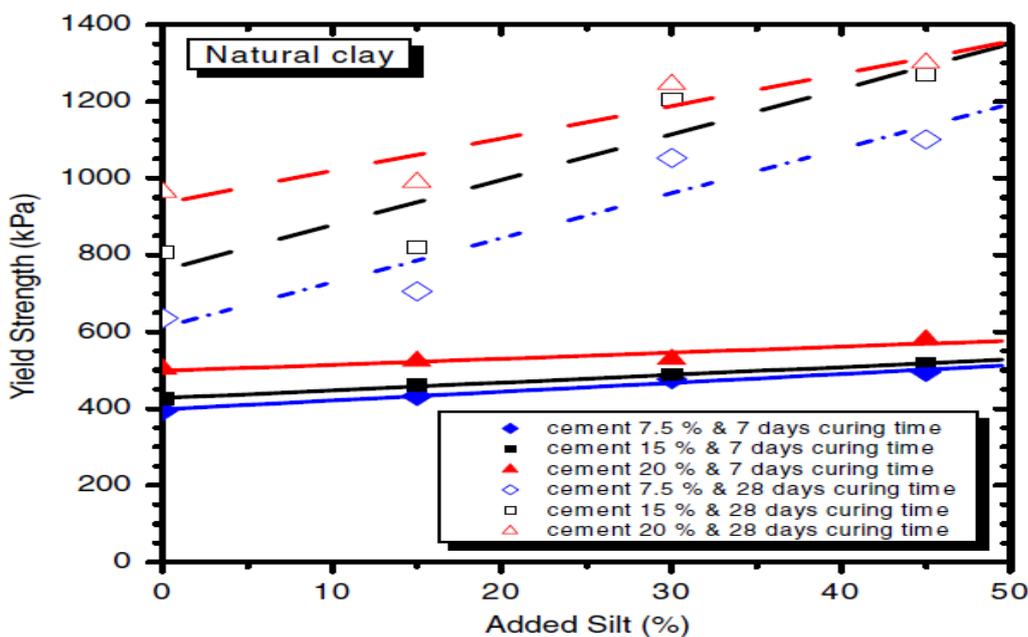


Figure 4.20 Effect of clay/silt content on strength (adopted after Teerawattanasuk and Voottipruex (2014))

Based on the proposal of the effect of soil aggregation, aggregates (or macropeds) developing from wet-soil mixing have been identified to control the behaviour of such soil compacts, with greater number of aggregates potentially developing from a higher clay system (Lawton *et al.*, 1992). Mitchell and Soga (2005) also identified the development of aggregates and described them as fundamental constituents controlling soil behaviour as they almost act as single particles and interact to produce strength and stiffness in clays. Croft (1967) argued that even if a soil-cement slurry is thoroughly mixed, clay particles would still form clusters surrounded by the cement slurry. These arguments suggest that because of aggregates formation a clay-silt sample with higher clay content could possibly better enhance engineering characteristics than silts. With potential greater number of clusters forming in the same order of increasing clay content in the present soil series:  $A < B < C$ , the trends in both Figure 4.18 and Figure 4.19 appear to demonstrate that the higher the clay content, the better the engineering performance. However, the relationships are nonlinear.

#### 4.3.3 Summary of UCS behaviour

Naturally, clay or silt soils do not exist individually but occur together in certain proportions and are referred to as fine-grained soils or soft soils. Typically, because silt has more enhanced engineering properties (e.g. hardness, durability) than clay, shear strength values are normally higher with reducing clay content. Also, in the treatment of soft soils, increase in strength and stiffness is usually the result of pore volume reduction and or cementing of the soil particles. The process adopted for precipitation of calcium carbonate into the silty-clay soils investigated here proved effective as it brought about inter-particle cementation and estimated void volume decreases and hence, the development of strength and stiffness.

At all levels of carbonate cementation (0%, ~12%, ~25%, and ~40%  $\text{CaCO}_3$  contents), higher clay contents have yielded higher peak stresses. The reason for this was suspected to be the impact of reduced void ratios rather than a texture since specimens existed in very low dry densities. There is the understanding also that greater numbers of clay macropeds resulting from mixing can enhance strength.

While void ratio decreased, strength and stiffness increased with increasing calcareousness (or  $\text{CaCO}_3$  content). However, this beneficial effect of increased carbonate content is assumed not to be without bounds.

Inter-particle bonding and its ease of elimination under loading/and or wetting conditions can be critical factors that define soils strength and collapse potential. As shown in the above

Figure 4.18 and Figure 4.19, UCS values (9 – 25 kN/m<sup>2</sup>) and stiffness values of the non-calcareous specimens are nearly insignificant (i.e., far below the upper limit of undrained shear strength of normally consolidated soft clays (Smith, 2014)). Whereas calcareous specimens revealed substantial compressive strengths increasing in the order of degree of calcareousness. The next section discusses the relationships between degree of calcareousness, debonding, and collapsibility of specimens.

#### 4.4 Collapse Behaviour of Samples

##### 4.4.1 Collapsibility of uncemented non-calcareous silty-clays

Low dry density uncemented, non-calcareous (i.e., 0% CaCO<sub>3</sub> content) samples were initially tested as control in order to enhance the understanding of the role of CaCO<sub>3</sub> in both the strength and collapse behaviours of calcareous soils. The impact of the wetting fluid type (in the case of collapse upon wetting) on these soils was also examined. These samples labelled A0, B0, and C0 have been described in Table 3.4, with A, B, and C representing clay/silt ratios of 1, 1.9 and 4, respectively while 0 denoting zero CaCO<sub>3</sub> content. The physical properties of test specimens prepared from these samples are shown in Table 4.3. At least three specimens were tested in each and mean final compressions at each loading stage and collapse potentials CP taken. Standard deviations between 0.1 and 1.0 were estimated for the CP values.

Table 4.3 Physical properties of uncemented non-calcareous test specimens.

<i>Specimen ID</i>	<i>Clay/Silt ratio</i>	<i>Specimen height (mm)</i>	<i>Dry density Mg/m<sup>3</sup></i>	<i>Void ratio, e</i>	<i>Porosity, n</i>
A0	1	19	1.01	1.62±0.002	61.80±0.03
B0	1.9	19	1.01	1.61±0.001	61.63±0.02
C0	4	19	1.01	1.57±0.002	61.16±0.03

Oedometric specimens with dry density approximately 1.01Mg/m<sup>3</sup> were wetted with distilled water or 5% acetic acid solution at 300kPa overburden stress in the single oedometer test (SOT) while only the acidic solution was used to pre-soak specimens in the double oedometer test (DOT). Loading and unloading sequences are detailed in section 3.5.2. Figure 4.21 and Figure 4.22 present deformation curves obtained from both the single and double oedometer collapse testing. In each of these figures, different colours distinguish soil types. In the single oedometer curves in Figure 4.21, full and broken lines represent distilled water wetting and 5% acidic solution wetting, respectively while in the double oedometer (Figure

4.22) full and broken lines represent wetting after loading and pre-soaked, respectively. The axial deformations of the samples due to loading before and after wetting, as well as CP and degree of severity, for the different wetting fluids, are summarized in Table 4.4.

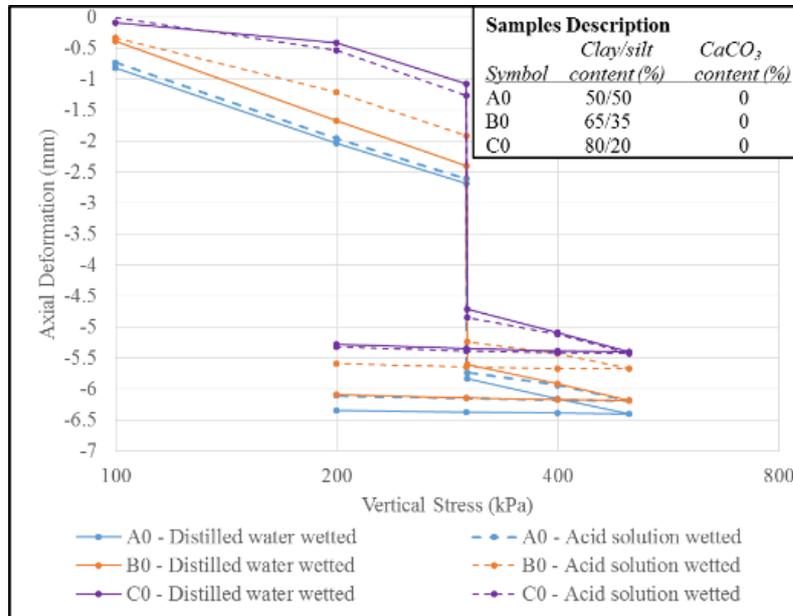


Figure 4.21 Single oedometer deformation curves of different uncemented non-calcareous silty-clay samples with two wetting fluids

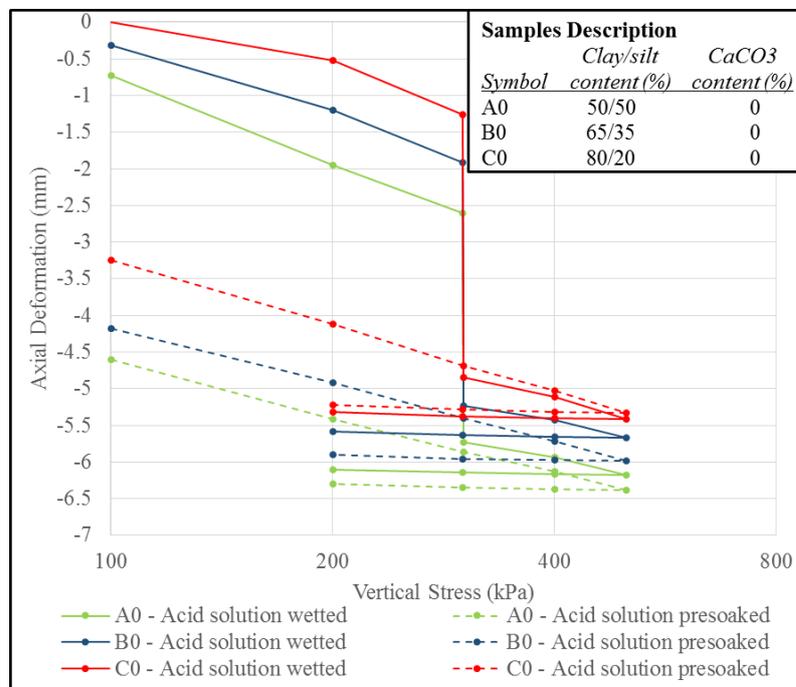


Figure 4.22 Double oedometer curves of different uncemented non-calcareous silty-clay samples with two wetting fluids

Table 4.4 Collapse potential and degree of severity of uncemented non-calcareous silty-clay samples

Sample	Initial Specimen height (mm)	Distilled water wetted at 300kPa (single oedometer)				5% acetic acid solution wetted at 300kPa (single oedometer)			
		Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Distilled water wetted)	Collapse Potential (%) (Distilled water wetted)	Severity	Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Acid wetted)	Collapse Potential (%) (Acid solution wetted)	Severity
A0	19	16.3	13.2	19.3±0.06	ST	16.4	13.3	19.1±0.25	ST
B0	19	16.6	13.4	19.3±0.18	ST	17.1	13.8	19.4±0.35	ST
C0	19	17.9	14.3	20.3±0.18	VST	17.7	14.2	20.2±0.32	VST
CP (%) at lower vertical stresses, from acid solution presoaked double oedometer									
		At 100 kPa	severity	At 200kPa	severity	At 300kPa	severity		
A0		21.2	VST	20.3	VST	19.9	ST		
B0		20.7	VST	20.9	VST	20.5	VST		
C0		17.1	ST	19.4	ST	19.3	ST		

In the SOT (Figure 4.21), curves for each pair of specimens (distilled water and acid solution wetted) were expected to show uniform deformations up until the wetting stage (300kPa) thereafter, they could differ depending on the effect of the wetting fluid type. However, minor variations have been recorded in some cases. These are not thought to be significant and can be attributed to experimental errors arising from the setting up of the apparatus and small operational differences.

Traditionally, clays are less easily densified compared to silts due to the diffuse double layer, flocculated structure and high water-retention capacity exhibited by clays. This was demonstrated by the amount of compressions obtained during pre-wetting loading. Both the early (pre-wetting stages) and the late (post-wetting) halves of the compression curves of single oedometers tests appear to indicate a higher deformation with lower clay/silt ratios in these uncemented non-calcareous samples (Figure 4.21). Similarly, presoaked curves of the DOT (Figure 4.22) show greater wetted deformations with decreasing clay/silt ratios at every stage of loading. Conversely, greater CPs (see Table 4.4 above) were recorded for higher clay/silt ratios in both wetting fluid types of the single oedometers. While CPs estimated from the double oedometers at 300kPa overburden stress, may closely relate (by a maximum difference of ~ 1 (Table 4.4)) to those of the single oedometers, at lower overburden stresses CPs have not followed a pattern but appear in many cases to show higher values compared to the single oedometers.

Following results in Table 4.4, especially from the single oedometers, there seems to be an indication of a textural (clay/silt ratio) control on both deformability and collapsibility. As noted earlier, clay resistance to deformation in a dry state leaves more room for samples containing higher clay content to settle or collapse when wetted. Again, clay minerals are known for their inter-particle attraction which in principle, contributes to bond strength in their unsaturated state. The type of bond strength or particle affinity, however, depends on the distance between the particles. Nevertheless, this bond strength resulting from particle attraction can easily be broken by factors, such as increased moisture content above a certain threshold. Increased water content in the diffused double layers of a clay structure increases distance between the layers, and the force of attraction holding layers together is broken, making the clay structure weaker (Mitchell and Soga, 2005). On the other hand, quartz silt particles possess no affinity for each other and only derive strength from frictional interlock. However, this depends on grain size.

Sample C0 contains 80/20 % kaolin clay/quartz silt which explains why it exhibits lower pre-wetting deformations in its unsaturated state relative to samples B0 and A0 having 65/35 and 50/50 % clay/silt proportions, respectively. Therefore, in the same way, B0 deforms less than A0 at pre-wetting stages and collapsed more post-wetting. However, it is important to note that the amount of structural collapse upon wetting may depend on the pore spaces available to accommodate such collapse as well as the amount of overburden load to enhance collapsing.

In addition, the initial void ratio and porosity, which are factors that determine how much a fine-grained soil may compress under loading, have shown decreasing values with increasing clay contents in samples before testing (Table 4.4). However, at the end of early loading (i.e. pre-wetting) the void ratio trend was reversed, increasing with higher clay content, confirming higher clay content leads to a higher resistance to densification. Since samples with higher silt contents compressed significantly at pre-wetting stages, they might have had their structure broken and are more packed with less void space available for any further densification.

Considering curves in Figure 4.21 and CP values in Table 4.4, it is evident that wetting fluid type (distilled water or 5% acid solution) did not influence the collapsibility of these non-calcareous samples indicating that collapse was therefore merely mechanically driven. Identical specimens wetted with either distilled water or the 5% acidic solution collapsed to approximately the same degree and compression trends after the collapse region were also similar to those in the pre-wetting region. This demonstrates the geochemical inertness of the samples to both the 5% acidic solution and distilled water. The mechanism of collapse in these

samples was possibly a result of broken physical or physicochemical force such as suction and molecular attraction, which provided the initial strength.

As is expected of non-expansive soils, the kaolin clay (with quartz silt) having relatively low water absorption capacity, exhibited irrecoverable behaviour after loading and unloading. This has been illustrated by relatively flat curves with close to zero or marginally negative slopes in all the rebound curves. In other words, no significant recovery or swelling pressures can be expected from these kinds of samples after densification. Beyond the collapse region, further settlements occurred indicating that samples still exhibited a loose state relative to the prevailing overburden stresses.

#### **4.4.2 Collapsibility of calcite cemented (calcareous) samples**

The key feature of samples here is that  $\text{CaCO}_3$  crystalizes in situ and acts as the inter-particle bonding material. Two sets of cemented samples were created. The first set of samples basically possessing the main criteria (low dry density, high void ratio and porosity) of a typical collapsing soil as described in section 2.3. These specimens were manufactured as would be tested, i.e., directly into oedometer rings with no further coring or trimming needed after production (see section 3.4.5). They exhibited very low dry densities ( $1 \pm 0.1 \text{ Mg/m}^3$ ) and exaggerated void ratios ranging between 1.5 - 1.8 and estimated porosities of 60 – 65 % post-carbonation. These values can be typical of young sedimentary deposits or normally consolidate clay (see Kokusho *et al.* (1982) and Shibuya *et al.* (1998)). Their stability can only be a consequence of chemical bonding as provided by  $\text{CaCO}_3$ .

The second set of specimens were those cored/trimmed into oedometer rings, from larger manufactured samples as described in Section 3.4.5 (3i). These specimens exhibited different dry densities, void ratios and porosities depending on soil type and compaction variables. These samples replicated lime-treated engineered earthworks that had undergone carbonation. Though their dry densities were quite high, it was appropriate to investigate the potential in situ effect of carbonation on the collapsibility of such engineered soils.

Each of the two sets of calcite-cemented samples described above contained calcite contents of varying degrees characterising them into *moderately*, *strongly* and *very strongly* calcareous samples. They were all oven-dried under temperatures between 45-50 °C to produce nearly completely dry or unsaturated samples before testing.

### Low dry density samples

The physical properties of the test specimens are presented in Table 4.5. Results obtained after testing are summarized in Table 4.6 with deformation and collapse curves presented in Figure 4.23 to Figure 4.28. The CP from single oedometer tests are those achieved at 300kPa overburden stress while those from the double oedometer (acidic solution presoaked) were estimated at 100, 200 and 300kPa overburden stresses.

Table 4.5 Physical properties of cemented calcareous oedometric test specimens with low dry densities.

Specimen ID	Clay/Silt content (%)	CaCO <sub>3</sub> content (%)	Specimen height (mm)	Dry density (Mg/m <sup>3</sup> )	Void ratio, e	Porosity, n (%)
A1	50/50	~12	19	0.93	1.87±0.004	65.12±0.05
A2	50/50	~25	19	0.96	1.72±0.002	63.25±0.02
A3	50/50	~40	19	0.99	1.53±0.003	60.54±0.03
B1	65/35	~12	19	0.93	1.84±0.002	64.75±0.02
B2	65/35	~25	19	0.96	1.70±0.002	63.01±0.02
B3	65/35	~40	19	1.00	1.59±0.005	61.42±0.07
C1	80/20	~12	19	0.93	1.82±0.003	64.61±0.05
C2	80/20	~25	19	0.96	1.68±0.004	62.65±0.06
C3	80/20	~40	19	1.00	1.54±0.003	60.72±0.05

Table 4.6 Specimen compressibility during loading, collapse potentials and degree of severity of low dry-density cemented calcareous samples

Sample	Initial Specimen height (mm)	Distilled water wetted (single oedometer)				5% acetic acid solution wetted (single oedometer)			
		Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Distilled water wetted)	Collapse Potential (%) (Distilled water wetted)	Severity	Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Acid wetted)	Collapse Potential (%) (5% Acid wetted)	Severity
A1	19	18.1	14.4	20.2±0.33	VST	17.8	13.6	25.2±0.19	VST
A2	19	18.8	18.6	1.3±0.09	MT	18.8	17.2	9.0±0.79	T
A3	19	18.9	18.9	0.2±0.04	NT	18.9	18.2	3.5±0.23	MT
B1	19	18.5	14.9	18.8±0.49	ST	18.6	14.2	23.5±0.79	VST
B2	19	18.9	18.7	0.8±0.17	NT	18.9	17.5	7.3±0.06	T
B3	19	18.9	18.9	0.1±0.14	NT	18.9	18.5	2.1±0.17	MT
C1	19	18.6	15.5	17.0±0.19	ST	18.7	14.6	21.9±0.53	VST
C2	19	18.9	18.8	0.4±0.01	NT	18.9	17.8	5.7±0.15	T
C3	19	18.9	18.9	0.1±0.02	NT	18.8	18.4	2.4±0.85	MT
<b>Collapse Potential (%) at different vertical stresses, from acid solution presoaked double oedometer</b>									
		At 100kPa	Severity	At 200kPa	Severity	At 300kPa	Severity		
A1		17.3	ST	22.8	VST	24.6	VST		
A2		4.0	MT	5.6	T	8.5	T		
A3		1.5	MT	2.3	MT	3.4	MT		
B1		13.5	ST	20.7	VST	24.0	VST		
B2		4.1	MT	6.0	T	8.2	T		
B3		2.9	MT	3.7	MT	4.5	MT		
C1		11.2	ST	19.4	ST	23.6	VST		
C2		2.7	MT	3.9	MT	5.6	T		
C3		1.6	MT	2.4	MT	3.1	MT		

Figure 4.23 to Figure 4.28 are pairs of single and double oedometer tests results. Each pair shows an identical group of samples. The groupings are based on calcite content (i.e., degree of calcareousness: *moderately*, *strongly*, and *very strongly* calcareous samples). In the single oedometer curves (Figure 4.23, Figure 4.25 and Figure 4.27) full lines represent distilled water wetted and broken lines signify 5% acidic solution wetted, while in the double oedometer curves (Figure 4.24, Figure 4.26 and Figure 4.28), full and broken lines represent wetting after loading and pre-soaked, respectively.

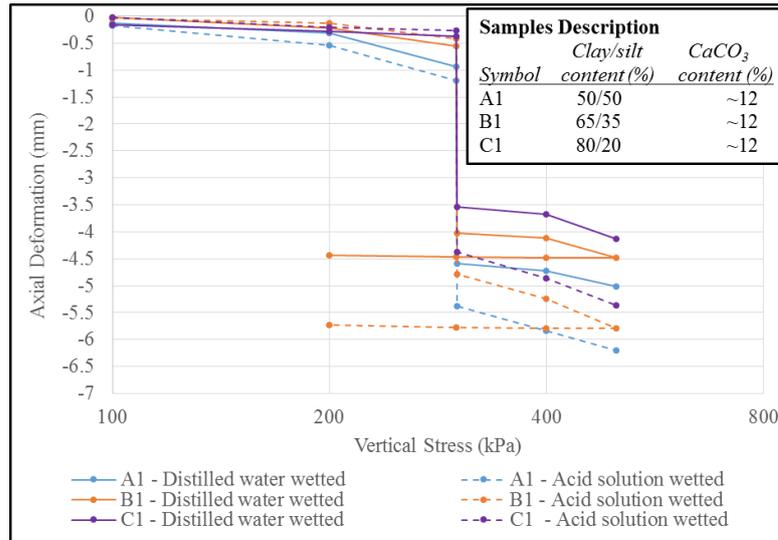


Figure 4.23 Single oedometer test results of different low dry-density cemented "moderately calcareous" samples

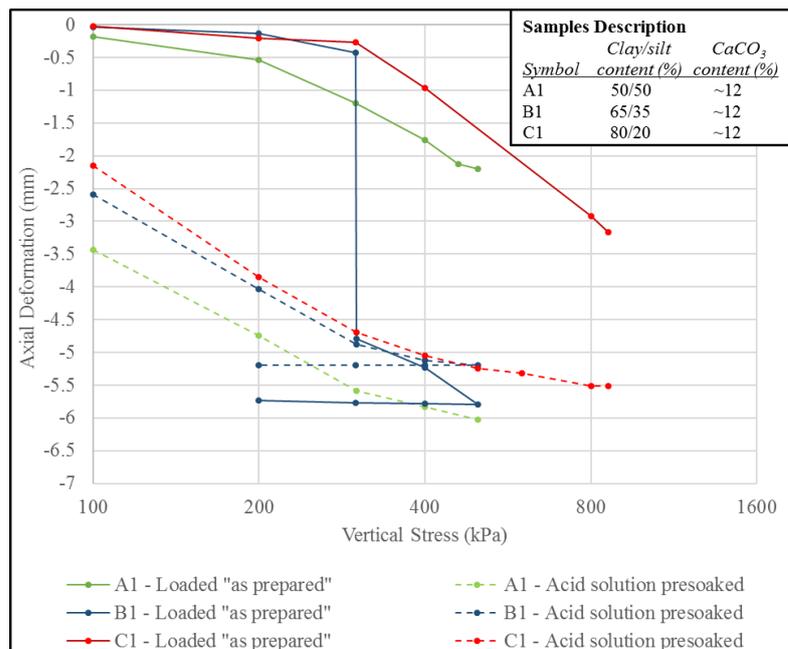


Figure 4.24 Double oedometer test results of different low dry-density cemented "moderately calcareous" samples

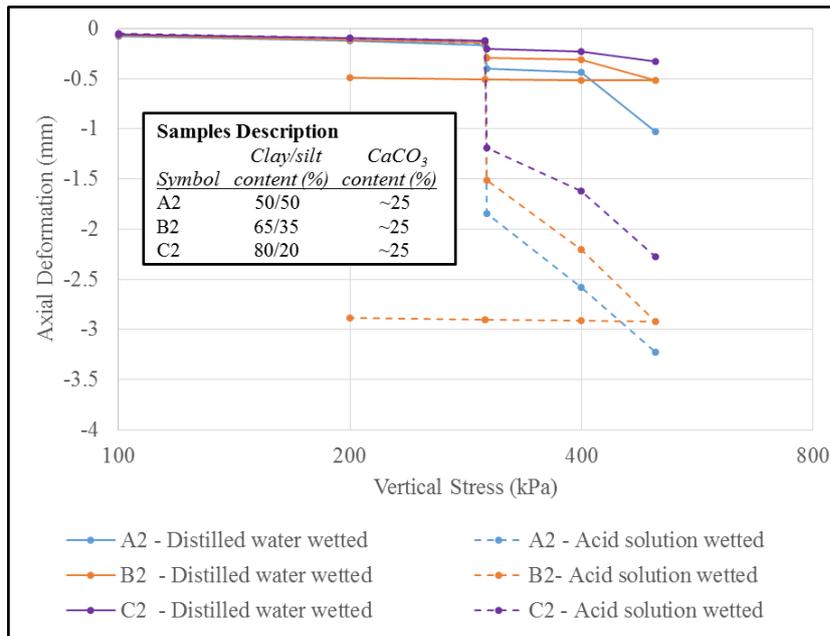


Figure 4.25 Single oedometer test results of different low dry-density cemented “strongly calcareous” samples

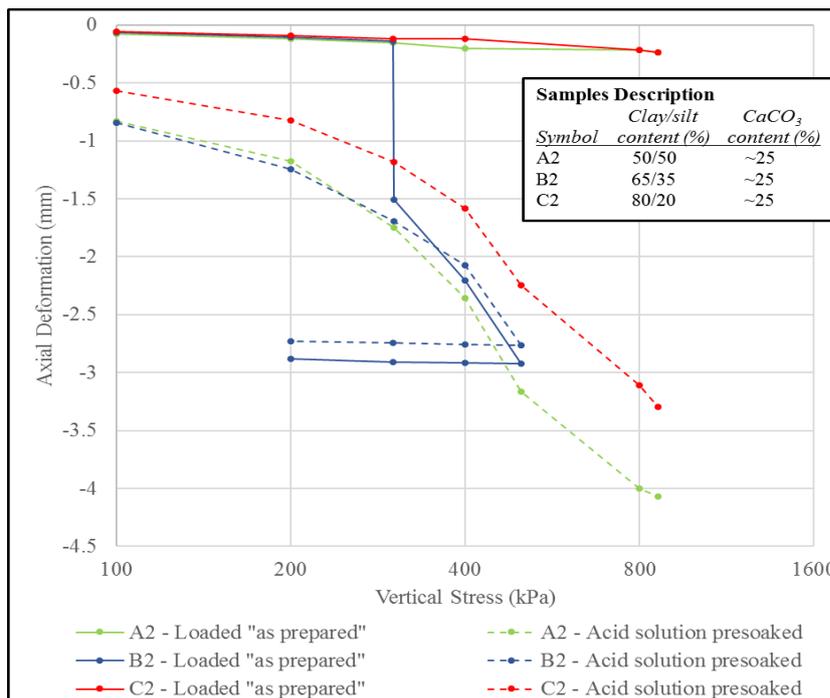


Figure 4.26 Double oedometer test results of different low dry-density cemented “strongly calcareous” samples

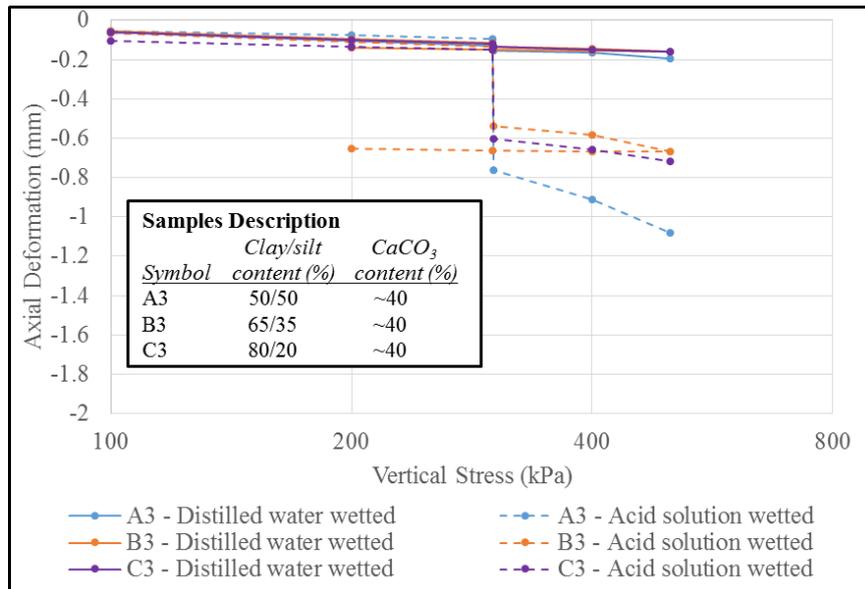


Figure 4.27 Single oedometer curves of different low dry-density cemented “very strongly calcareous” samples

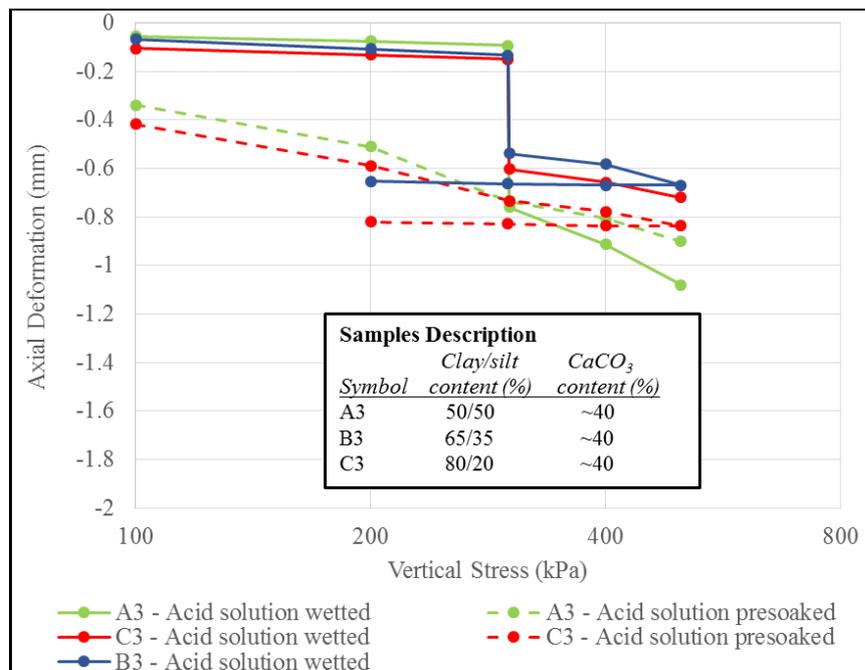


Figure 4.28 Double oedometer curves of different low dry-density cemented “very strongly calcareous” samples

As can be seen in Table 4.5, these specimens are highly porous and could only have exhibited structural stability through the CaCO<sub>3</sub> inter-particle cementation. This has been intended to enhance understanding of the impact of calcite content (or degree of calcareousness) and cement dissolution on both compressibility and the collapse phenomenon (sudden volume decrease due to wetting). The Figures above show relationships between different samples with similar calcite cementation levels. The impact of the degree of calcareousness is discussed later in section 4.5.1.

Generally, different samples with comparable degrees of calcareousness maintained similar physical properties, such as initial dry density, void ratio and porosity with very minimal differences following both calcite and clay contents. A small increase in dry density can be observed with increasing calcite content while initial void ratio and porosity have reduced with increasing clay and calcite contents. From a soil mechanics viewpoint, these parameters indicate the capacity for smaller compressions and CPs with increasing clay and calcite contents. This is evident in the Table 4.6.

Furthermore, considering collapse phenomenon as an aftermath of calcite-cement dissolution, it is reasonable to anticipate the degree of collapse following the amount of dissolution. That has been demonstrated by comparing the respective CP values obtained from distilled water wetting and acidic solution wetting. The results are as expected because acidic solutions ( $\text{pH} \leq 5$ ) readily dissolve  $\text{CaCO}_3$  in soils (Jackson 1956 in Al-Kaysi (1983)), increasing their pore volume and weakening inter-particle bonding and giving room for higher wetted deformations under loading. In the same vein, it is also expected to have lower CPs where distilled water applied as the wetting fluid since calcite remains insoluble or extremely weakly soluble in distilled water (Smalley (1971); Mansour *et al.* (2008); Li *et al.* (2016)).

Additionally, it is also logical to expect that higher  $\text{CaCO}_3$  contents could lead to higher pore volumes after dissolution occurred, with a consequent higher CP. Unfortunately, the present results have not corroborated this.

The pre-wetting compressions have also appeared to decrease with increasing carbonate content (calcareousness). That is, stiffness increased with  $\text{CaCO}_3$  content. For instance, pre-wetting compression (at 300 kPa) values approximating 1.2, 0.15 and 0.12 mm were recorded for the *moderately*, *strongly*, and *very strongly* calcareous samples, respectively but there was no identifiable trend relating to their clay/silt ratios. In some cases, higher clay contents yielded higher pre-wetting compressions, a situation in contrast with the non-calcareous samples. It is not clear if this is a consequence of other factors, which may include inter-particle bonding patterns. However, their CP values both for the distilled water wetted and acidic solution wetted tend to show similar influence of clay/silt ratios as in the non-calcareous samples. CPs (at 300kPa overburden stress) from single oedometers ranged between “*severe trouble to very severe trouble*” (16 to 26 %), “*no trouble to trouble*” (0.4 to 10 %), and “*no trouble to moderate trouble*” (0.09 to 3.6 %) for *moderately*, *strongly* and *very strongly* calcareous samples, respectively with higher values resulting from acidic solution wetting. Even though comparable classes of collapse severity were also estimated from the double oedometers at the same 300kPa overburden stress, discrepancies existed in terms of actual CP values. Double oedometer

revealed overestimated CP values in some cases and underestimated values in others. At lower overburden stresses CPs are obviously lower.

On the contrary, beyond the collapse region, amount of compression for both distilled water and acidic solution wetted conditions, trends appeared to align with clay/silt ratios. It is interesting to note that, irrespective of the degree of calcareousness, samples having lower clay/silt ratios exhibited higher settlements beyond the collapse region. A similar trend was displayed by the presoaked curves at all loading stages of the double oedometer testing with both presoaked curves and wetted-after-loading curves nearly converging from the collapsed stage onwards. This clearly demonstrates a lesser impact of wetting on the compression of more clayey soils compared to lesser clayey ones.

#### ***High dry density (well & poorly compacted) samples***

In geotechnical engineering, especially in road base and subbase constructions, soft soil subgrades are sometimes treated with lime. Whether quicklime or slaked lime, when lime-treated soils are exposed to carbon sources, e.g., atmospheric carbon dioxide, under favourable conditions they react and are transformed to calcareous/carbonate soils. Because of the growing concerns of the modifications to mechanical characteristics of lime-treated soils resulting from carbonation (e.g., development of cracks), it was considered worthwhile to investigate under the present theme of CaCO<sub>3</sub> impact on collapsibility, to understand to what extent of volume change upon wetting, would this lime conversion cause to both well-compacted and poorly compacted lime-treated subgrades.

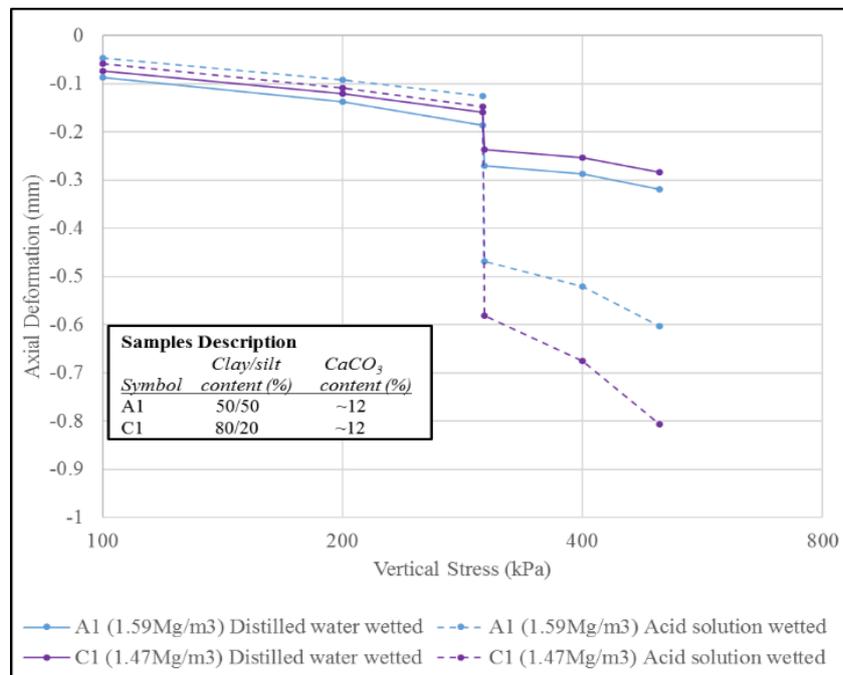
#### ***Samples well compacted (at OMC) and carbonated***

Samples compacted to their maximum dry densities (MDD) by means of the standard (lightweight) Proctor compaction method prior to carbonation have been adopted to represent well compacted lime-treated subgrades transformed to calcareous soils. These well-compacted samples have their physical properties presented in Table 4.7. While these parametric values are comparable to soils investigated by some authors, e.g., Fredlund and Gan (1995), dry density values fall above threshold recommended for collapsible soils according to Clevenger, 1958 (cited in Bell *et al.* (2003)). This notwithstanding, porosity and void ratio values qualify them as collapsible soils according to Fedas, 1966 classification (Okwedadi *et al.* (2014)). Moreover, certain conditions make nearly all compacted soils collapsible (Lawton *et al.*, 1992).

Generally, a well-compacted non-active clay soil existing at MDD can be said to be stable rather than metastable, but post compaction changes can alter as-compacted behaviours (Lawton *et al.*, 1992). For instance, carbonation of lime-treated soil is believed to compromise the structural integrity of such a system as lime volume expansion during carbonation of a dense lime-clay systems could likely result in the development of micro-cracks. On this basis Bagoniza *et al.* (1987) argued that carbonation reduces the strength characteristics of lime-treated subgrade. Again, the new mineral (calcite) formed can readily be dissolved when in contact with acidic solution, a process which could reduce bonding strength, density and increase void volume. Results obtained from the single and double oedometer testing of the well-compacted calcite-cemented samples are presented in Figure 4.29 to Figure 4.33 and summary of their CP shown in Table 4.8.

*Table 4.7 Physical properties of well-compacted, cemented calcareous oedometer test specimens*

<i>Specimen ID</i>	<i>Clay/Silt ratio</i>	<i>CaCO<sub>3</sub> content (%)</i>	<i>Specimen height (mm)</i>	<i>Dry density (Mg/m<sup>3</sup>)</i>	<i>Void ratio, e</i>	<i>Porosity, n (%)</i>
A1	1	~12	19	1.54	0.74	42.61
A2	1	~25	19	1.49	0.83	45.44
C1	4	~12	19	1.44	0.81	44.81
C2	4	~25	19	1.39	0.78	44.00
C3	4	~40	19	1.41	0.79	44.07



*Figure 4.29 Single oedometer curves of cemented “moderately calcareous” silty-clay samples compacted to their maximum dry densities*

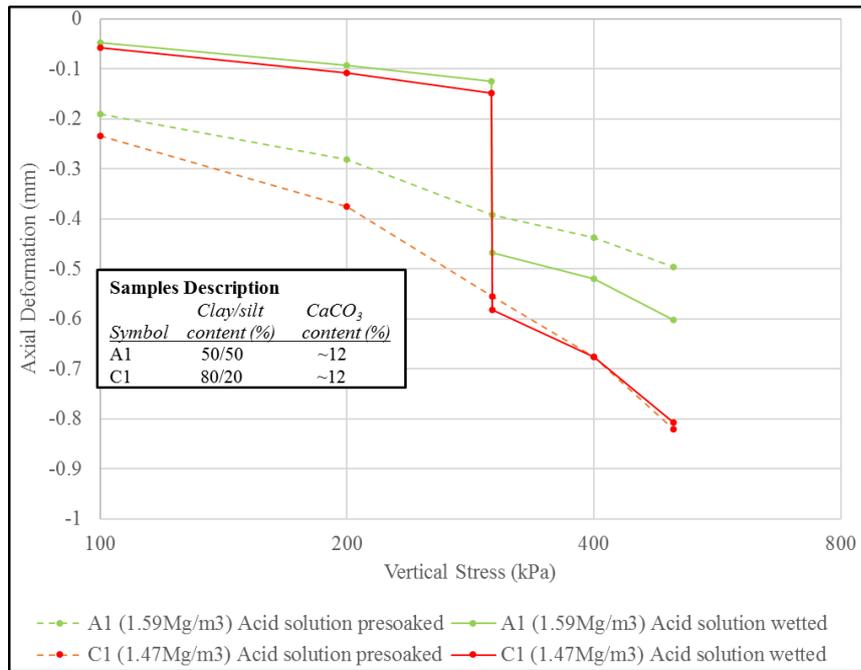


Figure 4.30 Double oedometer curves of cemented “moderately calcareous” silty-clay samples compacted to their maximum dry densities

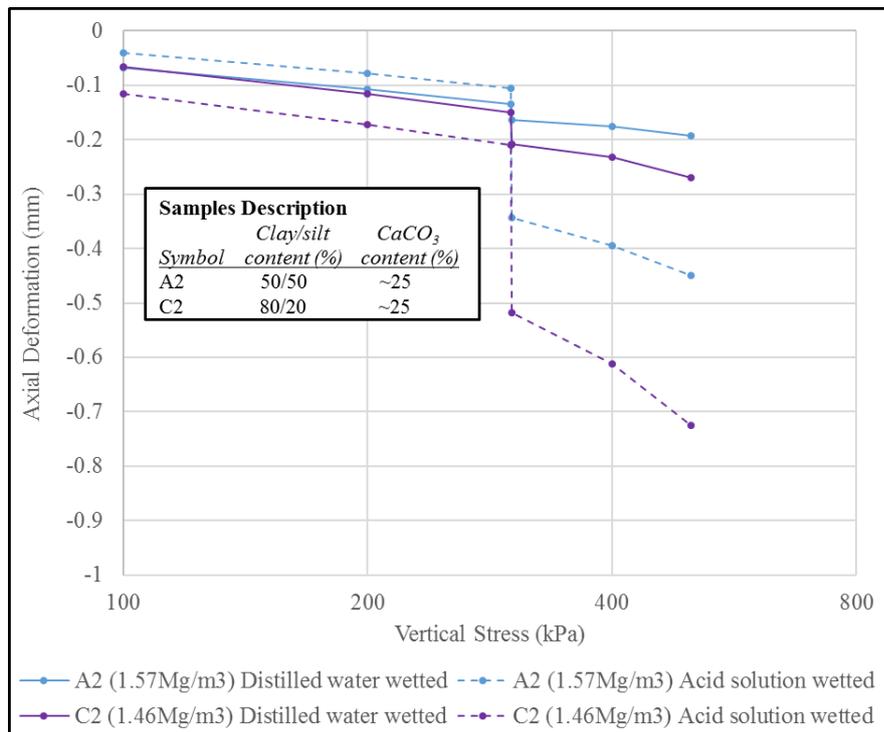


Figure 4.31 Single oedometer curves of cemented “strongly calcareous” silty-clay samples compacted to their maximum dry densities

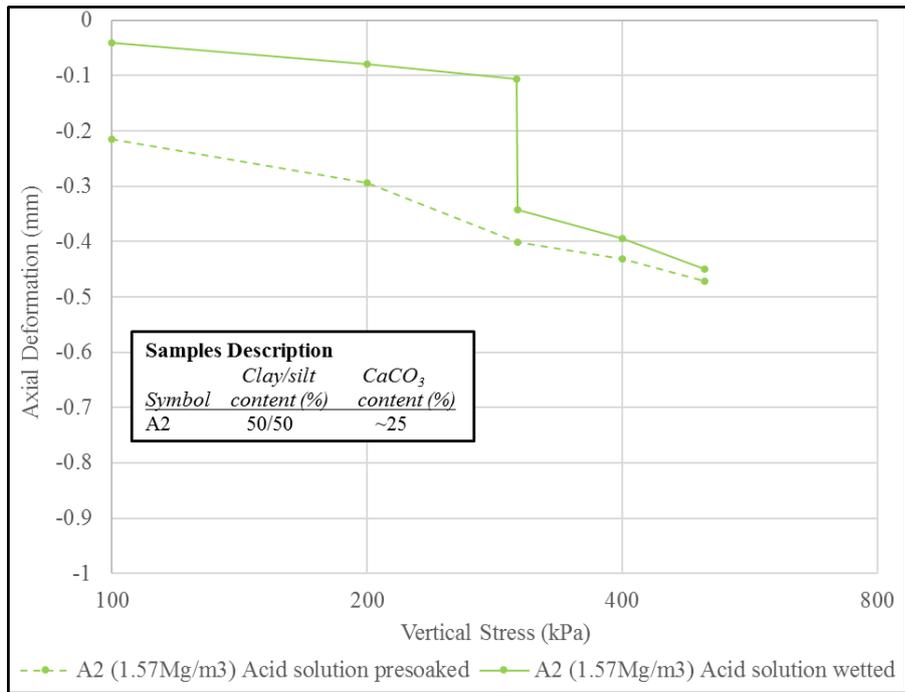


Figure 4.32 Double oedometer curves of cemented “strongly calcareous” silty-clay samples compacted to their maximum dry densities

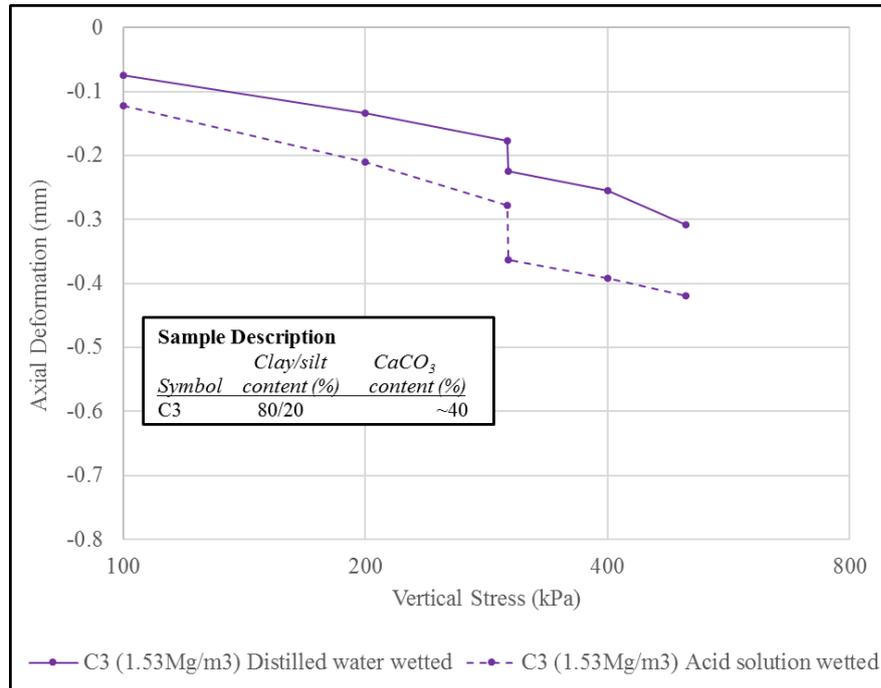


Figure 4.33 Single oedometer curves of cemented “very strongly calcareous” silty-clay sample compacted to their maximum dry densities

Table 4.8 Specimen compressibility during loading, collapse potential at different wetting stresses and degree of severity of well-compacted and cemented calcareous samples

Sample	Initial Specimen height (mm)	Distilled water wetted (single oedometer)				5% acetic acid solution wetted (single oedometer)			
		Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Distilled water wetted)	Collapse Potential (%) (Distilled water wetted)	Severity	Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Acid wetted)	Collapse Potential (%) (5% Acid wetted)	Severity
A1	19	18.8	18.7	0.5	NT	18.9	18.5	1.8	MT
A2	19	18.9	18.8	0.2	NT	18.9	18.7	1.3	MT
C1	19	18.8	18.8	0.4	NT	18.8	18.4	2.3	MT
C2	19	18.8	18.8	0.3	NT	18.8	18.5	1.7	MT
C3	19	18.8	18.8	1.2	MT	18.7	18.6	2.0	MT
<b>Collapse Potential (%) at different vertical stresses, from acid solution presoaked double oedometer</b>									
		<i>At 100kPa</i>	<i>severity</i>	<i>At 200kPa</i>	<i>severity</i>	<i>At 300kPa</i>	<i>severity</i>		
A1		0.8	NT	1.0	NT	1.4	MT		
A2		0.9	NT	1.1	MT	1.6	MT		
C1		0.9	NT	1.4	MT	2.2	MT		

As anticipated, these samples existing at their MDD with calcite cementation, have demonstrated insignificant pre-wetting compressions as shown by the single oedometers curves (Figure 4.29 to Figure 4.33 above). Though only five samples were investigated here, amounts of compression appeared to nominally increase with decreasing CaCO<sub>3</sub> and clay contents, with sample C3 not aligning to these trends. However, neither the amount of pre-wetting compressions nor composition seemed to influence the degree of wetting induced deformations. Unlike their counterparts with low dry densities, CP have not shown any clear trends from either wetting fluid. For instance, samples containing higher clay contents have shown greater CP in some cases but in other cases, those containing higher silt contents collapsed more. Conversely, samples with higher degree of calcareousness revealed higher CP values. Again, lower pre-wetting compressions did not translate into higher CPs as was the case with most of the low-density specimens.

From the foregoing, there appears to be an indication that for well-compacted lime-treated clay, carbonation properties could control the collapse behaviour rather than clay/silt proportions as was the case with the low-density specimens. Unfortunately, these carbonation properties as related to the structure of these well-compacted samples have not been adequately investigated here. Additionally, it is common knowledge that the denser a sample is the less

likely it is to compress, thus, sample C1 exhibited higher wetted compressions than sample A1 because of its lower density.

It is important also, to note here that all samples at their MDD, irrespective of their degree of calcareousness have not significantly collapsed. CP values falling within the “no trouble” and “moderate trouble” classes of severity for distilled water wetting and acidic solution wetting, respectively. From the CP values resulting from distilled water wetting (see Table 4.8), these samples would be classified as non-collapsible soil (Bell and Culshaw, 2001) but acidic solution wetting defines them as slightly collapsible at vertical stresses of 200 and 300 kPa. This implies that despite the stability of such soils in a neutral environment, their long-term stability can be of concern where the environment becomes acidic.

*Samples poorly compacted (at half OMC) and carbonated*

Compared to the samples discussed in the preceding subsection, these samples were compacted at approximately 50% of their optimum moisture contents (OMC) prior to carbonation. The samples represent poorly engineered lime-treated subgrades transformed to calcareous soils upon exposure to carbon dioxide. They are studied on the basis that all fills compacted at water contents far below/above OMC fall within the purview of collapsible soils according to Lawton *et al.* (1989); Lawton *et al.* (1992) Rogers (1995). Table 4.9 presents the physical properties of these samples. Their dry density, initial void ratio and porosity values are well within those recommended for classifying collapsible soils into large and small collapsibility according to Clevenger, 1958 and Feda, 1966 (cited in Bell *et al.* (2003) and Okwedadi *et al.* (2014)). They are also comparable to parametric values of collapsible samples investigated by authors such as Lawton *et al.* (1992) and Fredlund and Gan (1995). Deformation curves obtained from single and double oedometer tests are presented in Figure 4.34 to Figure 4.39 and CP summarized in Table 4.10.

*Table 4.9 Physical properties of poorly compacted (standard Proctor at half OMC), cemented calcareous oedometer test specimens*

<i>Specimen ID</i>	<i>Clay/Silt ratio</i>	<i>CaCO<sub>3</sub> content (%)</i>	<i>Specimen height (mm)</i>	<i>Dry density (Mg/m<sup>3</sup>)</i>	<i>Void ratio, e</i>	<i>Porosity, n (%)</i>
A1	1	~12	19	1.39	0.93	48.13
A2	1	~25	19	1.29	1.04	50.94
C1	4	~12	19	1.37	0.94	48.49
C2	4	~25	19	1.26	1.07	51.63
C3	4	~40	19	1.28	0.99	49.72

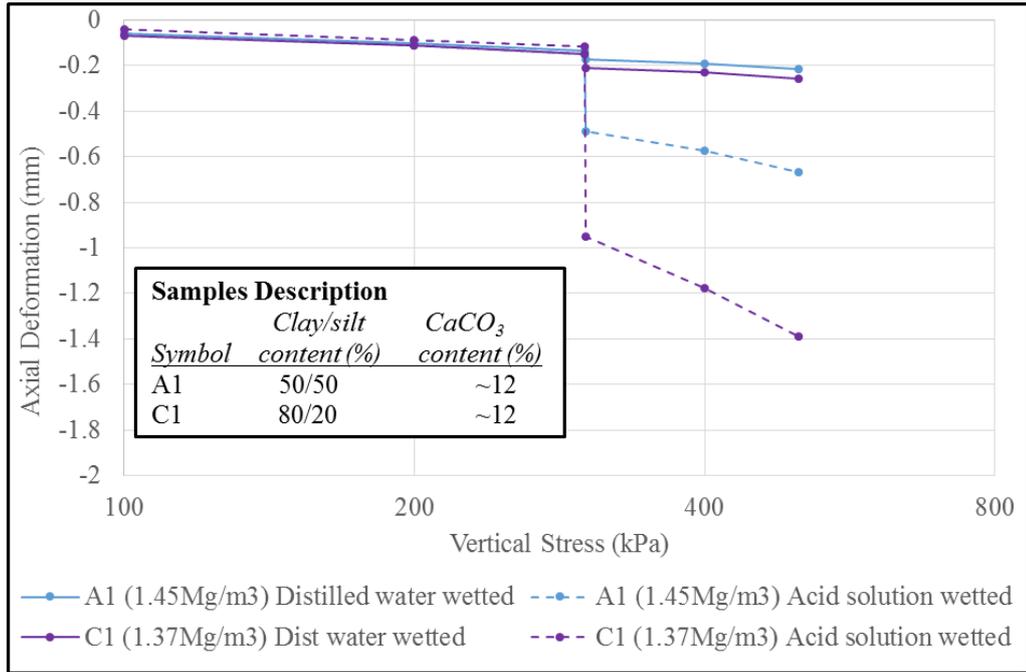


Figure 4.34 Single oedometer curves of cemented “moderately calcareous” silty-clay samples compacted at half their optimum moisture content

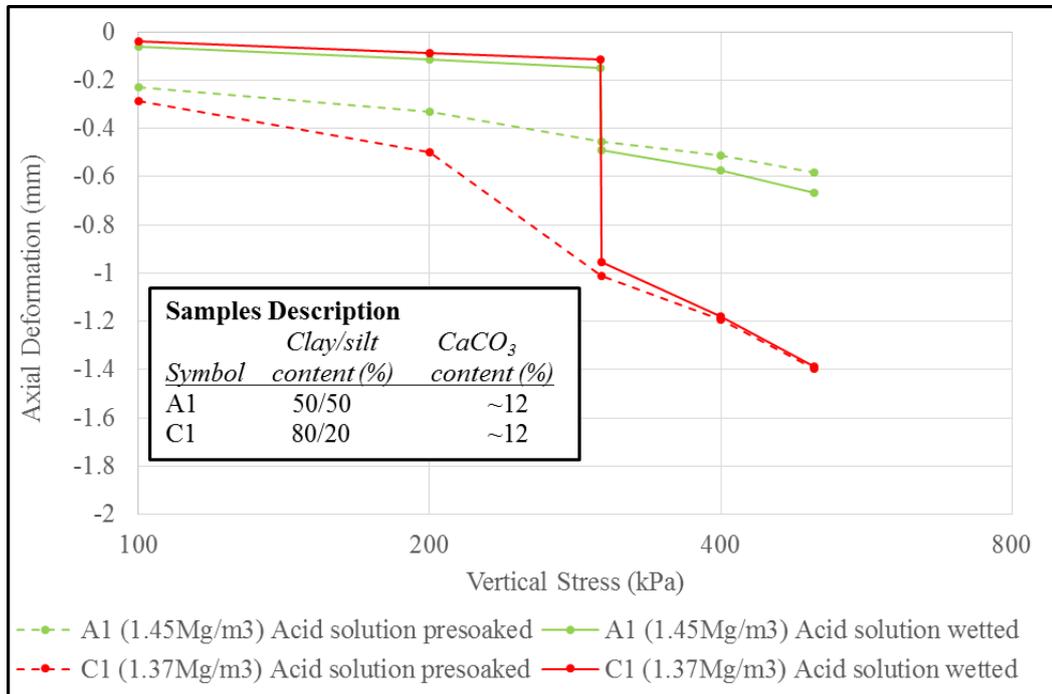


Figure 4.35 Double oedometer curves of cemented “moderately calcareous” silty-clay samples compacted at half their optimum moisture content

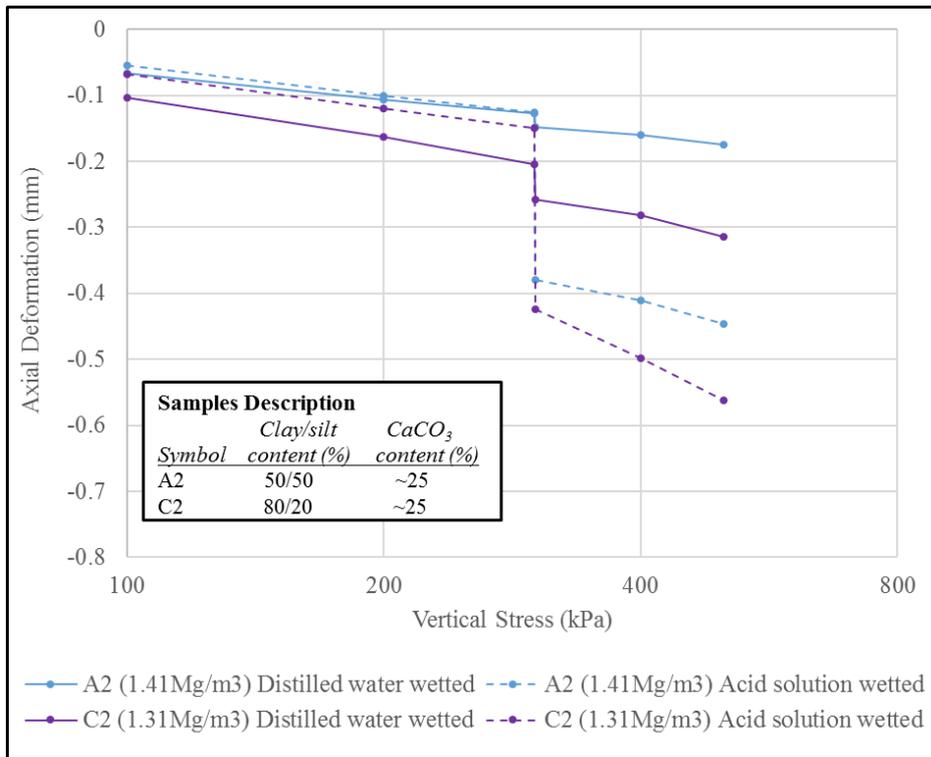


Figure 4.36 Single oedometer curves of cemented “strongly calcareous” silty-clay samples compacted at half their optimum moisture content

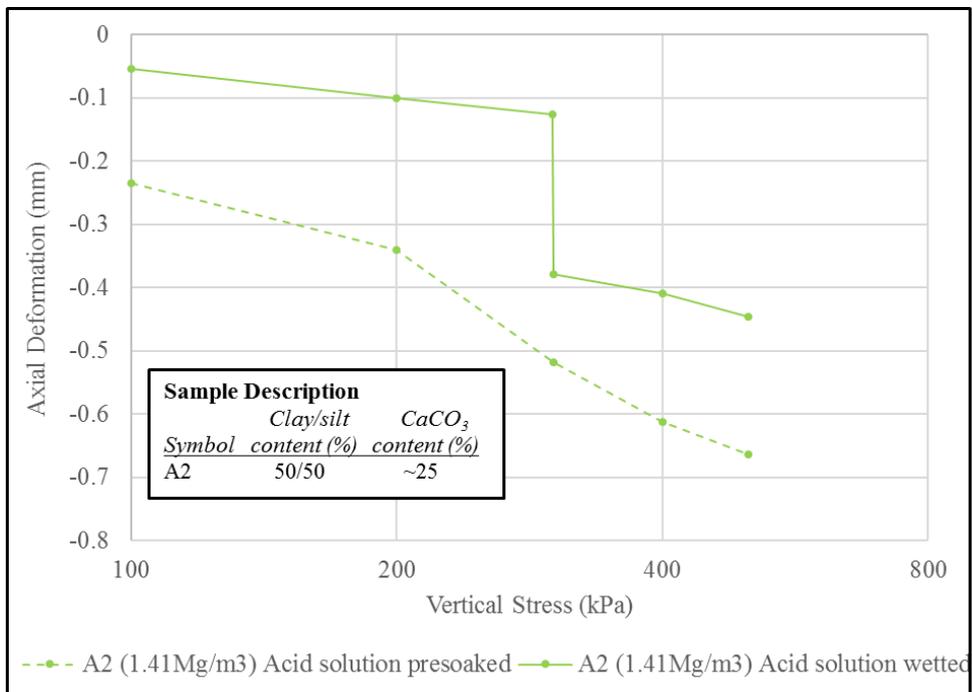


Figure 4.37 Double oedometer curves of cemented “strongly calcareous” silty-clay samples compacted at half their optimum moisture content

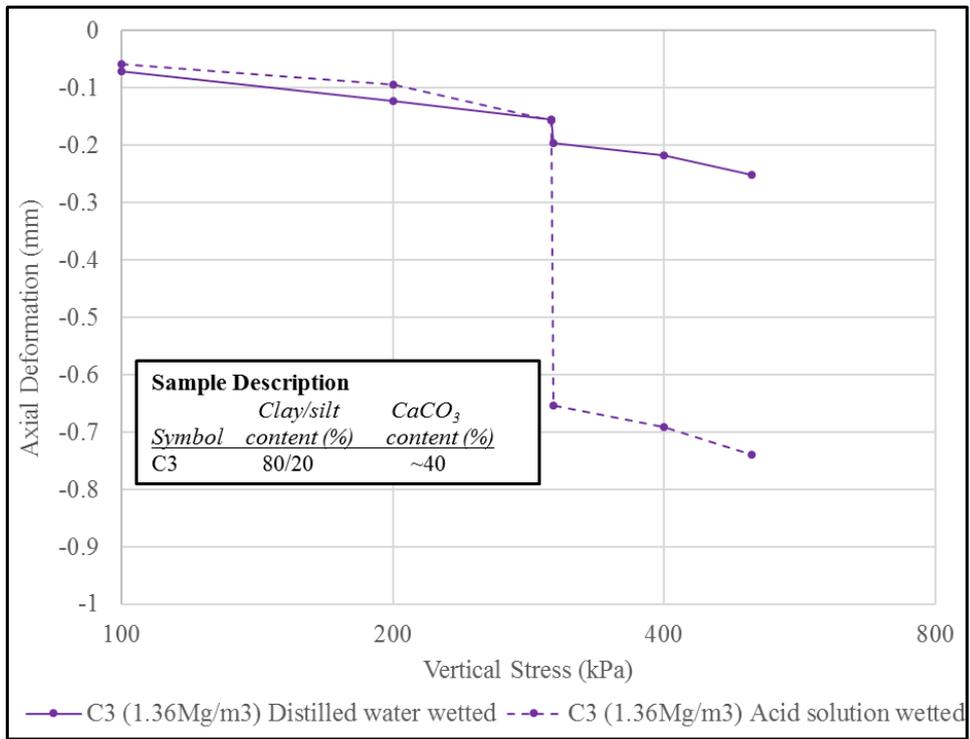


Figure 4.38 Single oedometer curves of cemented “very strongly calcareous” silty-clay samples compacted at half their optimum moisture content

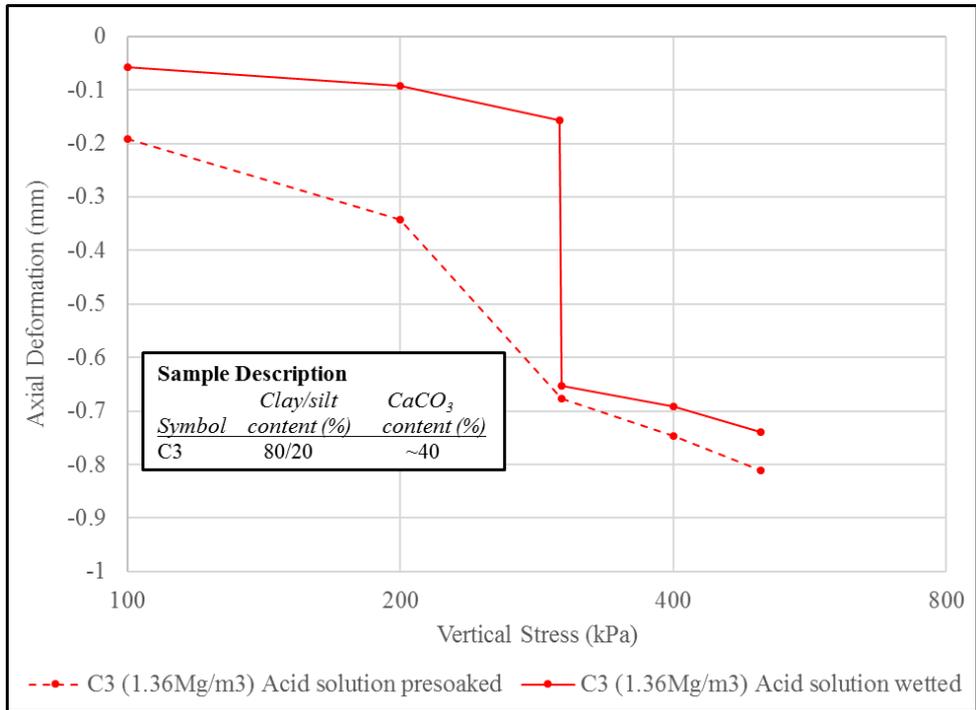


Figure 4.39 Double oedometer curves of cemented “very strongly calcareous” silty-clay samples compacted at half their optimum moisture content

Table 4.10 Specimen compressibility during loading, collapse potential at different wetting stresses and degree of severity of poorly compacted cemented calcareous samples

Sample	Initial Specimen height (mm)	Distilled water wetted (single oedometer)				5% acetic acid solution wetted (single oedometer)			
		Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Distilled water wetted)	Collapse Potential (%) (Distilled water wetted)	Severity	Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Acid wetted)	Collapse Potential (%) (5% Acid wetted)	Severity
A1	19	18.9	18.8	0.2	NT	18.8	18.5	1.8	MT
A2	19	18.9	18.8	0.1	NT	18.9	18.6	1.4	MT
C1	19	18.8	18.8	0.3	NT	18.9	18.0	4.5	MT
C2	19	18.8	18.7	0.3	NT	18.8	18.6	1.5	MT
C3	19	18.8	18.8	1.1	MT	18.8	18.4	3.5	MT
<b>Collapse Potential (%) at different vertical stresses, from acid solution presoaked double oedometer</b>									
		At 100kPa	severity	At 200kPa	severity	At 300kPa	severity		
A1		0.89	NT	1.2	MT	1.6	MT		
A2		0.5	NT	1.3	MT	2.1	MT		
C1		1.3	MT	2.2	MT	4.7	MT		
C3		0.7	NT	1.2	MT	2.8	MT		

Notwithstanding being compacted at half OMC, the curves in Figure 4.34 to Figure 4.39 also show only little pre-wetting compressions akin to their well-compacted counterparts, which indicates high load bearing capacity in their dry state. Pre-wetting compressions of between 0.12 and 0.21 mm were seen, with no definite patterns relating to either clay/silt ratio or degree of calcareousness (calcite content). Upon wetting as in the single oedometer, irrespective of the degree of calcareousness, low CP values were also recorded in all samples, with “no trouble” (0.11 – 1.05 %) and “moderate trouble” (1.35 – 4.47 %) classes of severity for distilled water wetting and acidic solution wetting, respectively.

In both wetting fluid types in the single oedometer, CP increase with decreasing clay and calcite contents. This was also corroborated by the CPs obtained from the acidic solution presoaked (double oedometer test) at all three stages of loading. Presoaked curves particularly in Figure 4.35, further demonstrate this claim. This agrees with the well-compacted samples reported earlier in this section but in contradiction with the results of low dry density calcareous samples reported in section 4.4.2.

Interestingly, CP values of the poorly compacted specimens fall into the same class of collapse severity as their well-compacted counterparts, proving that at both states of compaction

CaCO<sub>3</sub> cementation could support the silty-clay structure against wetting (especially, distilled water) induced sudden volume reduction.

***Collapse potential at different overburden stresses***

A few cemented *moderately* calcareous and *strongly* calcareous samples with physical properties identical to those presented in Table 4.5 (studied under Section 4.4.2) have been tested using both the single and double oedometer procedures with distilled water wetting and acidic solution wetting to investigate the effect of overburden stress at wetting on CP. Summaries of CP at various wetting stresses are presented in Table 4.11 (single oedometer) and Table 4.12 (double oedometer). Figure 4.40 and Figure 4.41 show compression (deformation) curves of two different *moderately* calcareous samples, acidic solution wetted at various overburden stresses. Figure 4.42 to Figure 4.45 show compression curves of two different *strongly* calcareous samples wetted with both distilled water and acidic solution. In all Figures colour distinguishes wetting stresses while broken lines and full lines denote acidic solution wetted and distilled water wetted, respectively.

*Table 4.11 Collapse potential at different vertical stress levels under single oedometer*

<i>Wetting (vertical ) stress (kPa)</i>	<i>Collapse Potential (%)</i>					
	<i>A1</i>	<i>C1</i>	<i>A2</i>		<i>C2</i>	
	<i>Acid solution wetted</i>	<i>Acid solution wetted</i>	<i>Distilled water wetted</i>	<i>Acid solution wetted</i>	<i>Distilled water wetted</i>	<i>Acid solution wetted</i>
200	20.3	18.0	-	5.9	-	4.7
300	25.2	21.9	1.3	9.0	0.4	5.7
460	18.7	-	5.2	12.9	3.9	10.1
860	-	13.9	14.6	19.0	11.5	17.0

Table 4.12 collapse potentials at different vertical stresses under double oedometer

Sample	CP (%) at various vertical stresses – under acid solution presoaked (double oedometer)						
	<i>At</i> 100kPa	<i>At</i> 200kPa	<i>At</i> 300kPa	<i>At</i> 400kPa	<i>At</i> 460kPa	<i>At</i> 800kPa	<i>At</i> 860kPa
A1	17.3	22.8	24.6	23.6	22.1		
A2	4.0	5.6	8.5	12.0	14.1	20.1	20.4
C1	11.2	19.4	23.6	22.6			
C2	2.7	3.9	5.6	7.2	10.3	15.4	16.3

Sample	CP (%) at various vertical stresses – under distilled water presoaked (double oedometer)						
	<i>At</i> 100kPa	<i>At</i> 200kPa	<i>At</i> 300kPa	<i>At</i> 400kPa	<i>At</i> 460kPa	<i>At</i> 800kPa	<i>At</i> 860kPa
A2	0.9	1.6		5.2		14.9	15.3
C2	0.4	0.5		1.5		9.2	9.9

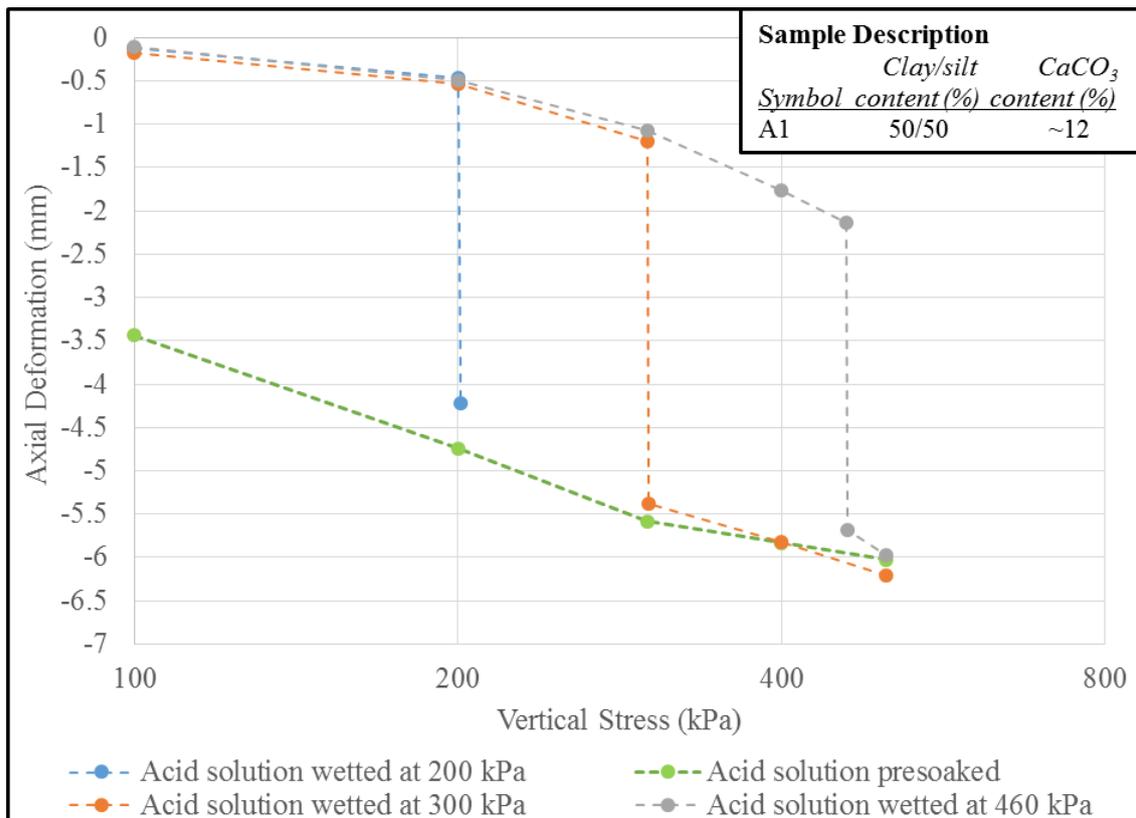


Figure 4.40 Collapsibility of cemented sample A1 at different wetting stresses under acid solution wetted single & double oedometer

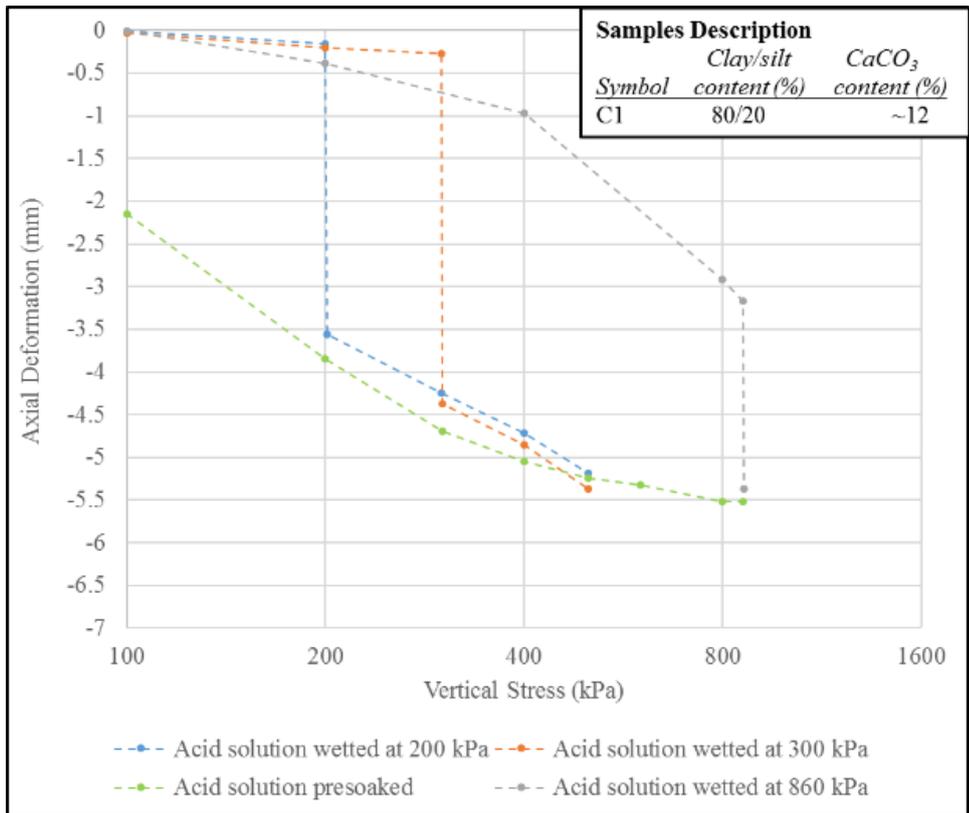


Figure 4.41 Collapsibility of cemented sample C1 at different wetting stresses under acid solution wetted single & double oedometer

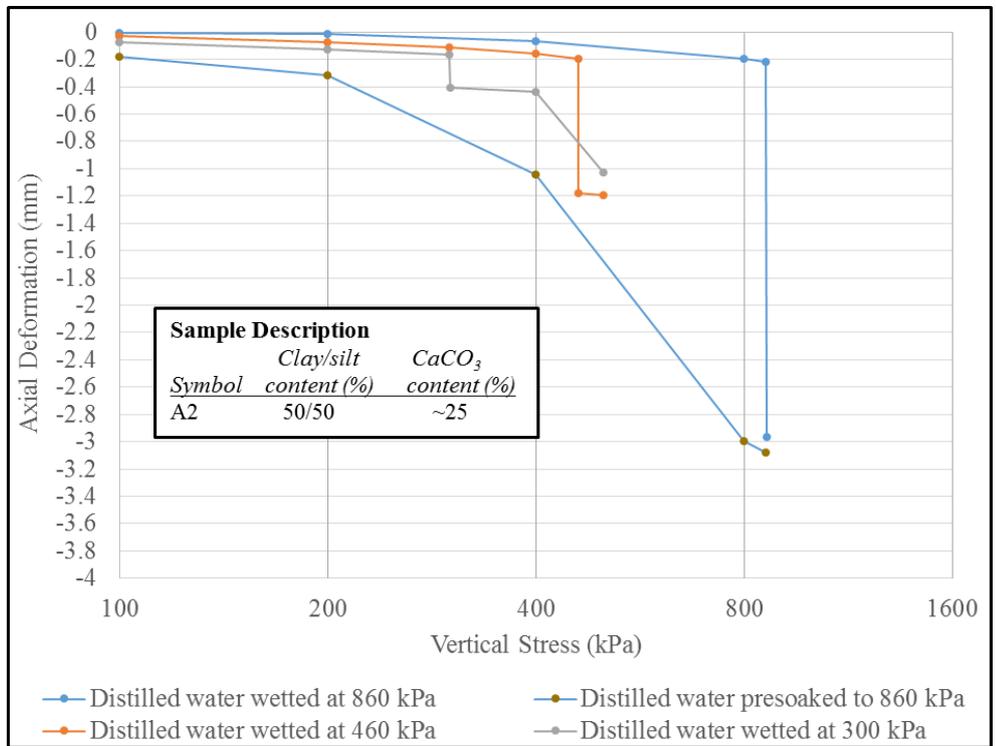


Figure 4.42 Collapsibility of cemented sample A2 at different wetting stresses under distilled water wetted single & double oedometer

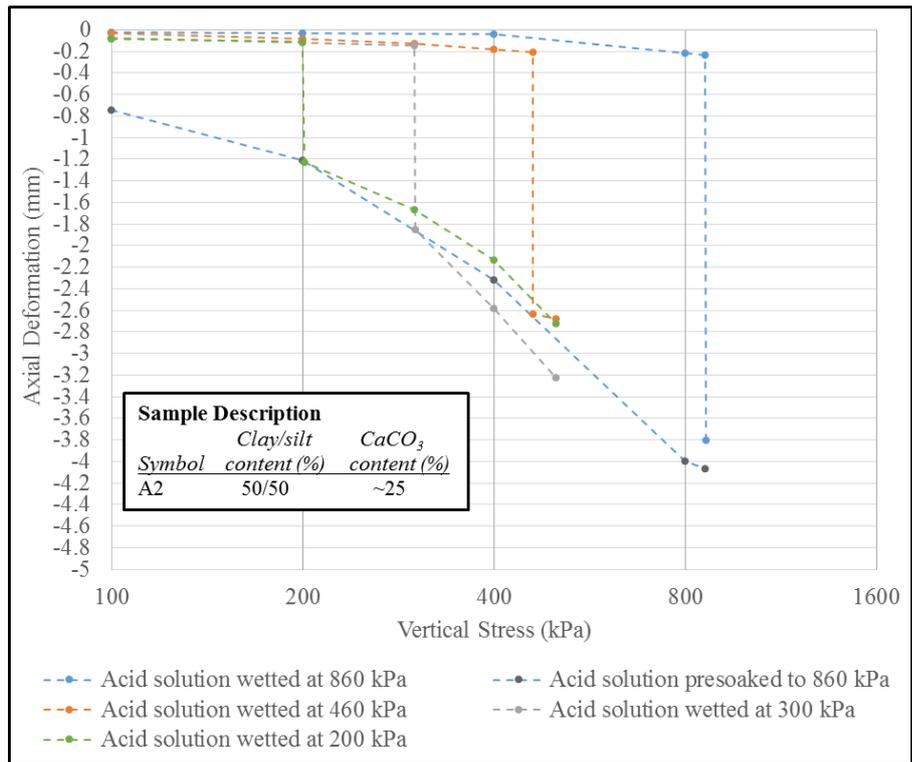


Figure 4.43 Collapsibility of cemented sample A2 at different wetting stresses under acid solution wetted single & double oedometer

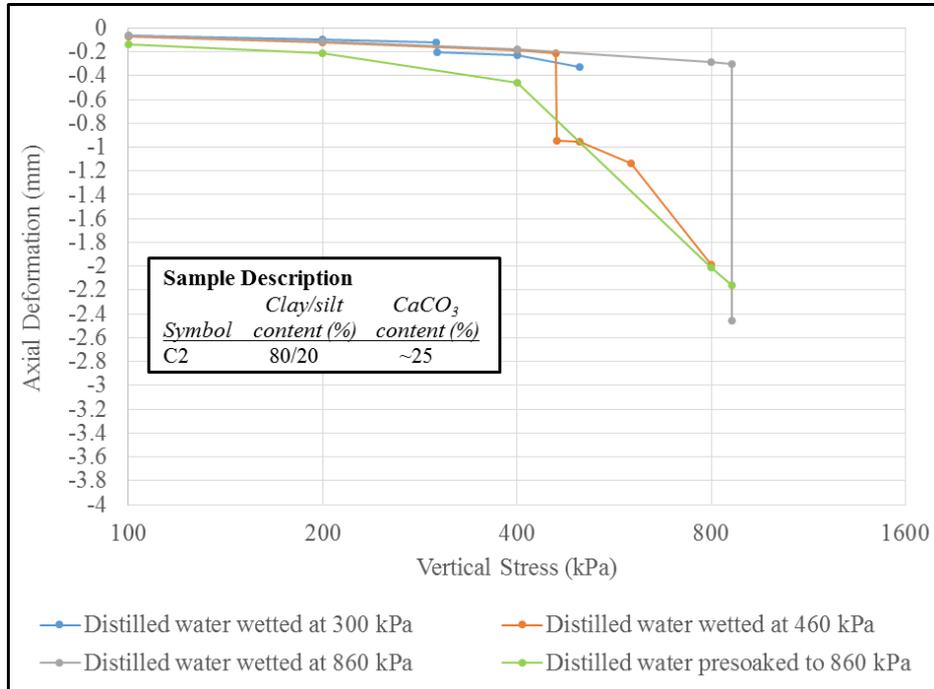


Figure 4.44 Collapsibility of cemented sample C2 at different wetting stresses under distilled water wetted single & double oedometer

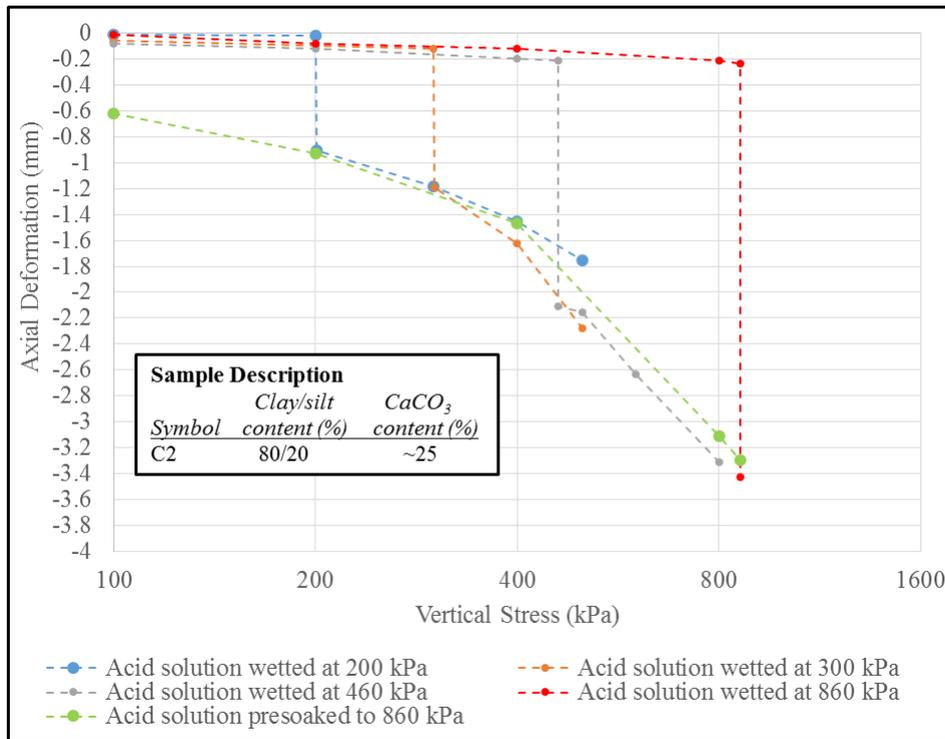


Figure 4.45 Collapsibility of cemented sample C2 at different wetting stresses under acid solution wetted single & double oedometer

Results presented in both Table 4.11 and Table 4.12 reveal that at all wetting stresses acidic solution wetting triggers greater collapses. In addition, despite the wetting fluid type CPs decrease with both increasing clay/silt ratio and calcite content. This influence by texture and calcite was observed earlier in section 4.4.2. Introduced in the present section is the distilled water presoaked (double oedometer procedure) test, in which wetted compression trends are similar to those obtained from the acidic solution presoaked. This somewhat validates the double oedometer procedure for collapse testing, in that results of a single oedometer and a double oedometer may remain comparable as far as both are wetted with similar fluid type.

Interesting to note from the CP values in Table 4.11 is the trend resulting from changing the overburden stress at wetting. CPs of double oedometer tests reported earlier and those in Table 4.12 above illustrate similar changes with overburden stress. These changes in CP upon change in overburden stress appear to be based on the degree of calcareousness (or cementation). However, different soils with similar degrees of calcareousness have not yielded identical magnitude of collapse. It is obvious here, that CP can be a function of stress history, degree of calcareousness and the overburden stress at wetting.

#### 4.4.3 Collapsibility of uncemented calcareous silty-clay samples

A handful of uncemented calcareous oedometric specimens with calcite contents and physical properties similar to their cemented counterparts reported in Table 4.5 have been similarly, tested under the single and double oedometer procedures. These specimens were prepared from silty-clay samples carbonated in loose condition, where the occurrence of calcite was assumed to be in two forms: (1) as individual grains within the soil structure, and (2) as particle-coating without inter-particle connection. It was aimed to provide understanding on the influence of mode of calcite occurrence on the collapsibility of calcareous soils. Distilled water and acidic solution wetting at an overburden stress of 300kPa were used in the single oedometer while only acidic solution was adopted in the presoaked (double oedometer procedure). CPs obtained are presented in Table 4.13 and deformation curves shown as Figure 4.46 to Figure 4.50.

Table 4.13 Specimen compressibility during loading, collapse potential at 300 kPa wetting stress and degree of severity of poorly compacted cemented calcareous samples

Sample	Initial Specimen height (mm)	Distilled water wetted (single oedometer)				5% acetic acid solution wetted (single oedometer)			
		Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Distilled water wetted)	Collapse Potential (%) (Distilled water wetted)	Severity	Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Acid wetted)	Collapse Potential (%) (5% Acid wetted)	Severity
A2	19	18.8	18.6	1.5	MT	18.8	17.2	10.4	ST
C2	19	18.9	18.7	1.1	MT	18.9	17.6	6.8	T
C3	19	18.8	18.7	0.5	NT	18.8	18.1	3.9	MT

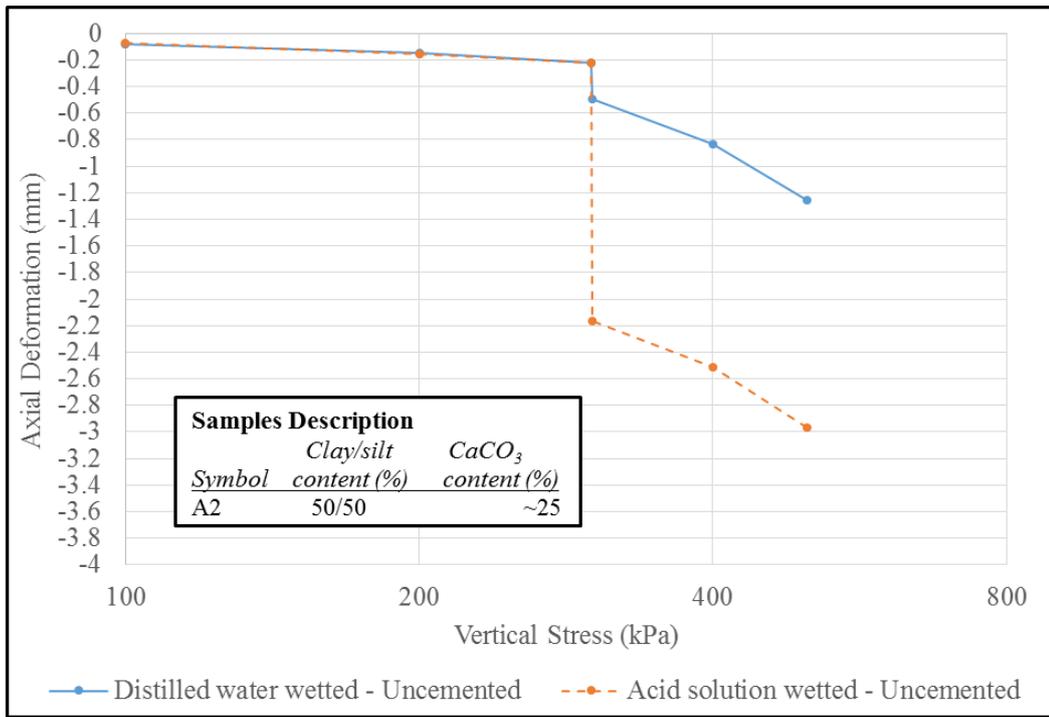


Figure 4.46 Single oedometer collapsibility curves of uncemented sample A2 with different wetting fluids.

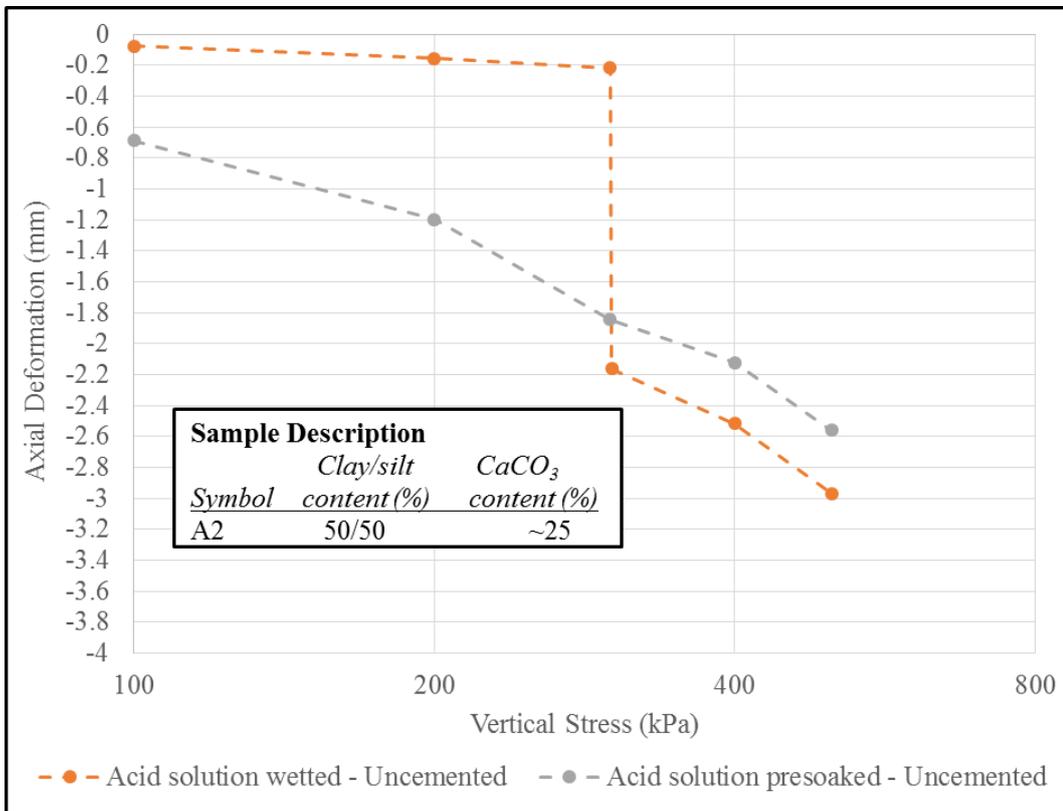


Figure 4.47 Double oedometer collapsibility curves of uncemented sample A2 with acid solution presoaked.

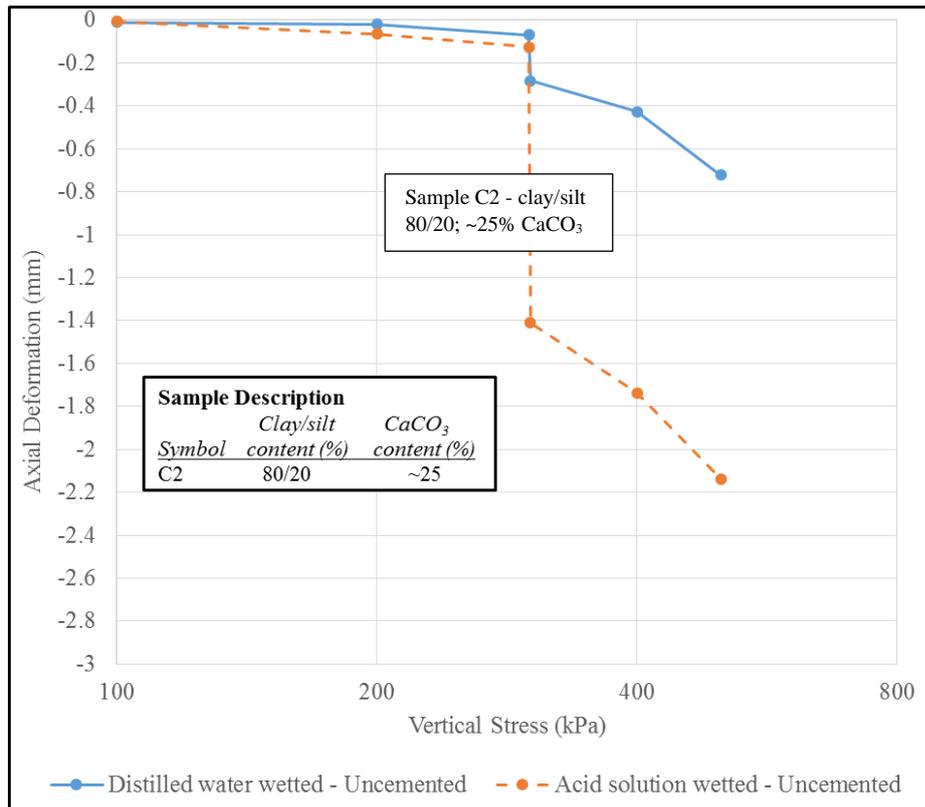


Figure 4.48 Single oedometer collapsibility curves of uncemented sample C2 with different wetting fluids.

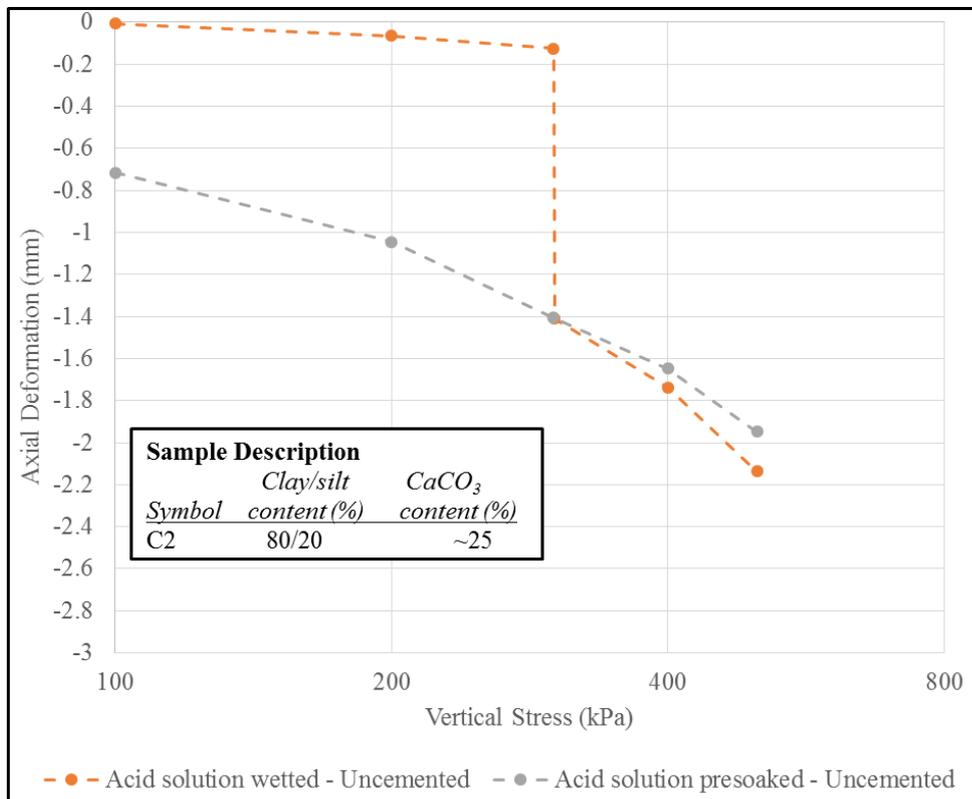


Figure 4.49 Double oedometer collapsibility curves of uncemented sample C2 with acid solution presoaked.

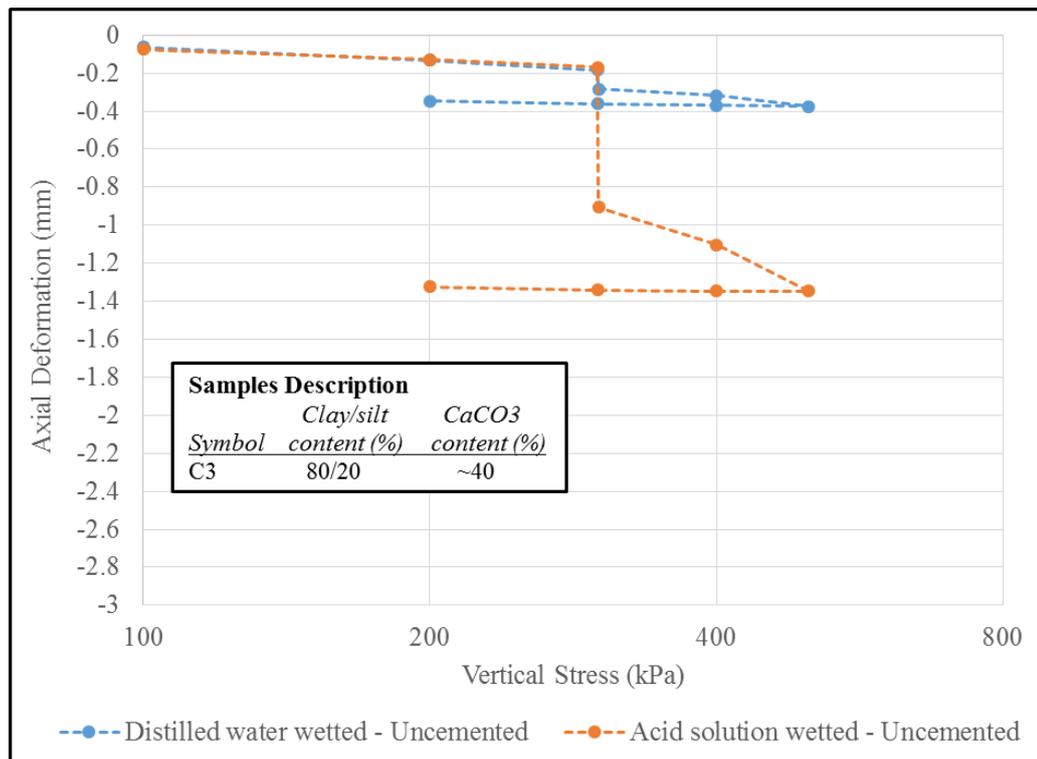


Figure 4.50 Single oedometer collapsibility curves of uncemented sample C3 with different wetting fluids.

While the volume of experimental data obtained as presented in the Table 4.13 might not be solely sufficient to draw concrete conclusions, there are fair indications that wetting with acidic solution also yields higher CPs than wetting with distilled water as expected. It also appears that clay/silt ratio and calcite content influenced the magnitude of collapse, with lower calcite contents and clay/silt ratios tending to show higher CP values. These observations, including trends of the presoaked (double oedometer) curves are fairly consistent with those of their cemented counterparts reported under section 4.4.2.

## 4.5 Discussion on Parameters Affecting Collapse

### 4.5.1 Effect of CaCO<sub>3</sub> content on collapse potential

There is a large body of knowledge available on the role of calcium carbonate on the collapse of collapsible soils. This knowledge is quite controversial due mainly to a poor understanding or not considering calcium carbonate to exist in different polymorphs (e.g., calcite, aragonite, vaterite) that exhibit varying chemical behaviours. For instance, Osipov and Sokolov (1995) referred to calcium carbonate and calcite as different salts of medium and low solubility, respectively. They estimated several hours to several months for dissolution of the calcite bonds. A position supported by Mansour et al (2008). Although several collapsible soils have been

reported to contain calcium carbonate contents ranging from 1 to 40 % (Penck (1931) and Grabowska-Olszewska (1988) cited in Langroudi (2014); Jefferson *et al.* (2005) Li *et al.* (2016)), and Northmore *et al.* (2008) claims that the calcareous layers of the British loess deposits in South East England were more collapsible than the non-calcareous layers. While Mellors (1995) was earlier sceptical about the effect of CaCO<sub>3</sub> bonds on the collapse of these British loess, Milodowski *et al.* (2015) supported Northmore *et al.*'s claim. Nevertheless, the literature fails to quantify the impact of CaCO<sub>3</sub> on the collapse potential of collapsible soils.

The crystal shape and structure of CaCO<sub>3</sub> in soils differ following the process of formation. These differences influence the wetted behaviour of such soils (Langroudi, 2014). Six basic structures in which CaCO<sub>3</sub> and other carbonates appear in soils are reported in section 2.3.3. In the present samples, SEM images (Figure 3.19 on page 132) roughly reveal calcite occurring as inter-particle meniscus cement supporting silt grains and clay clods in some samples. In others with larger amounts of clay content, calcite occur within clay matrix that overwhelmed the few silt grains, thus, reinforcing the clay structure. However, these microstructures were not clearly studied due to poor SEM images and was difficult to relate them to the behaviour of samples.

While it is debatable, increased CaCO<sub>3</sub> content relates to increased bonding as UCS results demonstrated in Figure 4.9 above. Figure 4.9 shows an increase in UCS with an increase in calcite content (degree of calcareousness) for all tested specimens. Expectedly, higher bonding and strength should lead to greater stability, thus, a reduced collapsibility under similar stress conditions. Milodowski *et al.* (2015) identified calcite cement occurring as reinforcement in meniscus clay bridges to reduce both the dispersion of the clay and collapse upon wetting. However, the relationship between strength and collapse potential (CP) identified in the present study appear to depend more on two variables. As illustrated in Figure 4.51 to Figure 4.53, CaCO<sub>3</sub> content and its degree of dissolution are critical in controlling the magnitude of collapse. Wetting with distilled water and a 5% acidic solution reveals that fluid pH impacts CP.

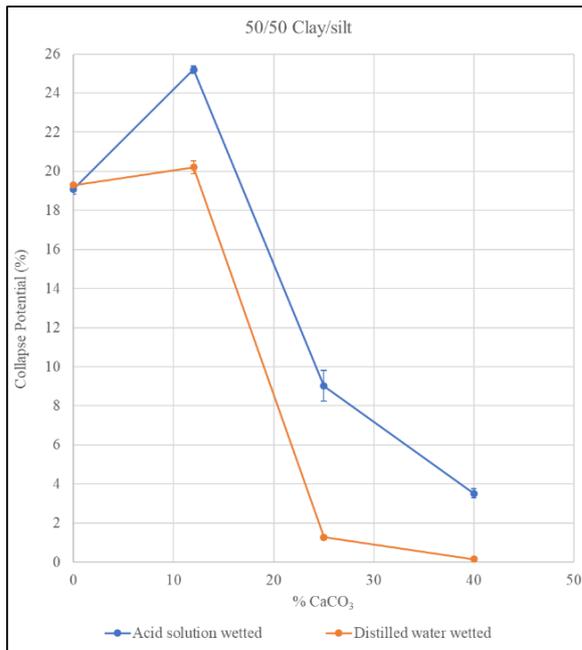


Figure 4.51 Relationship between calcite content and collapse potential of series A samples (low-density) under different wetting fluids

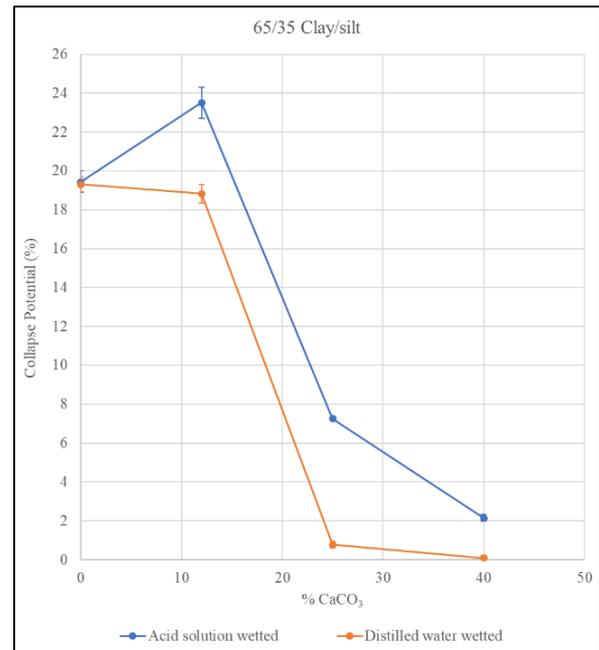


Figure 4.52 Relationship between calcite content and collapse potential of series B samples (low-density) under different wetting fluids

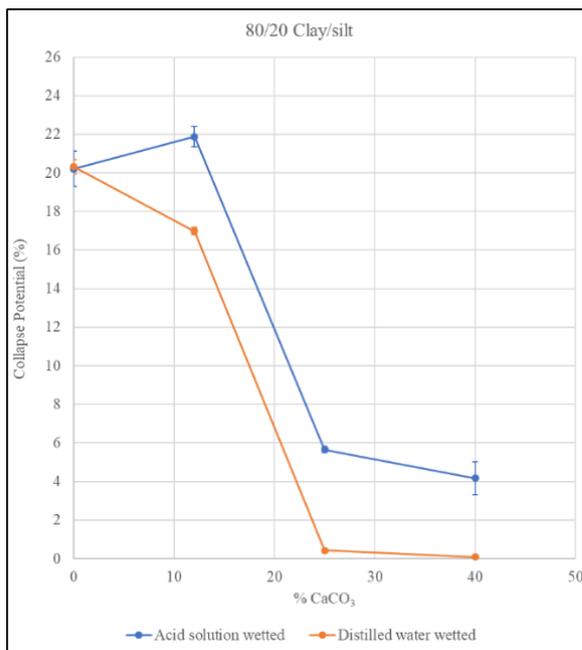


Figure 4.53 Relationship between calcite content and collapse potential of series C samples (low-density) under different wetting fluids

Regardless of higher UCS and stiffness exhibited by the *moderately* calcareous (~12% CaCO<sub>3</sub> content) samples compared to the non-calcareous (0% CaCO<sub>3</sub>) ones, they still yielded greater collapses in all acid solution wetted conditions and in one case of the distilled water wetted condition. While this occurrence could be explained by the different hypothesized collapse mechanisms of the samples, it is worth noting that at certain construction loads a more

stable ground could rather present greater trouble if in the long-term wetting fluids cause chemical dissolution of components of such a ground.

Firstly, the non-calcareous samples had undergone higher pre-wetting compressions due to loading because of their uncemented nature, reducing their capacity for further densification upon wetting. Secondly, while non-calcareous specimens seemed to undergo only a mechanical phase (particles packing) upon wetting, the *moderately* calcareous specimens were additionally subjected to a chemical bond dissolution phase, especially in the acidic solution wetting condition. In soil collapse, disaggregated clay bonds/bridges usually migrate into pore spaces during repacking (Klukanova and Frankovska (1995); Mellors (1995)). While this causes total soil volume reduction, it will not decrease the volume of solids. In the case of  $\text{CaCO}_3$  bonds, dissolution can result also in solid lose, thus, a greater total soil volume reduction. This could mean that in a typical field condition, soils containing higher  $\text{CaCO}_3$  content relative to others, would yield higher voids upon  $\text{CaCO}_3$  dissolution, thus exposing such soils to greater collapses.

Nevertheless, the oedometer experimental data here suggested otherwise. Increasing the  $\text{CaCO}_3$  content of the samples from ~12% to ~25% and to ~40% have rather significantly reduced the propensity of dissolution (even in acidic solution) and consequent collapse. For instance, increasing  $\text{CaCO}_3$  content from ~12% to ~25% reduced collapsibility by about 64 – 75 % and further increase of  $\text{CaCO}_3$  content to ~40%, further reduced collapse potential by 86 – 91 %. Extrapolations in Figure 4.54 suggest that for all samples tested, despite their very low dry density ( $1 \pm 0.1 \text{ Mg/m}^3$ ),  $\text{CaCO}_3$  contents reaching about 44% could reduce CP to about 0 %.

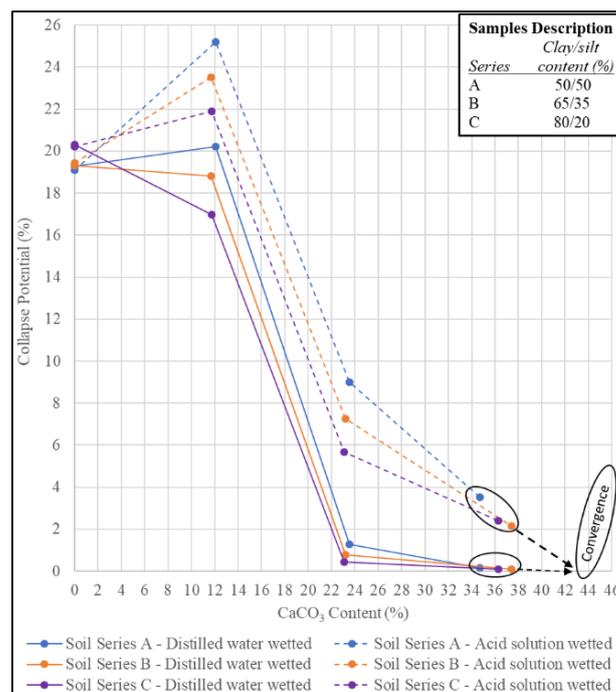


Figure 4.54 Extrapolating the impact of calcite content on the collapse potential of all low-density samples (series A, B & C)

The high degrees of calcareousness have rather strengthened (or stabilized) the samples, which though maintained a metastable structure, are not supportive of the sudden densification upon wetting phenomenon. That is, while those specimens still possess an open structure their inter-particle (and perhaps, inter-clods) bonding became too strong to be easily eliminated, particularly by distilled water, thus, yielding nearly negligible CP values. This is consistent with the collapse behaviour of the calcareous soils of Arizona and New Mexico reported by Gile et al 1965 cited in Demars and Chaney (1982). Gile and colleagues grouped those soils into five stages on their magnitude of collapsibility consequent of their levels of cementation. From weakly cemented (collapsible) to very strongly cemented (not influenced by moisture), and further to moderate hard rocks (requiring rock mechanics analysis).

However, wetting the *strongly* calcareous (~25% CaCO<sub>3</sub>) samples with the acidic solution results in quite substantial CP values but a further increase in carbonate content to about ~40% drastically reduced CP. It is important to note here that, it is common knowledge that the dissolution of increased carbonate contents would certainly require greater amounts of solvent. Unfortunately, the amount of dissolving fluid in the standard oedometer cell used cannot be varied. Perhaps, a leaching process could be used in future to investigate if higher carbonate contents could lead to higher collapses. However, Smalley (1971); Mansour *et al.* (2008); Li *et al.* (2016) have noted that a carbonate soil requires several cycles of wetting and drying to dissolve CaCO<sub>3</sub> before collapsing. On the contrary, Karakouzian et al. 1996 cited in Mansour *et al.* (2008) have described leaching process as differing from the principle of sudden collapse upon wetting. They termed such a process to result in what they referred to as chemical piping or leaching-and-collapse.

It is clearly demonstrated here in the Figure 4.54 that for CaCO<sub>3</sub> at certain contents the collapsibility of calcareous silty-clay soil increases. On the other hand, increasing carbonate content above certain levels becomes beneficial for the stability of the soil. However, this appears to be a function of the ratio of CaCO<sub>3</sub> content to dissolving fluid. Nonetheless, other factors, such as density, porosity, void ratio, etc. capable of affecting collapse potential need to be considered in conjunction with CaCO<sub>3</sub> content.

#### **4.5.2 Nature of CaCO<sub>3</sub> occurrence - cemented versus uncemented**

While carbonate content influences the collapse potential of a calcareous silty-clay, the nature of its occurrence may determine the level of influence. As illustrated in Figure 4.55, calcite occurring as inter-particle cementation provides greater stability and resistance to wetting induced collapse compared to calcite occurring as non-cementing material (i.e. in loose

calcareous soils). However, it is unclear why calcite dissolution would have been different in the two circumstances even though exposure time was the same. Possibly, structural differences could have been responsible for the difference in collapse potential between these two different types of calcareous soils.

Considering that wetting fluid contained in the oedometer reservoir was insufficient to provide a complete dissolution of the carbonate in the oedometer specimen, the stability originally provided by cementation in the cemented specimens is likely to be substantially retained post-wetting. On the other hand, the uncemented specimens can more easily undergo both densification and calcite dissolution because of easier access of wetting fluid to possible dissolution sites, as well as greater ease of particles re-arrangement.

An explanation for the incomplete dissolution of calcite even in acidic solvent is potentially a result of the small quantity of solvent contained in the oedometer reservoir becoming saturated and unable to progress the dissolution reaction.

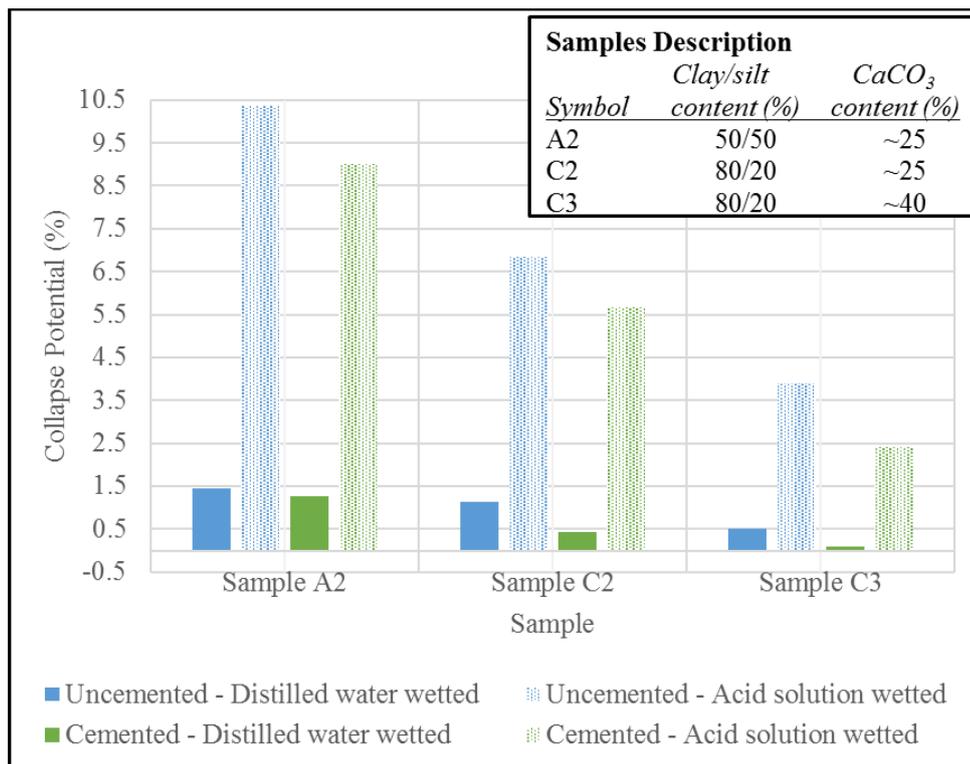


Figure 4.55 Influence of cementation and noncementation on the collapse potential of calcareous silty-clay soils

#### 4.5.3 Effect of dry density

Initial dry density and its related properties (void ratio and porosity) feature as some of the critical criteria controlling collapsibility of soils with increasing dry density resulting in reduced

collapse potential (Clevenger 1958 cited in Alan and Robert (1988); Barden *et al.* (1973); Lawton *et al.* (1989); Rogers (1995); Assallay (1998)). Additionally, densification has been successfully employed to mitigate collapse in some collapsible soils (Jefferson *et al.* (2005); Chris DF (2012); Langroudi (2014)). Figure 4.56 and Figure 4.57 present calcareous samples at three different initial dry densities. The two Figures respectively show collapses under acidic solution wetting and distilled water wetting conditions. Influence of wetting fluid on CP has previously been discussed.

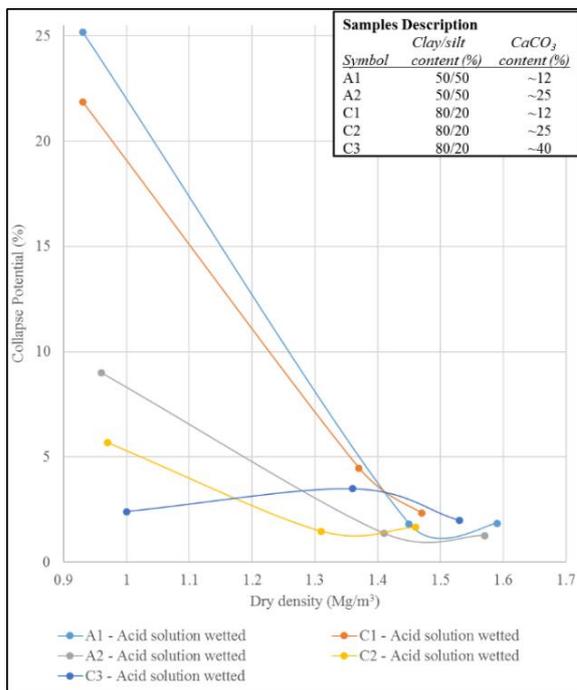


Figure 4.56 Effect of dry density on the collapse potential of calcareous silty-clay soils under acid solution wetting

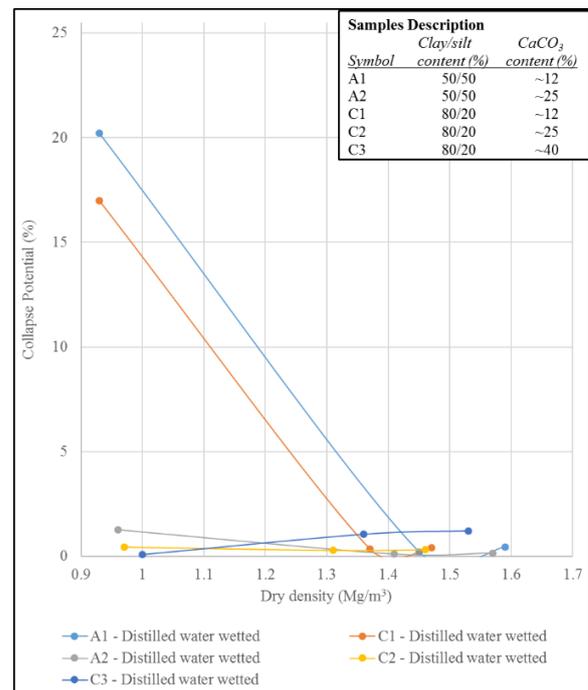


Figure 4.57 Effect of dry density on the collapse potential of calcareous silty-clay soils under acid solution wetting

In the Figure 4.56 and Figure 4.57, it can generally be observed that CP decreases with increase in dry density up to certain dissimilar levels for different samples, which is in agreement with claims by other authors (e.g. Clevenger 1958 in Alan and Robert (1988)). Conversely, above certain dry density levels, although collapses have not been significant, they appear to show a reversal of the trend. CPs tending to increase with higher dry densities (MDDs yielding higher CPs than samples at 50% OMC). Interestingly, this new trend is observable in both samples with low and high degrees of calcareousness confirming the effect to be that of density rather than carbonate content. Perhaps, the argument by some scholars, such as (Vance *et al.*, 2015) who postulated lime microcracking during carbonation can be valid. Cracking may

develop because lime-soil compacts at high density have relatively low pore volume, perhaps insufficient to accommodate the volume increase resulting from the conversion of lime to calcium carbonate. This cracking increases pore volume and as well weakens the compact structure (Bagoniza *et al.*, 1987).

#### 4.5.4 Effect of clay content

The impact of clay on the collapse phenomenon is commonly described based on micromechanics, regarding it mainly as the bond component of a collapsible soil that softens upon wetting, weakening shear resistance at particle contacts (Mellors, 1995). On this basis many researchers including Handy 1973 (cited in Bell *et al.* (2003)); Basma and Tuncer (1992); Lawton *et al.* (1992); Langroudi (2014); Assadi Langroudi and Jefferson (2015) have argued the impact of clay content on the collapsibility of soils, and some have proposed clay content thresholds to define collapse potential (CP). Unfortunately, a generalized definition of collapse based on clay thresholds negates the fact of some researchers (e.g., Lawton *et al.* (1992)) who claimed that a pure clay can also collapse under certain placement conditions.

Even though Handy 1973 suggested a reducing collapsibility with an increasing clay content in Iowa loess whose composition is unknown, on the basis that increased clay content results in reduced pore spaces and a denser material, the results presented here in Figure 4.58 for non-calcareous silty-clay samples have rather demonstrated an opposing trend. A higher clay content generally resulted in higher CPs for the two different wetting fluid types used. However, greater uncertainty exists in the results as clay content increased, and though dry density was identical in the three samples, there was marginal decrease in void ratio with increasing clay content. Notwithstanding, the marginal void ratio changes were not observed to influence CP.

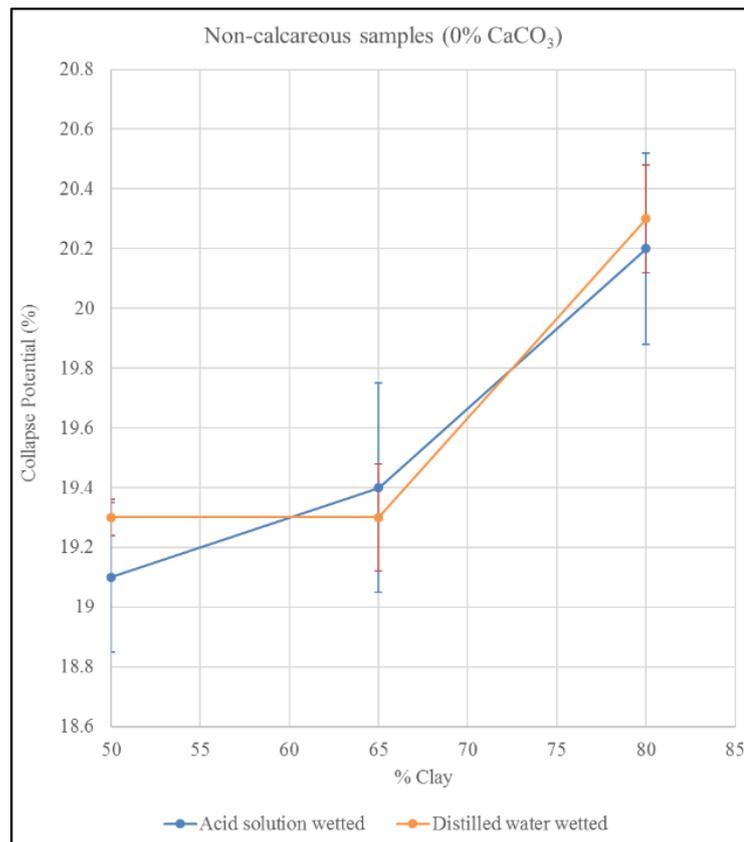


Figure 4.58 Effect of clay content on the CP of non-calcareous silty-clay soils with different wetting fluids

Again, with clay contents reaching 50 – 80 % and yet samples exhibiting significant collapse is inconsistent with many claims in the literature. There are claims that collapse occurs only where clay contents do not exceed 32% (Handy 1973 in Bell *et al.* (2003)) or 40% (Langroudi (2014) and references therein). Assadi Langroudi and Jefferson (2015) also demonstrated that the risk of collapse in loams can be eliminated with greater than 15% clay content. Figure 4.59 (modified from Langroudi (2014)) presents a comparison of data from both the literature and the current study showing clay levels in different collapsible soils where minimum and maximum collapses had occurred.

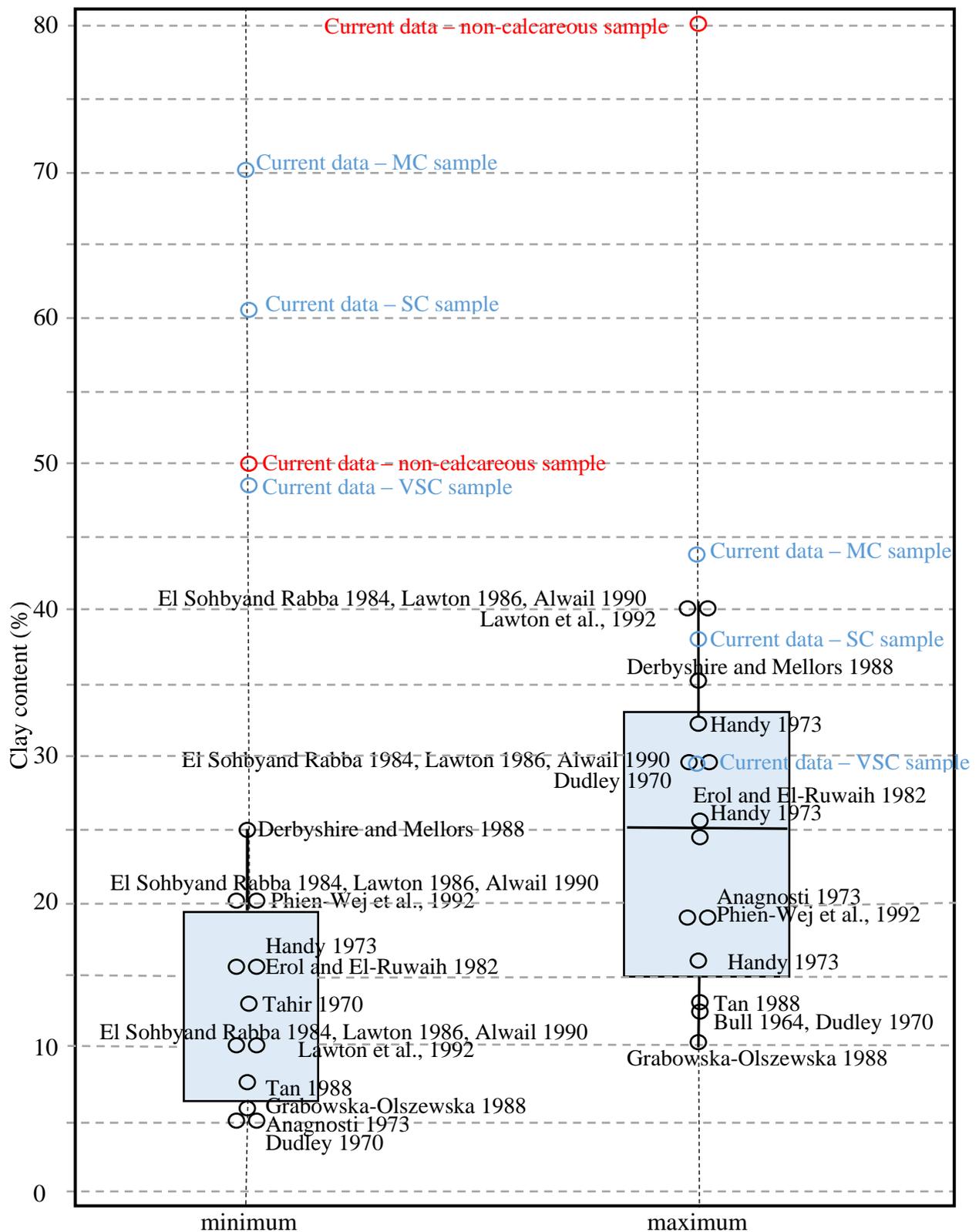


Figure 4.59 Thresholds of collapse potential based on clay content of different collapsible soils (Modified from Langroudi (2014)). M=moderately, S=strongly, VS=very strongly, and C= calcareous.

Lawton *et al.* (1992) reported El Sohby and Rabbaa (1984)'s data of collapse test on two mixes (sand-clay and silt-clay) (partly presented in the Figure 4.59 above) and reported an increasing CP with increasing clay content, but maximum collapses were at clay contents of

about 30-40 % and 10-20 %, respectively (see Figure 2.12 in section 2.3.3). Clay contents beyond those ranges resulted in swelling, thus, reducing CP. Another soil (Ottawa sand-Kaolin mix) studied by Lawton also revealed similar trend with maximum collapse occurring between 12 and 16 % clay content (same Figure 2.12). Although the present non-calcareous silty-clay samples are distinct in composition and dry density from those, they have shown similar trend but with the greatest CP occurring at the maximum clay content (80%) tested (Figure 4.58). Obviously, these contradictions indicate that different soils collapse differently, and clay content may not in itself adequately define the CP of a soil. The type of clay (e.g., expansive or nonexpansive) and structure of sample may need to be considered interdependently to gain a better understanding of the impact of clay. Therefore, it may be necessary to argue that clay thresholds previously used to define CP can only apply to those samples those thresholds were derived.

Lawton *et al.* (1992) have proposed a fundamental effect of macropeds on both initial strength and collapsibility of a remoulded clayey soil. An idea supported by Croft (1967); Mitchell and Soga (2005). From a laboratory collapse testing programme, which Lawton and colleagues undertook to understand the impact of macropeds in collapsibility of compacted clayey soils they proposed that during soil-water mixing, macropeds developed. Further, that a higher number and larger macropeds followed greater clay contents and provided better support for the soil systems. Although few macropeds/clods were observed from the present SEM images (Figure 3.19 on page 132), their consequent impact could not be adequately studied here due to the poor SEM images. However, the present oedometer results tend to agree with Lawton *et al.* (1992)'s argument. Figure 4.21 and Figure 4.22 for example, show samples with higher clay contents becoming less compressible prior to wetting, which can be the result of a better support provided by greater number of macropeds and clay bonding. Dry clay at particle contacts provide shear strength that resists densification (Basma and Tuncer, 1992). However, the reduced pre-wetting compressibility avails samples a greater pore space for the consequent densification upon wetting where clay bonds (inter-particle and inter-macropeds) are broken, thus, resulting in greater CP values. Evidence from Basma and Tuncer (1992) suggests that this effect increases along with increasing clay content ( see Figure 4.60 for example), but they however relied on Lawton *et al.* (1989) to note that there will be a limit to this effect; that beyond a certain clay content swelling will dominate collapse. However, this condition occurs where a clayey sample with significant plasticity is wetted at a confining pressure lower than its swelling pressure (Houston *et al.*, 2001).

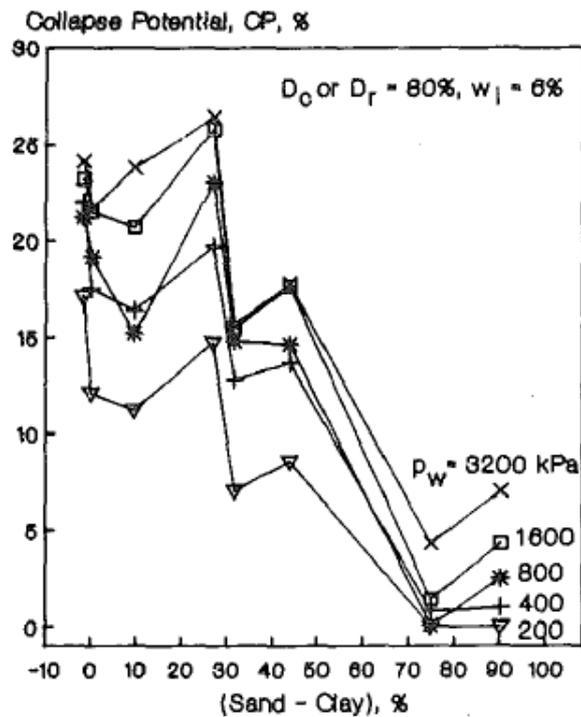


Figure 4.60 Effect of clay content and collapse potential (adopted from Basma and Tuncer (1992))

On the other hand, calcite cemented (calcareous) samples have demonstrated that lower clay content yields higher CP in both wetting fluid conditions (Figure 4.61 to Figure 4.63) contrary to their non-calcareous counterparts. Although this trend appears to be consistent with suggestions by Handy 1973 (cited in Bell *et al.* (2003)); Langroudi (2014); Assadi Langroudi and Jefferson (2015), their samples were not reported to have being impacted by calcite bonding as it appears to be the case here. Additionally, the literature reported similar trends only occurring at clay contents not exceeding specific thresholds that are below those of the currently studied samples. Contrary to the trend in Figure 4.61 to Figure 4.63, is that shown by the calcareous (10 -13 % CaCO<sub>3</sub>) brickearth of South-East England studied by Milodowski *et al.* (2015) where at Ospringe Pt. 1 with 17.5% clay recorded CPs averaging 0.5%, 4.2%, and 5.3% at wetting stresses of 100, 200 and 400 kPa, respectively but at Ospringe Pt. 2 with 12.8% clay, a CP of 3% was recorded at wetting stresses of 100 and 200 kPa but a 0.8% at 400kPa. Obviously, clay content has not solely influenced the CP of the samples. Milodowski *et al.* (2015) believed that the microstructure of clay and CaCO<sub>3</sub> bond controlled their behaviour and not necessarily clay content. Unfortunately, this was inadequately studied here.

Furthermore, as illustrated by the moderately calcareous samples according to Figure 4.23 and Figure 4.24, is a relative reduced pre-wetting compressibility with an increasing clay content. This implies that calcite cementation was more effective with higher clay contents, which is consistent with UCS results earlier discussed in section 4.3.2. Contrary to the behaviour of the non-calcareous samples, a lower pre-wetting compressibility in the calcareous

samples did not translate to higher collapse upon wetting, rather samples with higher clay contents exhibiting lower pre-wetting compressibility have shown lower CP. The reason for this may be explained in two ways.

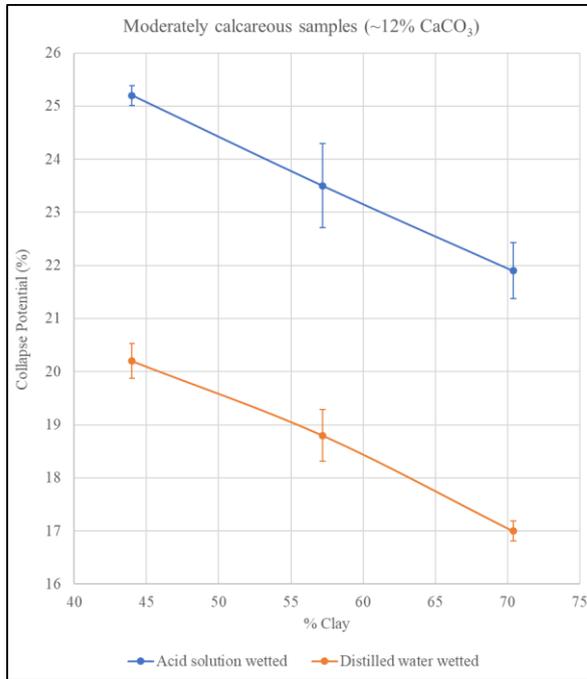


Figure 4.61 Effect of clay content on the CP of moderately calcareous silty-clay soils with different wetting fluids

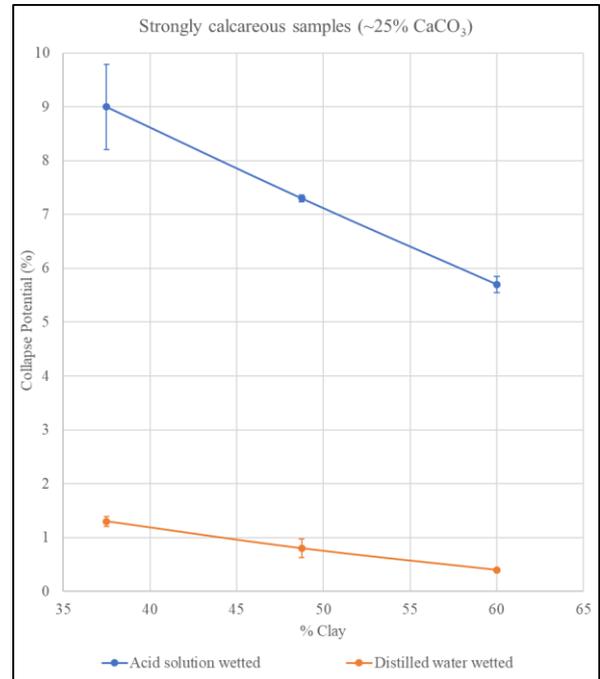


Figure 4.62 Effect of clay content on the CP of strongly calcareous silty-clay soils with different wetting fluids

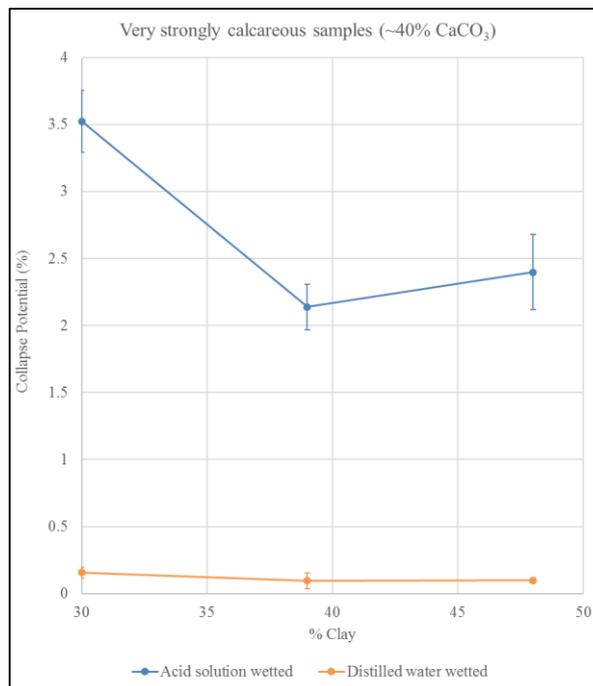


Figure 4.63 Effect of clay content on the CP of very-strongly calcareous silty-clay soils with different wetting fluids

Firstly, perhaps at lower clay content, the shear resistance provided at particle contacts by both clay and calcite bonds will consequently be lower. UCS and stiffness results presented in Figure 4.18 and Figure 4.19 agree to this. Accordingly, a pre-wetting stressing will more easily weaken the specimen's structure and perhaps increased microporosity and the propensity for consequent densification upon wetting.

Secondly, a higher clay content which perhaps favours macropeds formation and stronger calcite bonding will result in greater pre-wetting shear resistance. After wetting, because of the high clay content filling greater pore volume, deformation will be expected to be lower. Also, the stronger nature of bonds here may as well be more resistant to the softening effect of wetting. Relationships between UCS, stiffness and CP discussed in a later section (6.3.4) indicate that in calcite cemented clays, increasing UCS and stiffness result in decreasing CP. Again, because clays are less permeable than silts and calcite cementation further reducing infiltration, an impaired accessibility of wetting fluids to all potential bonded sites could be responsible for lower CP values recorded with higher clay (calcareous) specimens. This is, however, contestable considering that porosity was relatively high in all samples to allow fluid to permeate through sample during wetting period. Though, this could have been better understood through a permeability test but not considered in the scope of this study.

Furthermore, trends in Figure 4.63 appear to demonstrate that at high degree of calcareousness the influence of clay content on collapse becomes obscured. Although not proven here but considering the development of microcracking associated with volume increase of lime during its conversion to calcite, it can be assumed that the microstructure of calcite and its dissolution mechanism could have possibly control CP at this large calcite content. Lastly, it is worthy to note that the present study has not identified a threshold clay content to define collapse. Again, a critical clay content was not recognized, perhaps due to the large distances of clay contents between samples studied. Thus, these results may be considered inconclusive.

#### **4.5.5 Effect of wetting fluid type and rate of collapse**

The phenomenon of sudden volume decrease upon wetting commonly referred to as hydrocollapse, hydroconsolidation or hydrocompaction is greatly a function of time rate of inter-particle bond elimination and ease of particles repacking (or mechanism of collapse) under a constant load. Plots of axial deformation against time during the collapsing stage (i.e., wetted stage) of single oedometer tests are presented in Figure 4.64 to Figure 4.67. The shape of curves is comparable to that achieved from standard consolidation tests on fine-grained soils and evidently are three distinct regions of collapse. The first region is a time-lapse stage indicating the initial period when wetting fluid flows through bottom porous disc and enters the specimen

in one-dimension. The slope of the curves at this stage depended on the nature and initial strength of specimen where in some cases (especially in non-calcareous sample owing to near absence of cement bonding) a clear distinction from the second region appearing difficult.

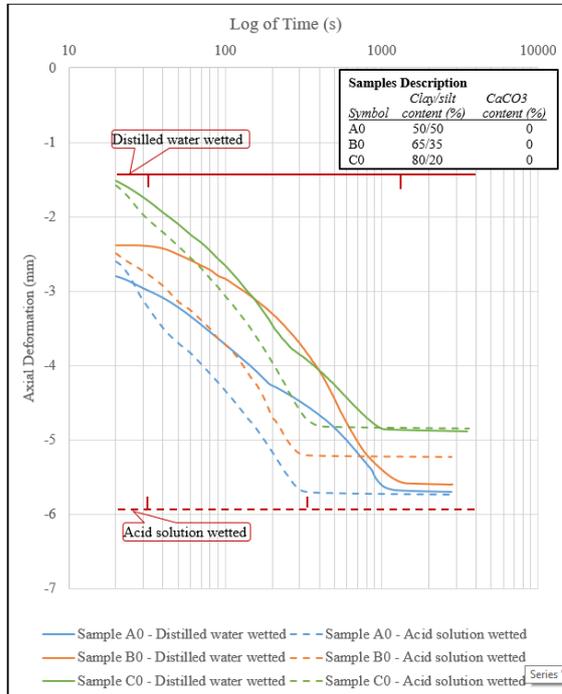


Figure 4.64 Time rate of collapse of non-calcareous silty-clay soils under different wetting fluid conditions

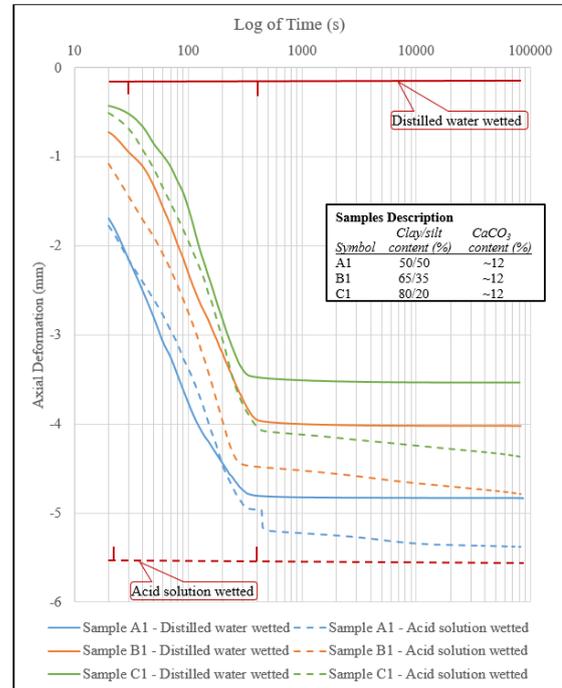


Figure 4.65 Time rate of collapse of moderately calcareous silty-clay soils under different wetting fluid conditions

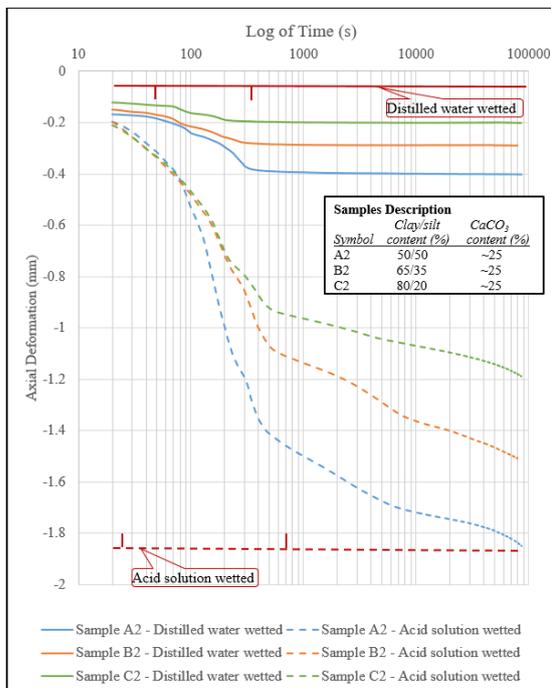


Figure 4.66 Time rate of collapse of strongly calcareous silty-clay soils under different wetting fluid conditions

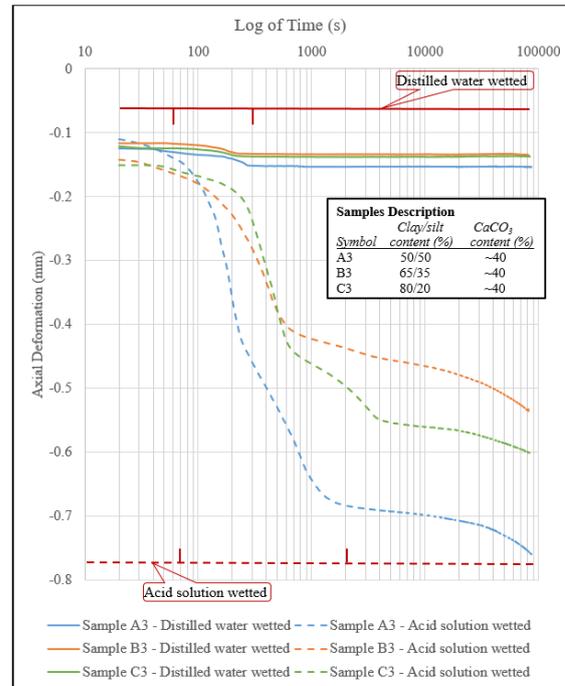


Figure 4.67 Time rate of collapse of very strongly calcareous silty-clay soils under different wetting fluid conditions

The second region represents period when the wetting fluid completely saturated the specimen and it is believed that at this stage inter-particle, inter-macropeds (or aggregates) and intra-macropeds bonding and bridges are weakened, reduced and/or eliminated. The bulk of collapse takes place at this stage. In cases of low initial bonding and where distilled water applied as wetting fluid, nearly complete collapse occurred here but in high calcite bonding collapse gradually continues in the acidic solution wetted condition.

Region 3 can be interpreted differently at certain degrees of calcareousness and differences in wetting fluid type. For a distilled water wetted test, this region represented the end of a complete collapse and the beginning of some sort of minor secondary compression. On the other hand, where an acidic solution applied as wetting fluid, this region witnesses either some minor or major secondary compression depending on specimen degree of calcareousness. This is therefore indicative that although sudden large settlement may occur very quickly, collapse may continue for longer periods perhaps until bond material dissolution is fully achieved.

There are reports in the literature arguing that collapse is only achieved quickly in laboratory flooded oedometer specimens, traditionally within several hours or less. Booth (1977) report three sands (soils A, B, and D) tested under a range of moisture, density, and stress conditions to have achieved 90% collapse in not more than 25 minutes. Huang, 1989 investigated a lean clay (Lawton *et al.*, 1992) and stated a variation in time required to achieve complete collapse depending on overburden stress at wetting. About 4 and 0.5 hours were noted for overburden stresses of 96 kPa and 383 kPa, respectively for a soil compacted to  $R_s = 80\%$  and  $S_i = 36\%$ . Lawton *et al.* (1992) investigated soil samples with different proportions of kaolin clay and Ottawa sand under marginally similar conditions with a 400kPa stress at wetting and reported time to achieve complete collapse ranging between 15 and 100 minutes. They found that the rate of collapse was increasing with increasing clay proportion up to around 40% kaolin, above which the rate of collapse was nearly constant. Additionally, that collapse occurred rapidly (not more than 2 hrs) even for higher clay proportions. Fookes and Best (1969) said a 95% collapse settlement upon wetting is achievable within 10 minutes while anything above 10 minutes is referred to as subsidence settlement.

In the present study, four key factors have been identified that control the time rate of collapse in the silty-clay samples (both calcareous and non-calcareous). They are calcite content, density, overburden stress at wetting, and wetting fluid type. Even though clay content did not appear to delay complete collapse as is illustrative of the *second region*, it is seen to limit deformation in terms of total settlement experienced by sample. The higher the clay content the less total deformation a specimen undergoes.

Even though in many cases, soaking of specimen in the oedometer test was sustained for about 24 hours, majority of collapse in all the samples occurred merely in not more than 1500 seconds (25 minutes). However, samples with high degrees of calcareousness demonstrated huge potential for further, but gentler continuous collapsing where they are wetted with acidic solution (see Figure 4.66 and Figure 4.67 in particular). This potentially demonstrated both collapse settlement (achieved in less than 10 minutes) and subsidence settlement (collapse achieved in more than 10 minutes) according to Fookes and Best (1969).

Surprisingly, non-calcareous samples wetted with distilled water achieved complete collapse at about 1000 seconds as against calcareous samples wetted under similar conditions achieving complete collapse in merely about 300 seconds. This therefore, alludes to the fact that while ordinary water may go through a process of gradual weakening of clay macropeds and clay bridges between macropeds and silt particles, it is nearly nonreactive with calcite cementation. Smalley (1971); Mansour *et al.* (2008); Li *et al.* (2016) have argued that only a prolonged period of sustained or several cycles of wetting and drying may relatively dissolve calcite.

Non-calcareous samples also appear to collapse quicker under acidic solution wetting than in distilled water wetting. Perhaps, this is due to the effect of acid on clay mineral. Clays treated with acid solutions generally cause clay particles disaggregation, elimination of mineral impurities, dissolution of alumina layers, and therefore altering their structure and chemical composition (Panda *et al.* (2010); Komadel (2016)). Acid activation of clays is an industrial process used to improve the surface and catalytic properties of clays used for certain purposes (e.g., petroleum cracking catalyst, sorbent, etc.). There are results in literature that confirm that this process results in reduction of clay  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{CaO}$  and  $\text{K}_2\text{O}$  contents (Thomas *et al.* (1950); Milliken (1952); Grim (1953); Rhodes *et al.* (1991); Panda *et al.* (2010); Komadel (2016)) thereby increasing porosity.

Another perspective to the time rate of collapse as related to overburden stress and density is been demonstrated in Figure 4.68 and Figure 4.69. The Figures present two samples wetted at different overburden stresses. Expectedly, it can be seen that increased overburden stress caused higher compressions to occur, thus resulting in higher densities pre-wetting. Though a higher overburden stress was envisaged to enhance collapsibility, however, with an increased delay to complete collapse occurring with increasing overburden stress, it is only reasonable to assume that the delay has been the result of an increased density. Because increased density reduces flow of fluid through a soil, and the process of inter-particle bond elimination and subsequent repacking might be slowed.

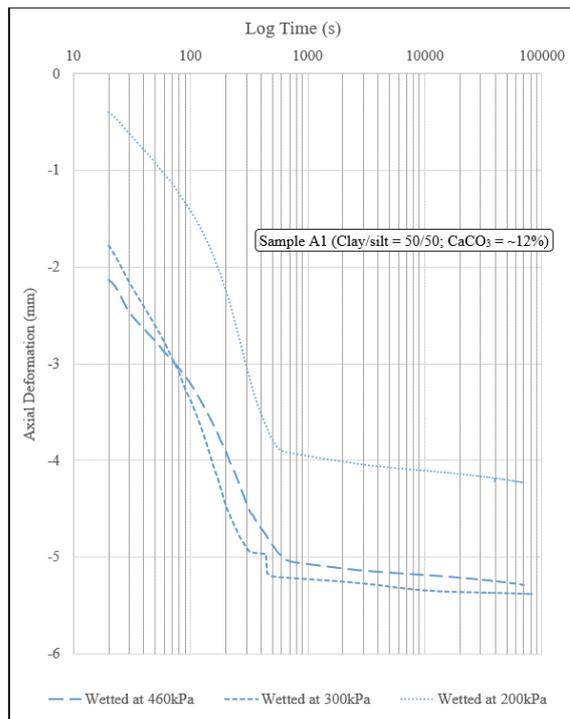


Figure 4.68 Impact of overburden stress at wetting on the time rate of collapse of a moderately calcareous silty-clay soil under acidic solution wetting condition

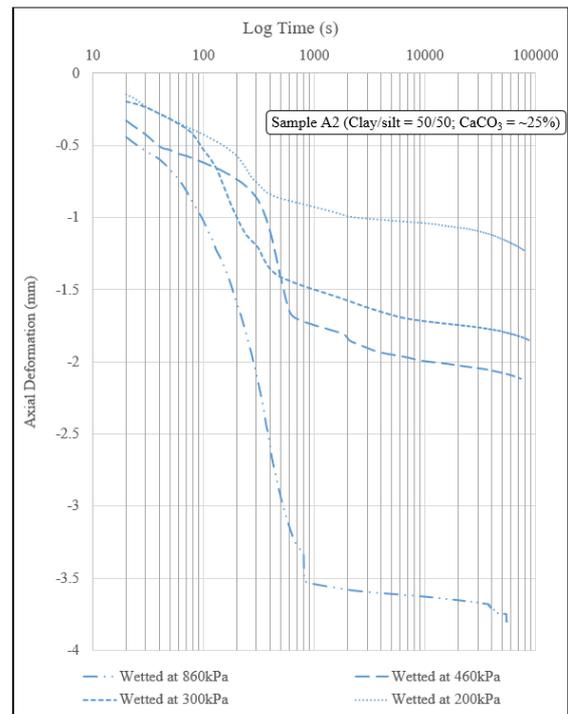


Figure 4.69 Impact of overburden stress at wetting on the time rate of collapse of a strongly calcareous silty-clay soil under acidic solution wetting condition

These experimental results demonstrate that sudden collapse at any location within a soil (natural or fill) can occur within a few minutes but there may also be a prolonged collapse, which is a result of slow inter-particle bond elimination. Therefore, the time needed for complete collapse to occur in the field is fundamentally a function of the method by which wetting occurs, type of wetting fluid, rate of infiltration, and type of bonding material. For example a number of deep filled canyons in southern California studied by Brandon *et al.* (1990); Lawton *et al.* (1992) were found to have water infiltrating from the surface as a result of precipitation and landscape irrigation, causing collapse in the fills. The collapse typically started many years post-compaction and continued for several years further. Conversely, Leonards and Narain (1963) reported collapse settlements in earth dams occurring virtually concurrently with the initial filling of the reservoir.

#### 4.5.6 Comparing single and double oedometer methods

Single oedometer test (SOT) and double oedometer test (DOT) methods are usually adopted in the oedometer. The DOT is however, a method commonly used to speedily estimate collapse potential (CP) at several stress points (Alan and Robert (1988); Fredlund and Gan (1995); Rogers (1995)).

Figure 4.70 and Figure 4.71 compare CP results of SOT and DOT obtained at different overburden stresses under acidic solution and distilled water wetting conditions. The results tend to display fair comparability of the two methods in high carbonate samples (*strongly* calcareous (A2 and C2)) at all stress points. In low carbonate samples (*moderately* calcareous (A1 and C1)), the techniques only yielded comparable CPs at some stress levels and show substantial differences at others.

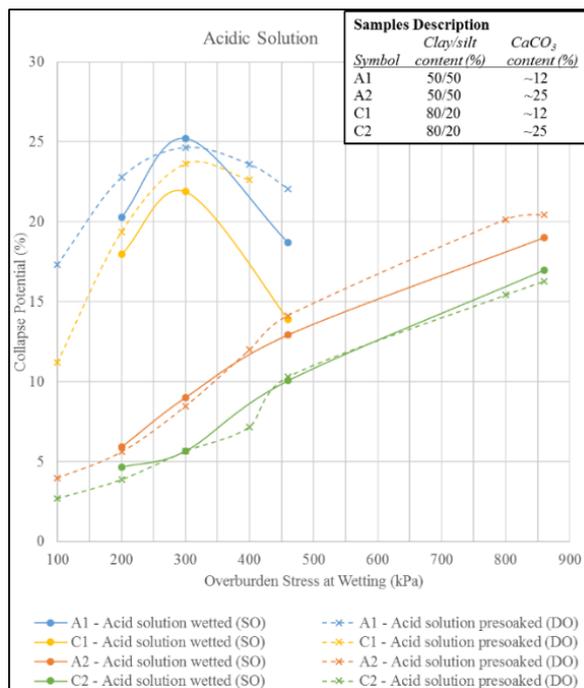


Figure 4.70 The influence of overburden stress and testing technique on the collapse potential of different calcareous silty-clay soils under acidic solution wetting condition

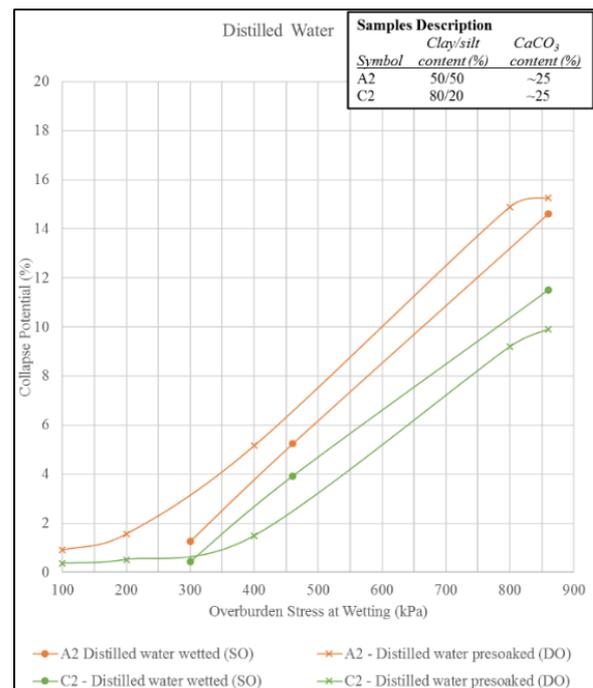


Figure 4.71 The influence of overburden stress and testing technique on the collapse potential of different calcareous silty-clay soils under distilled water wetting condition

A few published comparisons between SOT and DOT have claimed that they generally predicted collapses with only small differences (e.g. Booth (1977), reporting the DOT as overestimating CP by only about 10% compared to the SOT). However, Lawton *et al.* (1992) argues whether the DOT can accurately predict CP in all collapsible soil types. Lawton *et al.* (1992) reported that another work on the CP of cement- and lime-stabilised soils, conducted by Lawton has demonstrated that DOT predicted CP values were significantly greater than those measured by SOT and recommended that DOT be adopted only where confirmatory tests have been performed for the specific soil and the conditions to be assessed. Nevertheless, results presented here show both methods either overestimating or underestimating magnitudes of collapse at one stress level or the other in both acidic solution and distilled water wetted conditions. Therefore, it can be concluded in line with Lawton's recommendation that DOT

may be used only as a guide to estimate CP where anticipated field conditions are taken into consideration.

#### **4.5.7 Influence of wetting stress on collapse potential**

Another important criterion controlling the magnitude of collapse of a metastable soil is the overburden stress at wetting. This can be seen to differently impact on the magnitude of collapse obtainable in different soils depending mainly on their bonding and stress history. Generally, it is evident that collapse magnitude increases with increasing stress at wetting irrespective of wetting fluid type (Figure 4.70 and Figure 4.71). However, low bonded samples (*moderately* calcareous samples A1 and C1) appear to demonstrate that the increase in CP following increase in load is not without bound. The A1 and C1 curves in Figure 4.70 indicate that CP is a maximum at some critical value of overburden stress (300 kPa). Above this, CP decreases with increasing overburden stress. This may be true for most soils. A similar view was expressed by Lawton *et al.* (1992), who went further to say that the critical overburden stress for certain conditions and soil types may be so huge becoming of no practical consequence. In other words, where the critical stress level of the soil is far greater than the stress a proposed infrastructure would impose, then the CP at that critical stress is of no concern.

The decrease in CP at high stresses may be a result of pre-wetting densification. Milodowski *et al.* (2015) have also reported similar results obtained from collapse testing of the calcareous brickearth of South-East England (2.5m depth, Ospringe Pit 1). They presented CP values averaging 0.5, 4.2, and 5.3 % for wetting at 100, 200 and 400 kPa overburden stresses, respectively. At a second location (2.5m depth, Ospringe Pit 2) 100m north of Pit 1 their results revealed CP value of 3% for wetting at 100 and 200 kPa vertical stresses while a 0.8% was recorded at 400kPa and 0% at 800kPa overburden stress. Milodowski *et al.* (2015) argued that the reduction in CP values at 400kPa and further down to zero at 800kPa may appear to reveal ‘yielding’ or breaking of intergranular bonds as loading increased prior to wetting, apparently reducing the soil’s pore volume and its susceptibility to collapse.

#### **4.5.8 Summary on the collapse behaviour of samples**

Three major soil properties, which interdependently controlled the collapse behaviour of soil samples have been identified. These are dry density, clay content and degree or type of inter-particle bonding material. Also influencing the magnitude of collapse are overburden stress at wetting and wetting fluid type.

In situations where no CaCO<sub>3</sub> was present in the samples wetting fluid type did not significantly influence collapse potential. However, where CaCO<sub>3</sub> was present, the influence became obvious and depending on the degree of dissolution, a calcite-cemented (calcareous) soil may collapse to a greater degree than a non-calcareous soil upon acidic solution wetting.

Even though dry density was a collapse-controlling factor, it was rather complemented by the degree of bonding. Specimens with nominally comparable dry densities but varying degrees of cementation (or CaCO<sub>3</sub> content) exhibited collapse magnitudes relative to CaCO<sub>3</sub> content. On the other hand, samples with comparable CaCO<sub>3</sub> content but differing dry densities, showed collapse magnitudes relative to dry densities.

According to collapse classification criterion based on dry density, e.g. Clevenger 1958, the low dry density of samples ( $\sim 1 \pm 0.1 \text{ Mg/m}^3$ ) reported in this thesis should have all yielded significant CPs. However, this was not so. Many of the samples tested became nearly non-collapsible because of carbonate bonding. At certain CaCO<sub>3</sub> levels, bonding became the collapse-controlling factor rather than dry density. Although Rogers (1995) suggested low bonding strength as necessary for a soil to show collapse, it is unclear what low bonding strength could mean, since natural loessial samples with CaCO<sub>3</sub> contents well above 25% have also appeared in the literature as being collapsible ((Li *et al.* (2016); Jefferson *et al.* (2005)).

Notwithstanding the high clay contents in samples studied here, high collapses have been recorded in contradiction of the position of Okwedadi *et al.* (2014); Handy, 1973 (in Bell *et al.* (2003)), perhaps because of the exaggerated void ratios and related properties. The ratio of clay/silt in samples, especially the non-calcareous and *moderately* calcareous ones was found to be a collapse-controlling factor. Values of CP increased with decreasing clay/silt ratio in the aforementioned samples but in others with higher degrees of calcareousness, this trend was not observed.

It is clear from the results that no single soil parameter is adequate to predict the degree of collapse, and that the importance of inter-particle bonding material and anticipated field pore fluid type should not be ignored in site characterisation if collapse must be prevented.

Lastly, it was evident from CP values obtained by wetting at different overburden stresses, that the CP of a soil may be maximum at a critical overburden stress. That is, CP may increase with increasing stress level up to a maximum overburden stress, above which it begins to decrease.

## Chapter 5 Sodium Silicate Treatment

### 5.1 Introduction

In many ground improvements studies, the unconfined compressive strength (UCS) and stress-strain behaviours pre- and post-treatment are typically used to evaluate the improvement strategy. Even though soil collapse is usually a consequence of moisture intake, it is highly dependent on the structure and inter-particle cementing strength. As such, improving strength is necessary to mitigate collapse (Iranpour and haddad, 2016), and a relationship can be established between strength and collapse resistance. This is, however, most effective where a treatment strategy produces imperviousness or stability against moisture.

Sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) solution is reported to produce an impervious cement gel around soil grains when reacted with soils containing certain metal ions (e.g.  $\text{Ca}^{2+}$ ). This gel has the potential to act as a binder, stabilising soils prone to collapse upon wetting.

Previous research has inferred the effectiveness of  $\text{Na}_2\text{SiO}_3$  and other treatments to reduce collapse potential (CP) from the improvement in UCS (Sokolovich (1965); Sokolovich and Ibragimov (1971); Jefferson *et al.* (2005)). Thus, there is little direct evidence in the literature of treatments reducing CP. Although strength may relate to collapsibility, the resistance of sodium silicate-treated soil to wetting induced collapse is dependent, apart from bonding, on other factors relating to the chemistry and thermodynamics of the reactions between the silicate and the metal ions (e.g.,  $\text{Ca}^{2+}$ ).

According to Hurley and Thornburn (1972) the strength, durability and permeability of a bonding gel produced from the reaction of  $\text{Na}_2\text{SiO}_3$  and metal ions depended on factors which include  $\text{SiO}_2:\text{Na}_2\text{O}$  molar ratio and concentration, temperature, salts (acids and bases) present in reaction.

This chapter begins with a pilot study to select a suitable  $\text{Na}_2\text{SiO}_3$  formulation based on workability and effectiveness to bond without significant effect of syneresis. Thereafter, is the presentation and evaluation of laboratory results of UCS and CP tests conducted on treated samples. At the outset, treatment was intended only for samples that exhibited high collapse severity (*severe trouble (ST) and very severe trouble (VST)*) as presented in chapter four. However, treatment and compressive testing was further carried out on some low-collapsible and non-collapsible samples in order to allow evaluation the impact of carbonate content on the treatment (in terms of enhanced UCS).

## 5.2 Formulation of Sodium Silicate Solution and Treatment Strategy

### 5.2.1 Formulation of solution

The implication of Hurley and Thornburn (1972)'s finding is that, depending on soil composition, a sodium silicate solution with specific properties would need to be selected to suite the treatment of particular soils. Workability of the solution is another factor to consider. Therefore, following Hurley and Thornburn (1972),  $\text{Na}_2\text{SiO}_3$  solutions of different  $\text{SiO}_2:\text{Na}_2\text{O}$  (Si/Na) molar ratios and concentrations were formulated, and investigated at a pilot stage to ascertain which could be most suitable and effective to treat all the different samples using a non-destructive means (e.g. absorption).

Two forms of commercially available  $\text{Na}_2\text{SiO}_3$  solutions differentiated by their Si/Na molar ratios (1.71 and 3.81), supplied by *Ingessil S.r.l. Silicate Industry, Italy* (see their full commercial silicate compositions in Table A1-1 of Appendix A1) were used alongside with a colloidal silica from *Fishers Scientific, UK*. The colloidal silica was needed to alter the Si/Na ratio of the original  $\text{Na}_2\text{SiO}_3$  solutions to produce more forms of the solution. After confirming the unsuitability of the two original solutions (Si/Na = 1.71 and Si/Na = 3.81), proportions of the colloidal silica were mixed with proportions of the Si/Na = 1.71 original solution in order to increase its silica component thereby raising the solution's molar ratio and concentration to different levels. Table 5.1 presents four types of  $\text{Na}_2\text{SiO}_3$  solutions (original and formulated) examined at different concentrations.

Concentration expressed in molarity (M), determines the viscosity and workability or absorbability of the solution whereas, molecular ratio of silica to sodium oxide (Si/Na) together with concentration controls the chemical interaction and bonding gel types formed with the reacting inorganic substance (Hurley and Thornburn, 1972).

Because of the interest to understand the impact of calcite bond on the treatment, it was important to treat the specimens without having to destroy their original structure. Therefore, mixing was not an appropriate method. In practice, grouting is more common with sodium silicate treatments, but successful grouting will depend largely on the permeability of soil and grout viscosity. The following non-destructive strategies for applying the solution were considered at the preliminary level:

- (1) Grouting. This could be implemented only for compressive test (CT) specimens because the oedometer specimens were too thin and forcing the injector into them caused their breaking apart. For the CT specimens, a hole was drilled into the specimen using a handheld drill with a drilling bit of about 5mm diameter. It was attempted to pump the solution into the specimens with a syringe through the drill hole (as shown in Appendix A7 (A7-1)). This method proved unsuccessful for all specimens and solutions tried. The

process of drilling and forceful injection handoff the fluid resulted in the damage of weak specimens (i.e. *moderately* calcareous).

- (2) Pressurised absorption. A vacuum desiccator with a maximum pressure of 1 bar was used. This method was implemented on both CT specimens and oedometer specimens. Weak (*moderately* calcareous) and stronger CT specimens were half-soaked in the solution inside the vacuum desiccator chamber. The chamber was then pressurised (see setup in Appendix A7 (A7-2)) gradually to about 0.8 bar and allowed time for the solution to rise to the top of specimen. At certain solution concentrations (3 - 4), the solution could permeate weak oedometer and CT specimens. However, the weak CT specimens disintegrated at complete saturation (which was at ~20 minutes). The same solutions and process conducted on the stronger CT specimens resulted in wetting only a thin layer (i.e. solutions could not permeate the specimens even after applying the maximum 1 bar pressure for additional one hour). The success of the pressurised absorption method seen with the weak oedometer specimens may possibly be because they were treated within their ring confinement. Nevertheless, a consistent method was desired to treat both oedometer and CT specimens.
- (3) Impregnation by capillary absorption. Both oedometer and CT specimens were half-soaked in solution and allowed to absorb solution by capillary rise in one-dimension to minimize air entrapment. Capillary rise was slow in these low permeable soils, especially those with higher calcite contents, resulting in early formation of an impermeable layer that prevented the solution from reaching far into the core of the specimens. However, in weaker specimens (*moderately* calcareous), both oedometer and CT specimens were completely saturated in good time (less than an hour) but the edges of some CT specimens were observed to begin gradual disintegration before solution permeated to their core. As a result, some of the treated specimens were tested with somewhat irregular edges. The disintegration may be that the inter-particle bond was weakened by the solution.

From the above strategies, tried with various concentrations of the solutions as shown in Table 5.1, only strategy (3) performed relatively satisfactorily in both oedometer and compressive test specimens, notwithstanding the drawbacks identified.

Table 5.1 Sodium silicate solutions formulated and examined for suitability at pilot level (treatment by impregnation).

$SiO_2:Na_2O$ (Si/Na) Ratio	Concentration (M)	Density (g/ml)	Remarks
1.71*	5.52	1.54 (original)	Too viscous to be absorbed by any samples.
	3.2	1.32 (diluted)	A1 oedometer specimen treated. Setting only occurred at about 48 hours. Significant cracking and shrinking visible
	4.8	1.47 (diluted)	A1 and C1 oedometer specimens treated. Setting only occurred at about 48 hours. No cracking but significant shrinking.
3.01	5.2	1.47 (formulated)	Too viscous to be absorbed by any samples.
	2.0	1.18 (diluted)	C1 oedometer specimens treated. Setting occurred at about 24 hours. Significant cracking visible
	3.0	1.28 (diluted)	C1 oedometer specimens treated. Setting occurred less than 24 hours. Significant cracking visible  C3 & B3 compressive test specimens treated. Setting occurred less than 24 hours. Significant cracking and breaking of specimens
	3.5	1.32 (diluted)	C3 & B3 compressive test specimens treated. Setting occurred less than 24 hours. Solution did not penetrate sufficient depth. Significant cracking and breaking occurred after oven drying but did not when dried under room ambience
	4.0	1.36 (diluted)	C1 oedometer specimens treated. Setting occurred less than 24 hours. No shrinking, minor cracking only on the top face. Where the top face has direct contact with solution no visible cracking occurred
3.40	5.1	1.45 (formulated)	Too viscous to be absorbed by any samples.
	4.0	1.36 (diluted)	Too viscous to be absorbed by any samples.
	3.5	1.31 (diluted)	B2 compressive test specimens treated. Setting occurred in less than 24 hours. Did not substantially permeate but no visible cracking, except in one case where minor

The aim of this investigation was originally to determine an optimum amount of  $\text{Na}_2\text{SiO}_3$  solutions most appropriate to improve the present samples through a non-destructive treatment method. This could have been achievable by grouting but considering the nature (size, strength and permeability) of specimens grouting was unsuitable.

Despite the drawbacks from the tried strategies owing mainly to the low permeability of the specimens, impregnation by capillary rise (absorption) nonetheless was considered the most practicable non-destructive strategy to treat the specimens. The main disadvantage with this method was the difficulty to control both the quantity of absorbed solution and its distribution. It was assumed that a complete saturation could mean an even distribution of the solution. However, there is the possibility that the concentration of the solution might change along its absorption path.

Both CT and oedometer specimens adopted at the pilot treatment stage were impregnated with various forms of  $\text{Na}_2\text{SiO}_3$  solutions presented in Table 5.1 with two general assumptions made: (1) any solution type capable of satisfactorily improving weak specimens would also adequately improve stronger specimens; (2) solution type absorbable by an oedometer specimen would be absorbable by a comparable CT specimen and vice-versa.

For the oedometer specimens, treatment was done by placing individual oedometer specimens (inside oedometer rings) in a small porcelain dish (as shown in Figure 5.1) with a stainless porous disc placed at the base of specimen. The solution was then poured into the dish and the specimen allowed to absorb progressively from the bottom to the top until complete saturation was observed as evident in Figure 5.1a, b and c. CT specimens were placed on porous discs in a plastic bowl of capacity to contain several specimens. The solution was then poured into the bowl to soak the specimens without submerging so that air was not entrapped in the centre of specimens during the absorption process. However, a time-gap was determined after which the upper part of the specimen not in direct contact with the solution was turned into the solution leaving a minimal space for air escape. This can be seen in Figure 5.2a. In this treatment, a monitoring specimen was also soaked side-by-side and cut at time intervals to observe when absorption had reached the specimen core (Figure 5.2 b, c and d). In all impregnation treatment systems, as absorption proceeded, more solution was supplied until absorption was completed. Thereafter, wet treated specimens were weighed and placed on nonporous stainless-steel discs and allowed to set under ambient conditions for 24 hours before oven drying for another 24 hours at temperatures between 45 – 50 °C. Setting was considered to be the initial gel time, i.e. the period post-treatment when formation of a firm gel was achieved. This was estimated with a gentle finger press down.

Unfortunately, the solution suitable for impregnating the *moderately* and *strongly* calcareous specimens could not sufficiently permeate through the *very strongly* calcareous specimens. Reducing solution concentration to a certain level (about 3.5) permeated these *very strongly* calcareous samples but oven drying for 24 hours resulted in specimens fracturing.

Because of the adverse effects of specimens fracturing and shrinking (syneresis), which are common features of soils stabilized with certain  $\text{Na}_2\text{SiO}_3$  formulations, physical observations of extent of cracking and shrinkage occurrence post-drying were made (see Figure 5.3) in order to determine the most suitable solution type to be adopted in the main treatment programme.



Figure 5.1 Solution absorption in oedometer specimens: a, b and c are different stages of the absorption process

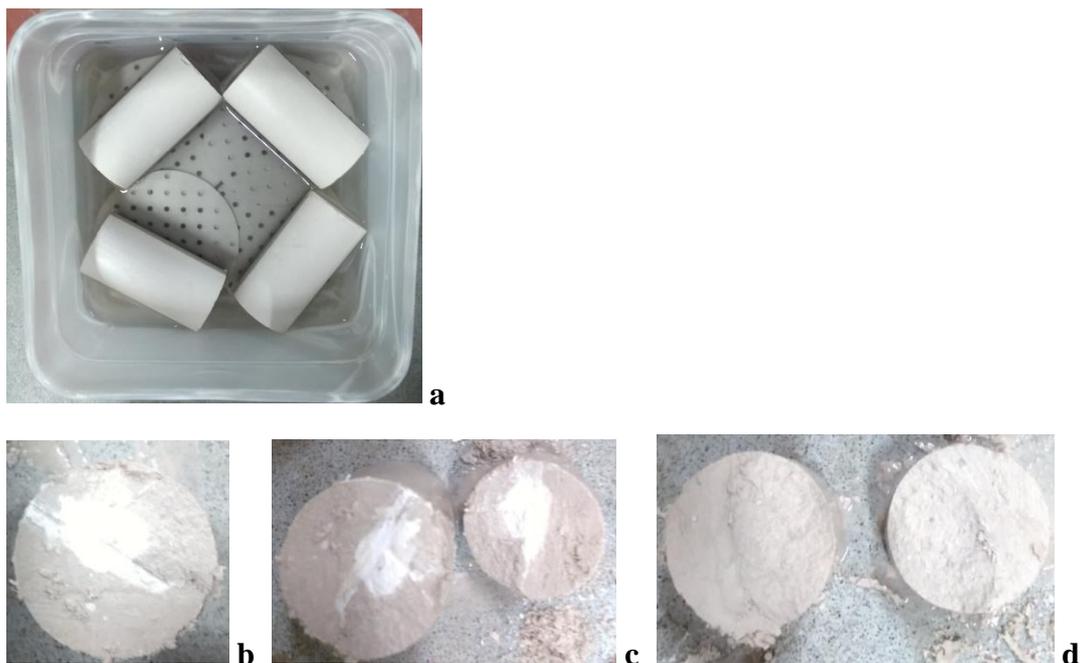


Figure 5.2 Solution absorption in compressive test specimens (a): b, c and d are monitoring stages of the absorption progress

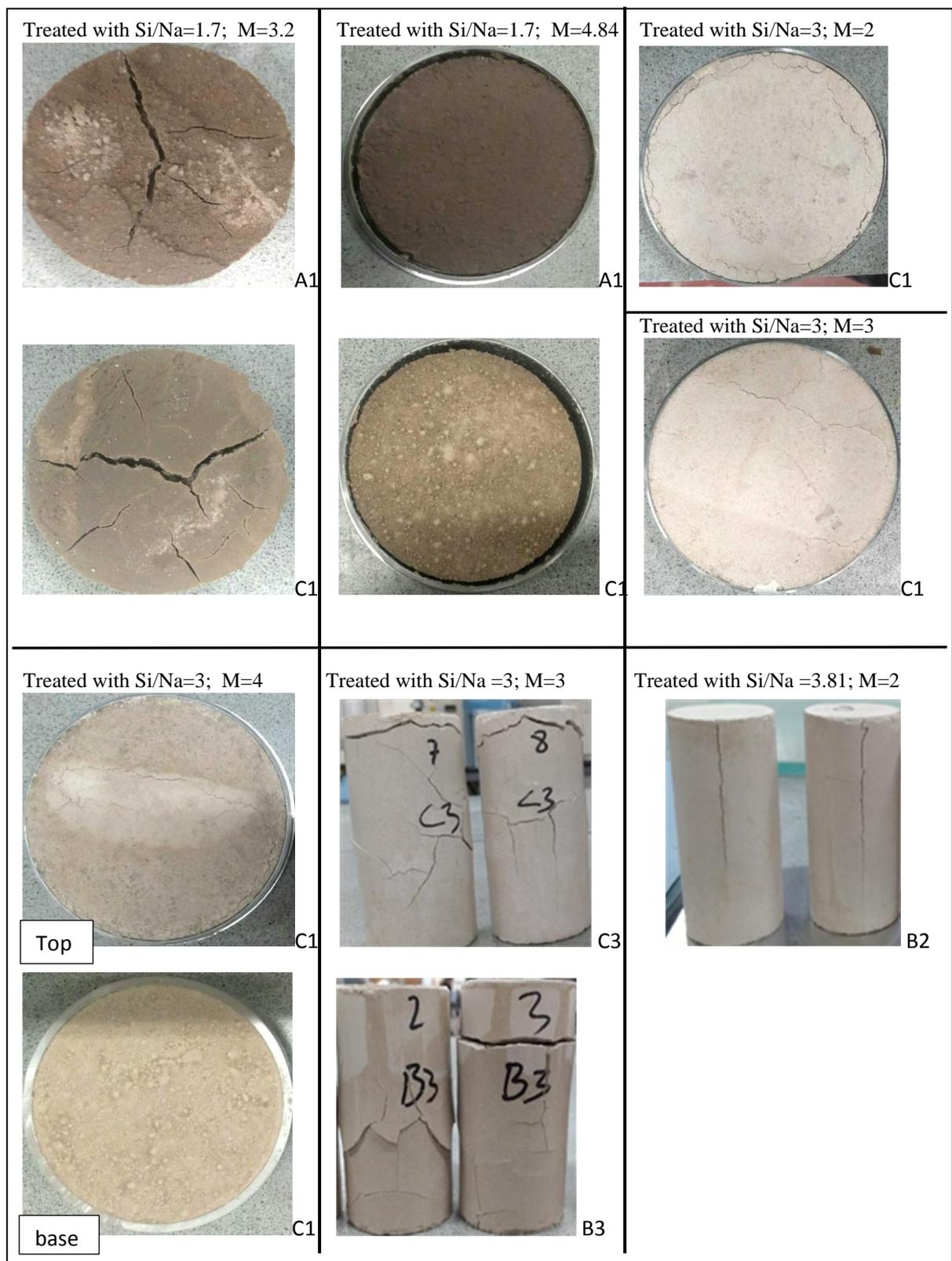


Figure 5.3 Observed specimens surface consistency after treatment (impregnation by absorption), setting and 2 days oven drying at 45 -50 °C

Even though the literature reports sodium silicate solutions with densities of 1.10 – 1.15 g/ml as adequate for treating certain collapsible loessial soils (Sokolovich (1976); Evstatiev (1988); Jefferson *et al.* (2005)), those treatments were only successful when enhanced with

admixtures, such as ammonia or formamide solutions and in other cases, an alternating injection of carbon dioxide and a silicate solution. The present treatment strategy employs a single-step treatment procedure where only sodium silicate solution was used. Consequently, only solutions with higher densities ( $> 1.3$  g/ml) appeared effective.

From investigations and observations of the performance of the various  $\text{Na}_2\text{SiO}_3$  solutions, the following preliminary conclusions were reached:

- Nearly all forms of  $\text{Na}_2\text{SiO}_3$  solution above certain concentrations demonstrated ability to agglomerate soil particles when treated and allowed to dry out. However, there are issues of durability, integrity and performance of agglomerated soils depending on the type and concentration of solution adopted.
- Treatment with high molar ratio (Si/Na) solutions, that is up to and above 3.0, allowed for reasonable setting time in all concentrations investigated and little or no shrinking was visible for treatment with concentrations (M) greater than 3 when applied on *moderately* and *strongly* calcareous specimens.  $M \leq 3$  resulted in cracking but only such were reasonably absorbable by the *very strongly* calcareous specimens.
- For treatments with Si/Na = 1.71 solution at all concentrations, a satisfactory set level was only achievable after about 48 hours and large shrinking occurred with concentrations (M) of 5.5 and 4.84. With lower concentration, significant cracking also accompanied shrinking.
- The viscosity of all forms of sodium silicate solution investigated relied on both molar ratio and concentration. The higher the molar ratio (Si/Na) or concentration the more viscous the solution was. However, a lower concentration of a higher molar ratio solution can be more viscous than a higher concentration of a lower molar ratio solution, implying that viscosity is mostly a function of molar ratio. Viscosity controlled the absorbability of the soil specimens.
- Solutions with molar ratio (Si/Na) of 3 and above at concentrations (M) of about 3.5 and greater were found to be appropriate. For absorbability and availability reasons a Si/Na = 3 at M = 4 and M = 3.5 was selected. However, these were only reasonably absorbable by the *moderately* and *strongly* calcareous specimens.
- Observations revealed that in those cases where minor cracks occurred in oedometer specimens where Si/Na = 3, M = 4 solution was applied, cracks were only visible on the upper surface. This is possibly because the solution that rises to the upper surface could have changed chemically (e.g., concentration) through the absorption process. After full

soaking as illustrated in Figure 5.1, turning the upper surface into direct contact with solution and allowing soaking for few minutes prevented cracks occurring.

- Because of the difficulty in controlling the amount of solution absorbable by the specimens, there is the concern as to whether it will be logical to compare the mechanical behaviour of different treated specimens, which perhaps absorbed different quantities of solution. A controllable treatment system was therefore necessary and so, treatment by mixing and kneading was subsequently considered.

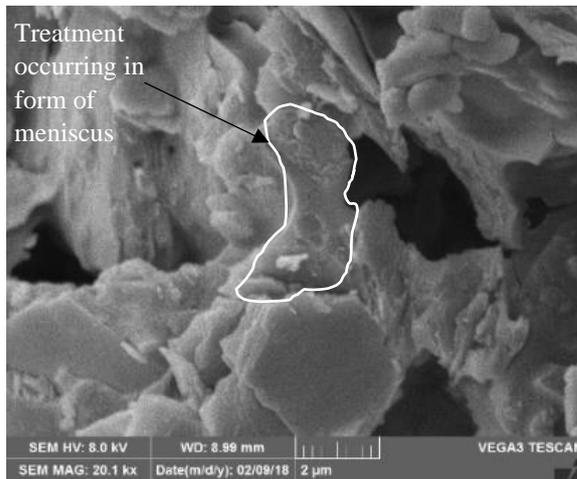
### 5.2.2 Main treatment of test specimens

Two treatment strategies have been employed using the most suitable solution ( $\text{Si/Na} = 3$ ,  $\text{M} = 4$ ). The same impregnation procedures described in the preceding section have been adopted to treat both oedometer and CT specimens while mixing and kneading (Appendix A8) was used to treat only a group of *moderately* calcareous CT specimens.

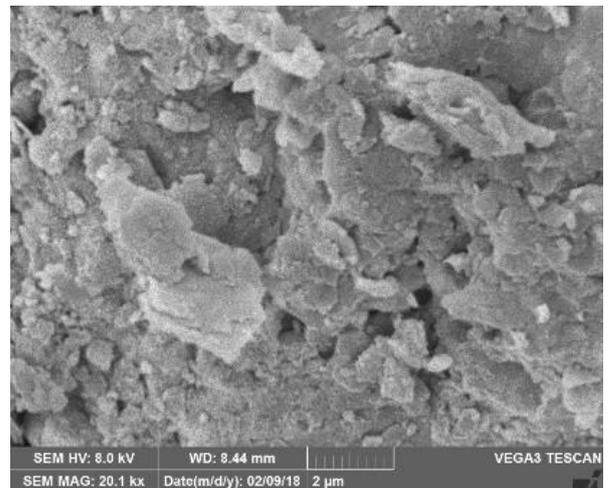
Though curing methods may differ in some ways, 7, 14 and 28 days curing periods are typical of many ground treatment programmes considering that portlandite (in lime, cement and concrete) hydration is nearly complete at 28 days curing (Domone and Illston, 2010). Sokolovich and Ibragimov (1971) have also corroborated an idea that the hardening process of the cement gel from a sodium silicate grout was a rapid solidification process. This was an earlier claim by Sokolovich (1965) who said that the hardening ends at 28 days. In the present study treated specimens have been cured either in an oven (heat curing) at temperatures between  $45 - 50\text{ }^{\circ}\text{C}$  for 24 hours or under room ambience (see Appendix A9) for 7, 14 and 28 days with room temperature and relative humidity being around  $18 \pm 3\text{ }^{\circ}\text{C}$  and  $25 - 35\%$  respectively.

Compressive test specimens followed both curing methods in order to compare strength development from both, whereas, oedometer specimens were only heat cured. Treated oedometer specimens were desired to be devoid of moisture at testing because of the impact pore fluid could have both on the compressibility of the specimens and on the chemistry and concentration of wetting fluid (acidic solution in particular).

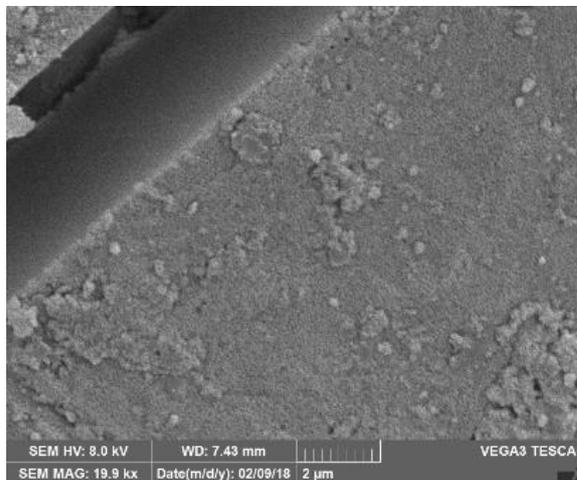
The microstructure of some of the treated specimens have also been investigated under the scanning electron microscope. See SEM images in Figure 5.4 below:



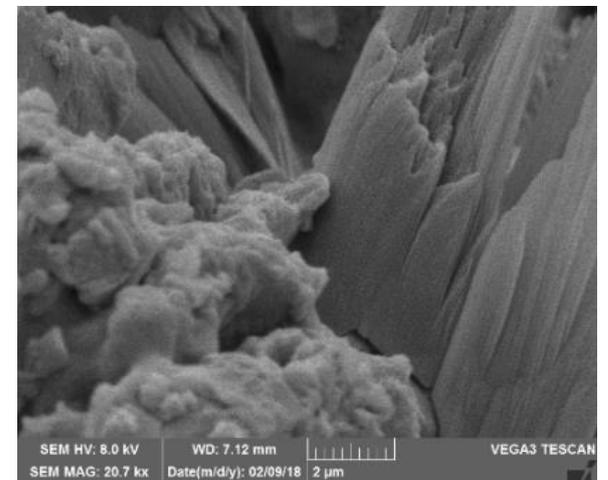
*Sample A1 – oedometer specimen treated by impregnation*



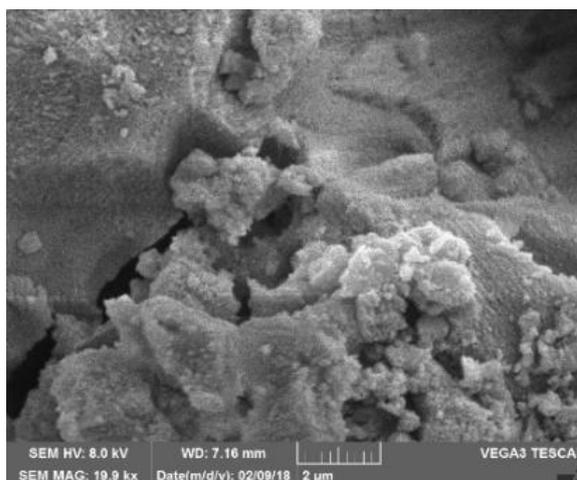
*Sample A1 – compressive test specimen treated by hand mixing*



*Sample A2 – compressive test specimen treated by impregnation*



*Sample B1 – oedometer specimen treated by impregnation*



*Sample C1 – oedometer specimen treated by impregnation*

*Figure 5.4 SEM Micrographs of specimens treated with sodium silicate solution ( $Si/Na=3$ ,  $M=4$ ) and oven dried*

As anticipated, the micrographs shown in the Figure 5.4 above appear to indicate that treatment by impregnation might have reasonably conserved the original structural framework of the specimens with the absorbed solution sticking to the surfaces of grains as well as filling micropores. Possibly, the crystalline calcite structure providing initial stability was retained and could likely positively contribute to the specimen post-treatment strength characteristics. It can be observed from micrographs of samples A1 and B1 that large voids were preserved after treatment, particularly in sample A1 with higher silt content. Solution can be seen forming menisci bonding at particle contacts, bridging them together. In specimens where initial large voids were minimal, the menisci bonding substance contributed to creating a dense mass structure, as is evident in samples C1 and A2. Clearly, the infilling of micropores by the silicate or its by-products will lead to a reduced porosity and permeability, thereby increasing strength and reducing collapsibility.

Treatment by mixing and kneading obviously destroyed the specimen's pre-treatment structure. The micrograph of a CT specimen of sample A1 treated by this method showed a different structural arrangement compared to that treated by impregnation, with a kind of dispersed appearance and somewhat even distribution of voids. Consequently, it is likely that in such specimens, strength would solely depend on the cementitious products resulting from the calcite-silicate reaction.

### **5.2.3 Stress-strain behaviour of samples treated by impregnation (capillary rise)**

Samples treated here were duplicates of the CT specimens tested under section 4.2 of chapter 4. No less than three specimens of each sample type were impregnated with a mean amount of absorbed solution for each batch given in Table 5.2. Since for each sample, a minimum of 12 specimens were required and the absorption container could only take a maximum of four specimens at a time, it was necessary to treat in batches. Thus, each treatment batch was used for each curing period to maintain some uniformity. There were discrepancies of absorbed proportions of solution ranging above 1% to about 10% for specimens treated (soaked) at different times. These errors were considered significant in some cases, thus, a setback for the study.

Table 5.2 Mean percent-absorbed treatment solution ( $Si/Na = 3, M = 4$  (\* $M=3.5$ )) for different sets of specimens and their curing time

Batch	Cured period	Mean percent absorbed solution (by mass of dry specimen)								
		A1	A2	A3*	B1	B2	B3*	C1	C2	C3*
1	7 days	53.1	39.0	19.9	50.4	53.4	20.6	50	35.8	45.3
2	14 days	57.8	52.3	41.6	54.7	53.4	46.3	53.6	53.1	48.3
3	28 days	57.8	52.3	42.8	50.4	53.4	39.7	57.8	53.1	48.3
4	48 hrs oven dried	63.0	52.2	49.3	57.8	55.3		55.3	54.4	50.8

The stress-strain relationships and summary of mean strengths (a maximum standard deviation of 0.3) and stiffness characteristics of at least three specimens for each curing period are presented as Figure 5.5 to Figure 5.7 and Table 5.3, respectively. Photographs showing failure patterns are presented in Appendix A10.

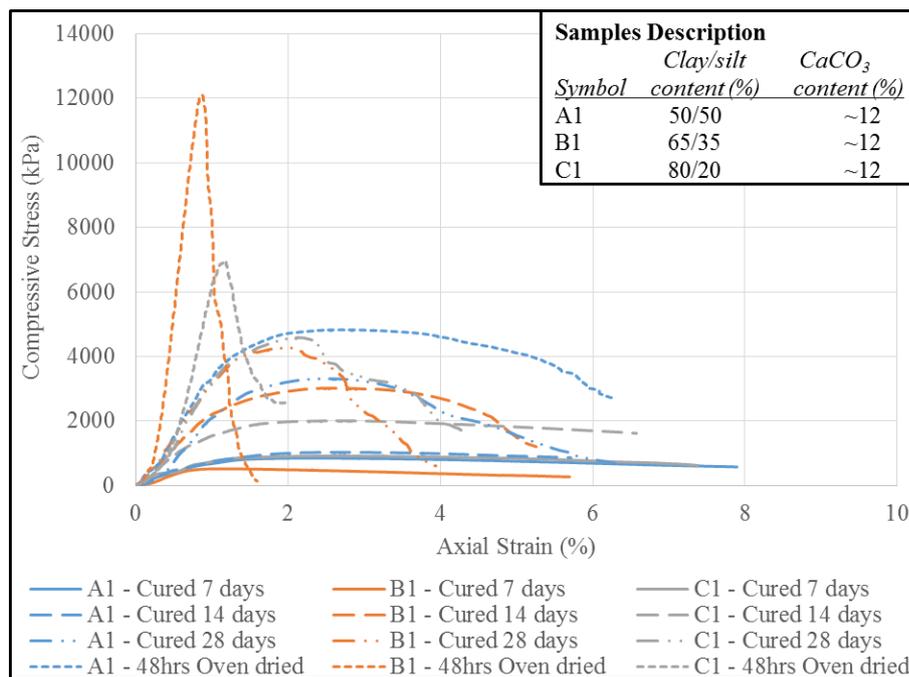


Figure 5.5 Stress-strain relationship of moderately calcareous samples impregnated with  $Si/Na=3, M=4$  solution, cured under room ambience for 7, 14 and 28 days, or oven dried 48 hrs under  $45-50^{\circ}C$

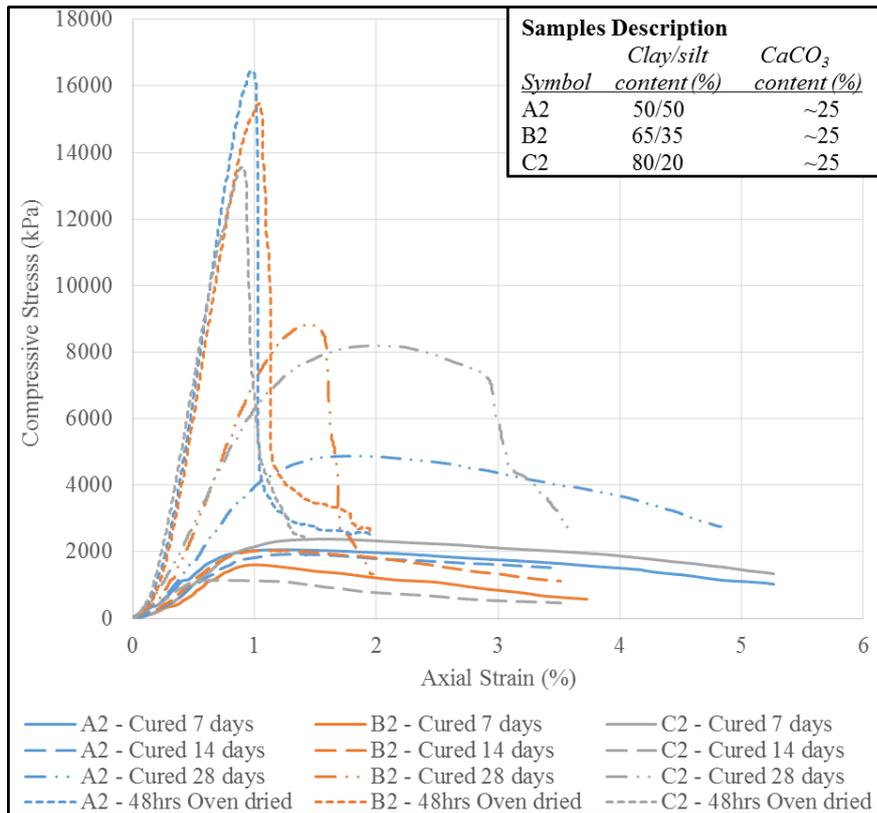


Figure 5.6 Stress-strain behaviour of strongly calcareous samples impregnated with Si/Na=3, M=4 solution, cured under room ambience for 7, 14 and 28 days, or oven dried 48 hrs under 45-50 °C

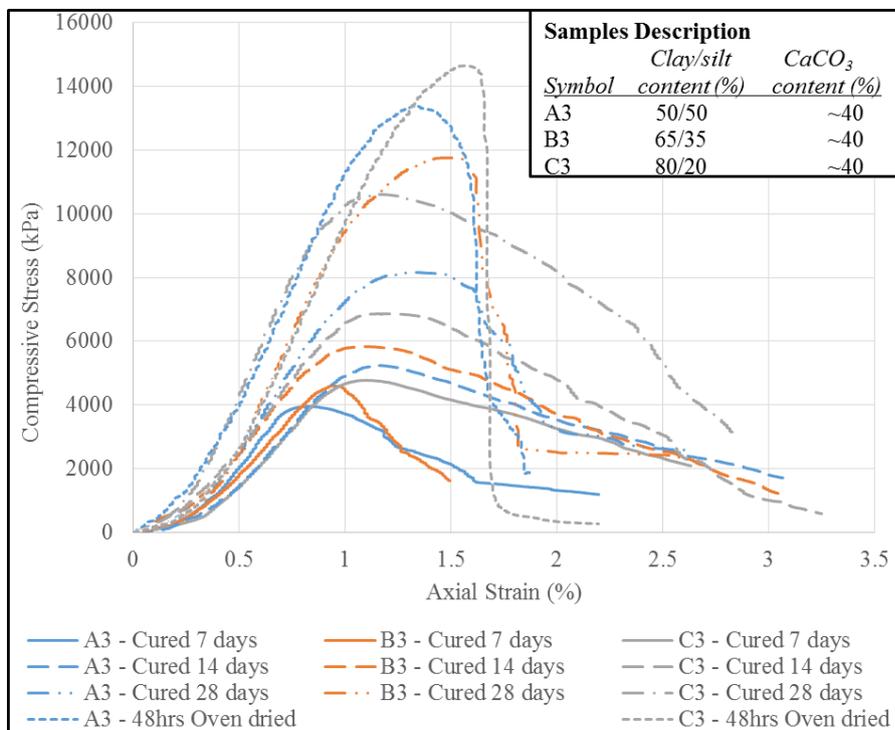


Figure 5.7 Stress-strain behaviour of very strongly calcareous samples impregnated with Si/Na=3, M=3.5 solution, cured under room ambience for 7, 14 and 28 days, or oven dried 48 hrs under 45-50 °C

Table 5.3 Mechanical properties of the calcareous silty-clay samples under unconfined compressive strength testing

Sample	Curing period	unconfined compressive strength (kPa)	Strain at peak stress (%)	Stiffness (MPa)
A1	7 days	853	2.3	0.9
	14 days	1041	2.6	1.1
	28 days	3314	2.5	2.6
	24 hours oven drying	4822	2.7	3.6
A2	7 days	2064	1.2	2.9
	14 days	1921	1.3	2.9
	28 days	4885	1.8	5.4
	24 hours oven drying	16460	1.0	25.0
A3	7 days	3949	0.8	8.9
	14 days	5246	1.2	7.7
	28 days	8167	1.3	10.3
	24 hours oven drying	13414	1.3	14.3
B1	7 days	523	1.2	1.2
	14 days	3033	2.6	2.9
	28 days	4256	1.9	4.3
	24 hours oven drying	12100	0.9	20.7
B2	7 days	1608	1.0	2.7
	14 days	2047	1.1	3.2
	28 days	8837	1.5	9.1
	24 hours oven drying	15484	1.0	21.7
B3	7 days	4603	0.9	7.1
	14 days	5846	1.1	8.6
	28 days	11756	1.5	10.0
	24 hours oven drying	-	-	-
C1	7 days	935	3.3	0.7
	14 days	2003	2.6	1.7
	28 days	4596	2.1	4.2
	24 hours oven drying	6959	1.2	10.0
C2	7 days	2381	1.6	3.3
	14 days	1135	0.7	3.3
	28 days	8205	2.0	7.0
	24 hours oven drying	13568	0.9	20.0
C3	7 days	4772	1.1	7.1
	14 days	6866	1.2	8.0
	28 days	14646	1.2	14.3
	24 hours oven drying	10598	1.6	14.0

Curves of both the treated *moderately* calcareous specimens (Figure 5.5) and *strongly* calcareous specimens (Figure 5.6) show that curing time up to 28 days still appear inadequate to define definite failure peaks. Notwithstanding the increase (nominal in some cases) in both UCS and stiffness with curing time, specimens continued demonstrating a ductile behaviour with curing time up to 28 days. Stress-strain curves showing broad peak surfaces. This being most conspicuous in younger specimens where cracking was nearly invisible as compressed specimens extensively bulged and yielded large strains (see Appendix A10). Testing was ended when minor fractures were observed.

On the other hand, *very strongly* calcareous specimens of all ages demonstrated stiffer behaviour with defined peaks (Figure 5.7) compared to the *moderately* and *strongly* calcareous specimens of similar ages. However, it is worthy of note that these *very strongly* calcareous specimens were impregnated with a lower solution concentration (3.5M). Again, the solution did not permeate more than about 1 mm depth of these specimens, only forming a thick gelatinous layer around specimen.

The behaviour of all heat-cured specimens evolved to a strain-hardening behaviour akin to soft rocks (Akai, 1977 cited in Adachi (1981); Vaughan (2009) ). However, failure shear planes were not identifiable on ruptured specimens. Surprisingly, on average, *moderately* and *strongly* calcareous samples appeared to be more brittle than their *very strongly* calcareous counterparts.

There was an assumption that pre-treatment bond strength could influence post-treatment strength because impregnation was not destructive of the original inter-particle connections. This implied that stronger untreated specimens should remain stronger post-treatment. However, in chemical soil treatment schemes post-treatment characteristics are usually influenced by the chemical interactions and resulting bonding products. Thus, the strength development after treatment with sodium silicate solution may be dependent on the amount of cementitious calcium silicate produced, which is also a function of the amount of absorbed solution and the availability of calcium ions in the system (Hurley and Thornburn, 1972).

Because of the differences in the concentration of solutions used to treat the *very strongly* calcareous sample and others, it may not be logical to compare their results. Nevertheless, Table 5.3 reveals an increase in both UCS and stiffness with an increasing degree of calcareousness for each curing period irrespective of the initial proportion of absorbed solution. However, there appears to be a better performance decreasing degree of calcareousness particularly with older and heat-cured specimens. For instance, at 28 days curing samples with ~12% CaCO<sub>3</sub> (A1, B1 and C1) showed a strength development of between 430% – 620% post-treatment; those with

~25% CaCO<sub>3</sub> (A2, B2 and C2) revealed strength growths ranging 212% – 327% while those containing ~40% CaCO<sub>3</sub> (A3, B3, and C3) grew just 139% – 242%.

The impact of this treatment strategy on improving strength may be interpreted as more favourable to samples with low calcite content. This seemingly lesser effectiveness on the *very strongly* calcareous samples may be attributable to the lesser concentration of their treatment solution. The shallow penetration of solution would also have resulted in strength increasing only in the outer parts of these *very strongly* calcareous specimens. Another probability is that effect may be related to the ability of samples to grow additional strength in relation to their levels of compaction.

Strength evolution post-treatment was simply considered to result from curing of the cementing gel since chemical evolution was not investigated. The findings here also reveal an increasing strength and stiffness with increasing curing time. However, 24 hours heat-curing resulted in greater strength and stiffness than even the 28 days curing period, indicating that strength evolution was merely a function of the degree of dryness and not necessarily a chemical process during curing. However, it is also a possibility that heat-curing could have enhanced the cementing reaction. This trend may be better appreciated where moisture retained after curing is related to UCS obtained, as illustrated in Figure 5.8 below.

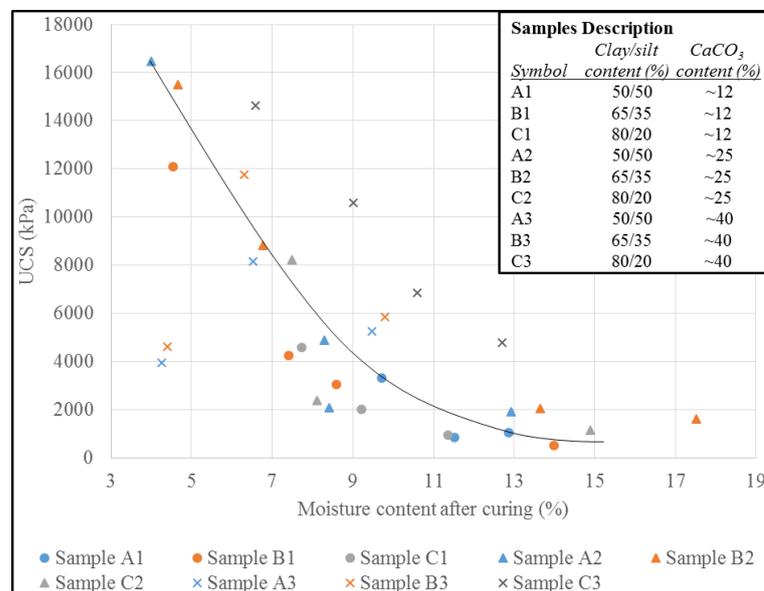


Figure 5.8 Relationship between moisture retained after curing and UCS

It is worthy of note that because of the uncontrollable nature of this treatment method, some identical specimens of the same samples have in some cases absorbed incomparable amounts of solution. In such cases, a lower retained moisture content was not necessarily an

indication of drying and such might as well yield lower UCS. An alternative treatment method was necessary to understand the impact of moisture content on UCS, thus section 5.2.4 below.

#### 5.2.4 Stress-strain behaviour of samples treated by mixing and kneading

For the purpose of controlling the amount of solution used, treatment by mixing & kneading was performed on duplicate *moderately* calcareous specimens (A1, B1, and C1) reported in section 5.2.3. Only these specimens were friable enough to be broken up as necessary for the mixing & kneading process. Those specimens containing higher calcite contents were relatively strong and not suitable for breaking and mixing.

Consequently, the *moderately* calcareous CT specimens were broken up, mixed with different proportions of the sodium silicate solution ( $\text{Si/Na} = 3$ ,  $M = 4$ ) and then remoulded into their original dry density. 30, 40, 50 and 60 % solution by mass of dry specimen were used. Treated samples were remoulded and then cured under ambient conditions for 7, 14 and 28 days before being subjected to compressive testing as described under section 3.5.3. For comparison sake, a few specimens were also prepared from a non-calcareous sample (C0) to get an insight of the potential impact of calcite. 30 and 40 % solution were adopted here, and the treated C0 then moulded into CT specimens with identical density as the C1 and cured for only 14 and 28 days.

Specimens mixed with 60% solution appeared too wet to be remoulded into neither a reasonable comparable density nor a consistent specimen. Remoulding was difficult because of excessive pore water pressures, which would not dissipate due to the high viscosity of the solution. Squeezing of material through the splits in the moulds was common during compaction, producing specimens with inconsistencies. Even though compressive testing was also performed on these specimens, it was considered illogical to compare their stress-strain characteristics with others. However, their stress-strain curves are presented together with the others in Appendix A11.

The stress-strain relationships of the calcareous specimens treated with 30, 40, and 50 % solution are presented as Figure 5.9 to Figure 5.12. Curves are grouped based on curing time. Figure 5.12 compares the treatment effect on the non-calcareous sample (C0) and its calcareous counterpart (C1), where  $\text{Na}_2\text{SiO}_3$  capacity to enhance the strength of clays even in the absence of calcite is demonstrated. However, the treatment performed more effectively, and better engineering characteristics were achieved where calcite was present. Similarly, curing time progressively stiffens and increases strength in the non-calcareous silty-clay. Table 5.4 is a

summary of mean strength and stiffness characteristics of all samples while photographs showing their failure patterns due to the compression are presented in Appendix A11.

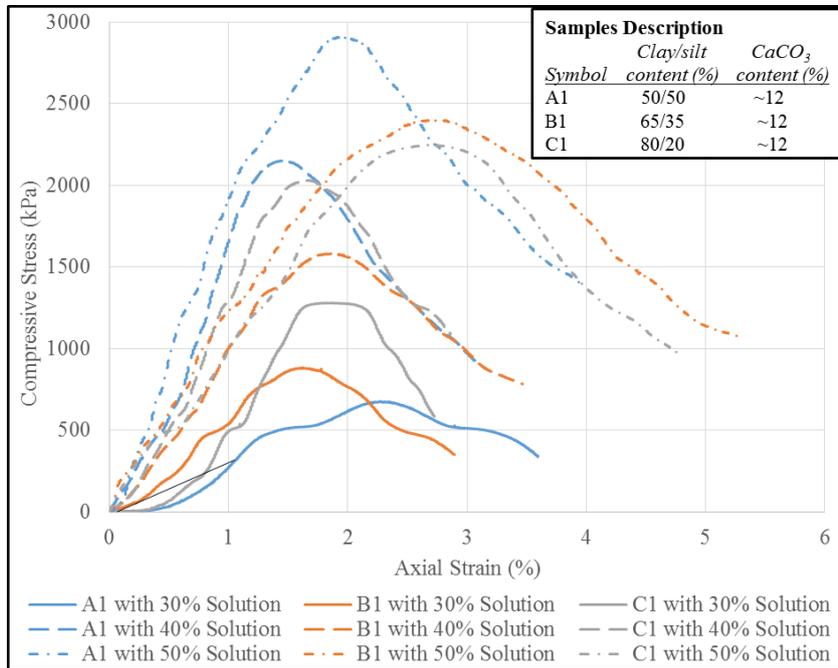


Figure 5.9 Stress-strain relationship of different moderately calcareous samples mixed with varying proportions of a  $\text{Na}_2\text{SiO}_3$  solution and cured 7 days

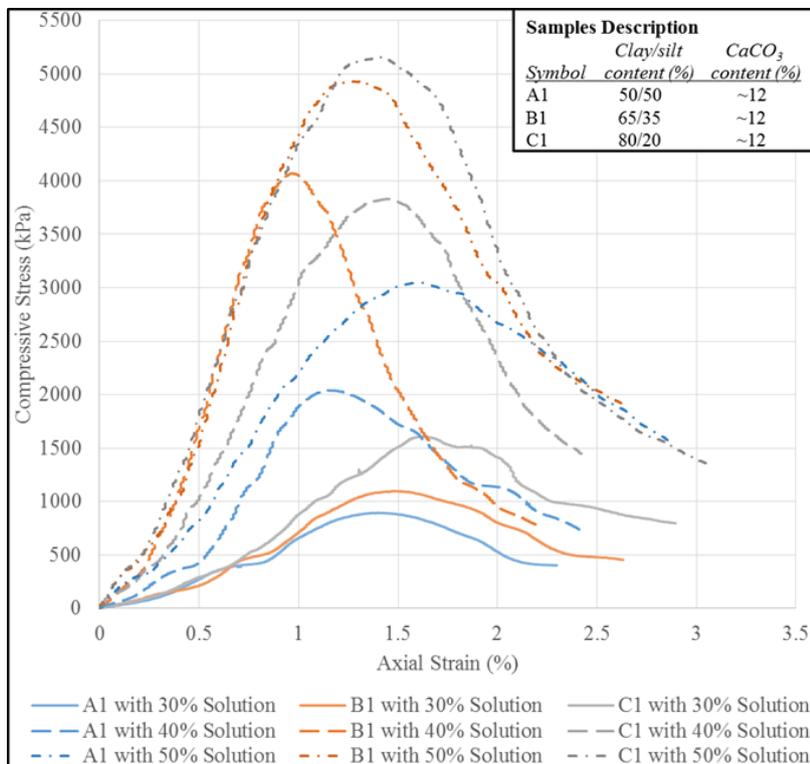


Figure 5.10 Stress-strain relationship of different moderately calcareous samples mixed with varying proportions of a  $\text{Na}_2\text{SiO}_3$  solution and cured 14 days

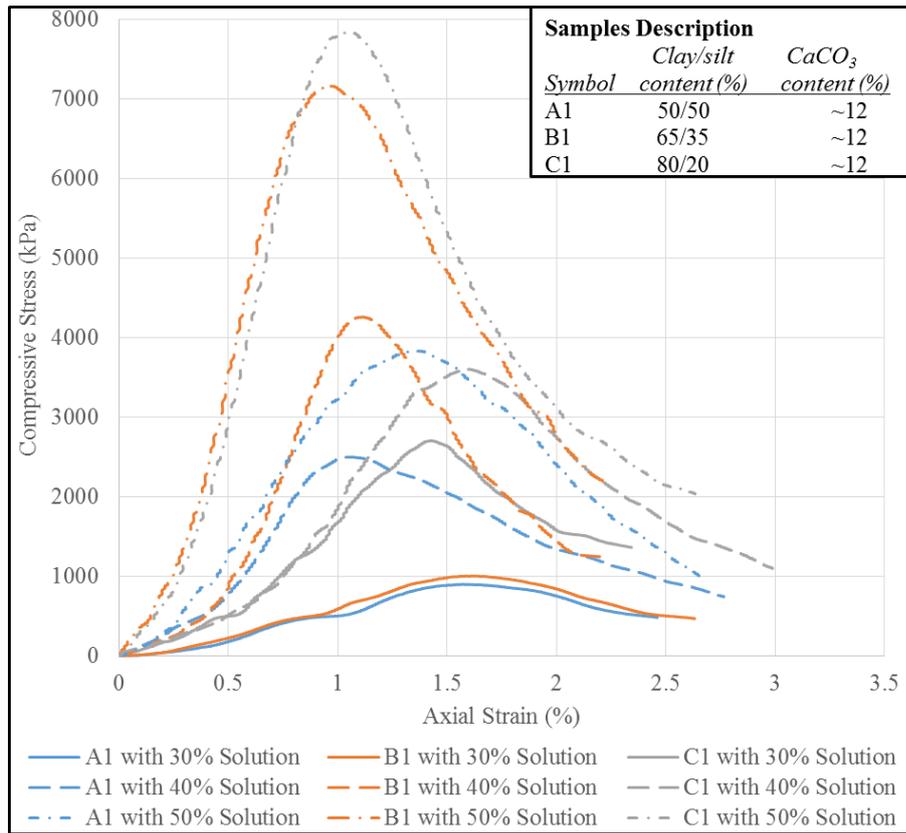


Figure 5.11 Stress-strain relationship of different moderately calcareous samples mixed with varying proportions of a  $\text{Na}_2\text{SiO}_3$  solution and cured 28 days

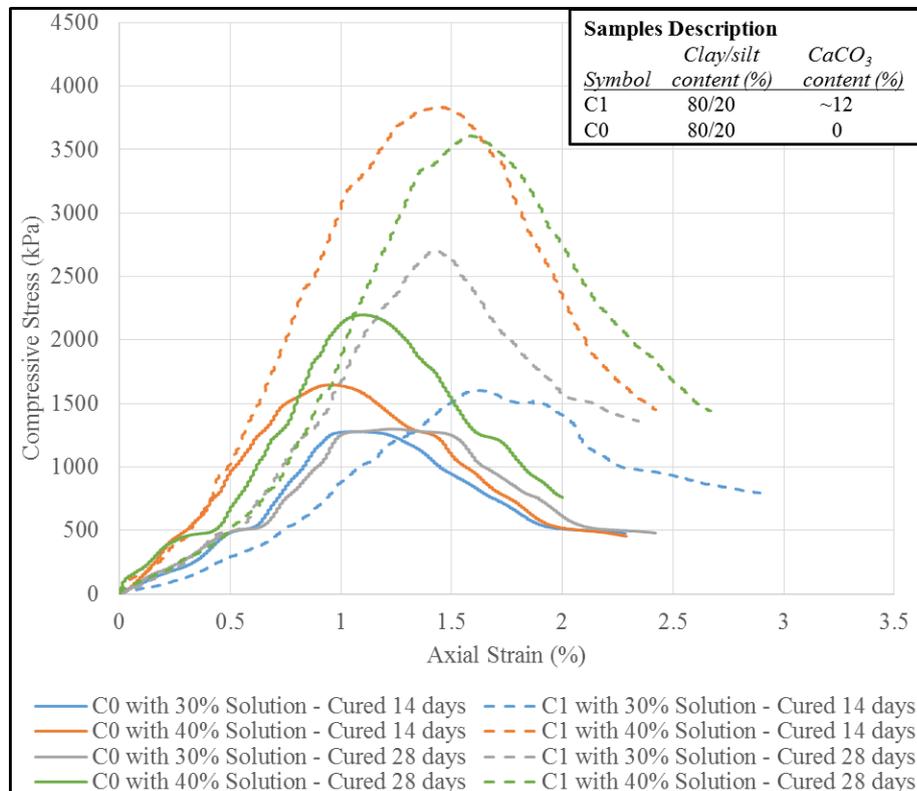


Figure 5.12 Stress-strain relationship of a non-calcareous and moderately calcareous samples mixed with two different proportions of  $\text{Na}_2\text{SiO}_3$  solution and cured 14 and 28 days

At each of the curing periods as in Figure 5.9, Figure 5.10 and Figure 5.11, values of peak compressive stresses summarized in Table 5.4 are seen to be increasing with increasing amounts of applied solution. However, up to 60% solution (results in Appendix A11), peak stress values declined drastically, especially in younger specimens. Nevertheless, aged (28 day) specimens showed high values where in one case, a higher peak stress than that recorded for 50% solution was recorded. However, due to the inconsistency of these 60% solution specimens, their UCS results were considered unreliable to be compared to others, just as their stress-strain curves demonstrated a broad peak with some undulating behaviour.

On the bases of strength, 50% solution can be recognized as the threshold content for effective improvement of these *moderately* calcareous soils. While at early (7 day) curing, all levels of treatment appeared to produce stress-strain curves with broad peaks, curing time progressively dried and stiffened specimens resulting in steeper peaks and higher UCS values (see section 5.3 below). However, curing time was not observed to significantly influence either strength or stiffness of specimens treated with 30% solution, except in an isolated case of sample C1 at 28 days curing. Notwithstanding this, treatment with higher amounts of solution produced higher post-cured moisture contents but with greater UCS values as can be seen in Figure 5.13 and Figure 5.14.

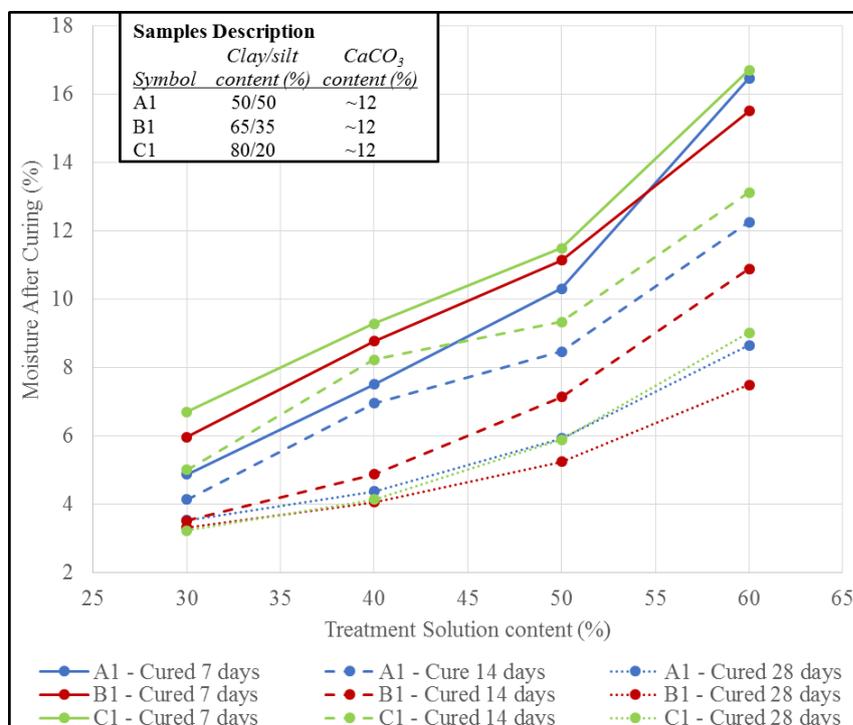


Figure 5.13 Relationship between amount of treatment solution and retained moisture content after curing

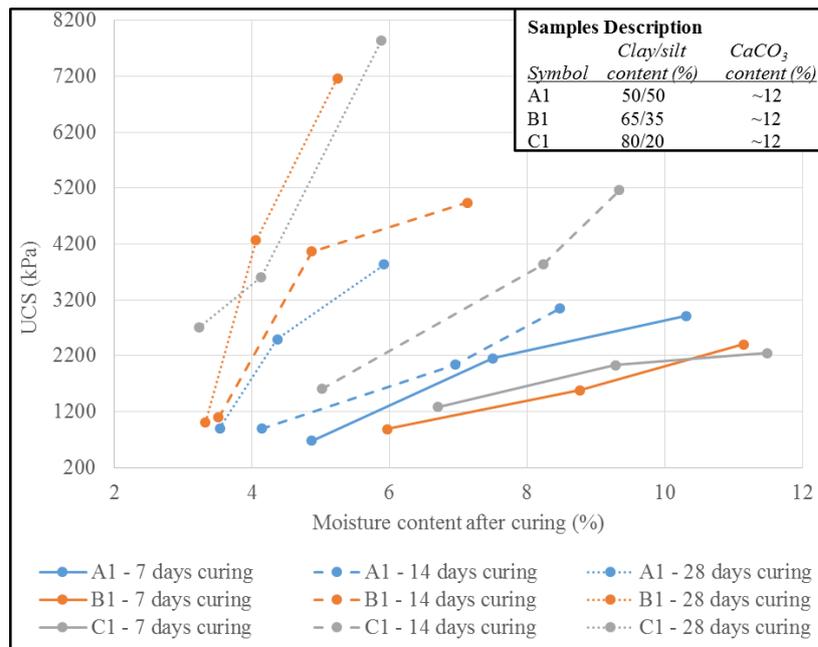


Figure 5.14 Relationship between retained moisture content after curing and UCS

Because of the method of treatment (mixing and kneading), soil macropeds appeared to develop. Thus, bonding was assumed to occur both between particles and macropeds. The deformational characteristics under compression observed from the photographs presented as Appendix A11 revealed the following for each level of treatment.

The treatment with 30% solution did not appear to substantially improve the engineering properties of specimens, such as bonding strength as can be seen from the failure patterns and relatively low UCS values. Irrespective of curing time, especially for samples A1 and B1, failure was observed in most cases to originate from both inter-particle and inter-macroped disintegration as indicated by the friability of the material showing little or no adhesion. A few small wedges also accompanied the friable failure, which occurred mainly at either top or bottom of the specimens. However, sample C1 at 14- and 28-days curing had failures originating through surface spalling that progressed inwards. The 14-day old C1 spalled near the middle while the 28-day olds spalled near an end platen, evolving into a somewhat cone-shaped failure.

Treatment with both 40% and 50% solution produced a considerable measure of adhesion between particles and between macropeds as demonstrated by the UCS values and failure patterns. Failure patterns of these specimens appeared similar irrespective of curing periods, forming a rough cone-like shape, which resulted from propagated shear cracks and wedging around one end of the compressed specimens. Cracks have also propagated longitudinally across the specimens but without a shear plane (seemingly tensile cracks). It is worth noting that specimens with 50% solution appeared to bulge slightly at early stages of compression.

This influenced their stress-strain curves, resulting in lower stiffness values in many younger specimens compared to those treated with 40% solution, as presented in Table 5.4.

*Table 5.4 Mechanical properties of the treated (by hand mixing) calcareous silty-clay samples under unconfined compressive testing*

Sample	% mass of solution applied (by mass of dry soil)	Curing period (days)	Peak unconfined compressive stress (kPa)	Strain at peak stress (%)	Stiffness (MPa)
A1	30	7	676	2.3	0.4
		14	894	1.4	0.8
		28	902	1.6	0.8
B1		7	883	1.6	0.7
		14	1098	1.5	0.8
		28	1007	1.6	0.7
C1		7	1278	1.9	1.2
		14	1605	1.6	1.3
		28	2707	1.4	2.7
C0		14	1279	1.1	2.1
		28	1301	1.2	2.0
A1		40	7	2151	1.5
	14		2041	1.1	2.7
	28		2495	1.1	4.0
B1	7		1582	1.9	1.1
	14		4067	1.0	6.0
	28		4266	1.1	7.0
C1	7		2028	1.7	1.7
	14		3832	1.5	3.5
	28		3606	1.6	3.8
C0	14		1648	1.0	2.2
	28		2200	1.1	3.2
A1	50		7	2914	1.9
		14	3046	1.6	2.6
		28	3836	1.4	4.2
B1		7	2405	2.8	1.1
		14	4938	1.3	6.0
		28	7168	1.0	11.3
C1		7	2245	2.7	0.9
		14	5161	1.4	5.0
		28	7843	1.1	11.7

### 5.3 Factors Affecting UCS and Stiffness of the Treated Samples

Though there is much report about the effectiveness of sodium silicate application in soil improvement, particularly for soils containing  $\text{Ca}^{2+}$  ions, there is not enough data in the literature in order to compare and understand these results. Therefore, a thorough analysis of all the factors that could affect the cured strength and stiffness of the material have been conducted and examined in the following sections.

#### 5.3.1 Effect of sample preparation method on UCS and stiffness

Two treatment methods were applied: mixing & kneading and impregnation by capillary rise. In the mixing & kneading method, 30, 40, and 50 % solution (by mass of dry soil) were used and only the *moderately* calcareous samples (A1, B1, and C1) could be treated by the method. On the other hand, impregnation involved absorption of relatively uncontrollable amounts of solution by nearly all the *moderately*, *strongly*, and *very strongly* calcareous samples. This may affect the consistency of specimens, thus, a major drawback for repeatability. Impregnation of a bulk sample from which specimens are cored could have improved consistency but it was difficult to achieve that because of absorbability issues.

Therefore, concerns were raised as to whether it was logical to compare results of the two different treatment techniques owing to the uncontrollable amounts of solution involved in one relative to the other. Bearing this in mind however, an attempt may be possible to compare the mix & kneading specimens of 50% solution with the impregnated *moderately* calcareous specimens, which absorbed a range of 50 to 58 % solution.

The two treatment techniques revealed significant variations in UCS and stiffness values when compared. It was anticipated that pre-treatment bonding could possibly enhance the post-treatment bonding strength development. That is to say, the non-destructive method (impregnation) could better improve samples' engineering properties compared to the destructive method of mixing and remoulding. Surprisingly, Figure 5.15 and Figure 5.16 proved to the contrary.

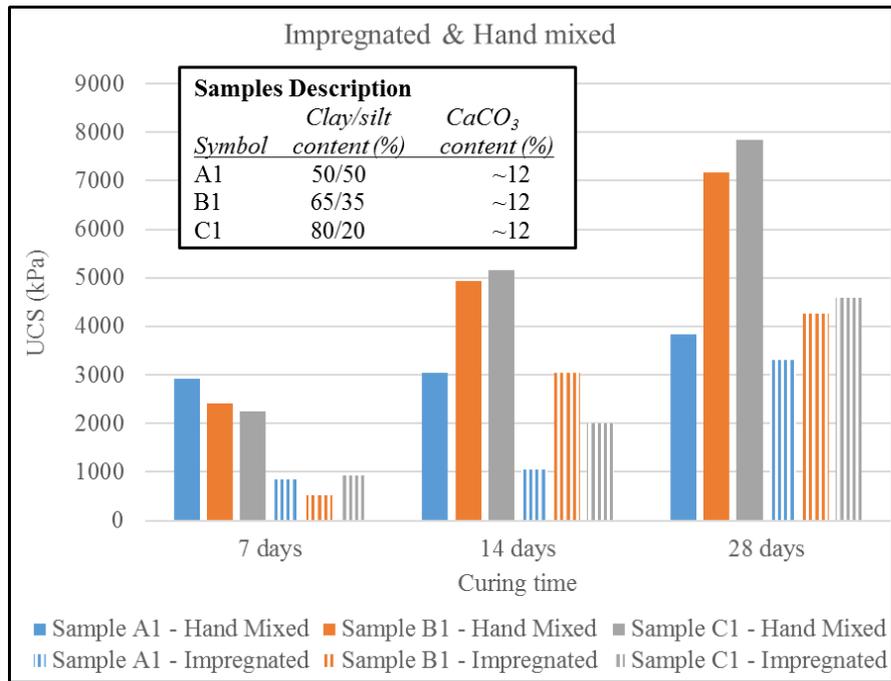


Figure 5.15 The influence of sodium silicate treatment method on unconfined compressive strength

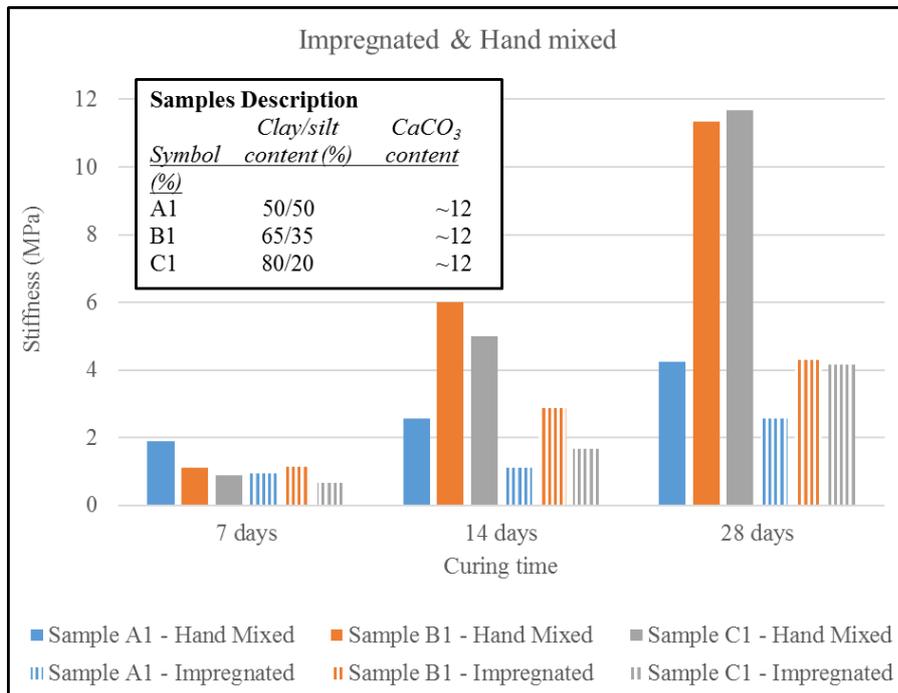


Figure 5.16 The influence of sodium silicate treatment method on stiffness

It is clear from the Figures above that at all ages, each sample type showed better enhanced UCS and stiffness resulting from the mixing & kneading method compared to the other. Two factors may be used to explain the reason for this occurrence:

- (1) It is true that polymerization/gelation of silicate solution depends highly on the amount of silica. Therefore, development of strength at all potential bonding sites within treated sample will depend on available silica to such bonding site. In calcareous clays with reduced permeability, it is likely that silica in the silicate solution could be ‘filtered out’ during the absorption process, starving specimen inner locations of silica and subsequent bond development. This could produce specimens that are strong around their outer layers but weaker within. Alekseev and Lipson, 1974 also recognized and referred to the silica filtering process as “mechanical absorption” but Sokolovich (1976) criticized this process noting that sodium silicate solution by nature is nearly a true solution. That is, silica cannot be separated (or filtered) from a silicate solution.
- (2) Mixing probably provides a more even distribution of treatment solution within sample. Thus, an even bonding system that is coherent was achieved, which is likely to produce greater compressive strength than an uneven bonding system. Sokolovich (1976) reported similar findings in an investigation of the impact of grouting and mixing in the silicization of loessial soil. Although Sokolovich acknowledged deficiencies in the experimental parts of the study, they identified structureless samples treated by mixing exhibiting highest strengths resulting from optimal distribution of the binder material.

### 5.3.2 Effect of calcite content on post-treatment strength and stiffness

It can be seen from the Figure 5.17 and Figure 5.18 (UCS) and Figure 5.19 and Figure 5.20 (stiffness) that  $\text{CaCO}_3$  presence in the soil-silicate systems has unarguably enhanced the effectiveness of the treatment. However, it is also evident that clays devoid of  $\text{CaCO}_3$  can also be bonded by  $\text{Na}_2\text{SiO}_3$  solution. This is consistent with Hurley and Thornburn (1972) standpoint that  $\text{Na}_2\text{SiO}_3$  solution has the potential to agglomerate any inorganic soil material but with the gel properties greatly depending on factors such as type of salts, acids or bases available in the reaction,  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio, concentration of solution, and temperature. Investigations by the Scientific-Research Institute of Foundations, on the effectiveness of silicization on carbonate and non-carbonate clayey soils have been reported in Sokolovich (1976) and findings also confirming silicization to better stabilize carbonate soils than non-carbonate ones.

Despite  $\text{Na}_2\text{SiO}_3$  capability to provide adhesion for any inorganic material, the present results corroborate the benefit of the  $\text{Ca}^{2+}$  ions in the exchange reaction that leads to the formation of a stronger cement ( $\text{CaSiO}_3$ ).

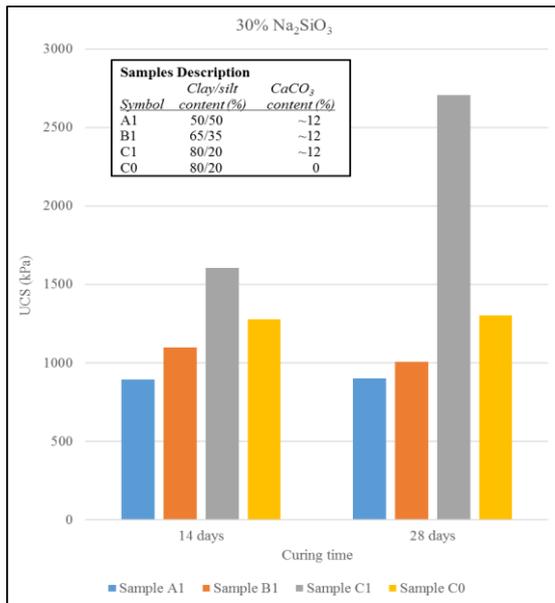


Figure 5.17 Impact of calcite presence on UCS of samples mixed with 30% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

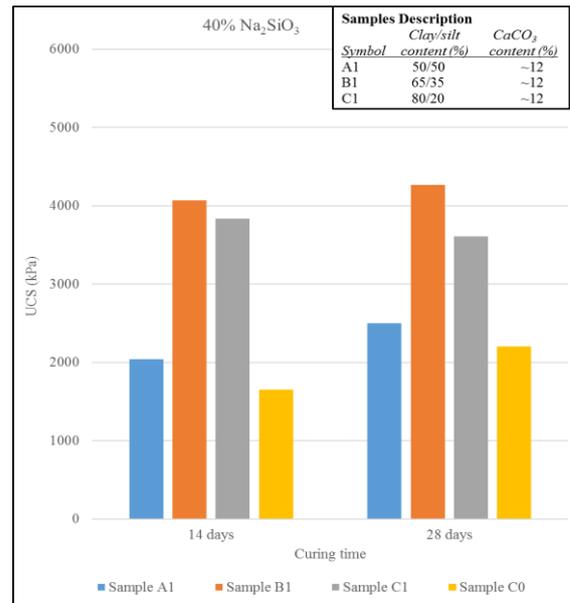


Figure 5.18 Impact of calcite presence on UCS of samples mixed with 40% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

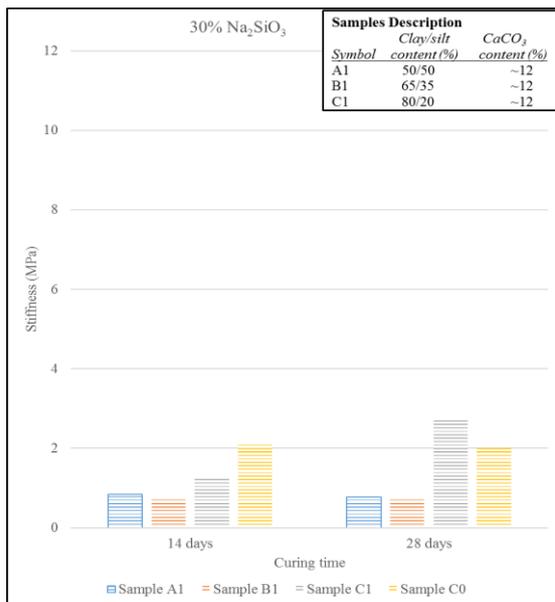


Figure 5.19 Impact of calcite presence on the stiffness of samples mixed with 30% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

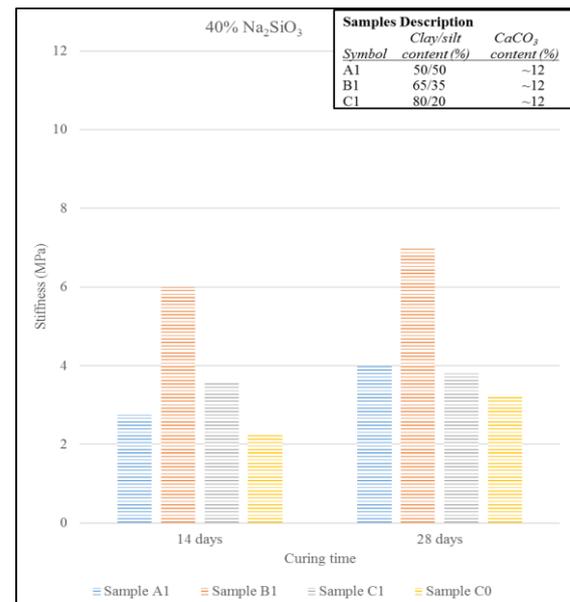


Figure 5.20 Impact of calcite presence on the stiffness of samples mixed with 40% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

Generally, treatment resulted in increases in peak UCS of specimens irrespective of their calcite content. Furthermore, as illustrated in Figure 5.21 to Figure 5.23, at all ages and curing methods, post-treatment UCS values appear higher with higher calcite contents except in few cases were lower values are recorded. This increase in UCS with increasing calcite content is akin to what was observed with the untreated samples. The cause for the drops in UCS, which occurred in few cases of the impregnated samples, was thought to be related to the amount of absorbed solution.

Because treatment by impregnation did not allow good control of the amount of solution absorbed by each specimen, some level of discrepancies was expected in those results. Each set of specimens absorbed significantly differing amounts of solution compared to others. The mean absorbed amounts (with variations ranging above 1 – 10 %) for each set of specimens are presented in Table 5.2. This undoubtedly, might have affected the UCS results.

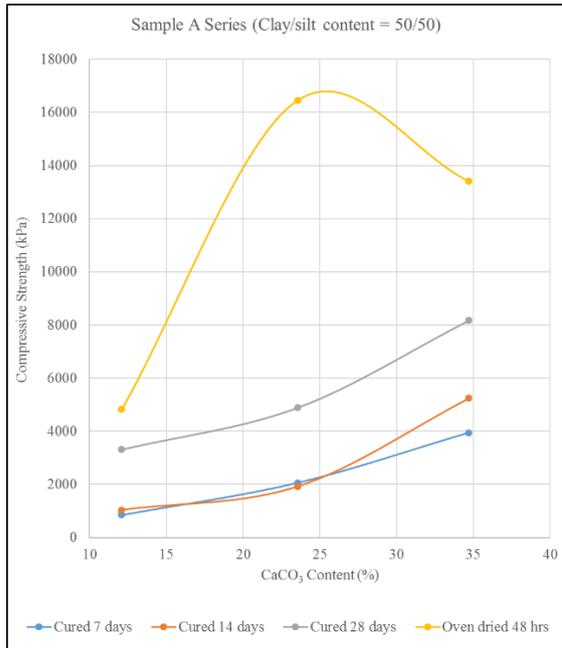


Figure 5.21 Impact of calcite content on the effectiveness of  $\text{Na}_2\text{SiO}_3$  treatment (impregnation) of samples.

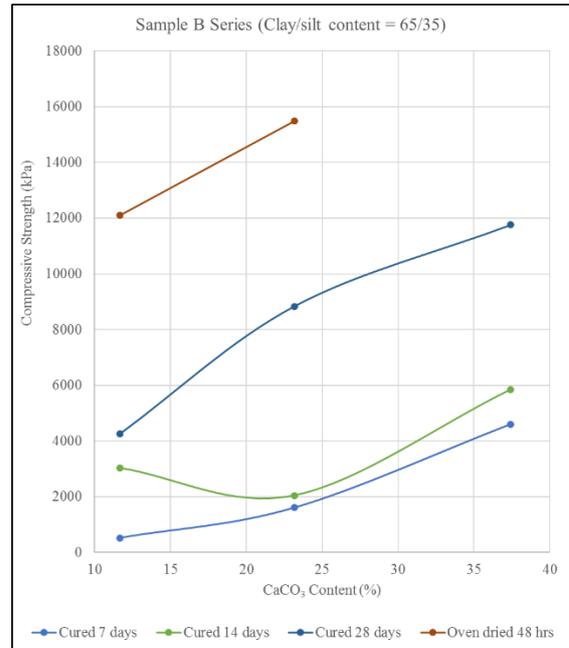


Figure 5.22 Impact of calcite content on the effectiveness of  $\text{Na}_2\text{SiO}_3$  treatment (impregnation) of samples.

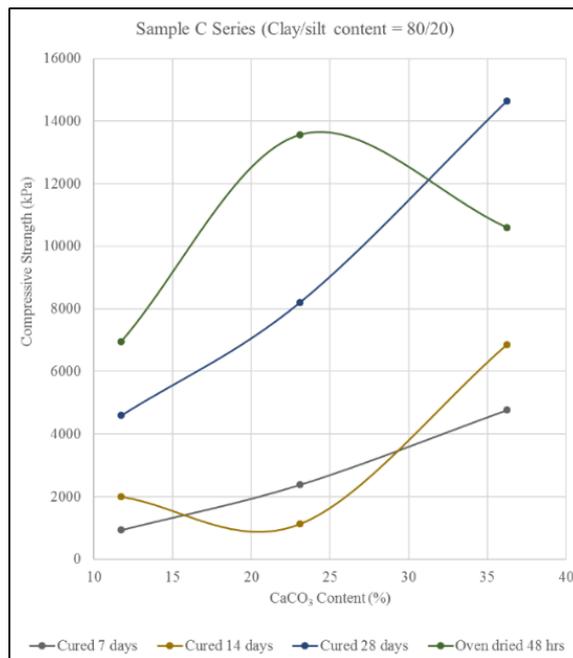
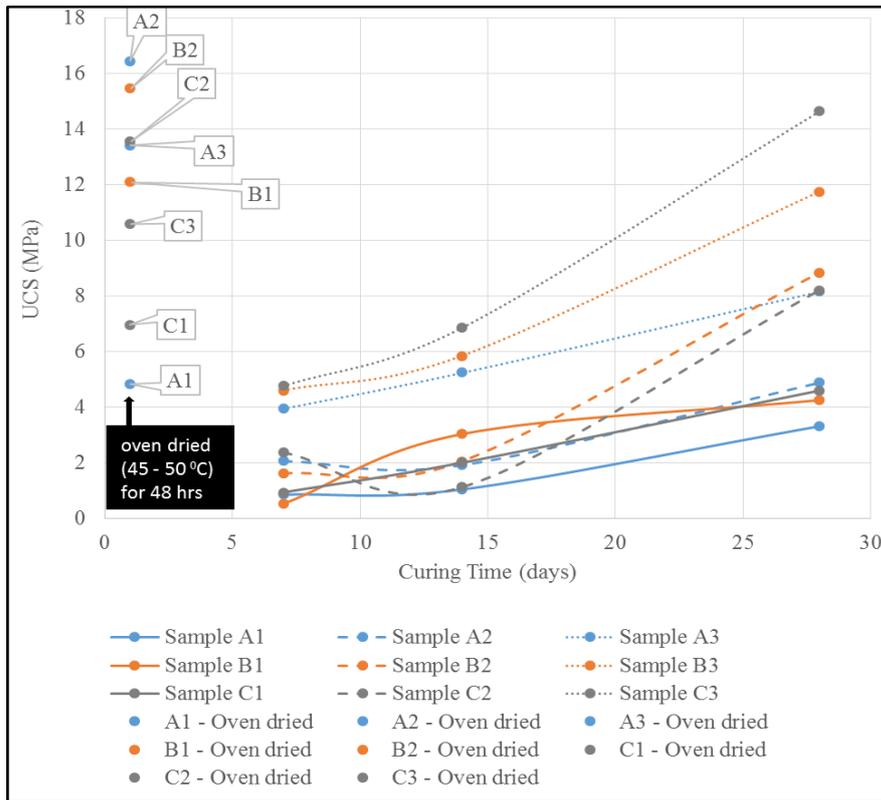


Figure 5.23 Impact of calcite content on the effectiveness of  $\text{Na}_2\text{SiO}_3$  treatment (impregnation) of samples.

According to McDonald and Thompson (2006), when an agglomerated material containing multivalent metal ions (e.g.  $\text{Ca}^{2+}$ ) reacts with aqueous silicates, an equivalent insoluble metal silicate cement is precipitated almost instantly. Hurley and Thornburn (1972) also reported that the strength of a silicized soil depends amongst other factors, on the amount of cementitious material produced, which is determined by the amount of absorbed solution and the availability of metal ions (e.g. calcium) in the soil. However, it is still unclear whether the discrepancies in amount of absorbed solution significantly affected the post-treatment strength of the present specimens. Nearly in all soil series (A, B, and C), it was observed (see Table 5.2) that higher calcite contents resulted in low absorbed proportions of the solution. This may be attributed to: (1) specimen pore volume reduction with increasing calcareousness, and (2) higher calcite contents resulting in quicker gel formation and impairing later absorption by the specimen.

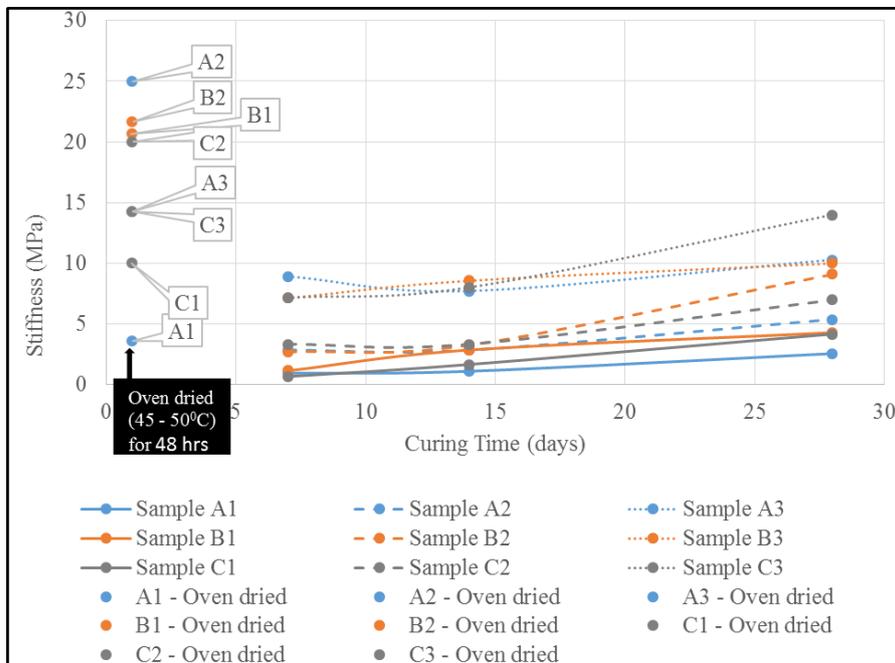
### **5.3.3 Effect of curing time and method on post-treatment UCS and stiffness**

The first set of examined specimens treated by both impregnation and mixing & kneading were cured under room ambience for periods of 7, 14, and 28 days. The moisture content retained post-curing directly related to the initial amount of applied solution and curing time (see Figure 5.13 for example). Even though, curing was not done in a strictly controlled room conditions, the nearly linear relationship between retained moisture content and amount of solution show that the curing ambient conditions did not widely influence the properties of samples. A second set of specimens only treated by the impregnation method were oven dried (or heat cured) under 45 – 50 °C. Figure 5.24 to Figure 5.28 below examine the impact of curing time on UCS and stiffness while Figure 5.24 and Figure 5.25 further compare the impact of the curing methods.



Symbol Description	
Symbol	Clay/silt content (%)
A	50/50
B	65/35
C	80/20
CaCO <sub>3</sub> Content	
1	~12%
2	~25%
3	~40%

Figure 5.24 Effect of curing method on the UCS of sodium silicate impregnated calcareous silty-clay samples



Symbol Description	
Symbol	Clay/silt content (%)
A	50/50
B	65/35
C	80/20
CaCO <sub>3</sub> Content	
1	~12%
2	~25%
3	~40%

Figure 5.25 Effect of curing method on the stiffness of sodium silicate impregnated calcareous silty-clay samples

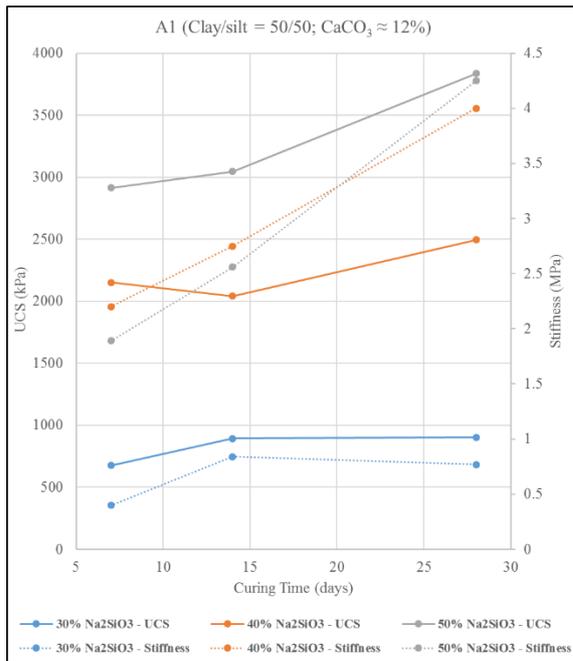


Figure 5.26 Effect of curing time on UCS and stiffness of sample A1

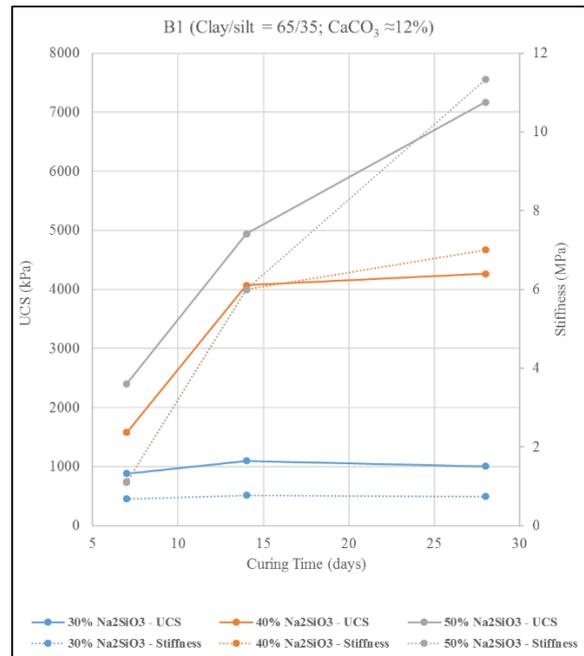


Figure 5.27 Effect of curing time on UCS and stiffness of sample B1

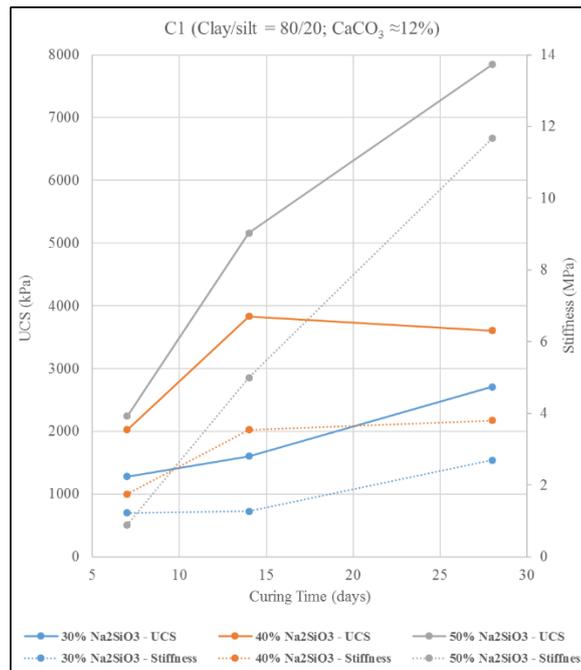


Figure 5.28 Effect of curing time on UCS and stiffness of sample C1

Generally, both UCS and stiffness improved with curing time. Increased curing time resulted in loss of moisture (see Figure 5.8 and Figure 5.14) and hardening, which gave rise to strength development and stiffening. Thus, heat curing having resulted in greater moisture loss provided higher UCS and stiffness values compared to room curing (Figure 5.24 and Figure

5.25). While this may be true, it is also possible that the higher temperatures will have favoured a cementing reaction.

For samples treated by impregnation, heat curing yielded average UCS values between approximately 5 – 12 MPa for *moderately* calcareous samples, and approximately 14 – 16 MPa for *strongly* calcareous and *very strongly* calcareous samples. Whereas, room curing times up to 28 days only yielded maximum UCS values of between 3 – 5 MPa for the former, and between 5 – 11 MPa for the latter. Surprisingly, at 7-days curing UCS values for the impregnated samples were only slightly higher than those of their untreated counterparts while stiffness values drastically reduced. Untreated calcareous specimens, which exhibited brittle behaviour, became ductile post-treatment irrespective of curing period. Only a few of the heat cured specimens still demonstrated brittle behaviour. It shows that treatment initially changes (weakens) the soil's bonding structure before subsequently developing stronger cementing bond with curing time or drying. Therefore, where quick soil strengthening is needed the curing process may require speeding up.

Even though, sodium silicate solutions are sticky in nature and get tackier upon drying with an adhesive capacity to hold soil particles together, the cementing effect of silicate solutions in soil improvement is still being debated. For instance Hurley and Thornburn (1972) believe that the strong bonding and strength development from a  $\text{Na}_2\text{SiO}_3$  solution treatment results from the hardening of an insoluble metallic silicate, calcium silicate ( $\text{CaSiO}_3$ ) cementitious gel precipitated from the cation exchange reaction between soil's  $\text{CaCO}_3$  and  $\text{Na}_2\text{SiO}_3$ . They went further to state that the polysilicate ions ( $\text{CaSiO}_3$  gel) are formed from the polymerization of the monomeric silicate ions of the highly alkaline  $\text{Na}_2\text{SiO}_3$  as the pH is lowered by the presence of the metal salt (e.g.,  $\text{CaCO}_3$ ). If this is true then calcium ions,  $\text{Ca}^{2+}$  from the present calcareous silty-clay soils will replace the sodium ion,  $\text{Na}^+$  of the  $\text{Na}_2\text{SiO}_3$  solution and attach themselves to the anions ( $\text{SiO}_3^{2-}$ ) of the silicate component to precipitate  $\text{CaSiO}_3$ .

Other views are slightly different. According to Weldes and Lange (1969); Iler (1979); McDonald and Thompson (2006), the cementing impact of  $\text{Na}_2\text{SiO}_3$  solution simply arise from the crosslinking of silica monomers forming polymer/gels round treated soil particles. This crosslinking process occurs as the solution dehydrates and silica concentration increases. From Weldes and Lange (1969)'s standpoint, the bonds formed by this process (i.e., by setting the silicate solution through a gelling action) are usually weaker than those gotten from drying of the silicate-soil system. Implying that drying is a quick means of strength development in silicized soils as polymerization requires longer periods of time depending on certain physical

and chemical conditions. This may explain why the present heat cured specimens appeared stronger than the room cured ones. However, in field conditions heat-curing, especially below the ground surface may not be practicable. This perhaps explains why many applications of sodium silicate treatment of soils have followed multi-step methods (i.e., the application of  $\text{Na}_2\text{SiO}_3$  solution and an additive) to enhance strength development.

Weldes and colleague further argue that because of the nature of some sodium silicates, their dry films can rehydrate and gradually dissolve. Therefore, heat-curing of those silicate films or bonds is not adequate to achieve complete insolubility. Iler (1979) holds similar views and claimed that bonds produced by polymerization (gelling action) have better moisture resistance. Suggesting, therefore, that for treatment of moisture sensitive soils (e.g. collapsible soils) polymerization process may be preferable to drying.

Further on the debate around drying and increased cementing strength of silicized soil, Figure 5.14 above suggests that drying in itself alone may not necessarily be responsible for strengthening of treated soils. The relationship between post-curing moisture content and UCS in the Figure 5.14 presented concerns about drying and strength direct linking. In that Figure, treated specimens with identical curing have rather revealed higher UCS with higher moisture condition. However, post-curing moisture content was directly related to the amount of applied solution (Figure 5.13). Possibly, the initial amount of solution applied has some influence on the development of bonding strength.

#### **5.3.4 Effect of quantity of solution applied on UCS and stiffness**

Traditionally, in soil treatment practice, for example, in soil-cement and soil-lime systems, increasing binder content results in better improved engineering properties such as compressive strength and stiffness. The experimental results presented here in Figure 5.29 and Figure 5.30 have also followed a similar trend for the  $\text{Na}_2\text{SiO}_3$  treatment. However, at a certain quantity of solution UCS and stiffness begin to decline. As illustrated in Figure 5.29, UCS improves with increasing mass of solution up to the 50% level but sharply diminishes at 60%. Even though, little has been discussed about the specimens treated with 60% solution, their quality suggested it unreasonable to compare those results with the others. For instance, moulding of specimens mixed with 60% solution produced specimens with inconsistent smoothness and some distortions because of excessive wetting (too much applied solution).

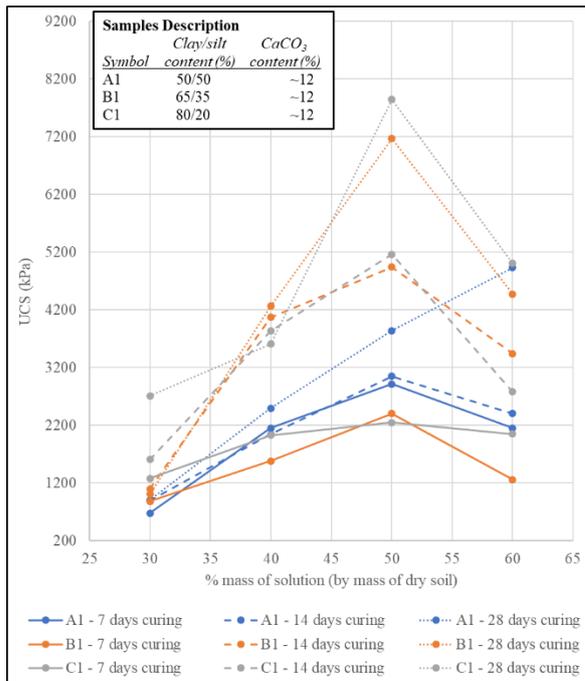


Figure 5.29 Relationship between % mass of solution (by mass of dry soil) applied and UCS

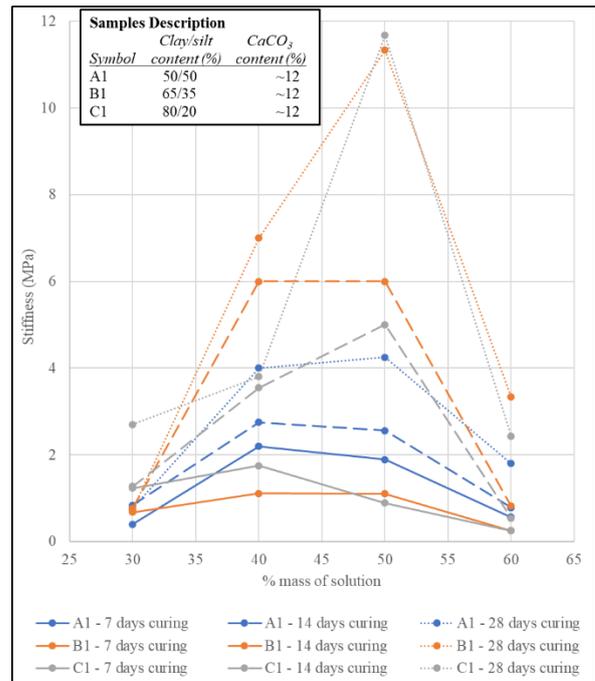


Figure 5.30 Relationship between % mass of solution (by mass of dry soil) applied and stiffness

As is obvious in Figure 5.29, the 50% solution recorded highest peak UCS in all soil types, except for an isolated case of sample A1 where a 60% solution produced greater peak UCS after 28 days curing. This notwithstanding, 50% solution can be regarded as the threshold or optimum for these soils. Like in the UCS, stiffness also showed increase with curing time for all levels of treatment, but stiffness did not grow with amount of solution beyond 40% in most cases. Stiffness growth however, continued up to the 50% solution level for specimens cured up to 28 days. Conversely, as earlier mentioned, younger (7- and 14-days) specimens rather revealed their highest stiffness values at 40% solution. Below and above this level they appeared more fragile.

Hamouda and Amiri (2014) directly related sol gel set time and strength. Consequently, since strength depends on gel setting, samples treated with very high amounts of solution could be expected to yield lower strengths than their counterparts treated with lesser amounts subjected to identical curing conditions. This is true because specimens with higher amounts of solution will normally take longer to expel their excess moisture.

The concentration of the metal ions (e.g., Ca<sup>2+</sup>) in a sodium silicate-soil system affects gel formation. Higher concentrations give rise to higher gelation (Krumrine and Boyce (1985) cited in Hamouda and Amiri (2014)). Effectively therefore, with very high amounts of solution such as the 60% used here, Ca<sup>2+</sup> concentration in the system would normally be reduced thereby delaying setting and gel precipitation. This is a possible reason why treatment with 60% solution produced lower UCS than the 40% and 50%. Silica content in the soil-silicate system



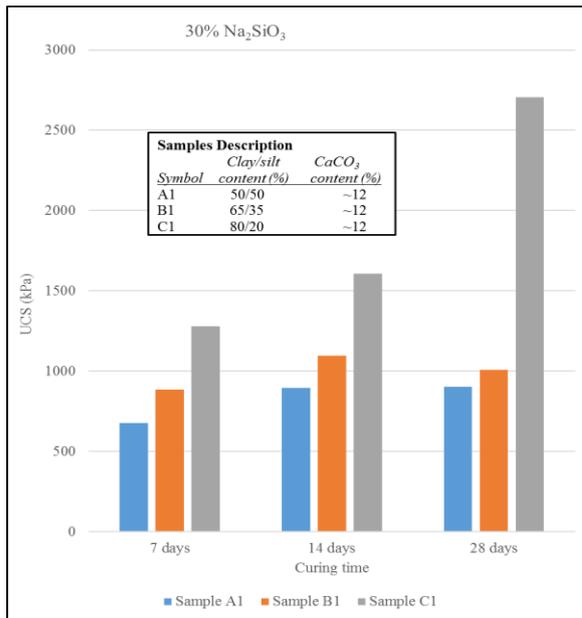


Figure 5.31 Impact of clay/silt ratio, and presence of carbonate in soil on UCS of samples mixed with 30% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

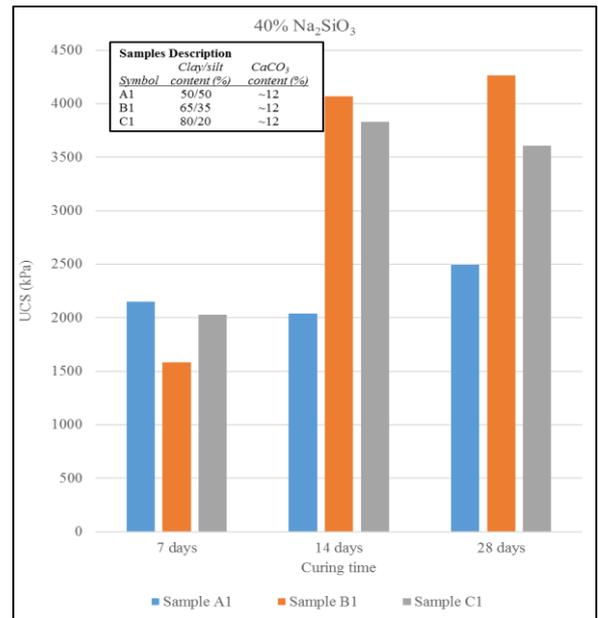


Figure 5.32 Impact of clay/silt ratio, and presence of carbonate in soil on UCS of samples mixed with 40% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

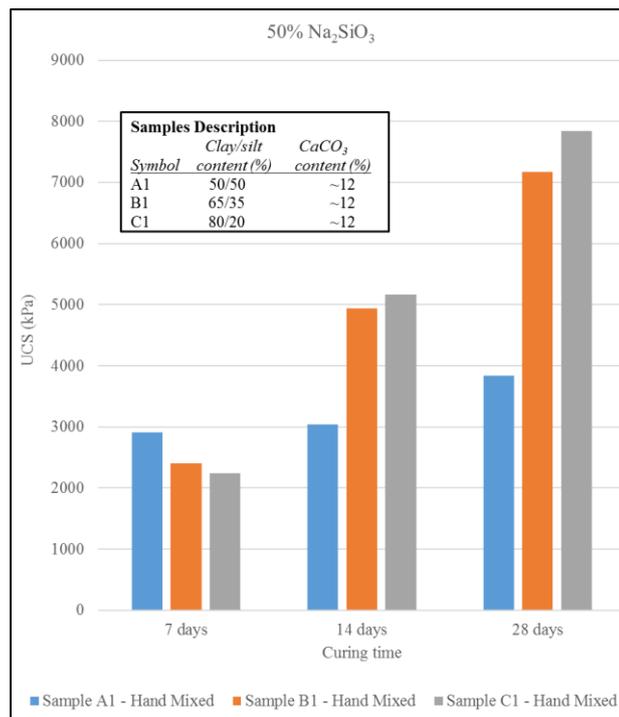


Figure 5.33 Impact of clay/silt ratio on UCS of calcareous samples mixed with 50% sodium silicate solution and cured for several days

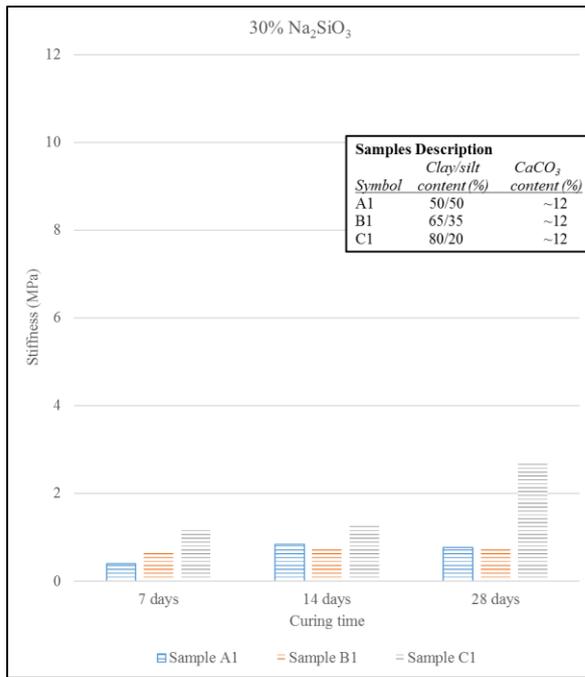


Figure 5.34 Impact of clay/silt ratio on the stiffness of calcareous samples mixed with 30% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

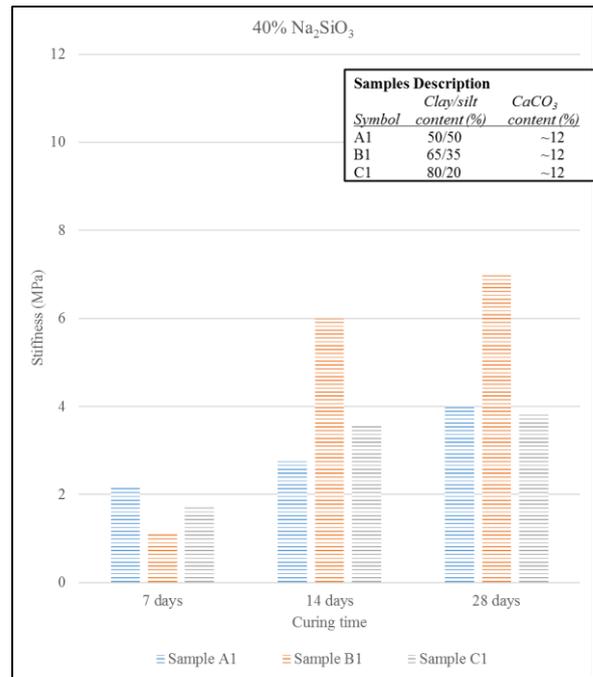


Figure 5.35 Impact of clay/silt ratio on the stiffness of calcareous samples mixed with 40% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

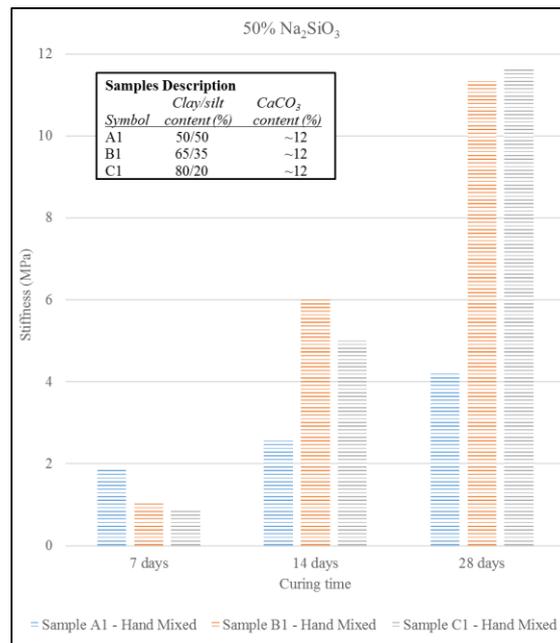


Figure 5.36 Impact of clay/silt ratio on the stiffness of calcareous samples mixed with 50% Na<sub>2</sub>SiO<sub>3</sub> solution and cured for several days

In all of these (Figure 5.31 to Figure 5.36), both UCS and stiffness have not showed a consistent trend relating to influence by clay/silt content. In some instances, at certain curing times, higher clay contents produced higher UCS and stiffness values while appearing to the contrary in other cases. In some other cases, no specific trend was even recognizable.

For example, samples treated with 30% solution demonstrated increasing UCS with increasing clay content at every age (curing period) but stiffness only aligned to this kind of trend at 7-days curing. Conversely, samples treated with 50% solution revealed a decreasing UCS and stiffness with increasing clay content at 7-days but reversed at both 14- and 28-days. No trend relating to clay/silt content was noticeable at any age for the specimens treated with 40% solution. These results are not clear on the influence of clay/silt ratio and are quite difficult to describe.

However, having identified 50% solution as the optimum amount for treating these calcareous samples, some explanations are proffered for the samples at this level of treatment (Figure 5.33 and Figure 5.36). At early curing (7-days) when bonding may yet not well formed, sample A1 having a clay/silt ratio of 1 developed a significantly greater peak UCS and stiffness than samples B1 and C1 with respective clay/silt ratios of approximately 2 and 4. Sample B1 being slightly stronger than sample C1. This is perhaps, due to better engineering properties of silt over clay. However, at later ages when bonding might have been well developed, cementation impact on strength became predominant and higher clay contents became better improved or more effective with the treatment. Compressive strength growth in sample A1 from its 7-days strength was only approximately 4.5% and 31.6% after 14- and 28-days curing periods, respectively. On the other hand, samples B1 and C1, which exhibited comparable early strengths, recorded significant higher proportions of compressive strength growths than sample A1. Strength growth for sample B1 was ~ 105% and 198% after 14- and 28-days, respectively while for sample C1 it was ~130% and 249% after the 14- and 28-days, respectively.

Recall that strength development of a silicized soil is dependent on the precipitation of cementitious material and subsequent hardening. These processes depending on pH, curing time, temperature and the degree of available metal ions ( $\text{Ca}^{2+}$ ) relative to the amount of applied solution. At early ages of curing where cement formation was presumably underdeveloped and hardening not reasonably achieved, treatment solution can possibly act as lubrication and an enhancement to sliding of soil grains and aggregates relative to one another under compressive loading. This will result in an easy structural breakdown, thus, low compressive strengths. Because clays are made up of plate-like particles whereas silts particles are angular in nature, in the event of such lubrication and absence of bonding, plate-like grains are potentially more likely to slide over one another compared to angular grains, which are capable of exhibiting some resistance to sliding through grains interlocking. This interlocking or friction brings about an increased strength, which might explain why at early ages, higher clay contents resulted in lower UCS. At this stage soil texture may be the controlling factor while, at later stages of curing, where cohesion would have developed, compressive strength would derive from both

soil texture and bonding. Contrary to other treatment methods such as cement-silty-clays, where higher strengths developed with higher silt contents (Teerawattanasuk and Voottipruex, 2014), here higher clay contents yielded higher strengths. Possibly, due to the electrostatic nature and higher surface area of clays, cementation may be more effective with clays than silts.

#### 5.4 Mitigating Collapse Potential

Treatment was carried out by impregnation as described under section 5.2.1 above using the sodium silicate solution ( $Si/Na = 3$ ,  $M = 4$ ). The samples with the worst-case collapse (“*very severe trouble (VST)*”) were treated and tested first using both the single and double oedometer procedures, with distilled water and the 5% acetic acid solution applied as wetting fluids in the single oedometer system and only acidic solution applied in the presoaked (double oedometer) system. Testing procedures were as described in section 3.5.2. Wetting was initiated at a vertical stress of 300kPa in the single oedometer, and CP values had estimated standard deviations between 0.1 – 0.3.

Because of the sticky nature of the sodium silicate solution, at the end of each testing process, both porous discs and oedometer ring were observed to adhere together with tested specimen and became problematic to disassemble these components. In many instances, efforts to detach them resulted in breaking porous discs or in other cases porous discs had to be soaked for days and sticking soil scratched hard to remove. This led to damage of equipment and thus further testing was not possible. Unfortunately, this limited the number of treated/tested samples to only those which exhibited “*very severe trouble*” collapsibility. The properties of the few samples impregnated, heat cured and tested are given in Table 5.5 below:

Table 5.5 Properties of sodium silicate treated oedometer samples (moderately calcareous)

Specimen ID	Clay/Silt ratio	CaCO <sub>3</sub> content (%)	Mean dry mass of untreated specimens (g)	Wet mass after treatment (g)	Dry mass after oven drying (g)	Dry density (Mg/m <sup>3</sup> )
A1	1	~12	81.37	153±0.7	130±3	1.51±0.02
B1	1.9	~12	81.52	151±1.6	124±3	1.45±0.02
C1	4	~12	80.95	152±1.4	125±2	1.46±0.02

Following treatment and heat curing, samples increased significantly in dry weight. Increases ranging between 52% to 60% are recorded, with sample A1 recording highest. These dry weight increases have followed the quantity of silicate solution absorbed by each specimen, which perhaps is a function of the samples' moisture holding capacity in terms of available void spaces. According to Table 4.5 showing properties of untreated samples, A1, B1, and C1 are in a decreasing order of void ratio and porosity as could also be observed from their open structure in the SEM images in Figure 5.4. The implication was a decreasing capacity to hold treatment solution in the same order.

However, a major setback of the treatment process was lack of effective control of the excess solution adhering to the surface of specimens. As a result, there were indiscriminate amounts of treatment solution held by specimens, leading to a lack of clear understanding of the influence of the samples' physical properties on absorbability.

With increase in mass and volume remaining constant, increase in dry density from about  $1.0 \text{ Mg/m}^3$  to approximately  $1.5 \text{ Mg/m}^3$ , i.e., nearly 50% increase were produced. Mechanically, this may imply improve strength and stability. The load-deformation relationships of these three treated samples under single and double oedometers comparing curves obtained pre-treatment are presented in Figure 5.37 to Figure 5.42. A summary of their CPs is shown in Table 5.6.

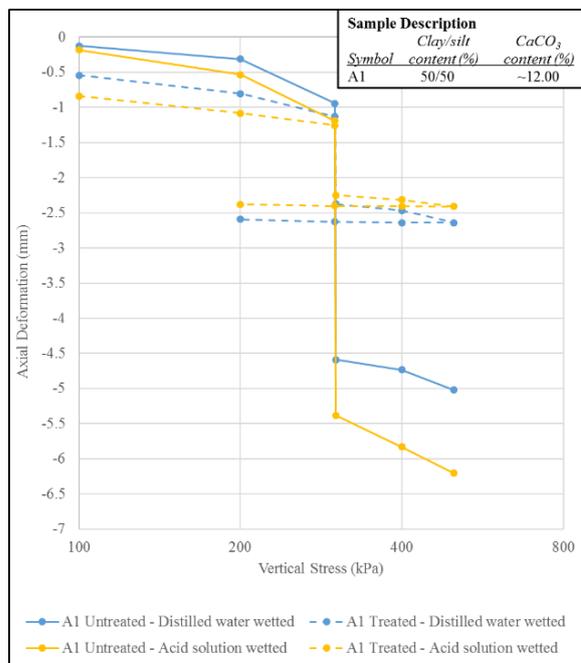


Figure 5.37 Single oedometer test results of  $\text{Na}_2\text{SiO}_3$  treated A1, low dry-density cemented "moderately calcareous" sample

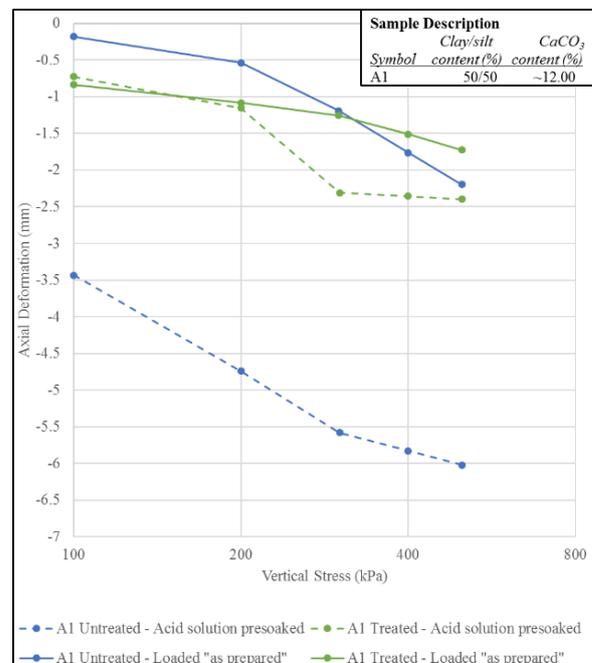


Figure 5.38 Double oedometer test results of  $\text{Na}_2\text{SiO}_3$  treated A1, low dry-density cemented "moderately calcareous" sample

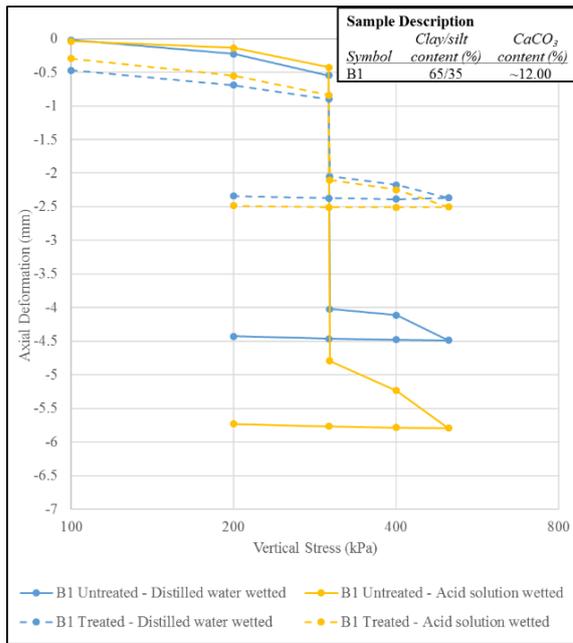


Figure 5.39 Single oedometer test results of  $\text{Na}_2\text{SiO}_3$  treated B1, low dry-density cemented "moderately calcareous" sample

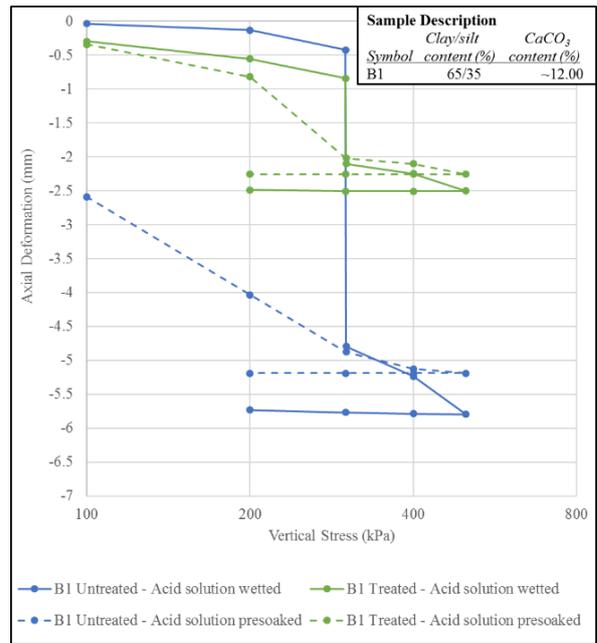


Figure 5.40 Double oedometer test results of  $\text{Na}_2\text{SiO}_3$  treated B1, low dry-density cemented "moderately calcareous" sample

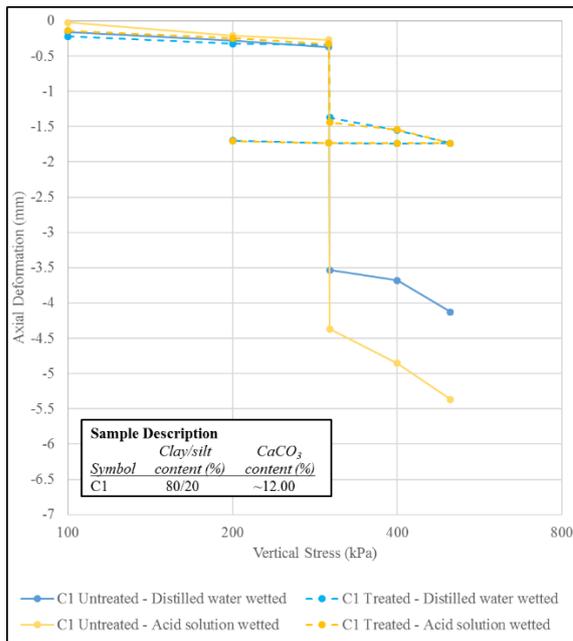


Figure 5.41 Single oedometer test results of sodium silicate treated C1, low dry-density cemented "moderately calcareous" sample

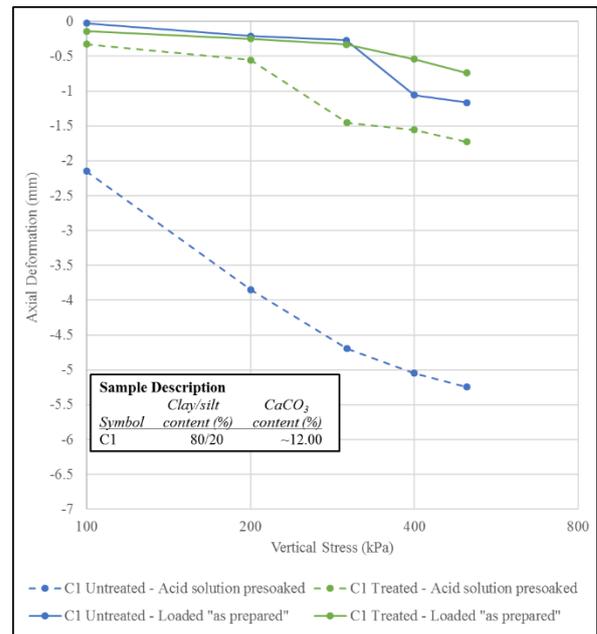


Figure 5.42 Double oedometer test results of sodium silicate treated C1, low dry-density cemented "moderately calcareous" sample

Table 5.6 Treated specimens - compressibility during loading, collapse potentials and degree of severity of low dry-density cemented calcareous samples

Sample	Initial Specimen height (mm)	Distilled water wetted (single oedometer)				5% acetic acid solution wetted (single oedometer)			
		Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Distilled water wetted)	Collapse Potential (%) (Distilled water wetted)	Severity	Height before wetting (mm) (at 300kPa Stress)	Height at end of collapse (mm) (Acid wetted)	Collapse Potential (%) (5% Acid wetted)	Severity
A1	19	17.9	16.6	7.0	T	17.7	16.7	5.6	T
B1	19	18.1	16.9	6.4	T	18.2	16.9	6.9	T
C1	19	18.7	17.6	5.5	T	18.7	17.6	5.9	T
<b>Collapse Potential (%) at different vertical stresses, from acid solution presoaked double oedometer</b>									
		<i>At 100kPa</i>	<i>Severity</i>	<i>At 200kPa</i>	<i>Severity</i>	<i>At 300kPa</i>	<i>Severity</i>		
A1		-0.6	NT	0.4	NT	5.9	T		
B1		0.3	NT	1.4	MT	6.5	T		
C1		1.0	NT	1.6	MT	6.0	T		

Treated specimens also became denser than their untreated forms and appearing stiffer under loading; only compressing slightly at pre-wetting loading stages. Although specimens with lower clay contents absorbed marginally higher amounts of treatment solution, their pre-wetting deformations (compressions) and CPs at 300kPa (SOT) remained greater with A1 > B1 > C1, similar behaviour to their untreated counterparts.

Conversely, CPs estimated at different wetting stresses from the double oedometer testing revealed otherwise. At both 100kPa and 200kPa wetting stresses, CP values were rather higher with increasing clay content while at 300kPa, CP values did not align to any trend. The presoaked curves have demonstrated a noteworthy deformation pattern at every load increase. A fairly consistent amount of deformation is seen at each stress increment except at 300 kPa where a disproportionate deformation occurred. This is an indication of a possible threshold stress at which collapse is maximum in these specimens. However, it cannot be confirmed as it was not investigated further. The nearly zero gradient of the rebound curves attest to a permanence in the deformations that occurred.

The influence of wetting fluid type on the magnitude of collapse observed appeared to be similar. Apart from a possible increase in strength resulting from the treatment, treated specimens also exhibited moisture-repelling behaviour. At the end of the oedometer wetting period only part of the specimen became saturated. In single oedometer tests, wetting lasted for about 26 hours while it was 28 hours soaking in the double oedometer system.

### 5.4.1 Concluding discussion

Results presented in Figure 5.43 and Figure 5.44 demonstrate substantial positive effect of the treatment strategy in mitigating collapse properties of the soils. In Figure 5.43, single oedometer (at 300kPa wetting stress) results of both distilled water and acidic solution wetting conditions generally revealed mitigation levels approximating between 59% – 73% and 70% – 78%, respectively.

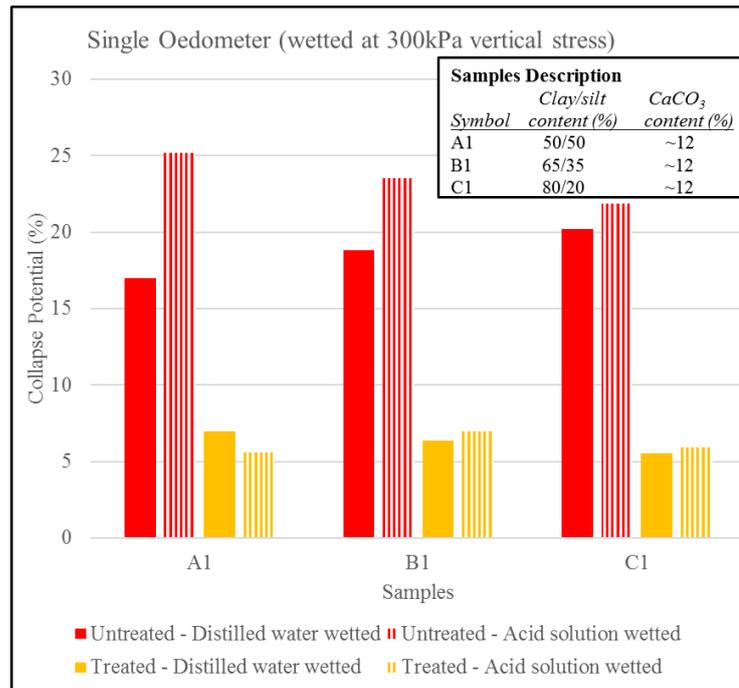


Figure 5.43 Calcareous silty-clay collapse mitigating impact of a sodium silicate solution tested under acidic solution and distilled water wetting conditions of the single oedometer.

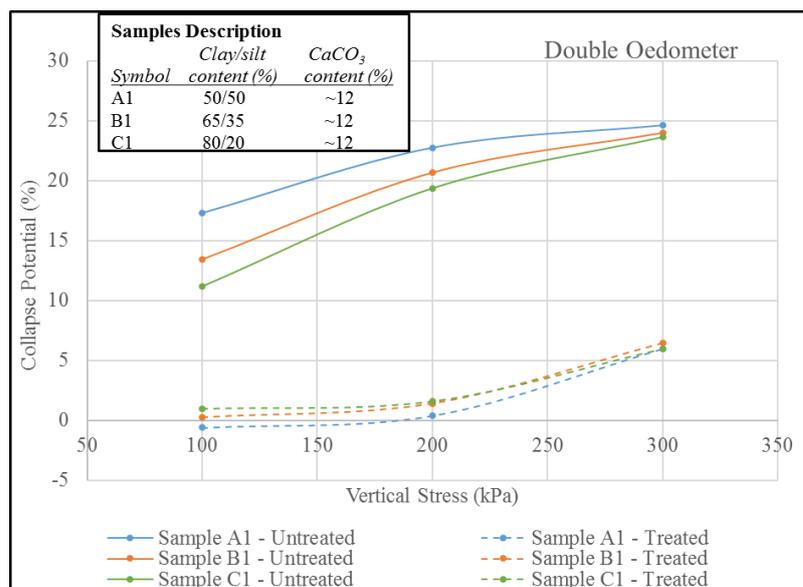


Figure 5.44 Calcareous silty-clay collapse mitigating impact of a sodium silicate solution tested under distilled water presoaked (double oedometer).

Double oedometer results (distilled water pre-soaked) in Figure 5.44 revealed about 73% – 75% mitigation impact at a 300kPa wetting stress but reaches to between 91% – 98% at 100 and 200 kPa stresses. In one case (sample A1 treated), some swelling was observed at a 100kPa stress.

As stated in chapter two, the reaction of sodium silicate with calcium is an ionic exchange reaction believed to precipitate water resistant (insoluble) silicate sheaths that surround the soil grains, producing cementation effect (Wooltorton (1955); Hurley and Thornburn (1972)). While  $\text{Na}_2\text{SiO}_3$  treated samples remained fairly collapsible, treatment has obviously reduced the severity of collapse from “*very severe trouble*” to “*trouble*” under acidic solution wetting and from “*severe trouble*” to “*trouble*” under distilled water wetting. Both at 300kPa wetting stress. Below 300kPa, collapse reduction was from “*very severe trouble*” to “*moderate trouble*” at a 200kPa and from “*severe trouble*” to “*no trouble*” (non-collapsible) at a 100kPa. See Table 3.9 on page 138 for collapse classification.

Results in Figure 5.43 clearly indicated a greater damaging influence of distilled water on silicized soils than acidic solution. It can also be interpreted that acidic solution has the potential to limit the magnitude of collapse obtained of a silicized soil compared to distilled water. This agrees with Iler (1979) argument that an acidic environment enhances the gelation or polymerization process of  $\text{Na}_2\text{SiO}_3$  treatment. Thus, perhaps the silicized samples still underwent some further improvement during the acidic solution wetting in the course of the oedometer testing.

Despite that heat curing, as adopted here demonstrated the potential to produce stronger bonds than room curing, Weldes and Lange (1969) have claimed that it is not an adequate process to achieving complete insolubility of silicized soils. Weldes and Lange also noted that curing by polymerization better produces insolubility. This may explain why the treated samples remained collapsible at some levels of overburden stresses. However, it is also common knowledge that at high shear stresses above the bonding strength of a soil, bond breaking occurs, and densification becomes probable upon wetting and/or loading. Furthermore, since silicate treatment by impregnation has earlier been identified to produce weaker samples than mixing & kneading, it is possible that mixing and standard compaction followed by polymerization could have better improved the collapse properties of these samples.

## 5.5 Chapter Summary

Results from both strategies of treatment employed demonstrated the ability of a well-selected (or well formulated) solution of sodium silicate to improve the engineering properties of fine-

grained soils in a “single-step” treatment system, whether  $\text{CaCO}_3$  was present or not. However, better performance was achievable in the presence of  $\text{CaCO}_3$ .

The factors that influence the workability and effectiveness of a sodium silicate treatment of calcareous clays included  $\text{SiO}_2/\text{Na}_2\text{O}$  molar ratio and concentration, degree of soil calcareousness, and curing temperature. These factors affect the rate of formation and hardening of silicate bonding gel and its loss of water and cracking. Performance of a well-selected solution increases with increasing solution amount up to an optimum (50% by mass of dry soil for *moderately* calcareous soils), and with curing time (or drying). However, at young ages treatment reduces stiffness, which recovers with drying. High amounts of solution take a longer time to achieve full performance.

Treatment by impregnation revealed that a certain type of  $\text{Na}_2\text{SiO}_3$  solution might be suitable to treat calcareous clays of up to about 12%  $\text{CaCO}_3$  content, above which there is an early gelation process preventing further penetration of solution. Avoiding such quick early gelation by lowering solution concentration results in post-treatment cracking. This suggests that, an inappropriately selected solution type applied by single-step method can be detrimental to a treated calcareous silty-clay after drying.

Finally, the single-step treatment application of a sodium silicate solution of  $\text{Si}/\text{Na} = 3$ ,  $M = 4$  proved effective for both enhancing strength properties, reducing perviousness to water and acidic solutions and thereby mitigating collapse potential of fine-grained calcareous soils. This notwithstanding, treatment did not completely eradicate collapse at overburden stress up to 300kPa. Perhaps, post-treatment modifications (e.g., compaction) could further reduce collapse.

## Chapter 6 **General Discussion**

### 6.1 **Introduction**

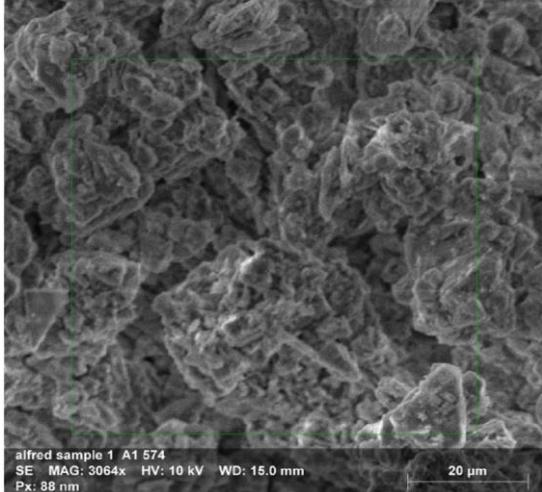
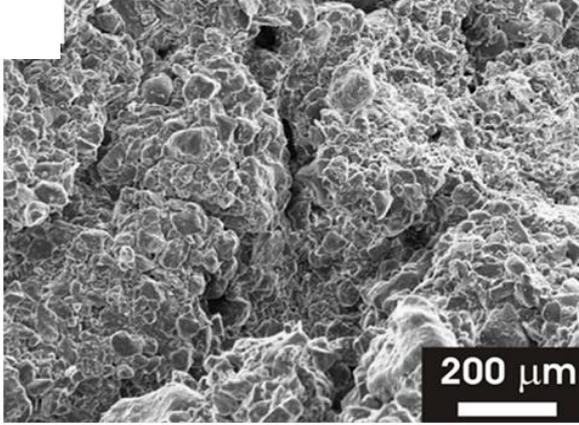
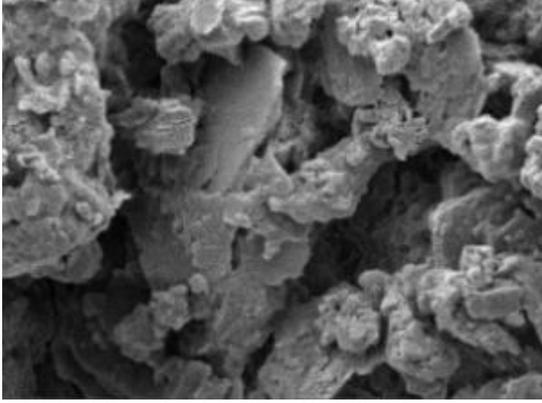
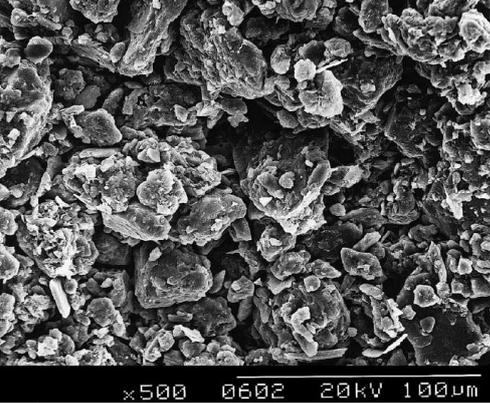
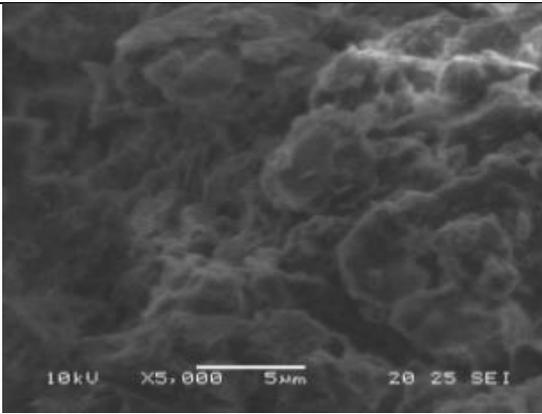
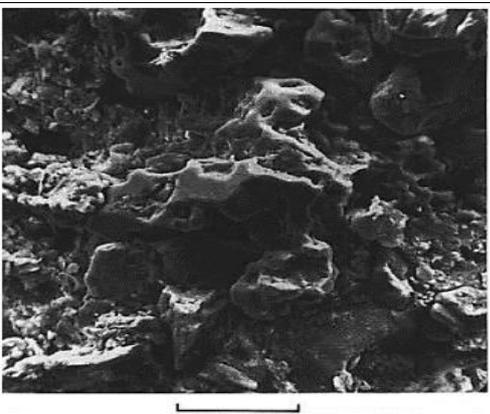
To achieve the objectives of this research, the study investigated three broad themes, which include manufacture of potentially metastable calcareous silty-clay samples; evaluation of their collapse behaviour with relation to their compressive strength; and the potential of a sodium silicate improvement. This chapter is structured to discuss areas where key contributions, impact and significance are made by the thesis as well as the scaling up of the laboratory experimental work.

### 6.2 **Manufacture of Collapsible/Metastable Samples**

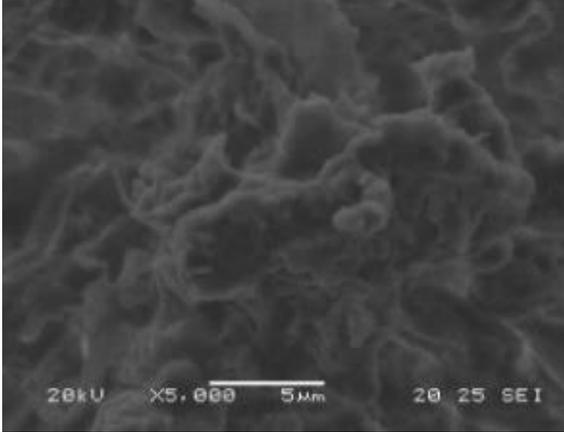
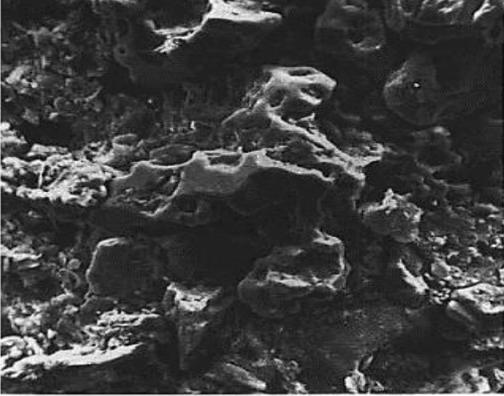
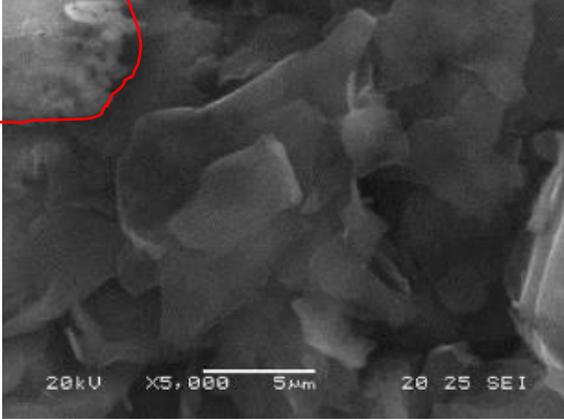
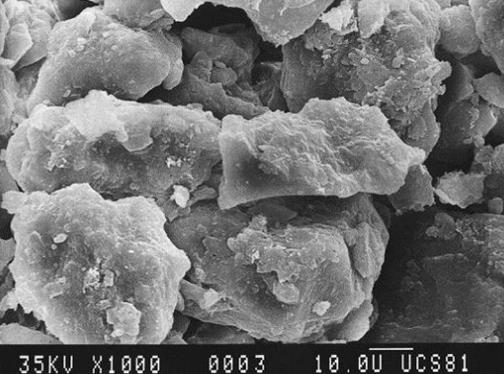
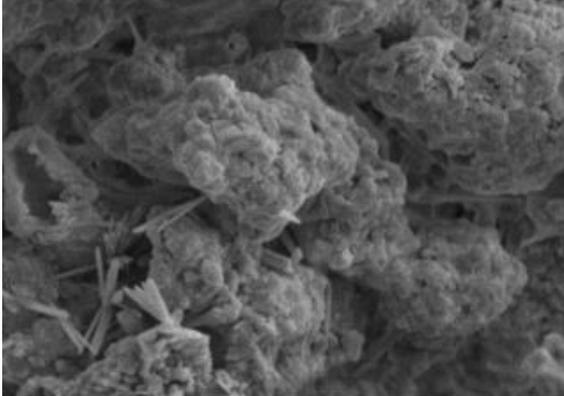
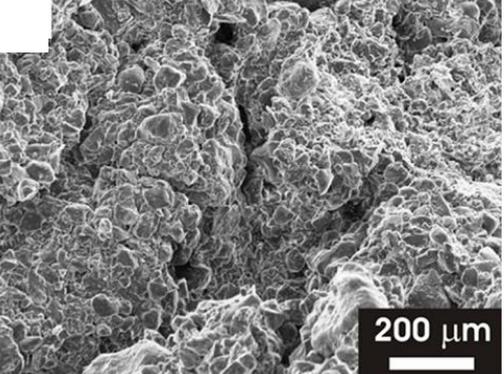
This research has provided a simple and effective technique to produce collapsible calcareous silty-clay samples akin to natural marls through a controlled process. Processes for production of marls have not hitherto existed in the literature. Some factors influencing the efficiency of the process have also been identified. The technique successfully precipitated predetermined amounts of calcite into silt-clay mixes. Because this technique is cheap and easy to operate, it will enhance further studies on calcareous soils. This is important to advance the knowledge of the impact of calcite on soil behaviour.

Characteristic features of soils that undergo volume change upon wetting (i.e. collapse) as detailed in section 2.3 have been somewhat replicated artificially in the present samples with a calcite bonding. In order to evaluate the production procedures, empirical and analytical methods, such as scanning electron microscope (SEM) imaging, analysis of the images using PCAS (Particles (Pores) and Crack Analysis System), and the determination of void ratio and porosity have been used to characterise the samples. Unfortunately, the quality of the SEM images was not good enough, and that adversely impacted their microstructural study. However, through observation, it was revealed that some of the SEM images showed microfabric comparable to a range of micrographs of naturally occurring collapsible soils as presented in Table 6.1 below.

Table 6.1 (a & b) Comparable microstructures of SEM images of some present samples and collapsible soils found in the literature

Present SEM Image	Comparable Image from Literature and Reference
 <p><b>A1 (clay/silt ratio of 1 with ~12% CaCO<sub>3</sub>)</b> – An open structure of silt grains and clay clods supported by CaCO<sub>3</sub></p>	 <p>SEM photomicrograph of typical macroporous pelletised brickearth fabric, comprising pellets of compact silt grains (with interstitial clay) in an open-packed arrangement. Calcareous brickearth, 2.39 m, Ospringe Pit 1 (adopted from Milodowski <i>et al.</i> (2015)).</p>
 <p><b>A3 (clay/silt ratio of 1 with ~40% CaCO<sub>3</sub>)</b> – A loosely packed silt fabric interconnected by abundant CaCO<sub>3</sub>-clay matrix</p>	 <p>SEM of undisturbed Malan loess. Vertical face illustrating the characteristic fabric of Upper Pleistocene loess from the Lanzhou region, western Loess Plateau of north China (after Derbyshire <i>et al.</i> (1995) and Derbyshire (2001))</p>
 <p><b>C1 (clay/silt ratio of 4 with ~12% CaCO<sub>3</sub>)</b> – Clay and CaCO<sub>3</sub> overwhelmingly surrounded the minor silt grains.</p>	 <p>Carbonate connectors under scanning electron microscope- authigenic calcite in Melan loess, China (adopted from Derbyshire and Mellors (1988))</p>

(a)

Present SEM Image	Comparable Image from Literature and Reference
 <p data-bbox="240 656 804 741"><b>C2 (clay/silt ratio of 4 with ~25% CaCO<sub>3</sub>)</b> – CaCO<sub>3</sub> bonding with the clay platelet overwhelmingly surrounded the minor silt grains.</p>	 <p data-bbox="836 629 1340 741">Carbonate connectors under scanning electron microscope- authigenic calcite in Melan loess, China (adopted from Derbyshire and Mellors (1988))</p>
 <p data-bbox="240 1189 804 1261"><b>D1 (clay/silt ratio of 0.11 with ~12% CaCO<sub>3</sub>)</b> – CaCO<sub>3</sub> globules visible at top left corner but more calcite could be embedded within silt grains.</p>	 <p data-bbox="836 1144 1340 1193">Clean edge-to-edge fabric in angular coarse silt Malan loess (Derbyshire and Mellors, 1988)</p>
 <p data-bbox="240 1675 804 1758"><b>D3 (clay/silt ratio of 0.11 with ~40% CaCO<sub>3</sub>)</b> – D3-Needle shaped CaCO<sub>3</sub> outgrowth from the CaCO<sub>3</sub> globules, coating and bridging silt particles.</p>	 <p data-bbox="836 1653 1340 1825">SEM photomicrograph of typical macroporous pelletised brickearth fabric, comprising pellets of compact silt grains (with interstitial clay) in an open-packed arrangement. Calcareous brickearth, 2.39 m, Ospringe Pit 1 (adopted from Milodowski et al. (2015)).</p>

(b)

Observations of the SEM images generally show that microfabric of the samples vary from one to another, except in few cases where some similarities were noticed. These variations are perhaps, due to the differences in proportions of constituent materials. Microfabric is one key feature that defines the collapsibility of a soil (Briscoland and Chown (2001); Rezaei *et al.* (2012)). As such, the collapse potential (CP) of the present samples, changes with change in proportion of soil constituent materials. However, in natural collapsible soils, not only the proportioning of constituent materials but a combination of sedimentation mechanism and grain type influence the structure of metastable soils (Rogers, 1995). These factors determine the packing state, thus, dry density, void ratio and porosity.

Apart from degree of saturation or suction, the literature identifies low dry density, high void ratio and porosity as common features of collapsible soils (Barden *et al.* (1973); Lawton *et al.* (1989); Rogers (1995); Assallay (1998)) and have been used to fix thresholds to classify collapsibility of soils. For example, Clevenger (1958) cited in Alan and Robert (1988) suggested greater than  $1.44\text{Mg/m}^3$  and less than  $1.28\text{Mg/m}^3$  as marked dry density thresholds for small and large collapse potentials, respectively. Porosity greater than 40%, silt content greater than 30%, and small clay contents are some other reported thresholds for collapse occurring (see Okwedadi *et al.* (2014)).

In the present study, one set of the samples were statically compacted to predetermined low dry densities (approximating  $1\pm 0.1\text{Mg/m}^3$ ), exaggerating both void ratio and porosity to enhance a clearer understanding of the impact of inter-particle cementation on their mechanical behaviour. Nevertheless, specimens with comparable values of void ratio and porosity have earlier been studied (see section 3.4.1). A second set of samples with higher dry densities (above the lower threshold ( $1.28\text{Mg/m}^3$ ) according to Clevenger, 1958) were also prepared.

Obviously, the first set of samples exhibited a large open structure (see Figure 3.19 on page 132) with estimated void ratios,  $e$  above 1.5 and porosities,  $n_0$  greater than 60%, satisfying the “> 40%” criterion for collapse to occur according to Fedas, 1966 (cited in Alan and Robert (1988)) while the second set of samples exhibited significantly lower  $e$ , with  $n_0$  still greater than 40%, satisfying the collapse criterion.

X-ray diffraction analyses confirmed the calcite phase (with its strongest spectrum at a diffractive angle of  $29.5^\circ$  theta), as the calcium carbonate cement precipitated in the artificially simulated calcareous samples as illustrated in Figure 3.17.

At different compaction levels, each set of samples exhibited higher void ratios and porosities with decreasing calcite and clay contents. However, there has also existed a nominal

increase in dry density with higher calcite contents, which might be a result of the mass increase of lime following its transformation.

While there are arguments that volume expansion of lime by carbonation of a lime-soil system produces microcracks or surface exfoliation (Vance *et al.*, 2015) and adversely affects engineering properties of such soils (Bagoniza *et al.*, 1987), others believed carbonation increases strength (Van Balen (2005); Despotou *et al.* (2016)). Consequently, varying degree of calcareousness (or calcite content), dry density and related properties, and perhaps, clay/silt ratio are considered as some influential factors to control the mechanical behaviour of samples. These are further discussed in the following sections.

### **6.3 Mechanism of Collapse and Relationship with Strength**

The strength and collapsibility of any fill, whether natural or anthropogenic depends on certain factors, such as material properties, placement conditions, and secondary conditions operating in-situ. These provide baselines for engineered fill designs and specifications. Some workers, e.g., Lawton *et al.* (1992); Watts and Charles (2015) have thoroughly assessed these factors, and the present study has reviewed and re-examined them in artificially produced calcareous silty-clay samples.

#### **6.3.1 Material property and composition**

The impact of soil properties such as clay content, dry density, porosity, void ratio, have hitherto been individually used to classify CP. Notwithstanding their effect, they have been identified in this study not to independently define CP, especially for samples bonded with calcite. Rather, bonding material type and content, and wetting fluid type are the critical factors controlling collapse. This study reveals that even an open structure on itself may not yield significant collapse in the presence of strong inter-particle bonding. That is, an open structure may not directly mean metastability. This knowledge is therefore important for engineers to design safe and economic foundations on soils that may exhibit an open structure.

#### ***Implication of soil composition (clay content)***

Watts and Charles (2015) identified non-engineered fills as the most complex to deal with because of their widely varied composition. Watts and Charles (2015) provide an elaborate review of material types and the significance of particle strength on collapsibility of such fills. However, it is generally agreed that all soil types can be collapsible provided certain structural conditions are met (Lawton *et al.* (1992); Jefferson and Rogers (2012); Watts and Charles

(2015)). Nevertheless, the literature appears to identify clay content as a major criterion for defining collapse potential (Handy, 1973 cited in Bell *et al.* (2003); Langroudi (2014); Okwedadi *et al.* (2014)), but seems to ignore the impact of chemical bond material components in describing collapsible soils despite many (e.g., Northmore *et al.* (2008); Milodowski *et al.* (2015)) identifying CaCO<sub>3</sub> for example, as a key contributor to collapse occurring.

This study demonstrates that clay content influences the magnitude of collapse beyond those levels ( $\leq 40\%$ ) traditional known or used by previous authors to define CP. Also, that even above 40% clay content, CP can still increase with increasing clay content depending on placement conditions and type of bonding available.

The effect of clay content in reducing compressibility of collapsing soils was reported in Handy, 1973 cited in Bell *et al.* (2003); Jefferson *et al.* (2005); Langroudi (2014). Handy suggested a collapse criterion in relation to clay content, noting higher collapsibility with reducing clay contents. However, Handy only cited a high probability of collapse for samples having less than 16% clay (supported by Okwedadi *et al.* (2014); Langroudi (2014)), and no collapse for samples with more than 32% clay content. See Figure 4.59 in chapter 4 for more proposals with collapse reported only at not more than 40% clay content. Conversely, samples studied here contained clay contents reaching 80% in some cases, and yet have exhibited very high CP values, particularly for *moderately* calcareous and non-calcareous samples.

Jefferson *et al.* (2005) appear also to support the idea that increasing clay content can be useful in mitigating CP in granular soils where a clay suspension is injected to clog pore spaces, thus eliminating compressibility. While this technique has been effectively used to treat collapsible soils, it does not literally describe collapsibility by clay content. Rather, the technique improves density and eliminates in-situ open structure. An increase in clay content, according to the current study, produces higher CP. This, however, was identified to result from the effect of macropeds arising from clay mixing and remoulding (Basma and Tuncer (1992); Lawton *et al.* (1992)). Despite these inconsistencies, the current study suggests that clay content should not be a sole criterion for predicting collapse since a pure clay soil can still be liable to collapse (Barden *et al.* (1973); Lawton *et al.* (1992)). The effect of clay content can be related to the fill's placement conditions and type of bonding available.

### ***Calcite content and its implication***

Despite the much mention of the impact of CaCO<sub>3</sub> on collapsibility in the literature, there is fairly no quantification of the magnitude of collapse that may arise in relation to the amount or type of CaCO<sub>3</sub> present in a collapsible soil. This work is first to quantify the impact of calcite

(as a polymorph of  $\text{CaCO}_3$ ) content on both the CP and rate of collapse of calcareous clayey soils. It demonstrates that calcite reduces CP with an increasing content up to about 40% (maximum studied here). Additionally, the study reveals that wetting with a low pH solution produces higher magnitudes of collapse, and a prolonged period to achieve total possible settlement. It also yields greater degree of collapse in moderately calcareous clay than in a non-calcareous clay of similar placement conditions. These contributions are quite important for geotechnical engineers dealing with similar soils because they inform the need for geotechnical site characterization to consider  $\text{CaCO}_3$  contents and field wetting fluid type prevalent, when assessing long term impacts.

### 6.3.2 Placement conditions

Placement conditions, e.g., water content and compactive energy determine the density and related properties of a fill. Engineered clay fills placed at low water content will experience excessive settlement upon wetting (Watts and Charles, 2015). Lawton *et al.* (1992) reported similar fate in the long-term if placement water content was too high and the fill later experiences drying. Such placement conditions usually produce open structure, low dry density and high void ratio and porosity.

To understand this impact, though not strictly relying on water content, the present study prepared samples to dry densities ranging from very low (akin to normally consolidated clays) to high (standard Proctor compacted) but with some calcite bonding. It was confirmed here that lower dry density is responsible for large CP but higher compactive energy can increase dry density and reduce pore volume as well as minimise CP. Watts and Charles (2015) agree with Lawton *et al.* (1989) that standard compaction at optimum water content or slightly wet of optimum can completely mitigate collapse in all forms of collapsible soils. Watts and Charles (2015) also show that proctor compaction to 95% of the maximum dry density at slightly dry of optimum can mitigate collapse in mudstone and colliery spoil (i.e. coarse-grained) fills, yet, may cause a maximum of 2% CP in clayey (i.e. fine-grained) fills.

However, while the foregoing argument by Watts and Charles (2015) may be true and their samples unbonded, the present study reveals that calcite content at certain levels can also mitigate collapse of clayey fills compacted to as low as about 65 – 90 % of their maximum dry density. At about 40% calcite content, clayey samples are rarely collapsible irrespective of the density. Increasing calcite content increases compressive strength and stability of the clay samples because of the stronger chemical cementation.

However, it was also revealed here that fill mineral composition and pH of service pore fluid can influence the rate and magnitude of collapse a fill may experience in its service life.

### 6.3.3 Stress history prior to wetting

Traditionally, it is believed (Watts and Charles, 2015) that where a fill is preloaded to a stress level beyond the structural design load, stiffness would be enhanced and therefore less settlement becomes probable. However, the results presented herein indicate that merely preloading beyond design load may not necessarily impact the CP of cemented unsaturated clayey soils unless the preloading stress is up to or above the critical stress of the soil. This is the point where cement bonds are broken, and lesser CPs begin to follow increased wetting stress, which is a reverse of the behaviour before the critical stress. This study reveals that the critical stress defers for each soil depending on bond strength and stiffness. It was as low as about 300 kPa for samples exhibiting stiffness between 1.2 – 2.8 MPa, and over 800 kPa for stiffness between 4.7 – 6.2 MPa. Lawton *et al.* (1992) believe that the critical stress is of no practical consequence where it is hugely larger than the structural design load.

Watts and Charles (2015) have also argued that the stress state of a fill can change with age due to changes in its environment. This affects its mechanical behaviour, such that, its response to a constant load becomes different in the long term. Aging may result in either strengthening (e.g., precipitation of chemical bonding or grain slippage and better interlocking) or weakening (e.g., stressing and coalescence of microcracks). The present studied samples, which are analogous to carbonated lime-treated clay fills, appear to show concerns associated with dissolution where low pH fluid wets such carbonated fills.

### 6.3.4 Relationship between UCS and collapse potential

The study has also revealed that UCS can be directly proportional to CP in certain soils, but difference in bonding element in relation with certain wetting fluids can revert that trend.

Collapsible soils are known to exhibit a metastable structure (Rogers *et al.* (1994); (1995); Assallay (1998)) with their collapsibility depending largely on the nature of soil structure (Clemence and Finbarr (1981); Iranpour and haddad (2016)). Despite collapse being a wetting induced phenomenon (Barden *et al.* (1969); (1973); Alan and Robert (1988); Lutenegger and Hallberg (1988); Tadepalli and Fredlund (1991)) some scholars have claimed that the treatment of such collapsible property can be achieved by improving strength related properties, such as increasing bonding, increasing density, etc. (Jefferson *et al.* (2005); Iranpour and haddad

(2016)). Iranpour and colleague believed that enhancing the resistance of clay and silt bridges, and cementation factors in collapsible soils structure could mitigate the collapse danger.

UCS and CP results shown in Figure 6.1 and Figure 6.2 establish two facts. On the one hand agreeing with the argument that increasing bonding strength can reduce collapsibility as is illustrated by the curves beginning from an approximately 12% CaCO<sub>3</sub> content and beyond. An increased calcite cementation content resulted in increased UCS and reduced CP values. Implying that UCS is somewhat inversely proportional to CP, negating the argument in some quarters that high carbonate contents can promote collapse (e.g. El-Sohby *et al.* (1988); Milodowski *et al.* (2015)). On the other hand, cemented specimens (with about 12% CaCO<sub>3</sub> content) yielded higher CP values compared to non-calcareous uncemented weaker specimens. This tends to claim that increased UCS is rather directly proportional to CP. This is a contradiction revealing that despite the importance of bond strength factor in the mitigation of collapse, it is critical also to consider perhaps other important factors, such as dissolution resistance of the bonding agent with respect to anticipated wetting fluid types, and other prevailing soil conditions just before wetting. As discussed earlier in this chapter, the magnitude of collapse of a soil depends on several factors.

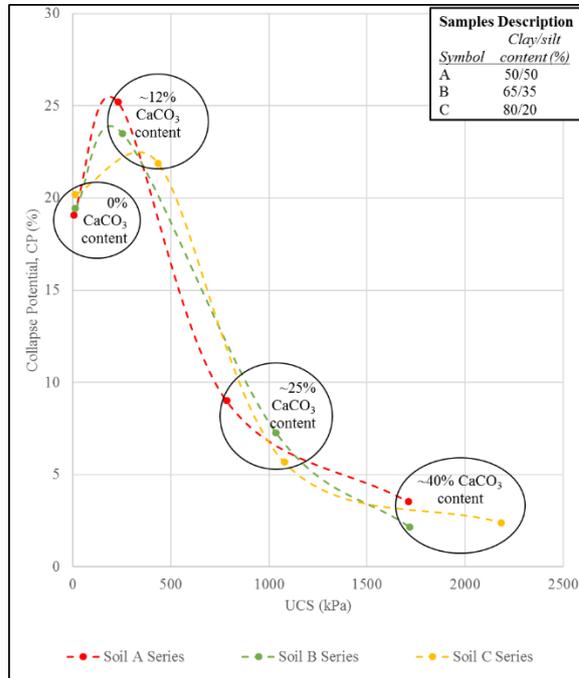


Figure 6.1 Degree of calcite cementation versus UCS and CP under acidic solution wetted condition

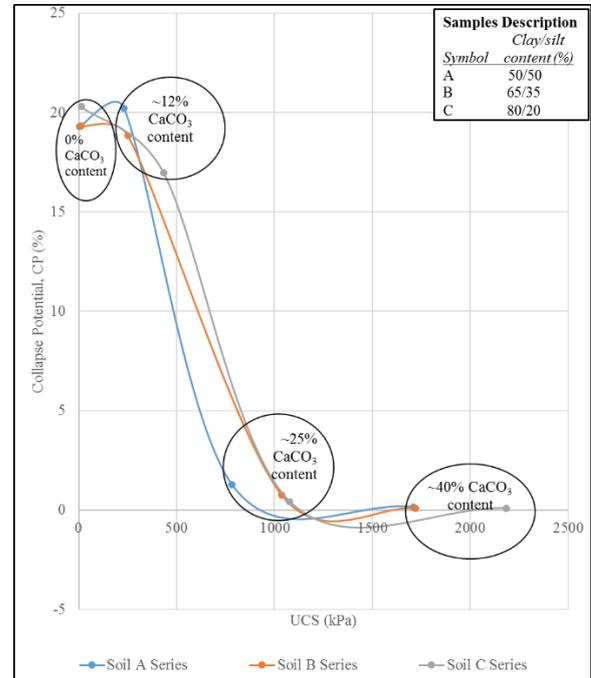


Figure 6.2 Degree of calcite cementation versus UCS and CP under distilled water wetted condition

## 6.4 Sodium Silicate Improvement for Earthworks

As stated earlier, the current samples are comparable to natural clay marls, which are commonly used in the construction of embankments and earth-dam cores (Lamas *et al.* (2002); (2011); Mansour *et al.* (2008)). However, Lamas identifies problems arising from dissolution, piping, and internal erosion to be associated with marls used in dam construction. Although these parameters were not specifically assessed in the current study, the effect of dissolution was observed in the oedometer testing to arise where a low pH (< 5) fluid wetted the samples. Nevertheless, the treatment of samples with a Na<sub>2</sub>SiO<sub>3</sub> solution mitigated this effect. It improved compressive strength through enhanced chemical cementation, as well as developed an impervious layer that prevented samples from getting wet/soaked.

While Na<sub>2</sub>SiO<sub>3</sub> treatment is a recurrent technique, it has mainly been applied as an additive or in conjunction with other substances: a process referred to as multi-step treatment (see Terzaghi 1947 and Riedel 1952 in Hurley and Thornburn (1972); Sokolovich and Gubkin (1970); Sokolovich and Ibragimov (1971); Ou *et al.* (2009); Maaitah (2012); Chien *et al.* (2014)). Nonetheless, the current investigation found that a well-selected Na<sub>2</sub>SiO<sub>3</sub> solution with molar ratio  $\geq 3$  and concentration (M)  $\geq 3.5$  was reasonably effective for a single-step (without an additive) treatment of the calcareous silty-clay samples. Thus, the quality of clayey marls can be enhanced by this treatment for the construction of embankments and dams.

However, it was more effective when applied by mixing rather than impregnation (capillary rise). Thus, suitable for preconstruction treatments. Furthermore, samples increased in strength with increasing amount of solution applied, clay and calcite content as well as curing time. Nevertheless, this treatment appears satisfactory only for long term improvement needs. An interesting finding here is that, contrary to claims in the literature, the present study identifies cementation to produce better performance with higher clay contents rather than silt.

## 6.5 Implication for Scale Up and Application in Practice

### 6.5.1 Scale up

Two sets of samples analogous to non-engineered and engineered fills with calcite bonding were investigated in this study. Nevertheless, because of the controlled manner of production and size of specimens tested, specimens are considered more homogenous than actual fills, thus, may not show true representation of field conditions. Again, in the case of collapse testing, wetting conditions in the lab can be quite different from field conditions (Houston *et al.* (1995a); Jefferson and Rogers (2012)). These are some of the reasons why field tests are recommended

in order to tie with laboratory results where possible (Jefferson *et al.* (2005); Watts and Charles (2015)). For example, Zhang and Zhang, 1995 stated that in China, the actual field collapse potentials are merely one-seventh of the laboratory determined full-wetting collapse (Al-Rawas, 2000). Houston *et al.* (1995a) argued that this is possible where a soil is only partially wetted, and only a portion of the full CP is achieved. Therefore, the laboratory results must be considered with caution and high factors of safety are recommended if the parameters are used in design calculations.

The non-engineered fills were characterised by their low dry-density, high void ratio and high porosity (see Table 4.5) similar to those of natural sediments of young geologic age like the Italian soils reported in Table 2.5. The study identified that 300kPa load, an equivalent construction load of a 15m high embankment, placed on such soils may not cause instability in their unsaturated state, but can result in serious collapse dangers when wetted, except where calcite content reached over 25%. However, where a low pH fluid wets, even a 200 kPa load causes severe collapse concerns at least for soils containing no more than 25% calcite content.

The engineered fills on the other hand, represented two different construction fills compacted, one at optimum moisture content (Table 4.7) and the other at dry of optimum (Table 4.9). This study has revealed on a general note, both set of samples to be non-collapsible. This is obviously due to calcite bonding because it is well reported that compaction at dry of optimum is a collapse precursor. Thus, calcite bonding can potentially mitigate the effect of poor compaction.

### 6.5.2 Quantifying the amount of silicate for treatment

A key factor to consider in chemical treatment of soils will be the chemical equilibrium between reactants and expected product(s). This understanding is useful at the design stage in order to be able to design an effective treatment programme. Based on the chemistry of the reacting components of a treatment system, an optimum amount can be estimated for the applied chemical in order to maintain an equilibrium. However, the present study reveals, for instance, in the case of  $\text{Na}_2\text{SiO}_3$  solution applied to a calcite-clayey system, that the amount of solution needed for an effective treatment must exceed that which satisfies the chemical equilibrium (see Equation 5.1). This is because the dry clayey component of the calcite-clayey system absorbs part of the solution, preventing it from reaching the calcite reaction fronts except an excess solution was added to complement. Thus, strength increased with increasing excess up to a maximum.

Laboratory treatability studies using the proposed site soils are, therefore, necessary prior to designing for a field trial and subsequent full-scale field implementation (Al-Tabbaa and Evans, 1999). However, laboratory application methods, especially mixing and kneading usually produces greater homogeneity in treated soils than in an in-situ field scale application. Also, because of in-situ soil variations compared to laboratory soils, it is sometimes difficult to correlate results of treatability studies and those from in-situ application (Al-Tabbaa and Evans (1999); Al-Tabbaa *et al.* (2012)). Nonetheless, they are good guide if applied with caution.

Even though the silicate improvement strategies (mixing or impregnation) studied here proved effective to achieve increased compressive strength and reduced collapsibility, it is doubtful if impregnation, for instance, could be applied at a large scale typical of field condition.

## 6.6 Summary

Procedures adopted to produce artificial metastable calcareous clay soils yielded positive results in terms of ability to precipitate predetermined amounts of calcite and replicating collapse feature, e.g., an open fabric, low dry density, high void ratio and porosity, etc. The calcite content appearing to exert the most influence on both UCS, CP and rate of collapse. The magnitude of collapse of samples could be reasonably related to their compressive strength. Generally, factors such as placement conditions, material properties and others have been found to also control the behaviour of samples.

The study further reveals the potential of a well selected sodium silicate solution to improve both UCS and CP, making these soils suitable for engineering construction of embankments and dams. Conclusions reached and areas recognized for further investigation are presented in the subsequent chapter.

## Chapter 7 Conclusions and Further Research

### 7.1 Introduction

The present study provides an understanding of the collapse and strength behaviour of metastable calcareous silty-clay soils to complement existing body of knowledge on soil collapsibility.

The aim of this project is to understand how different soil parameters (particularly calcium carbonate bonding and clay content) and wetting fluid type affect collapsibility in calcareous silty-clay soils and the potential of sodium silicate to mitigate collapsibility of these soils. In order to achieve this aim, a number of specific objectives were set as outlined in section 1.2. This chapter summarises the conclusions drawn from this study with reference to the original objectives and sets out a pathway for future research.

### 7.2 Conclusions

#### 7.2.1 Key characteristic features of collapsible soils and their improvement (objective 1)

The literature concludes that every soil type can potentially be collapsible under the right conditions. The prerequisite for collapse being an open metastable structure, with main soil features including low dry density, high void ratio and porosity, enough inter-particle bonding, and unsaturated nature (except quick clays).

It also indicates that densification and to some extent, chemical treatment (e.g., lime and cement) are the common improvement strategies used to mitigate collapse potential of collapsible soils. However, there are concerns around the use of some of these chemicals and the effectiveness of densification processes on collapsible calcareous soils. The following concerns led to the investigation of a sodium silicate treatment:

- Environmental impact and cost of cement production
- Inability to produce impervious layer to prevent water access
- Ineffectiveness of lime on calcium rich material
- Compaction crushes calcite bonds of calcareous soils and increases susceptibility to dissolution.

### **7.2.2 Development and evaluation of an effective system of production of artificial collapsible calcareous soils (objectives 2 and 3)**

This study concludes that an open metastable structure can be replicated in the laboratory through a controlled process that produces fixed amounts of precipitated calcite as bonding element. This was possible through volume-controlled compaction of lime-clay mixes followed by CO<sub>2</sub> gassing.

The process of lime conversion to calcite by gassing was found to be instantaneous. However, its success for a clay-lime system is controlled mainly by density, water content, and lime content. Certain combinations of dry density and water content hinder access of gas to potential reaction sites whereas, high lime contents are suspected to bring about the effect of surface passivation.

The microfabric of manufactured samples can be comparable with that of natural collapsible soils. In the artificial samples, microfabric varies from one sample to another due to differences in proportions of constituent materials. There were no clear trends in this variation, but pore volume was observed to broadly increase with decreasing clay and calcite content.

### **7.2.3 Characteristics of manufactured samples (objectives 4 and 5)**

#### ***Unconfined compressive strength and stiffness of untreated soils***

Calcite cementation is beneficial to the strength properties of fine-grained soils. UCS and stiffness improve with increasing calcite content. However, this effect showed a pattern of diminishing return indicating that there could be a level of calcite content where UCS increase may be zero or UCS decreasing. A similar effect was reported by Demars *et al.* (1976); Agarwal *et al.* (1977) who argued that strength parameters increase with increasing CaCO<sub>3</sub> content but start decreasing at above about 40% to 45% content.

The study shows that calcite bonding has a greater positive effect on soils with higher clay/silt ratios contrary to cement and lime bonding as reported in the literature (e.g., Currin *et al.*, 1976 cited in Sariosseiri and Muhunthan (2009); Teerawattanasuk and Voottipruex (2014)). For instance, an ~12% calcite content in clay/silt of 50/50, 65/35, and 80/20 resulted in UCS of 460, 550, and 867 kPa, respectively. It is possibly due to the higher surface area of clay compared to silt particles that allows for more effective bonding reaction of calcite and clays as suggested by Al-Kaysi (1983) and Nel *et al.* (2006).

The calcite-cemented soils demonstrated a brittle failure suspected to be the result of bond breakage under compressive stress as supported by Fedá (1995b) and Amann *et al.* (2011).

### ***Collapse potential and rate of collapse of untreated soils***

Collapsibility is controlled by a combination of material properties (calcite content, clay content, and dry density) and experimental/external factors (wetting fluid pH, wetting stress, and method of testing). It is apparent from the following conclusions reached, that no single criterion is adequate to predict magnitude of collapse in soils, and that the specifics of particles bonding material type and pH of pore fluid need to be defined in order to provide a complete site characterisation of collapse susceptibility.

Clay/silt content (50/50, 65/35, and 80/20) controlled the collapsibility of non-calcareous, *moderately*, and *strongly* calcareous samples but at higher degree of calcareousness, calcite becomes the influencing factor. Higher clay contents produced higher CP in non-calcareous samples but the trend reverses following the introduction of calcite bonding.

In non-calcareous (0% calcite) samples, collapse is practically entirely mechanically controlled, and the time lag between wetting and complete collapse is instantaneous. The mechanics of collapse is controlled mainly by broken clay bonds. The more clayey the sample, the lesser the pre-wetting compression, which availed greater void space for consequent settlement upon wetting.

In the present study, clay content has not been found to define collapse based on threshold values as suggested in the literature. It was evident that samples with clay contents up to the maximum (80%) studied here are still susceptible to collapse where dry density or bonding is inadequate. On the other hand, high calcite contents up to about 40 % have been found to eradicate collapse in the silty-clay soils irrespective of their physical properties. For instance, notwithstanding the relatively low dry densities ( $1\pm 0.1\text{Mg/m}^3$ ) and high void ratios of samples, insignificant collapses were recorded in the high  $\text{CaCO}_3$  samples.

Although dry density was a collapse-controlling factor, it was complemented by the degree of calcareousness. Specimens with nominally comparable dry densities but varying calcite contents collapsed with increasing calcite content reducing CP. Although the reason why samples with high calcite contents did not exhibit as high collapse as anticipated was attributed to there being insufficient volume of fluid in the oedometer cell to dissolve adequate amounts of calcite. On the other hand, samples with comparable degree of calcareousness but differing dry densities, collapsed with increasing dry density reducing CP.

A comparison of uncemented calcareous and cemented calcareous samples reveals a higher CP being produced by the former. This indicating that the mode of calcite occurrence can influence magnitude of collapse.

Increasing wetting stress increases CP, but only up to a maximum, above which it begins to decrease with increasing wetting stress.

CP results have demonstrated that both the single oedometer test (SOT) and double oedometer test (DOT) can produce comparable results under certain conditions. However, outside those conditions, variances occur. Therefore, it can be concluded that the DOT, which normally applies as a quick method for estimating CP may be used only as a guide where anticipated field conditions are taken into consideration. This, nonetheless, appears to be well established in the literature.

#### **7.2.4 Relationship between UCS and collapse potential (objective 6)**

The UCS and CP results have demonstrated two facts. First, in calcareous samples, an increasing UCS reduces CP, implying that UCS is somewhat inversely proportional to CP. On the other hand, however, due to the effect of calcite dissolution (in low pH wetting), a stronger sample (moderately calcareous) can collapse more than a weaker one (non-calcareous). That is, UCS was directly proportional to CP. This means that in certain conditions, strength should not be exclusively used to define collapsibility.

#### **7.2.5 Evaluation of the impact of wetting fluid pH (objective 7)**

Generally, acidic solution wetting produces higher collapse potentials in calcareous soils than distilled water wetting because of the effect of calcite dissolution.

Where distilled water applies as wetting fluid, complete collapse is achieved faster in calcareous soils than in non-calcareous ones because of calcite support and its inertness to water dissolution.

Additionally, wetting of non-calcareous samples with low pH fluid result in quicker collapses compared to wetting with distilled water. This perhaps is due to the ability of acids to destroy the chemistry and structure of clay (Panda *et al.* (2010); Komadel (2016)).

Results revealed that depending on certain conditions (e.g., dry density and calcite content) acidic solution wetting will produce higher collapse potential (CP) in a calcareous soil compared to a non-calcareous one of similar physical properties. For example, the three samples

with ~12% calcite content yielded CPs higher than their counterparts having 0% calcite by a range of approximately 10 to 30 %.

Calcite content and wetting fluid type defined collapses as either rapid (< 10 minutes) (“*collapse settlement*”) or slow (> 10 minutes) (“*subsidence settlement*”) according to Fookes and Best (1969). The findings here showed that in high calcite contents ( $\geq 25\%$ ) subsidence settlement will continue for a long time where soil was soaked with a low pH solution.

#### 7.2.6 Potential of sodium silicate treatment (objective 8)

From the results of the two strategies of treatment employed (impregnation, and mixing & kneading), it is concluded that a well-selected formulation of a  $\text{Na}_2\text{SiO}_3$  solution (defined in terms of  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio and concentration) improves the UCS and CP of calcareous and non-calcareous fine-grained soils even in a “single-step” application. However, better performance (UCS) are achieved in calcareous ones.

Calcareous samples develop a lesser susceptibility to wetting by both distilled water and acidic solution post treatment. Nonetheless, specimens still exhibited some level of collapsibility, possibly due to their initial low dry density and ductility of the cured silicate. Collapse mitigation was between 59 and 78 %.

The effectiveness of  $\text{Na}_2\text{SiO}_3$  solution is controlled by  $\text{SiO}_2/\text{Na}_2\text{O}$  molar ratio and concentration as well as the degree of soil calcareousness. Other factors are curing time and method, and treatment technique. These factors affect the rate and type of silicate bond formation and the sample’s syneresis (i.e. moisture loss and shrinking). The degree of calcareousness influenced the type and concentration of solution suitable for a good workability (or absorbability) and effective bond development.

Treatment by mixing & kneading enhances UCS better than treatment by impregnation. This is perhaps due to a more even distribution of solution caused by mixing. UCS and stiffness increase with curing time and increasing amount of applied solution. 50% (by mass of dry soil) solution was found to be the optimum for the present calcareous soils.

Furthermore, 24-hour heat curing produces higher UCS than curing 28 days under room-ambience conditions. At early curing (7-days), treatment reduces stiffness (brittleness), which subsequently redeveloped with curing time. The greater the amount of solution applied, the longer the curing time necessary for samples to achieve full performance (in terms of maximum possible strength and stiffness). Higher amounts of solution also result in increased ductility and strain to failure. However, insufficient amounts of solution give rise to the development of

large cracks following drying, causing lower than anticipated strength and increases brittleness due to stress concentration.

Finally, it was concluded that for an effective stabilization to occur with  $\text{Na}_2\text{SiO}_3$  treatment, an amount of solution exceeding that which satisfies the chemical equilibrium of reactants and products is needed. Therefore, it is recommended that a full-scale field application of  $\text{Na}_2\text{SiO}_3$  should be preceded by a laboratory treatability studies and a field trial.

### 7.3 Further Research

Because the conventional oedometer cell used in the present study holds only a few millilitres of fluid, the fluid may become chemically saturated and not able to fully dissolve calcite out of the soil system. Thus, CP may be underestimated for high carbonate samples. To fully understand the impact of calcite on the CP of soils, a modified oedometer system that allows draining and replacement of wetting fluid, is recommended for future testing to replicate field flow conditions.

In this thesis, the collapse mechanism has been mainly analysed based on volume change occurring at time intervals. This volume decrease was believed to arise from structural breakdown due to softening/weakening of bonds and clay clods (macropeds) upon loading and wetting. Although interpretations fit in to the micromechanics and microstructural frameworks of unsaturated collapsible soils, there were no real-time measurements to understand the effect of the internal forces on the process of structural breakdown. The traditional oedometer system adopted here did not allow for the real-time measurement of internal forces acting at particle contacts, which is useful in both the micromechanics and effective stress frameworks for fully understanding collapse mechanism of partially saturated soils.

Langroudi (2014) identified measurement of stiffness (resistance against deformation by applied load) in terms of the small-strain shear modulus,  $G_{\max}$  at particle contacts using an integrated Bishop triaxial testing apparatus equipped with bender elements. An alternative method is the use of a hybrid oedometer cell integrated with bender elements (i.e., acoustic oedometer). Also, suction-controlled wetting tests similar to those reported in Fredlund and Gan (1995); Gallipoli *et al.* (2003); Wheeler *et al.* (2003) could be used to determine the relationship between loss of suction and collapse for these soils. It is suggested that any of these alternative systems could be adopted in future research that further investigates artificial calcareous soil behaviour to provide real-time measurements of inter-particle forces to

supplement the results presented in section 4.4 of this thesis for further understanding. The results could then be used to develop a constitutive model for collapsible calcareous soils.

The carbonation of lime-treated soils is reported by some researchers to cause micro-cracking (Vance *et al.*, 2015) and weakening of such soils due to the expansion of lime molecules (Bagoniza *et al.*, 1987). However, findings in the present study indicated strengthening with carbonation. Perhaps, it is due to the low density of samples investigated herein. These samples had enough voids to accommodate the lime molecules expansion without causing cracking. It is suggested that a more robust testing of samples with greater densities than the present will possibly provide clearer understanding of carbonation impact on compressive strength vis-a-vis cracking of engineered lime-soil systems.

Even though the system used to produce samples here proved effective in precipitating calcite, it was too simplistic and did not provide room for the control of surrounding conditions (e.g., temperature and humidity), which have capacity to impact on the lime-carbon dioxide reaction. It is recommended that future research should consider such factors. Perhaps they could be controlled to produce calcium carbonate polymorphs other than calcite (e.g. aragonite and vaterite).

Calcite precipitations higher than theoretically expected amounts were observed in few cases. This may have been the result of carbonation of minor compounds occurring in the natural silt material used to constitute samples. Alternatively, the hydrated lime used also consisted of about 3 – 5 % other metal compounds and oxides, such as magnesium hydroxide ( $Mg(OH)_2$ ), iron oxide ( $Fe_2O_3$ ), alumina ( $Al_2O_3$ ), Lead (Pb), and Manganese (Mn), which can react with  $CO_2$  to form other minor carbonates. Some of these are heavier than  $CaCO_3$  so could have resulted in the excess amounts of carbonate noted. Advanced chemical testing methods (e.g. XRF) should be deployed or artificial materials could be adopted to clearly understand the cause of the apparent excess carbonate precipitations.

While some of the SEM images of studied specimens revealed structures comparable to natural soil materials, the image quality was deemed unsatisfactory. Apart from the inability to adequately identify the mode of calcite occurrence from the SEM images, poor contrast was another issue. Alternative image acquiring techniques such as XRCT should be investigated for their ability to produce better images for use in microstructural analysis.

Considering that a single-step sodium silicate treatment of oedometric specimens proved inadequate to eradicate collapse at relatively high overburden stress (300kPa), investigation into post-silicization modifications, leading to multi-step treatment strategy such as those used by

Terzaghi 1947 and Riedel 1952 (Hurley and Thornburn, 1972); Sokolovich and Gubkin (1970); Sokolovich and Ibragimov (1971) or subsequent compaction should be investigated for greater effectiveness.

Despite the successes of silicatization in mitigating collapse here, concerns may be raised as to whether the successes are due to the static nature of wetting fluid and confinement of treated specimen in the oedometer system, which prevents it from the chemical deterioration effect of wetting fluid. A modified collapse testing system that simulate field conditions allowing fluid flow can be used to further investigate the impact of silicatization on collapse.

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## Appendices

### A1 – Typical Commercial Sodium Silicate Compositions

Table A1-1 Compositions of sodium silicate as produced by Ingessil S.R.L, supplier of the sodium silicate solution used in the present study

R	SiO <sub>2</sub> %	Na <sub>2</sub> O %	Conc. %	d <sub>20</sub> g/ml	°Bé <sub>20</sub>	pH	Visc. <sub>20</sub> mPa·s
1,6	26,25	16,40	42,65	1,526	50	13,26	270
1,6	27,50	17,19	44,69	1,559	52	13,27	550
1,7	27,04	15,90	42,94	1,526	50	13,15	260
1,7	28,31	16,66	44,97	1,559	52	13,16	550
1,8	27,79	15,43	43,22	1,526	50	13,04	255
1,8	29,09	16,16	45,25	1,559	52	13,06	530
1,9	28,50	15,00	43,50	1,526	50	12,94	280
1,9	29,83	15,70	45,53	1,559	52	12,95	560
2,0	27,86	13,93	41,79	1,495	48	12,82	180
2,0	29,19	14,60	43,79	1,526	50	12,83	330
2,1	28,50	13,57	42,07	1,495	48	12,72	200
2,1	29,85	14,22	44,07	1,526	50	12,73	330
2,2	29,11	13,23	42,34	1,495	48	12,62	200
2,2	30,49	13,85	44,34	1,526	50	12,63	400
2,3	29,70	12,91	42,61	1,495	48	12,51	215
2,3	31,10	13,51	44,61	1,526	50	12,53	460
2,4	30,26	12,61	42,87	1,495	48	12,41	270
2,4	31,68	13,20	44,88	1,526	50	12,42	600
2,5	28,68	11,47	40,15	1,450	45	12,29	140
2,5	30,81	12,32	43,13	1,495	48	12,31	400
2,6	29,17	11,22	40,39	1,450	45	12,19	150
2,6	31,34	12,09	43,39	1,495	48	12,21	550
2,7	26,07	9,66	35,73	1,381	40	12,07	50
2,7	27,49	10,18	37,67	1,408	42	12,08	80
2,8	26,49	9,46	35,95	1,381	40	11,97	55
2,8	27,93	9,97	37,90	1,408	42	11,98	90
2,9	26,89	9,27	36,16	1,381	40	11,87	65
2,9	28,35	9,77	38,12	1,408	42	11,88	115
3,0	26,54	8,85	35,39	1,368	39	11,77	65
3,0	28,01	9,34	37,35	1,394	41	11,78	120
3,1	26,91	8,68	35,59	1,368	39	11,67	75
3,1	28,40	9,16	37,56	1,394	41	11,68	150
3,2	26,50	8,28	34,80	1,355	38	11,57	50
3,2	28,01	8,75	36,76	1,381	40	11,58	150
3,3	26,84	8,14	34,98	1,355	38	11,47	100
3,3	28,36	8,60	36,96	1,381	40	11,48	250
3,4	25,65	7,54	33,19	1,330	36	11,37	65
3,4	27,17	7,99	35,16	1,355	38	11,38	155
3,5	24,42	6,98	31,40	1,306	34	11,26	50
3,5	25,95	7,41	33,36	1,330	36	11,27	120
3,6	23,93	6,65	30,58	1,295	33	11,17	40
3,6	25,47	7,07	32,54	1,318	35	11,18	100
3,7	23,42	6,33	29,75	1,283	32	11,07	25
3,7	24,97	6,75	31,72	1,306	34	11,08	50
3,8	22,11	5,82	27,93	1,261	30	10,97	20
3,8	23,67	6,23	29,90	1,283	32	10,98	50

Table A1-2 A typical commercial composition of sodium silicate (after Iler (1979))

$SiO_2$ (%)	$Na_2O$ (%)	Weight ratio $SiO_2:Na_2O$	Density ( $g/cm^3$ )	Viscosity ( $Pa.s$ )
25.3	6.75	3.75	1.32	0.22
29.9	9.22	3.25	1.41	0.83
28.4	8.7	3.25	1.38	0.16
27.7	8.6	3.22	1.37	0.1
32.0	11.1	2.87	1.48	1.25
32.1	12.5	2.58	1.5	0.78
26.5	10.6	2.5	1.4	0.06
33.2	13.85	2.4	1.55	2.10
29.2	13.3	2.2	1.49	-
29.4	14.7	2.0	1.53	0.4
36.0	18.0	2.0	1.69	70.0
28.5	15.0	1.9	1.52	-
24.1	13.4	1.8	1.43	0.06
31.5	19.7	1.6	1.68	7.0

## A2 – Soil Preparation and Moulding of Samples

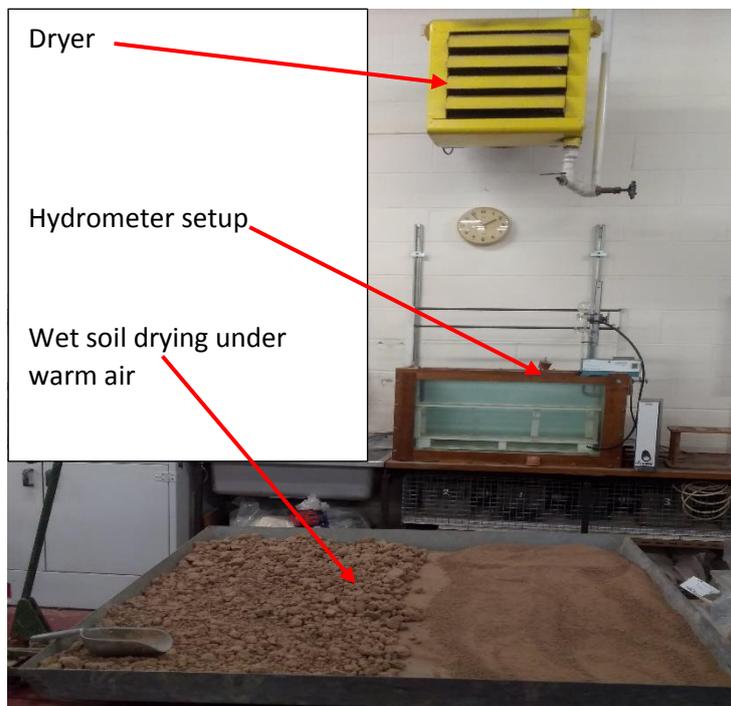


Figure A2-1 Drying of Lanton alluvium before extraction of sub  $63\mu m$  portion, and subsequent hydrometer experiment

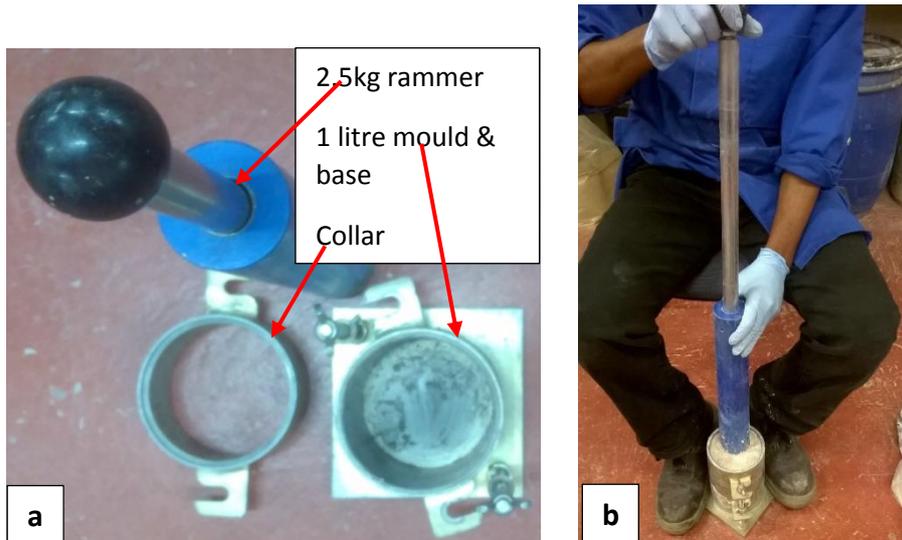


Figure A2-2 Standard light Proctor – determining dry density/moisture content relationship (a) apparatus components (b) compaction in progress

### A3 – Preliminary Carbonation Setup Leading to the Advent of Innovative Setup

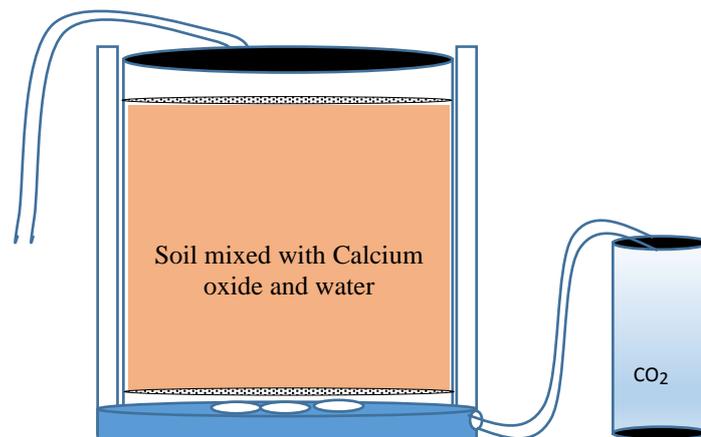


Figure A3-1 Preliminary carbonation setup (reacting calcium oxide and carbon dioxide)

A preliminary study was initiated where availability of materials informed an initial reaction of quicklime (CaO) and carbon dioxide (CO<sub>2</sub>) using a reaction system shown diagrammatically as Figure A3-1 above. The aim was to assess both efficiency of the reaction system as well as the effectiveness of the reactants (CaO and CO<sub>2</sub>) to precipitate relatively, predictable amounts of calcium carbonate. The following pilot test was carried out. 20% by mass of dry soil (i.e. approximately 16.67% by total mass) CaO was carefully mixed with kaolin in dry state by hand, and then 20% water (by mass of soil-lime mix) was added and thoroughly kneaded for several minutes and stored in sealable plastic bags to hydrate overnight. A reasonable amount of water

from the mix had condense on the plastic bag. Therefore, the mix was re-kneaded. Theoretically, the reaction of lime and CO<sub>2</sub> as shown in Equations 3.1 and 3.2 is independent of water. However, water was only thought to allow placement of the soil-lime mix to desired density. The wet soil-CaO mix was then placed in a 9.12x10<sup>-4</sup>m<sup>3</sup> (150mm height by 88mm diameter) compaction cylinder by tapering with a small wooden bar in approximately three equal layers. A relatively low density was thought appropriate for the sample to avoid such factor influencing the CO<sub>2</sub> flow. Two porous discs were placed on top and base of the cylinder fitted with a gas inlet and an outlet, then CO<sub>2</sub> gas was continuously passed through at an ~50kPa pressure for about six hours. In spite of the fact that higher gas pressures may be necessary for effective CO<sub>2</sub> diffusion for this reaction, because of the nature of reaction setup a relatively low pressure was thought to avoid creation of vents in the carbonated sample. After carbonation, sample was then oven dried at temperatures ranging between 105 and 110 °C for about 24 hours, cooled and then fizzing test using dilute HCl was initially applied to confirm presence of CaCO<sub>3</sub>. Three specimens were then taken from top, middle, and bottom of the cylindrical sample for quantitative determination of the CaCO<sub>3</sub> contents. The results from a Leco experiment revealed CaCO<sub>3</sub> contents less than expected, and also varying from one location to the other. 18.3, 17.4, and 16.3 % were reported from the sample top, middle and bottom, respectively, instead of an approximately 26.26 % expected. Recall from basic molar mass calculation, theoretically, a mole of CaO reacting with CO<sub>2</sub> and producing CaCO<sub>3</sub> would have a molar mass conversion ratio of 1:1.78. That is, every 1g of CaO carbonated should yield 1.78g of CaCO<sub>3</sub> (78% mass increase) in the case of a 100% degree of conversion. Therefore, carbonation of a 16.67% CaO should have been converted to ~26.26% calcium carbonate by total mass of carbonated soil. The obvious discrepancies noted above are thought to have resulted from incomplete and uneven carbonation process which may have been due to the ineffectiveness of reaction setup and or short reaction period.

The following drawbacks were identified from the above soil-CaO-CO<sub>2</sub> reaction as well as reaction setup:

- Though this reaction is known to be instantaneous in nature, the big question is whether the reaction apparatus effectively distributed permeating CO<sub>2</sub> gas to reach all possible reaction sites within the soil-lime system. On the other hand, can it be possible that CO<sub>2</sub> escaping from the outlet pipe could have only passed through weak zones developed within the sample?
- The longer the permeation time allowed, the more CO<sub>2</sub> is lost or wasted assuming vents were created in the sample. This results to cost ineffectiveness and air pollution

- The carbonation of quicklime will first go through the slaking process forming hydrated lime before proceeding to precipitate calcium carbonate. The slaking process requires large volumes of water whereas large amounts of water may impede carbonation reaction, which is diffusion controlled. Therefore, if quicklime is to be used in this process, complete slaking should first be achieved before proceeding to carbonation. Complete slaking can be complex to evaluate within the scope of this study.
- Apart from the health and safety, challenges identified with the usage of CaO, the heat generation from the exothermic reaction during slaking causes significant loss of water through evaporation and makes it difficult to estimate the amount of water used to prepare samples. Moisture content is necessary to determine samples initial dry density
- The increased temperature from the exothermic reaction would reduce CO<sub>2</sub> density making it easier to permeate through (possible weak zones) the soil-lime system to escape. In addition, CO<sub>2</sub> becomes even less diffusive and thus, delaying the carbonation process.

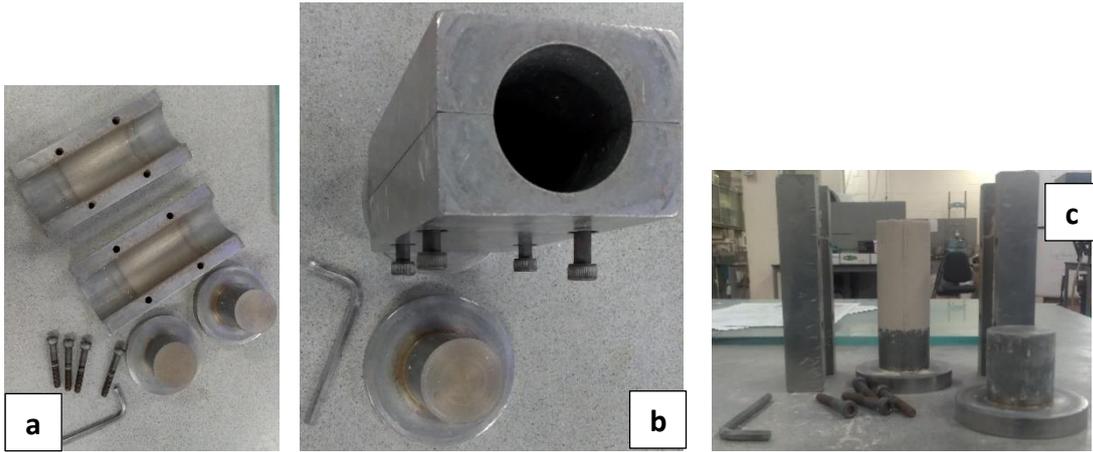
#### A4 – Preparing of Oedometric and Cylindrical UCT Specimens



Figure A4-1 Calcareous oedometric specimen shaved into ring from a bulk-carbonated high-density sample



Figure A4-2 Split mould components for moulding low dry density oedometric specimens (a) components (b) assembled (c) specimen compacted into ring



*Figure A4-3 Split mould components for moulding cylindrical compressive specimens (a) components (b) assembling mould (c) disassembling mould post specimen compaction*

## **A5 – Principle and Procedure for the Leco Technique of Determination of Calcium Carbonate Content in Samples**

### **Method S003: Soil Analysis - Decarbonisation of Soil for Determination of Organic Carbon**

#### **1) Principle of Method**

Approximately 0.1 g of sample, in a porous crucible, is treated with sufficient hydrochloric acid, 4 mol/L, to remove carbonates. After the acid has drained from the crucible, the crucible and sample are dried overnight at 65°C. The organic carbon is determined as described in Method I004

#### **2) Apparatus**

Filtering/porous crucibles, Leco part no. 528-028

Crucible tray(s)

Crucible tongs

Balance, readable to  $\pm 0.1$  mg

Pipette, 10 mL graduated in 0.1 mL divisions

Laboratory Oven No. 2, set at  $65^{\circ}\text{C} \pm 5^{\circ}\text{C}$

Aluminium foil

#### **3) Reagents**

**Refer to material safety data sheets.**

**Warning: refer to COSHH risk assessment S003**

**Note:** *Procedures for the preparation of reagents are given in the Appendices indicated.*

**Hydrochloric acid, 4.0 mol/L** (Appendix A)

#### **4) Sample Preparation**

Take a sub-sample of approximately 20 g of air-dried soil, previously crushed to pass a 2 mm sieve, and grind using a ball mill until the whole sub-sample passes through a 250 $\mu\text{m}$  sieve. Use a portion of this sub-sample to determine the dry matter factor as described in Method S002.

#### **5) Quality Control**

Analyse a portion of Inorganic Laboratory Reference Soil No. S003.

Use approximately 0.1 g for analysis

6) **Procedure**

**Warning:** refer to COSHH risk assessment S003

**Warning:** *Safety glasses and rubber gloves must be worn at all times while using hydrochloric acid.*

*All procedures using hydrochloric acid solutions must be performed in a fume cupboard.*

**Note:** *To minimise the risk of contamination, always use tongs when handling crucibles, and do not make identification marks on the crucibles.*

**Note:** *To minimise the risk of contamination, cover the crucibles and crucible tray with aluminium foil.*

Carry out a method blank determination.

- i) Place a crucible on the balance pan.
- ii) Press the tare button to set the scale to zero.
- ii) Weigh approximately 0.1 g of sample into the crucible.
- iv) Record the weight of sample, in g, to the nearest 0.1 mg.
- v) Place the crucible on the crucible tray.
- vi) Record the position number, of the crucible, on the tray.
- vii) Repeat steps 1 to 6 for each sample.
- viii) For the Method Blank determination, place an empty crucible on the crucible tray.
- ix) Record the position number, of the crucible, on the tray.
- x) Record the weight of "Method Blank" as 0.1000 g.

**Note:** *To minimise the risk of contamination, cover the crucibles and crucible tray with aluminium foil.*

- xi) Remove the crucibles from the tray and place in the well of the fume cupboard.

**Note:** *Place the crucibles in the fume cupboard in the same relative positions as the positions on the tray.*

- xii) Cautiously, add 1.0 mL of hydrochloric acid 4.0 mol/L, to each crucible.

**Caution:** *To avoid sample loss, add the acid to the sample, a drop at a time, initially, as vigorous effervescence will occur, due to the production of carbon dioxide, if carbonates are present. the acid may have to be added in several aliquots to avoid loss of the sample.*

- xiii) Allow the acid to drain from the crucible for 4 hours.
- xiv) Place the crucibles on the crucible tray, making sure that they are in their original positions.
- xv) Place the crucible tray in the oven at 60 to 70°C.
- xvi) Leave the crucible tray in the oven for 16 to 24 hours.
- xvii) Remove the crucible tray from the oven.

**Warning:**     *The crucible tray will be hot. Wear heat resistant gloves.*

- xviii) Allow the crucibles to cool.
- xix) To prevent contamination or loss of sample, cover the crucible tray with aluminium foil until the samples are analysed.
- xx) Calibrate the Leco CS230 Carbon/Sulphur Analyser, as described in Method I003, using a minimum of 5 standards.
- xxi) Measure the organic carbon content of Reference Soil No. S003, as described in Method I004.
- xxii) Record the organic carbon content, %, of Reference Soil No.S003.
- xxiii) Measure the organic carbon content of the method blank, as described in Method I004.
- xxiv) Record the organic carbon content, %, of the method blank.
- xxv) Check that the organic carbon value of Reference Soil No.S003 is within the limits stated.
- xxvi) Measure the organic carbon content of the samples, as described in Method I004.
- xxvii) Record the organic carbon content, %, of the samples.

## 7) Calculation

The organic carbon content is calculated as follows:

$$\text{Organic Carbon, \%} = C_s - C_{bl}$$

where

$C_s$  is the measured carbon percentage of the sample,  $C_{bl}$  is the measured carbon percentage of the blank

To calculate the organic carbon content to an oven dry basis:

$$\text{Organic Carbon, \%} = \frac{C_s - C_{bl}}{D}$$

where

D is the dry matter factor, determined as described in Method S002.

### 8) Repeatability

The results of duplicate determinations should be within the limits stated below.

CARBON CONTENT %		ACCEPTABLE VARIATION
GREATER THAN	UP TO AND INCLUDING	
0.0	0.25	0.025 % ABSOLUTE
0.25	7.50	10% RELATIVE
7.50		0.75 % ABSOLUTE

### 9) Preparation of Hydrochloric Acid Solution, 4.0 mol/L

#### i) Apparatus

500 mL measuring cylinder

500 mL amber glass bottle labelled "Soil Analysis, Method S003, Determination of Organic Carbon, Hydrochloric acid solution, 4.0 mol/L"

#### ii) Reagents

Refer to material safety data sheets.

**Warning:** refer to COSHH risk assessment S003A

**Note:** Procedures for the preparation of reagents are given in the Appendices indicated.

**Hydrochloric acid, S.G.1.18, 35%**

#### iii) Procedure

**Warning:** refer to COSHH risk assessment S003A

**Warning:** *Safety glasses and rubber gloves must be worn at all times.*

*This procedure must be performed in a fume cupboard.*

- 1) Add 325 mL deionised water to the 500 mL measuring cylinder.
- 2) Cautiously, add 175 mL hydrochloric acid, S.G.1.18, 35%, to the measuring cylinder.
- 3) Transfer the solution to the 500 mL amber glass bottle.
- 4) Stopper the bottle securely.
- 5) Invert the bottle several times to thoroughly mix the solution.
- 6) Store the bottle in a 'Safepak' container in a storage cabinet.

#### **Method S004: Soil Analysis - Determination of Total Carbon and Sulphur**

##### **1) Principle of Method**

Approximately 0.1 g sample is ignited in a stream of oxygen, oxidising carbon to carbon dioxide, and sulphur to sulphur dioxide. The CO<sub>2</sub> and SO<sub>2</sub> produced are quantified by infra-red detection, using a LECO Carbon/Sulphur Analyser, previously calibrated with a standard reference soil, of known carbon and sulphur concentration.

##### **2) Apparatus**

Crucibles, Leco part no. 528-018

Crucible tray

Crucible tongs

Balance, readable to  $\pm 0.0001$  g, Mettler AE 163, calibrated as described in Method I001.

Aluminium foil

##### **3) Reagents**

**Iron chip accelerator, Leco part no. 501-077**

**Leco cell II accelerator, Leco part no. 763-266**

##### **4) Sample Preparation**

Use air-dried soil, crushed to pass through a 0.5 mm sieve.

##### **5) Quality Control**

Analyse a portion of Inorganic Laboratory Reference Soil No. 1.

## 6) Procedure

**Note:** *To minimise the risk of contamination, always use tongs when handling crucibles, and do not make identification marks on the crucibles.*

At least 2 replicate determinations should be performed.

The sample should be thoroughly mixed by shaking the sample container for 5 minutes, and then left to settle for 2 minutes.

- i) Place a crucible onto the balance pan.
- ii) Press tare, to set the display to zero.
- iii) Weigh approximately 0.1 g of sample into the crucible.
- iv) Record the weight of sample to the nearest 0.1 mg.
- v) Place the crucible on the crucible tray.
- vi) Record the position number, of the crucible, on the tray.
- vii) To prevent contamination of the samples, cover the crucible tray with aluminium foil.
- viii) Calibrate the Leco CS244 Carbon/Sulphur Analyser, using the Carbon/Sulphur standard soil as described in Method I003, using a minimum of 5 standards.
- ix) Add a scoopful of both accelerators to the crucibles.
- x) Measure the total carbon and sulphur content of the reference soil, as described in Method I004.
- xi) Check that the carbon/sulphur readings are within the tolerances stated for the reference soil.
- xii) Measure the total carbon and sulphur content of the samples, as described in Method I004.
- xiii) Analyse a Carbon/Sulphur standard soil after every 5 samples.
- xiv) Check that the carbon/sulphur readings are within the tolerances stated for the Carbon/Sulphur standard soil.

## 7) Calculation

To calculate the total carbon/sulphur content to an oven dry basis:

$$\text{Total carbon/sulphur, \%} = \frac{C}{D}$$

where

C is the measured Carbon/Sulphur concentration, D is the dry matter factor, determined as described in Method S002.

**8) Repeatability**

The results of duplicate determinations should be within the limits stated below.

Carbon Content %		Acceptable Variation
Greater than	Up to and including	
0.0	0.25	0.025 % absolute
0.25	7.50	10% relative
7.50		0.75 % absolute

**References**

BS7755, Section 3.8, 1995; ISO 10694, 1995; Soil Quality, Part 3, Chemical Methods, Section 3.8, Determination of organic carbon and total carbon after dry combustion (elementary analysis)

Leco Corporation, Instruction Manual CS 230

Method I003 - Instrument Operation – Calibration of Leco CS230 Carbon - Sulphur Analyser

Method I004 - Instrument Operation – Operation of Leco CS230 Carbon - Sulphur Analyser

## **A6 – Thermal Analysis of Carbonated Samples to Determine Calcium Carbonate**

### **Content**

**Procedures and methods** - The samples (in dry powder form) were analysed using thermogravimetry (TG) and differential scanning calorimetry (DSC).

A subsample (ca. 38mg) was accurately weighed into an alumina crucible and analysed using a Netzsch Jupiter STA 449C TG-DSC (thermogravimetry-differential scanning calorimetry) system connected by a heated capillary transfer line to a Netzsch Aeolos 403C quadrupole mass spectrometer (QMS). Samples were heated from 25°C to 1000°C at a rate of 10°C min<sup>-1</sup> in an atmosphere of 20% oxygen in helium (purge gas, flow rate 30 ml min<sup>-1</sup>). The protective gas was helium (flow rate 30 ml min<sup>-1</sup>). Adapter heads (Jupiter and Aeolos) and transfer line were at 150°C.

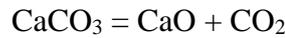
The QMS system was not functioning at the time of analysis: it would have been used to monitor water and carbon dioxide content of the gases evolved during heating of the sample e.g. from the thermal decomposition of organic carbon compounds and carbonate.

TG and DSC data were acquired and processed using Netzsch Proteus 61 software.

The STA 449C is essentially a furnace which surrounds the sample contained in an alumina crucible. The furnace can be heated at a specified rate to temperatures up to 1700C. The atmosphere within the furnace can be selected according to the analysis required, and in this case it comprised of 20% oxygen in helium (i.e. it was an oxidizing atmosphere). The sample crucible rests on a pedestal attached to a micro-balance such that changes in weight during the analysis can be monitored. The crucible rests directly on a thermocouple which monitors changes in sample temperature during furnace heating and is used to monitor heat generated or consumed during any exothermic or endothermic reactions which may occur. Variation in sample temperature is calculated against that of a reference (empty) crucible positioned adjacent to the sample crucible.

The thermal decomposition of carbonate is characterized by weight loss in the temperature range ca. 600 – 800 °C accompanied by an endothermic reaction. Weight loss at ca. 100°C is typically due to adsorbed water and multistage weight loss in the temperature range 200 – 550°C typically reflects oxidative decomposition of the thermally labile, recalcitrant and refractory fractions of the organic carbon component of natural soils.

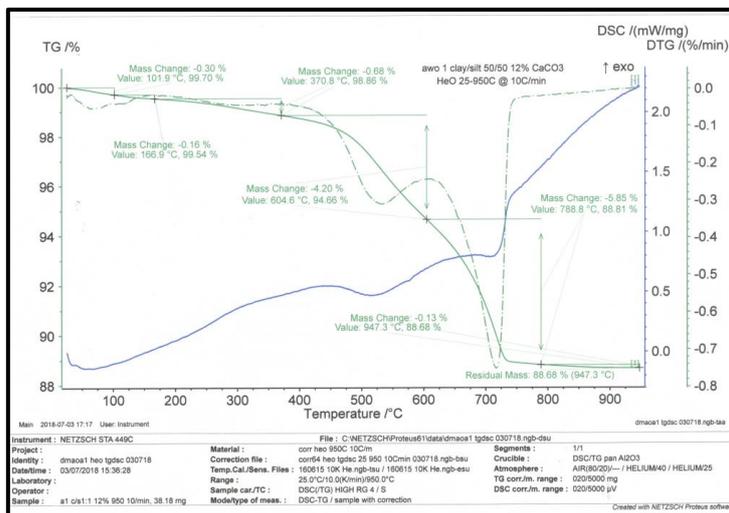
**Calculation of carbonate content** – The proportion of carbonate in the analysed specimen was estimated from calcium carbonate to carbon dioxide (molecular masses) ratio as a fraction of the CO<sub>2</sub> mass lost between 600 – 800 °C, based on the following reaction:



Relative molecular mass (RMM) – 100.09 = 56 + 44

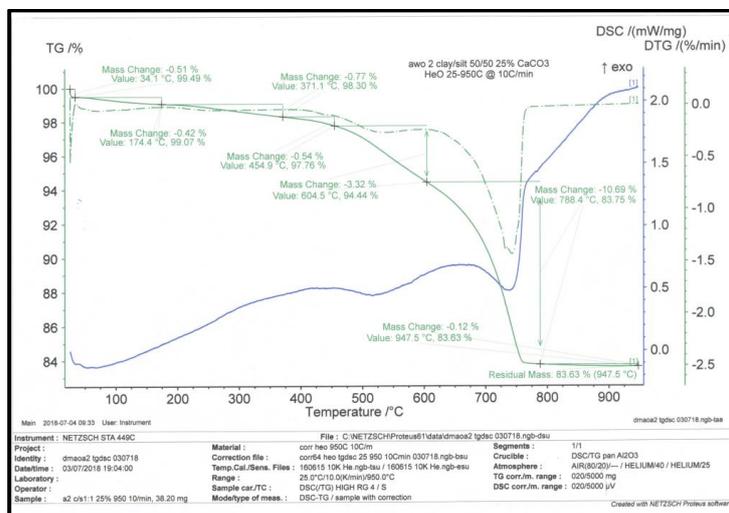
If the CO<sub>2</sub> mass is known, then the amount of CaCO<sub>3</sub> can be calculated. Hence, the following expression was used estimate calcium carbonate content from CO<sub>2</sub> mass loss obtained from Figures A5-1 to A5-6:

$$\text{CaCO}_3 \text{ Content (\%)} = \text{CO}_2 \text{ lost} \times \left( \frac{\text{RMM of CaCO}_3}{\text{RMM of CO}_2} \right) \quad 0$$



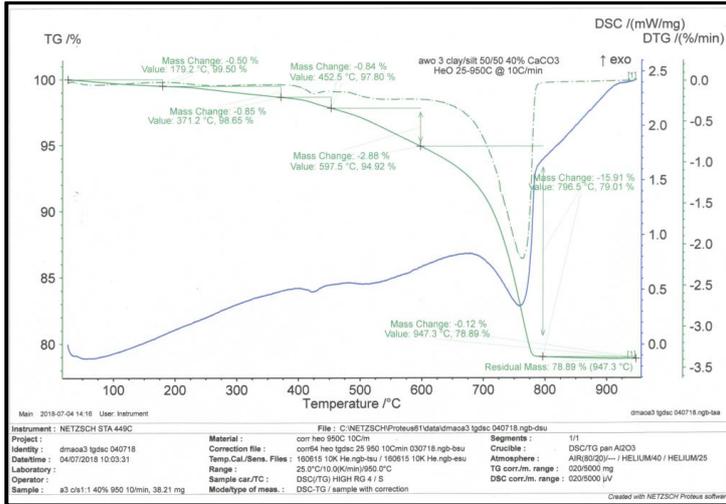
$$\text{CaCO}_3 \text{ Content} = 5.85 \times \left( \frac{100.09}{44.01} \right) = 13.30$$

A6-1 Sample A1 (clay/silt ratio = 1)



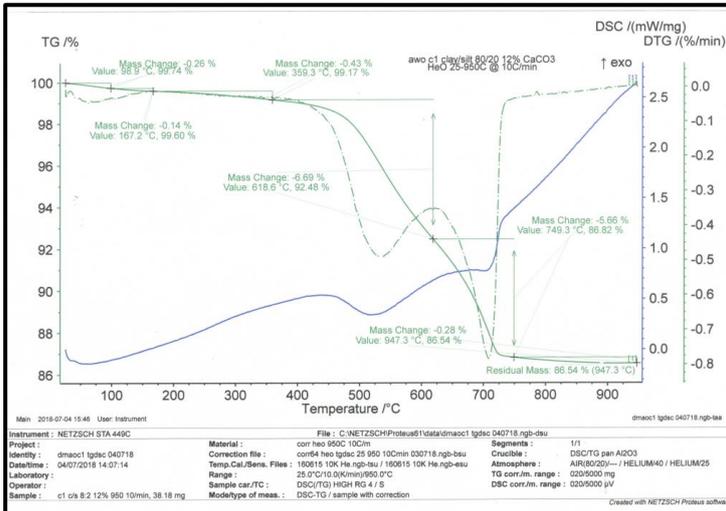
$$\text{CaCO}_3 \text{ Content} = 10.69 \times \left( \frac{100.09}{44.01} \right) = 24.31$$

A6-2 Sample A2 (clay/silt ratio = 1)



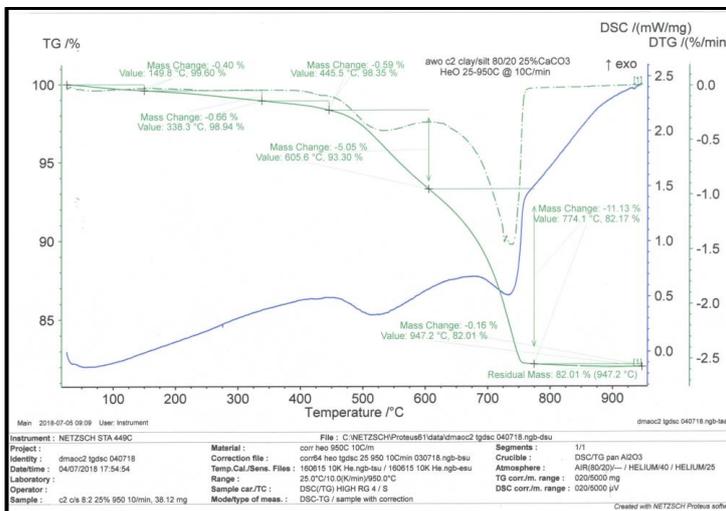
$$\text{CaCO}_3 \text{ Content} = 15.91 \times \left( \frac{100.09}{44.01} \right) = 36.18$$

A6-3 Sample A3 (clay/silt ratio = 1)



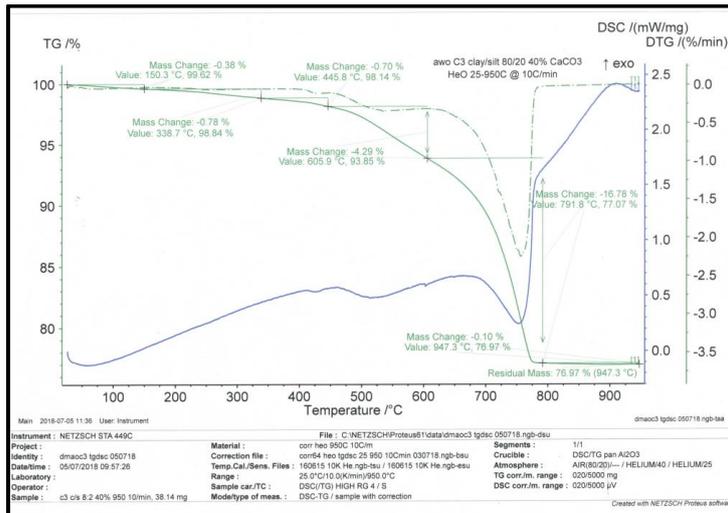
$$\text{CaCO}_3 \text{ Content} = 5.66 \times \left( \frac{100.09}{44.01} \right) = 12.87$$

A6-4 Sample C1 (clay/silt ratio = 4)



$$\text{CaCO}_3 \text{ Content} = 11.13 \times \left( \frac{100.09}{44.01} \right) = 25.31$$

A6-5 Sample C2 (clay/silt ratio = 4)



$$\text{CaCO}_3 \text{ Content} = 16.78 \times \left( \frac{100.09}{44.01} \right) = 38.16$$

A6-6 Sample C3 (clay/silt ratio = 4)

### A7 – Other Methods Tried to Impregnate Specimens



Figure A7-1 Procedures of the treatment by grouting



Figure A7-2 Setup of treatment by pressurized absorption

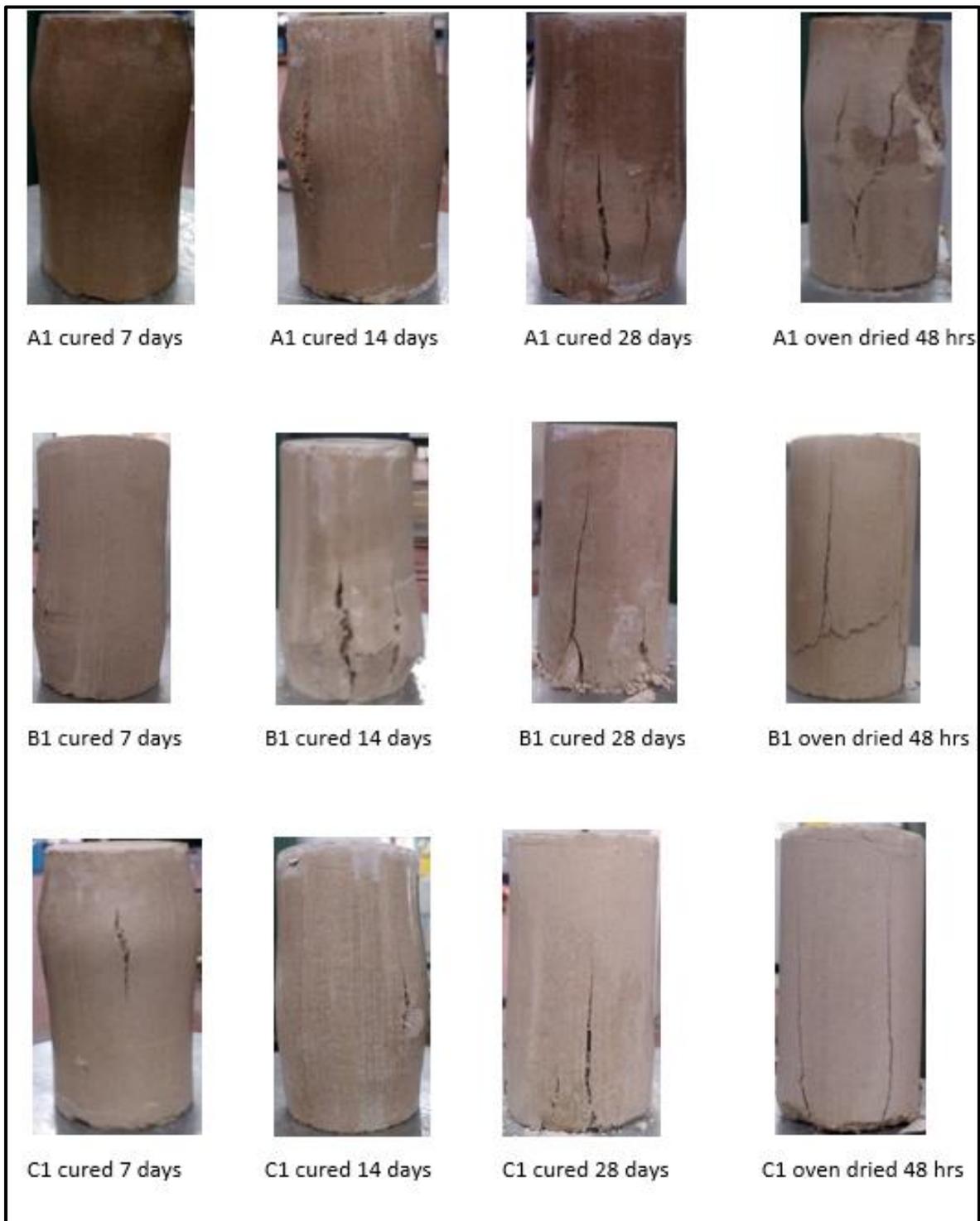
### A8 – Treatment by Hand Mixing and Kneading



### A9 – Curing Specimens Under Room Ambient Conditions



## A10 – Deformation Patterns of Representative Specimens Treated by Impregnation





A2 cured 7 days



A2 cured 14 days



A2 cured 28 days



A2 oven dried 48 hrs



B2 cured 7 days



B2 cured 14 days



B2 cured 28 days



B2 oven dried 48 hrs



C2 cured 7 days



C2 cured 14 days



C2 cured 28 days



C2 oven dried 48 hrs



A3 cured 7 days



A3 cured 14 days



A3 cured 28 days



A3 oven dried 48 hrs



B3 cured 7 days



B3 cured 14 days



B3 cured 28 days



C3 cured 7 days



C3 cured 14 days

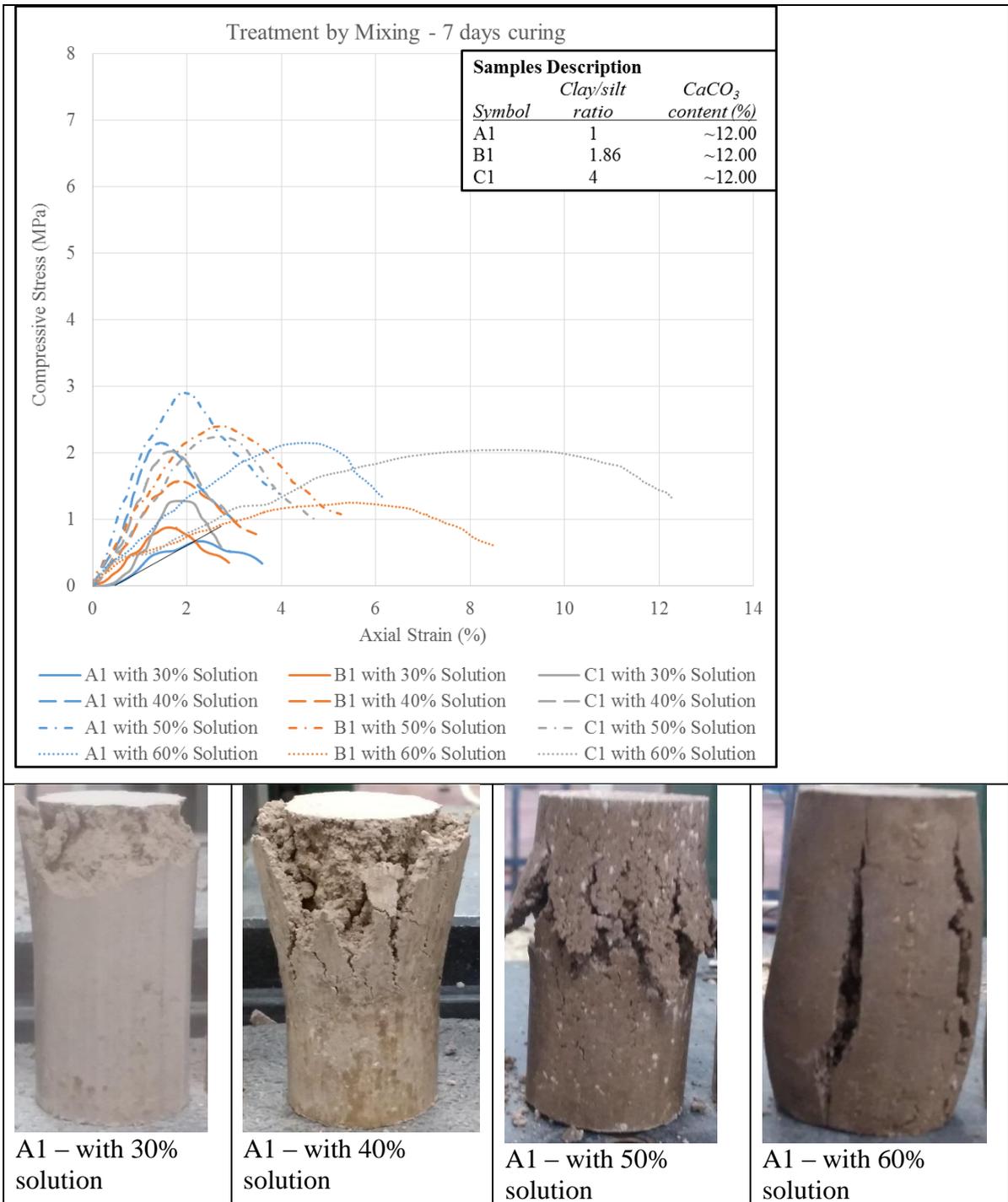


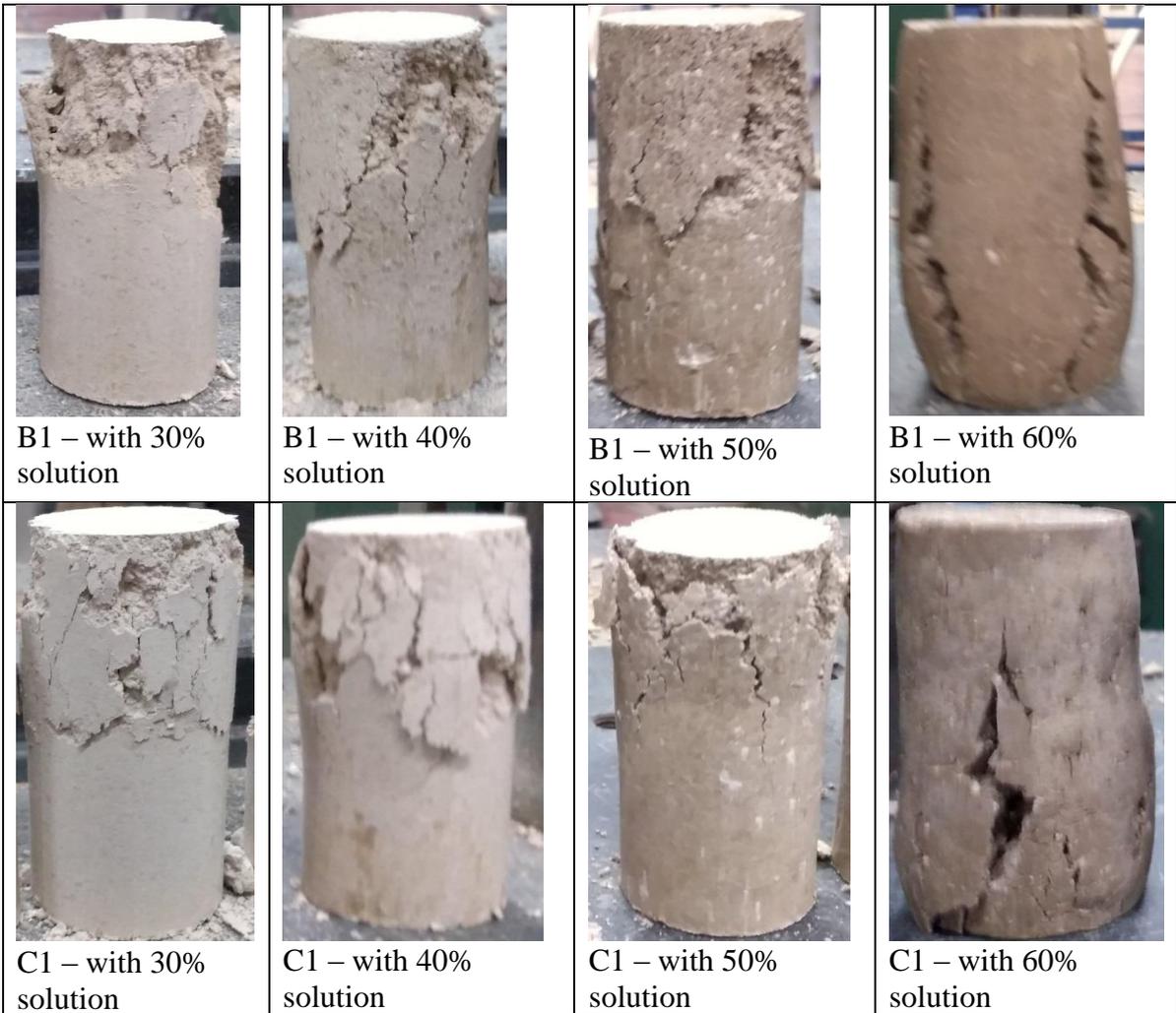
C3 cured 28 days

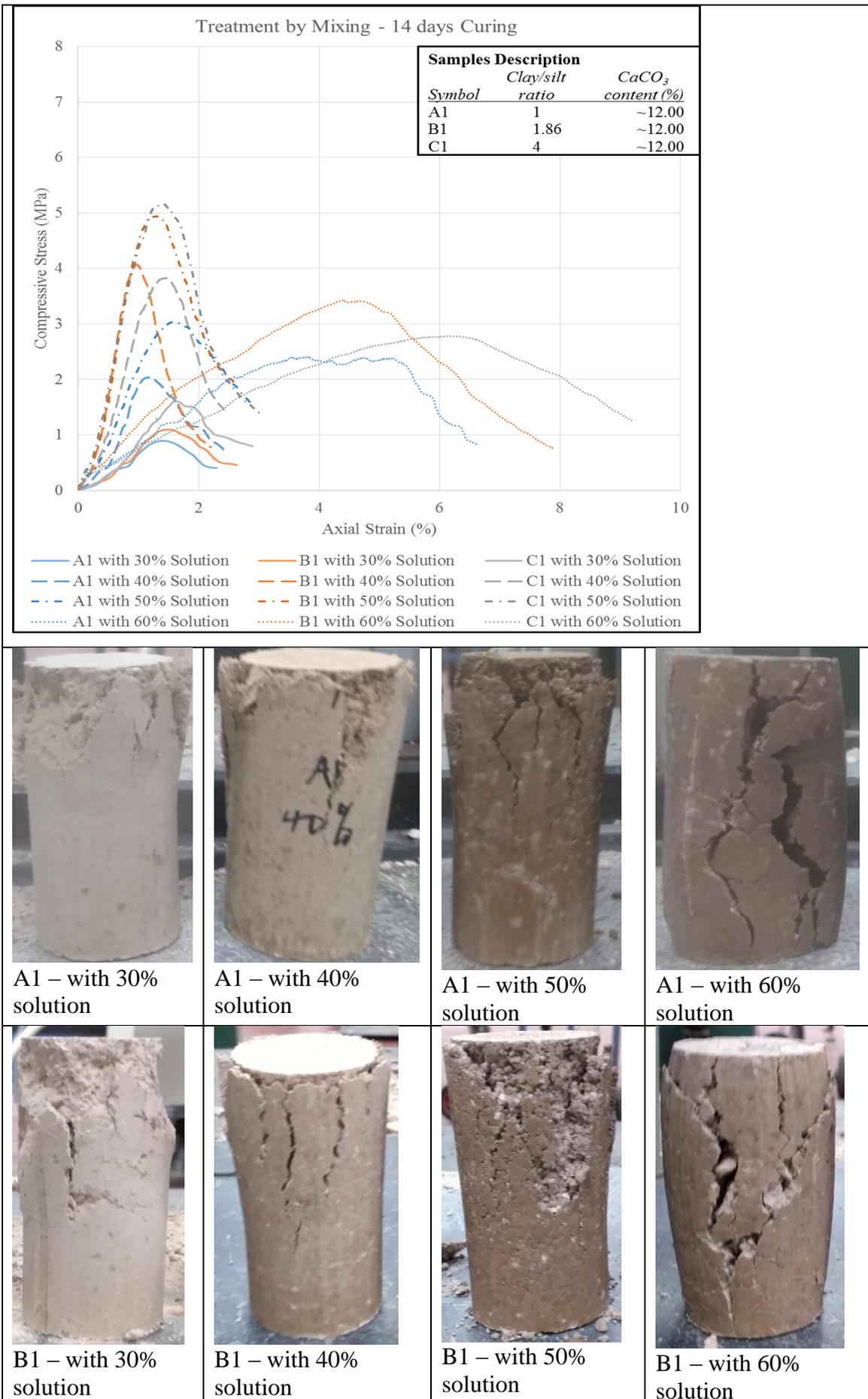


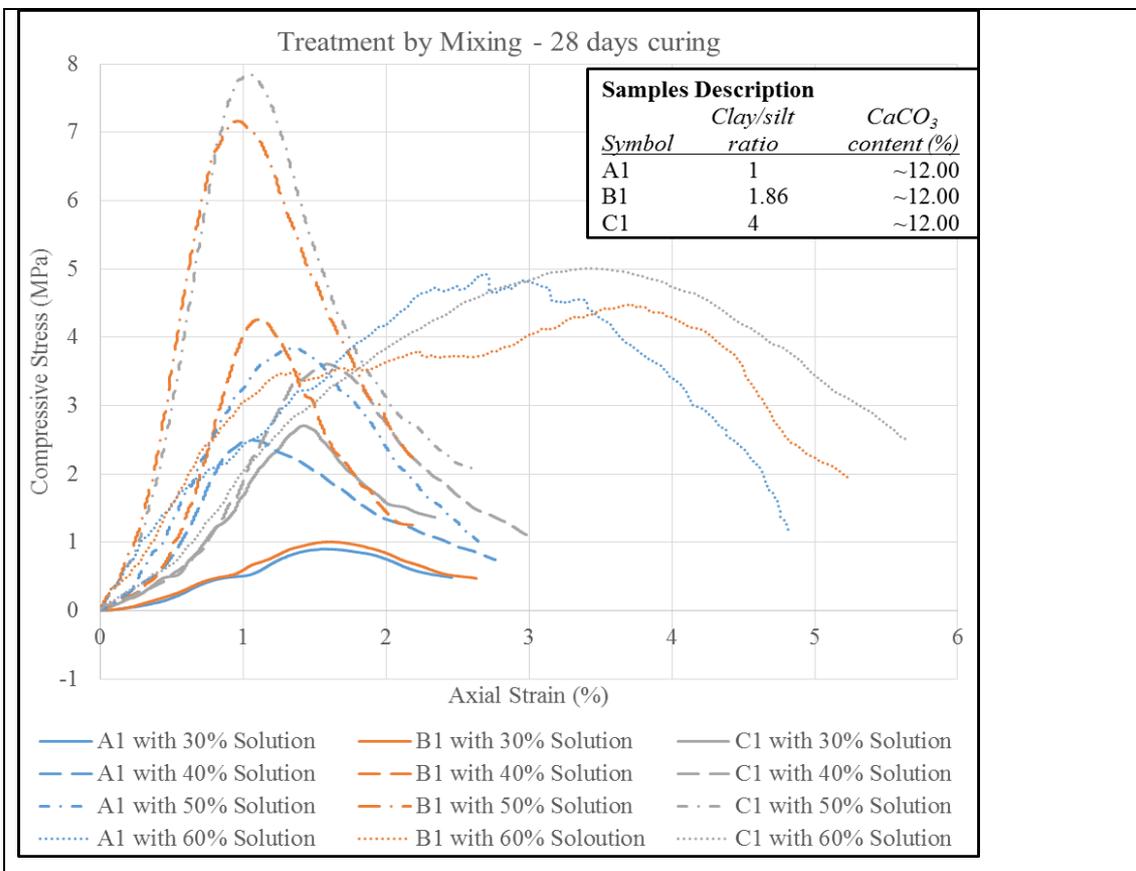
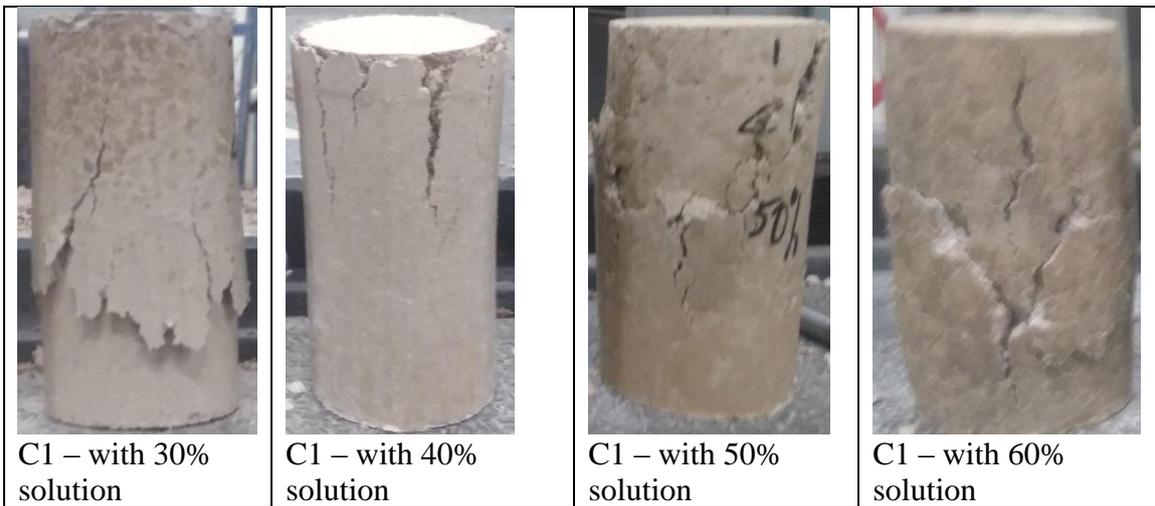
C3 oven dried 48 hrs

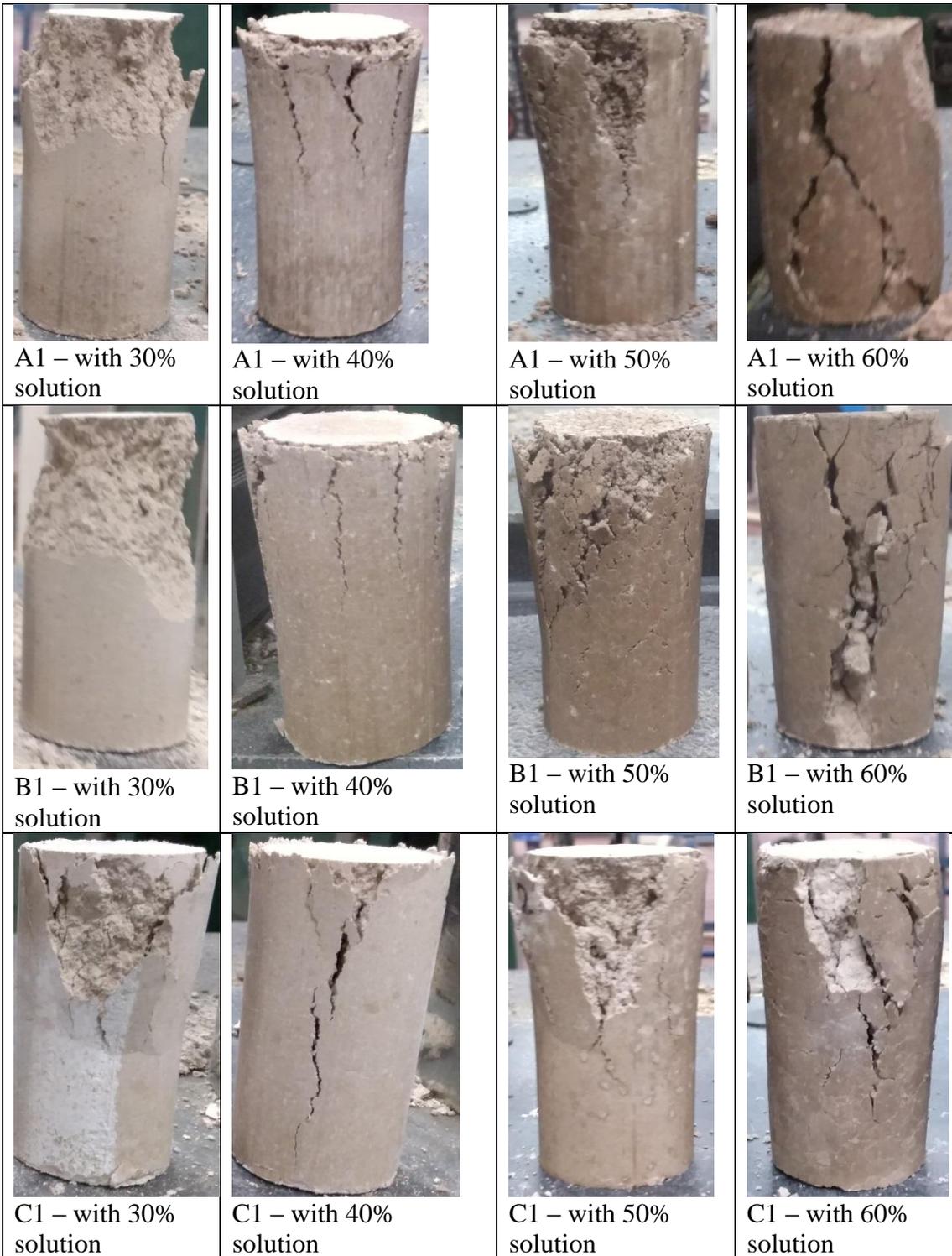
## A11 – Stress-Strain Relationship and Deformation Patterns of Specimens Treated by Hand Mixing and Kneading











## **A12 – Output (List of Disseminations)**

### **Conference presentation/paper**

#### *Impact and potential of calcareous Soils on Earthworks*

Opukumo, A.W. *et al.* (2016). 14<sup>th</sup> BGA Young Geotechnical Engineers Symposium. University of Strathclyde, Glasgow, 5<sup>th</sup> – 6<sup>th</sup> July 2016.

#### *Collapse impact of calcium carbonate in unsaturated clay soils*

Opukumo, A.W. *et al.* (2018). The 7<sup>th</sup> International Conference on Unsaturated Soils (UNSAT2018), Hong Kong, 3<sup>rd</sup> – 5<sup>th</sup> August 2018.