

**Faculty of Science and Engineering
Department of Civil Engineering**

**An Experimental Investigation on Stabilisation of Clay Soils
with Fly-ash Based Geopolymer**

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**This thesis is presented for the Degree of
Doctor of Philosophy
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DECLARATION

To the best of my knowledge and belief, this thesis contains no material previously published by any other person except where due acknowledgement has been made.

This thesis contains no material which has been accepted for the award of any other degree or diploma in any university.

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ABSTRACT

This thesis focuses on the use of fly-ash based geopolymer incorporating ground granulated blast furnace slag (GGBFS) for stabilisation of clay soils. In the last decades, several environmental issues stemmed from the use of ordinary Portland cement (OPC) and lime as traditional binders; the carbon footprint and high-energy consumption associated with the manufacturing of these binders form environmental limitations. Where bulk soil stabilisation is required, the consumption of these traditional binders is relatively high. Hence, the industry immensely needs new alternative binders featuring low environmental footprint without compromising soil stabilisation capabilities. Fly-ash based geopolymers through alkali-activated cement constitute such binders as they involves useful recycling/diversion of discarded aluminosilicate industrial waste material.

Usually, geopolymers necessitate curing at elevated temperature (60-80°C) to accelerate the formation of the cementitious products, thus, limiting the use of this technique in soil stabilisation. However, ambient curing is possible by the addition of a calcium-based component such as GGBFS, which is also a by-product waste material. The wide utilisation of fly-ash based geopolymer incorporating GGBFS in soil stabilisation at ambient temperature entails carrying out an extensive investigation into the engineering properties of treated soils and requires proper selection of the mixing proportions. Practical guidelines are also needed to gain an economic confidence and to support broader use of this technique for soil stabilisation; these guidelines should consider the soil specimen preparation, soil type, curing condition, testing procedure and mixing criteria. To date, relevant research on geopolymers related aspects is limited and a comprehensive study is warranted.

In this thesis, an experimental program was carried out to study the behaviour of clay soils treated with fly-ash based geopolymer incorporating GGBFS. The experimental program was performed in three stages. In the first stage, unconfined compressive strength tests and scanning electron microscopy analysis were utilised to optimise the geopolymer ingredients (i.e. fly-ash, GGBFS and activator) needed for the soil mixture. Given its well-defined characteristics and availability, kaolin clay was selected as the control soil for this purpose. In the second stage, engineering properties of different clay soil types (engineered and natural) were investigated, including

compaction, plasticity, strength, durability, pH performance and pulverisation. The third stage of investigation focused on understanding the undrained shearing behaviour of geopolymer-treated clays, where extensive triaxial tests under static loading conditions were conducted.

The results indicated that introducing partial replacement of fly-ash with GGBFS, when synthesised and activated in certain ratios, assists in achieving mechanical properties of geopolymer-treated clay comparable to those of OPC stabilised clay. However, the results also identified several influencing factors governing the enhancement of the engineering properties of geopolymer-treated clays, including geopolymer content, curing time and pulverisation, whereas other factors were relevant to mineralogical, plasticity/activity and pH level of clay. The study on undrained shearing behaviour of geopolymer-treated clays revealed the significant influence of some factors such as geopolymer content, confining pressure and loading type on stress-strain behaviour of treated clays. The direct influence of these factors was reflected in the yield strength, initial stiffness, failure envelope, shear strength parameters and contraction–dilation response. Within the range of the clay soils tested, an enhancement in the undrained shear performance was found to be highly driven by the clay mineralogy. Based on the thesis findings, practical procedures were quantitatively developed aiming at examining the suitability of certain clay types for geopolymer treatment and determining the optimum mixture ingredient for an effective treatment. The proposed practical procedures are expected to provide a significant contribution to the current stabilisation practice of clay soils.

LIST OF PUBLICATIONS

The following publications are resulted from the work undertaken in this thesis:

Refereed published journal papers:

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LIST OF NOMENCLATURE

A	Activity index
B	Skempton's coefficient
CD	Consolidated drained
CSR	Cyclic Stress Ratios
CU	Consolidated undrained
c'	Effective cohesion intercept
E_{50}	Modulus of elasticity
EDS	Energy-dispersive X-ray spectrometer
GGBFS	Ground granulated blast furnace slag
I_B	Undrained brittleness index
K_o	Coefficient of earth pressure at rest
LL	Liquid limit
MDU	Maximum dry unit weight
OMC	Optimum moisture content
OPC	Ordinary Portland cement
p	Mean total stress
p'	Mean effective stress
PI	Plasticity index
PL	Plastic limit
pH	Potential hydrogen (acidity and alkalinity scale)
q	Deviator stress
SEM	Scanning electronic microscopy
UCS	Unconfined compressive strength
XRD	X-Ray Diffraction
ε_a	Axial strain
ε_v	Volumetric strain
σ_1	Major (axial) principal stress
σ_3	Minor (radial) principal stress
σ'_1	Effective axial stress
σ'_3	Effective radial stress
Δu	Change in pore water pressure
ϕ'	Effective friction angle

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF THE STUDY

Damage to structures founded on problematic soils (e.g. soft and expansive) have been reported in different locations worldwide and have incurred significant financial losses (Al-Rawas et al. 2006). This problem is particularly significant in Australia, among other countries, where almost 20% of the Australian land is covered by problematic soils (Delaney et al. 2005). The problematic nature of such soils is typically related to their weak structure due to lack of sufficient cementation within the soil particles and because of the presence of expansive clay minerals (Mitchell et al. 2005). Stabilisation of clay soils prior to construction is often necessary, and maybe carried out by either mechanical (e.g. soil reinforcement, densification and dewatering) or chemical (e.g. addition of binders) means (Han 2015). Chemical stabilisation has proven its success as a cost-effective alternative to more expensive methods (e.g. soil reinforcement, densification, and dewatering) and involves in-situ soil mixing with binders that react chemically through hydration and pozzolanic reactions, to promote durable (in ideal cases) alteration of the soil engineering properties (Little et al. 2009). The most widely used stabilising binders are ordinary Portland cement (OPC) and lime; however, in recent decades, the carbon footprint associated with the manufacturing of these traditional binders has raised serious environmental concerns. Globally, the manufacturing of OPC and lime is responsible for 8-10% of the anthropogenic CO₂ emissions per year (Oates 1998; Garcia-Lodeiro et al. 2014a). Considering such emissions along with other detrimental environmental effects related to the sourcing and over exploration for non-renewable raw materials (Tayibi et al. 2009; Garcia-Lodeiro et al. 2014a; Sargent 2014), it is necessary to find new and more sustainable binders able to replace these traditional binders without compromising on the clay/soil stabilisation capabilities.

The use of binders obtained through alkaline activation of aluminosilicate materials represents a possible eco-friendly and low-energy alternative to traditional binders for soil stabilisation since it allows useful recycling/diversion of industrial waste materials (e.g. fly-ash and granulated ground blast furnace slag, GGBFS) (Cristelo et al. 2011; Sargent 2014). Alkaline activation binders derived from low calcium fly-ash,

popularly known as geopolymers, is currently the focus of research for soil stabilisation. Fly-ash based geopolymers have been widely observed to give high strength improvement in treated sand (e.g. Verdolotti et al. 2008; Sargent et al. 2013; Phummiphan et al. 2016; Rios et al. 2016b; Rios et al. 2016a; Rios et al. 2017) and clay soils (e.g. Cristelo et al. 2011; Cristelo et al. 2012; Cristelo et al. 2013; Zhang et al. 2013; Zhang et al. 2015; Liu et al. 2016; Arulrajah et al. 2018). Upon mixing with an alkaline activator, a cementation product called Sodium Aluminate Silicate Hydrate (N-A-S-H), forms from the fly-ash through a three-phase chain of chemical multi-reaction called “geopolymerisation”, leading to cementation of soil particles (Cristelo et al. 2012; Liu et al. 2016; Phummiphan et al. 2016). The use of fly-ash based geopolymer for soil stabilisation is particularly significant in Australia, where coal-fired power plants generate approximately 14 million tonnes of fly-ash per year, with over 58% of it is dumped directly into landfill (Sing et al. 2015). The utilisation of fly-ash based geopolymer may contribute to 80% reduction in CO₂ emissions (Van Deventer et al. 2006; Duxson et al. 2007b), while also consuming 70% less energy compared to OPC manufacturing (Tempest et al. 2009). With the reduced emission and energy consumption, fly-ash based geopolymer has an excellent potential as a sustainable binder for stabilisation of soils including applications of direct surface mixing (e.g. Zhang et al. 2013; Zhang et al. 2015; Phummiphan et al. 2016) as well as deep mixing (e.g. Cristelo et al. 2013; Sargent et al. 2013; Phetchuay et al. 2016).

Despite being energy-efficient and environmentally friendly, geopolymers must overcome practical challenges around curing temperature and the use of high activator content which currently restrict the broad utilisation of this technique in soil stabilisation. To maximise the short-term strength, geopolymers require elevated curing temperatures up to 80°C (Palomo et al. 1999; Sindhunata et al. 2006; Liew et al. 2012), while in the field where geopolymers are used for soil stabilisation, curing occurs at ambient temperatures. At such a low temperature, the rate of geopolymerisation is significantly slower than at elevated temperatures; hence, the strength enhancement of geopolymer-treated soil becomes lower and occurs over a longer timeframe compared to OPC-treated soil (Cristelo et al. 2012). Therefore, to increase the feasibility and competitiveness of fly-ash based geopolymers compared with OPC for soil stabilisation applications, large concentrations of the activator is necessary. However, increasing the activator content increases the overall cost of this

stabilisation method. To reduce the required amount of activator content (i.e. improve cost-effectiveness), while maintaining an effective ambient temperature curing, research has focused on enhancing the reactivity of the fly-ash based geopolymer by adding calcium-based by-product components, such as GGBFS. The addition of calcium oxide forms auxiliary cementation product, i.e. Calcium Aluminate Silicate Hydrate (C-A-S-H), along with the N-A-S-H geopolymer network (Granizo et al. 2002; Yip et al. 2005). Such addition increases the viability of geopolymers for bulk applications such as soil stabilisation; however, limited literature exists on this topic. Accordingly, there is a significant need to conduct studies that can carefully consider the use of GGBFS as a calcium-based component to enhance the performance of geopolymer-clay mixtures at ambient temperature, leading to more reliable utilisation of such a promising technique.

In addition to the above-mentioned restrictions, the underutilisation of geopolymer for specifically clay soil stabilisation may also attribute to the lack of understanding of the different engineering properties of geopolymer-treated clay, compared with other soil types (i.e. sand). For instance, limited research evidence exists in the literature to understand the compaction and plasticity properties of geopolymer-treated clay soil. Most of the available studies focus primarily on improving the unconfined compressive strength (UCS) of geopolymer- clay soil mixture (e.g. Cristelo et al. 2011; Cristelo et al. 2012; Cristelo et al. 2013; Zhang et al. 2013; Zhang et al. 2015; Liu et al. 2016; Phummiphan et al. 2016). However, these studies do not provide insights into the impact of soil/clay mineralogy and plasticity on the treatment results, both of which are known to have a significant impact on the behaviour of soil in general (Mitchell et al. 2005) and particularly on the performance of soil stabilisation (Little et al. 2009). Furthermore, the existing UCS studies were limited to addressing the long-term strength performance under weather effects (i.e. wetting-drying, freezing-thawing and leaching due to constant moisture ingress into the treated soils). However, the UCS test has very limited utility in advanced analyses of geotechnical problems as it fails to address the effects of pore pressure and confining pressure, both of which are known to influence the shearing performance of treated soil greatly. Despite these limitations, only very few studies have investigated the strength behaviour of geopolymer-soil mixtures beyond the UCS. Since the short-term stability involving undrained conditions is most frequently the governing criterion for any geo-

infrastructure supported on saturated clay soils, it is critically important to understand the undrained shear behaviour of both the stabilised and in-situ clays. However, related studies focusing on geopolymers are scarce and mostly dedicated to treating sands rather than clays (e.g. Rios et al. 2016b; Rios et al. 2016a). To effectively utilise geopolymers for clay stabilisation, it is thus essential to extensively evaluate the engineering properties of the geopolymer-clay mixtures, especially given implications of varying clay mineralogy; clay mineralogy is known to have a significant influence on the engineering behaviour of soils in general and particularly clay (Mitchell et al. 2005)

To gain an economic confidence and to support broader use of any binder for stabilisation of clay soils, practical procedures are required; these should consider the specimen preparation, soil type, curing conditions, testing procedures and specific mix design criteria. Such practical procedures are currently non-existent for geopolymer-treated clay soil. There is an immense need to develop practical procedures that assist engineers in applying this binder for the stabilisation of different clay types, including the determination of a proper dosage required for an effective treatment.

To address the gaps mentioned above, the research presented in this thesis will conduct extensive experimental work on fly-ash based geopolymer-incorporating GGBFS for the treatment of different clay soil types.

1.2 OBJECTIVES AND SCOPE OF THE THESIS

Inspired by the challenges in adopting geopolymers for soil stabilisation, this thesis aims to optimise an ambient-cured fly-ash based geopolymer mixture for stabilisation of clay soils, and to investigate and quantify the engineering properties of geopolymer-treated clays, through an extensive experimental program. This research also aims to enhance understanding and provide valuable insights into the shearing behaviour of geopolymer-treated clays. The outcomes of the thesis will be synthesised into practical procedures and guidelines that can be easily used by geotechnical engineers for routine stabilisation practice of clay soils. The specific aims of this research are to:

1. Understand the mechanism and effects of basic mix proportioning of fly-ash geopolymer utilising GGBFS as a calcium-based additive material with the aim to improve the short-term strength properties of the geopolymer-treated mixture at ambient temperature.

2. Optimise mixture components for an effective clay treatment, considering the soil plasticity, strength and durability studies.
3. Analyse and quantify the effects of geopolymer dosage, curing time, clay mineralogy and clay pulverisation on the engineering properties of geopolymer-clay mixtures.
4. Investigate the undrained and drained shearing behaviour of geopolymer-stabilised clay using triaxial tests under static loading conditions.
5. Develop rational practical procedures and guidelines to facilitate successful utilisation of the proposed geopolymer in the stabilisation of clay soils.

1.3 SIGNIFICANCE AND IMPORTANCE

This thesis is primarily concerned with the utilisation of geopolymer for stabilisation of clay soils of different mineralogy and broad plasticity ranges. This project is significant for the following reasons:

- 1- The findings of the study will provide an effective technique to utilise waste materials such as fly-ash and GGBFS as an effective alternative to traditional binders in clay stabilisation, providing economic and environmental benefits.
- 2- The investigation will contribute toward developing a geopolymer recipe for an effective and durable treatment of clay soils at ambient temperature.
- 3- The outcomes of the research will lead to better use of geopolymer in the stabilisation of clay soils, with an improved understanding of the shearing performance of geopolymer-treated clay subjected to static loading, considering the effect of soil mineralogy.
- 4- The results of the research will provide relevant data on the mechanical performance of geopolymer-treated clays, to help in developing the current databases for future studies when dealing with similar clay soils.
- 5- Based on an extensive experimental program, the project will establish rational practical procedures and guidelines that will provide insight to expand the use of geopolymer for clay stabilisation.

1.4 ORGANISATION OF THE THESIS

This thesis is organised into eight chapters; an overview of the work presented in each chapter is described below.

Chapter 1: Introduction. This chapter has introduced the research topic, constructed the rationale for the study and developed the objectives of the research.

Chapter 2: Literature review. This chapter provides an overview of the chemical stabilisation of soils and the mechanism of treatment using lime and cement as traditional binders, with emphasis on their environmental impacts. In this chapter, current knowledge, basic composition, performance and control variables of the geopolymer-soil mixture are also reviewed, and an attention is given to the application of geopolymer to soil stabilisation. Finally, gaps in geopolymer-clay knowledge related to the curing at ambient temperature and the suitability of different clay soil types for treatment are highlighted.

Chapter 3: Materials and methods. This chapter is devoted for providing details on the materials and equipment utilised in this study, sample preparation method and testing procedures. It presents the properties of the tested soils (i.e. engineered and natural) and geopolymer ingredients (i.e. fly-ash, GGBFS and activator), as well as the testing program adopted in the current research. The testing program includes compaction, Atterberg limits, pH, Unconfined Compressive strength (UCS), durability, permeability, microstructural analysis using scanning electron microscopy (SEM) technique and static loading triaxial tests.

Chapter 4: Optimisation of geopolymer mixture-incorporating GGBFS. This chapter presents the results of the UCS tests and SEM analysis outlined in Chapter 3, which were conducted on an engineered clay treated with geopolymer to determine the optimum geopolymer mixture. From these results, the effect of geopolymer composition (i.e. fly-ash and activator) were evaluated, and the role of GGBFS in enhancing the strength performance of geopolymer-clay mixture at ambient temperature was determined. In this chapter, a recipe that yields fly-ash based geopolymer with adequate mechanical properties were determined for the use in the subsequent soil stabilisation investigation (i.e. Chapters 5 and 6).

Chapter 5: Engineering properties of geopolymer-treated clays. This chapter summarises the results obtained from the extensive experimental investigation outlined in Chapter 3, to evaluate the effects of optimum geopolymer recipe on the engineering properties of different clay types. The testing of this chapter includes compaction, plasticity, pH, durability, permeability and unconfined compressive

strength. A thorough investigation of the impact of various controlling factors (i.e. geopolymer content, curing time, clay pulverisation and clay mineralogy) on the treatment of clay is also presented.

Chapter 6: Undrained shear behaviour of geopolymer-treated clay. This chapter summarises and discusses the test results obtained from the comprehensive series of the static triaxial tests outlined in Chapter 3. The influence of some salient factors affecting the static undrained shear strength of geopolymer-treated clay such as geopolymer dosage, confining pressure and clay type are presented. The effect of the stress path on the undrained shearing behaviour of the geopolymer-treated clay is also presented. Moreover, results from a limited series of isotopically consolidated drained triaxial testing are critically analysed against the undrained performance of geopolymer-treated clay.

Chapter 7: Practical procedures for geopolymer stabilisation of clay soils. This chapter utilises the outcomes of the experimental program presented in Chapters 4-6, to define appropriate mixing criteria for effective treatment of clay soils with geopolymer, before the implementation into rational practical procedures.

Chapter 8: Summary, conclusion and recommendations. The most important results of this thesis are summarised, and directions for further research are also identified. A list of references and appendices follow Chapter 8.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Chemical stabilisation has been extensively utilised in the geotechnical industry as a means to improve inherent problematic properties of clay soils; this method includes the use of traditional binders such as ordinary Portland cement (OPC) and lime. As highlighted in Chapter 1, OPC and lime have been identified with severe environmental impacts related to high CO₂ emissions and difficulties in sourcing raw materials. Consequently, increasing value is placed on useful recycling of industrial by-product waste materials through alkali-activated binders as possible environmentally-friendly and low-energy alternative binders to traditional cement. Geopolymers have developed in response to this demand, making use of fly-ash collected from the flue gases of furnaces at pulverised coal power plants. The addition of fly-ash based geopolymers modifies the engineering properties of the host soil significantly. Curing conditions, specifically temperature and curing time, have been observed to affect the behaviour of geopolymer-treated soils significantly and due to the wide range of chemical components incorporated in geopolymers mix, chemistry also plays a role. As such, significant investigations have occurred in the literature into the behaviour of geopolymer-treated soils aiming to understand how they behaves and how they can be utilised most effectively.

To advance the use of geopolymers as an eco-friendly binder for chemical stabilisation of clay soils, detailed chemical and geotechnical cross-disciplinary knowledge is needed. This requires an understanding of the fundamentals of chemical stabilisation of problematic soils using both traditional binders and geopolymers for ground improvement. Accordingly, this chapter aims to provide an overview of the relevant literature to understand the behaviour and properties of geopolymer-treated soils and factors on which these depend. First, the concept of problematics soils is outlined, highlighting the structural and mineralogical behaviour that uniquely identifies such soils. Then traditional chemical stabilisation technique is discussed, highlighting the mechanisms by which successful chemical stabilisation occurs, as well as the limitations, ultimately leading to a discussion of the potential of geopolymer which

addresses a number of limitations but is not yet widely utilised. This review is not intended to summarise the entirety of literature on chemical stabilisation; but rather aims at a review of the more important aspects of chemical stabilisation, highlighting areas within this field that require further research to support broader adoption of geopolymer for clay stabilisation, in accordance with the overall aims and objectives of this thesis.

2.2 PROBLEMATIC SOILS

The term “problematic soils”, in the geotechnical engineering context, refers to soils of potential low bearing capacity, excessive compressibility and high volume changes with moisture variation during service (Han 2015). Examples of problematic soils are expansive and soft soils. Damage from expansive soils, due to their excessive volume change behaviour, has the potential to cause significant cracks to low-rise buildings and roads (Gromko 1974; Petry et al. 1989). Soft soils give rise to problems of stability (i.e. soil movement) and settlement even under small-superimposed load (Bergado et al. 1996). However, the problematic nature of such soils is typically related to their weak structure and involved mineralogy (Mitchell et al. 2005). In order to understand these soil types and their impact on engineering activities, it is essential to understand the structure and the primary clay mineralogy of such soils and their impact on the engineering properties and thus their behaviour. The following sections discuss these factors.

2.2.1 Soil structure

During their geological history, soils acquire a certain structure, a term referring to a combination of soil particle arrangement (fabric) and forces between particles (bonding) (Mitchell 1976). According to Cotecchia et al. (2000), natural soil structure can be assigned to two basic classes, ‘sedimentation’ and ‘post-sedimentation’ structures. Sedimentation structure refers to the elements of structure developed during sedimentation and consolidation process, whereas post-sedimentation structure refers to the elements of structure developed after sedimentation. The geological processes that control the development of the natural structure of the soil can be mechanical unloading (overconsolidation), a change in physical or chemical composition (diagenesis) and cementation (Sorensen et al. 2007). Cementation has significant effects on the properties and stability of different soils such as clays, sands and soft rocks (Mitchell et al. 2005). It may develop naturally at the inter-particle connections

of soil particles from precipitation of calcite, silica, alumina and iron oxides (Mitchell et al. 2005). The mechanical strength of the cementitious bonds binding the soil particles together defines the degree of stability of soil structure and its ability to withstand the effects of applied forces. It has thus been recognised that natural cementation within soil structure has a significant influence on the geotechnical properties (i.e. strength and stiffness) and thus, the stability of soil and supported structures (Mitchell et al. 2005).

2.2.2 Clay mineralogy

Mineralogy is an essential factor that control the physical and chemical properties of problematic soils. Therefore a prior knowledge of what minerals are in the soil provide insight into its behaviour (Mitchell et al. 2005). In general, clay can refer to the class of minerals in soils derived from the chemical decomposition of rock constituents. More specifically, the term ‘clay’ represents a family of minerals, primarily hydrous aluminium silicates known as phyllosilicates, which are small in size (< 0.002 mm). The particles of the clay minerals are primarily plate-shaped, although, in some cases, needle or tubular-shaped (Mitchell et al. 2005). Silicon tetrahedron (SiO_4) and aluminium octahedron ($\text{Al}(\text{OH})_3$) are two simple units that form the structure of common clay minerals (i.e. phyllosilicates family). The stacking arrangement of these units (sheets) with different types of bonding forms different clay mineral groups (Figure 2.1).

The mineral kaolinite, for instance, consists of one tetrahedral and one octahedral layer held tightly by hydrogen bonding (Figure 2.1). Montmorillonite, as a member of the smectite clay mineral group, is an aluminous mineral in which silicon almost exclusively occupies the two tetrahedral layers. It has relatively weak bonds compare to kaolinite. Depending on the bonding forms of clay minerals, the individual layers may be charge deficient, resulting in a permanent electrical negative layer charge that is balanced by the absorption of cations from natural in-situ voids solution (i.e. pore water), causing a tendency in soil towards plasticity (rapid deformation without rupture) when interacting with water (Velde 1995). Expandable clay minerals such as montmorillonite (the most problematic) are capable of changing their volume (swelling) due to water uptake through the hydration of interlayer cations (Müller 2005). Such problematic behaviour of clay minerals within the soils may pose serious challenges to the service life of infrastructure projects.

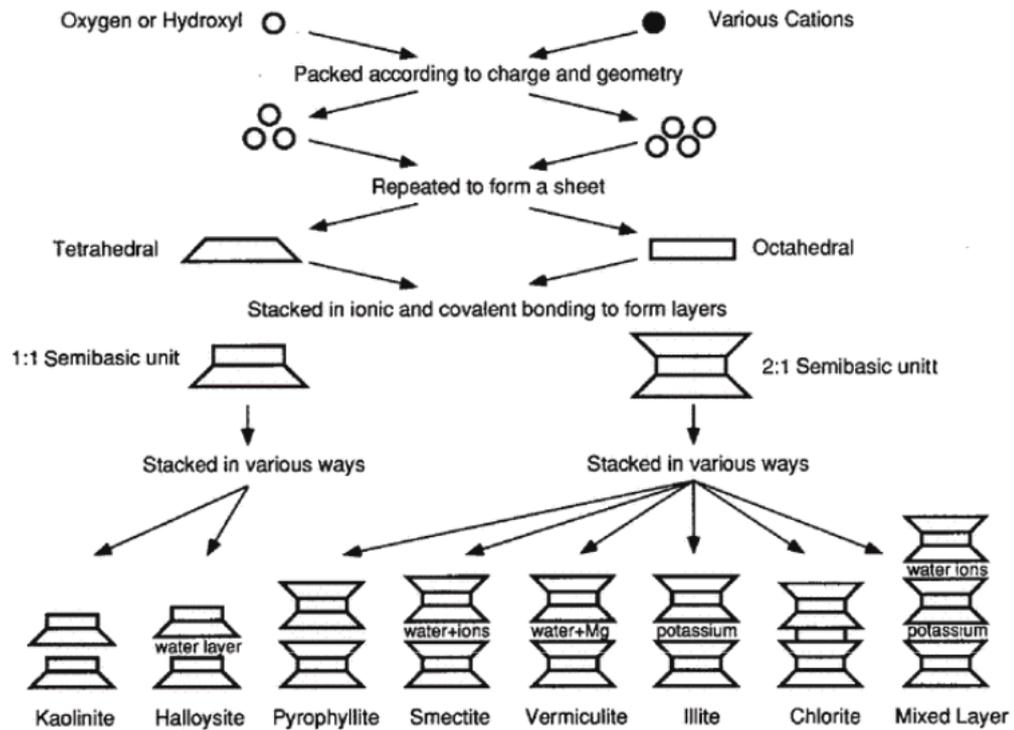


Figure 2.1: Synthesis pattern for clay minerals (Mitchell et al. 2005).

It is well documented in the literature that some cations (e.g. Ca^{2+}) naturally exist in pore water may interact through pozzolanic reaction with silica and alumina of clay minerals to form natural cementitious products, namely Calcium Silicate Hydrates (C-S-H) and Calcium Aluminate Hydrates (C-A-H) (Duxson et al. 2007a). The amount of cementitious products depends on the type of clay minerals and concentration (Ca^{2+}). However, the natural pozzolanic reaction usually produces low cementation within treated soils, and occurs at a slow rate and can last for years. However, such natural processes motivate the utilisation of industrial calcium-based products and other components through chemical stabilisation to alter the engineering properties of problematic clay soils (Little 1995) through similar mechanisms. In the following section, the fundamental concept of chemical stabilisation of soils is presented.

2.3 FUNDAMENTALS OF CHEMICAL STABILISATION OF SOILS

Chemical stabilisation is an in-situ soil technique used to improve the engineering properties of problematic soils. Most specifically, chemical stabilisation seeks to achieve an increase in strength, reduction in compressibility, improvement in stability characteristics and increase in the durability of problematic soils (Bergado et al. 1996; Karol 2003; Moseley et al. 2004; Kirsch et al. 2012; Nicholson 2014). This technique has been found to be a beneficial and cost-effective alternative to more expensive soil

replacement, or deep foundations (Nicholson 2014) used widely by the construction industry especially in Japan, United States, China and UK, among other countries (Moseley et al. 2004). Chemical stabilisation introduces a chemical binder to the in-situ soil through the mixing process. Shallow (surface) or deep mixing is usually carried out mechanically in the presence of sufficient moisture (Karol 2003; Kirsch et al. 2012). After in-situ mixing with soil, the chemical binder promotes various chemical reactions which cause durable (in ideal cases) alteration of the soil engineering properties (Little et al. 2009). The interaction of soil with a binder includes dissolution and disassociation of binder and soil particles, an exchange of ions, changes in pH of the pore water and precipitation of artificial cementations products. Such cementitious products bind the soil particles together, hence altering both the structure and mineralogy (i.e. mineral bonding and the cation exchange process) of the host clay soil (Section 2.2) and resulting in an overall improvement in soil behaviour. Long term property changes in the soil are referred to as soil stabilisation (e.g. permanent strength improvement), whereas short term enhancements are referred to as soil modification (e.g. plasticity reduction and limited strength enhancement) (Little et al. 2009). In the following sections, the use of traditional binders for chemical stabilisation is discussed, specifically, ordinary Portland cement (OPC) and lime additives, outlining the chemical reactions and mechanisms in which artificial cementation is developed.

2.3.1 Traditional binders

There are many different chemical stabilising agents (known as binders) available and in current use for soil stabilisation, including OPC, lime, fly-ash, pozzolans, GGBFS, cement kiln dust and bitumen; with the most traditional/common being OPC and lime (Das 2010). While the other less traditional options are becoming increasingly popular (Das 2010; Murthy et al. 2016), primarily for environmental reasons which will be discussed subsequently, further research into the behaviour of soil stabilised with such binders is necessary. As discussed in Chapter 1, this thesis seeks to contribute to this process through its investigation of geopolymer to stabilise clay soils. Thus, the performance of other non-traditional additives for soil stabilisation is beyond the scope of the current research and this literature review. Instead, this literature review focuses on the usage of lime and OPC as more popular binders used for chemical stabilisation of soils and interaction of soil with these binders to highlight the mechanism of

artificial cementation. The following sections briefly discuss the interaction of soil with lime and OPC to highlight the mechanism of artificial cementation.

2.3.1.1 Lime-treated soil

Chemical treatment of soils with lime has been in existence over 70 years ago (Little 1995). Lime is added to the soil either in the form of quicklime $\text{CaO}_{(s)}$ or as hydrated lime $\text{Ca}(\text{OH})_{2(s)}$. Immediately upon mixing with soil in the presence of water, hydrated lime $\text{Ca}(\text{OH})_2$ is formed, causing flocculation of clay particles to occur within the soil. Such reactions provide no strength improvement but cause immediate enhancement (i.e. modification) to the soil physical properties such as increased compactability and reduced plasticity. When a significant amount of lime is added to the soil, high concentration of hydroxyl ions are released from hydrated lime and the pH of the mixture is elevated forming a robust basic solution ($\text{pH} > 12$). The basic solution dissolves the silica and alumina sheets from the soil (i.e. clay minerals) which reacts with the calcium liberated from the hydrated lime, in a process referred to as a pozzolanic reaction (Little 1995). This reaction forms several cementation components, Calcium Silicate Hydrates (C-S-H) and Calcium Aluminate Hydrates (C-A-H), which adhere to soil particles, thereby stabilising the soil (Das 2010). The pozzolanic reaction is time-dependent, and the (C-S-H) product from the reaction is primarily responsible for the long-term enhancement in the soil mechanical properties (i.e. strength and stiffness). The critical element for adequate soil treatment with lime is the presence of reactive clay minerals (i.e. siliceous or aluminous material) as a source of pozzolans (Little 1995; Boardman et al. 2001).

2.3.1.2 OPC-treated soil

Ordinary Portland cement (OPC) is arguably the most popular soil stabilisation additive. The hydration and pozzolanic reactions are typical for the OPC-soil interaction. When OPC comes in contact with moisture in treated soil, the hydration phase begins immediately promoting rapidly enhanced engineering properties. The reaction products are mainly C-S-H and C-A-H, similar to those in the lime reaction but higher in produced quantity and rate of formation. Such products stabilise the soil by forming a solid structure around the soil particles, as shown in Figure 2.2, thereby reducing plasticity and increasing bearing load (Das 2010; Sargent 2014). Comparing the overall effects of OPC versus lime for soil stabilisation reveals that OPC is likely to develop higher strength enhancement at a faster rate (Das 2010).

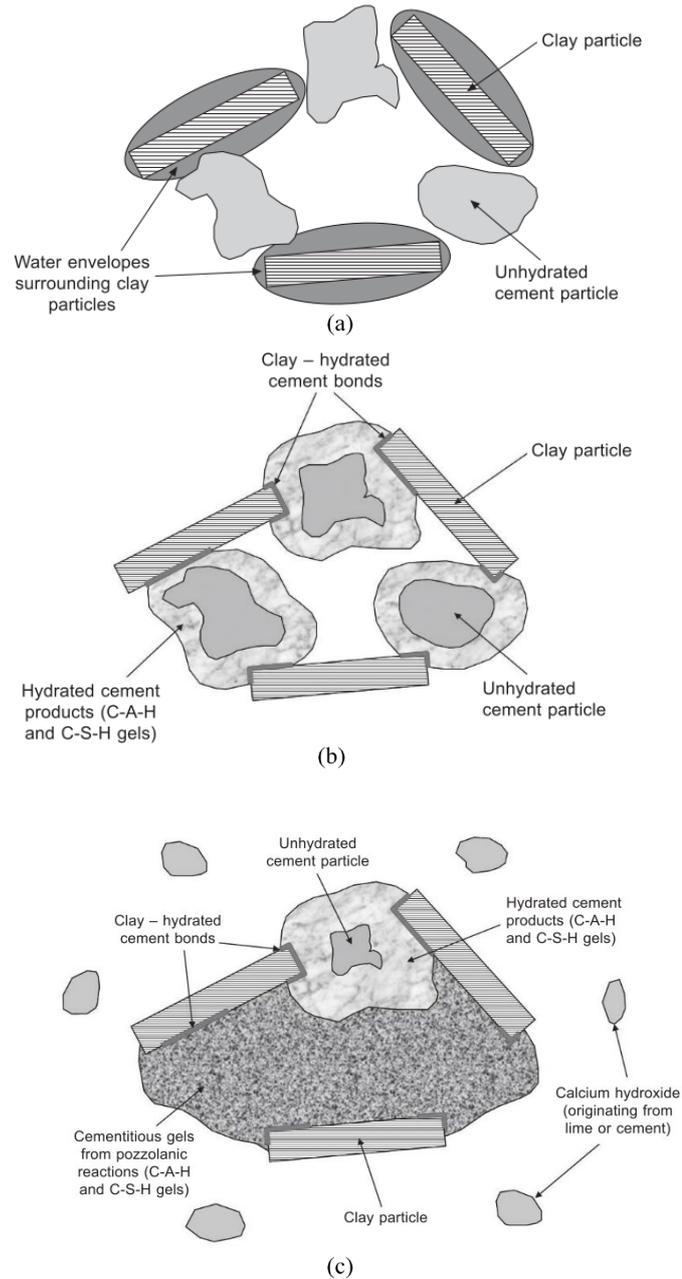


Figure 2.2: Typical state of soil treated with OPC: (a) before hydration phase; (b) hydration reaction state at short-term curing; and (c) hydration and pozzolanic reactions at long-term curing (Sargent 2014).

Four major components are responsible for the OPC reactivity; tri-calcium silicates (C3S), di-calcium silicates (C2S), tri-calcium aluminates (C3A) and tetra-calcium aluminoferrite (C4AF) (where C = Ca, S = SiO₂, A = Al₂O₃ and F = Fe₂O₃) (Sargent 2014). C3S is the most important strength-enhancing component on a short-term scale. In pozzolanic soils (i.e. clays and silts), secondary cementitious products of C-S-H and

C-A-H can also be formed through pozzolanic reaction, which provides further strength gain. This reaction can take place between silica/alumina of soil and calcium from the calcium hydroxide produced from initial cement hydration (Rafalko et al. 2008; Sargent 2014). However, in comparison to the OPC hydration reaction, the pozzolanic reaction is much slower and may last for years.

2.3.2 Mechanical behaviour of artificially cemented (structured) soils

When considering the purpose of chemical stabilisers, it is crucial to examine the mechanical behaviour of artificially cemented soil, particularly with regards to the impact of modified soil structure induced by artificial cementation and how this impacts the engineering behaviour of cemented soils regarding strength, stiffness, consolidation and failure. These are discussed here concerning traditional OPC and lime stabilisation. It should be noted that while the focus remains on artificially cemented soils, relevant literature related to natural cemented soils is also discussed where appropriate to address any knowledge gaps resulting from the scarcity of the literature related to artificially cemented soils.

2.3.2.1 Shearing behaviour

As stated in the previous sections, artificial cementation increases the strength of treated soils. The enhanced shear behaviour of cemented soils depends on the initial resistance of cemented structure. The damage of artificial bonding (i.e. de-structure) within treated soils leads to degradation of soil structure and subsequent changes in the soil stress-strain response. The change in strength, associated with the development of irrecoverable plastic strain, is marked by the yield point under static stress changes. However, the stress-strain failure mode can be significantly affected by the initial state of the microstructure of the cemented soils, which is governed by the pre-shear confining pressure level. Based on the initial state of the specimen, with respect to the stress-strain curve of the cemented bonds, three failure modes exist upon shearing of cemented soils (Coop et al. 1993), these are shown in Figure 2.3. At confining pressures lower than the yield strength of the cementitious bonds (Case-1, Figure 2.3), the stress-strain behaviour is elastic up to the yield point after which strain-softening behaviour is dominant due to the de-structure of the material upon further shearing. Such failure mechanism is well supported by different experimental studies conducted on cemented-soil mixtures (e.g. Uddin et al. 1997; Horpibulsuk et al. 2004). At the intermediate range of confining pressures where the cemented soil is not significantly

de-structured, the stress-strain behaviour is governed by the combined effect of cementitious bonds and the frictional response of partially de-structured material (Case-2, Figure 2.3). At this stage, yield occurs at low strains followed by strain hardening response. At high confining pressure (Case-3, Figure 2.3), the stress-strain behaviour is ductile as the microstructure of the cemented soil and the cementitious bonding are significantly influenced during consolidation. The stress-strain behaviour is essentially frictional and tends towards that of un-cemented soil with no yield point. The effect of high confining pressure on the mode of failure is also supported by the different experimental tests conducted on the cemented soils (Uddin et al. 1997; Porbaha et al. 2000; Horpibulsuk et al. 2004).

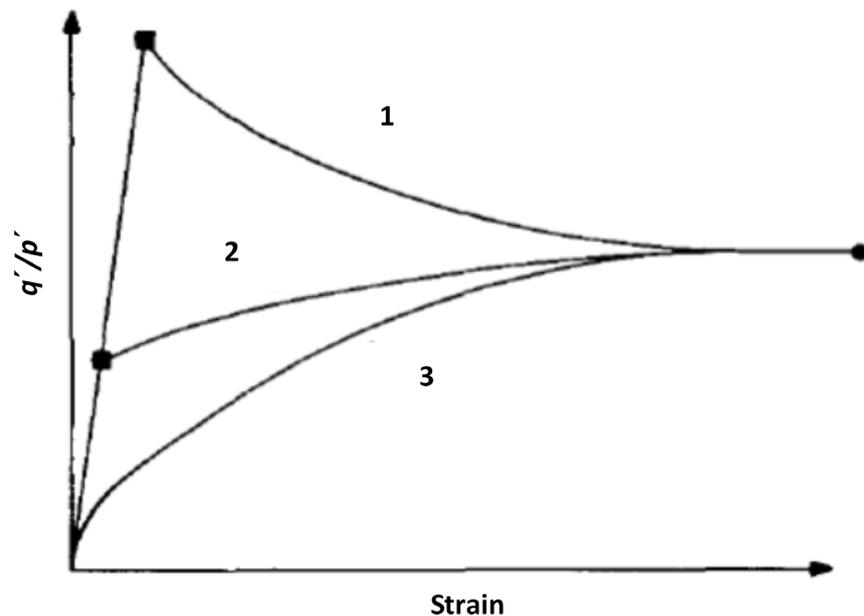


Figure 2.3: Idealised stress-strain behaviour of cemented soils : (1) confining pressures lower than the yield strength; (2) confining pressure at the mid-range; and (3) confining pressure higher than the yield strength (Coop et al. 1993).

Bergado et al. (1996) proposed a framework for the evaluation of undrained stress-strain behaviour of artificially cemented clay. According to his framework, three shearing response zones can be identified in the deviator stress versus mean effective stress or ($q-p$) space, as shown in Figure 2.4.

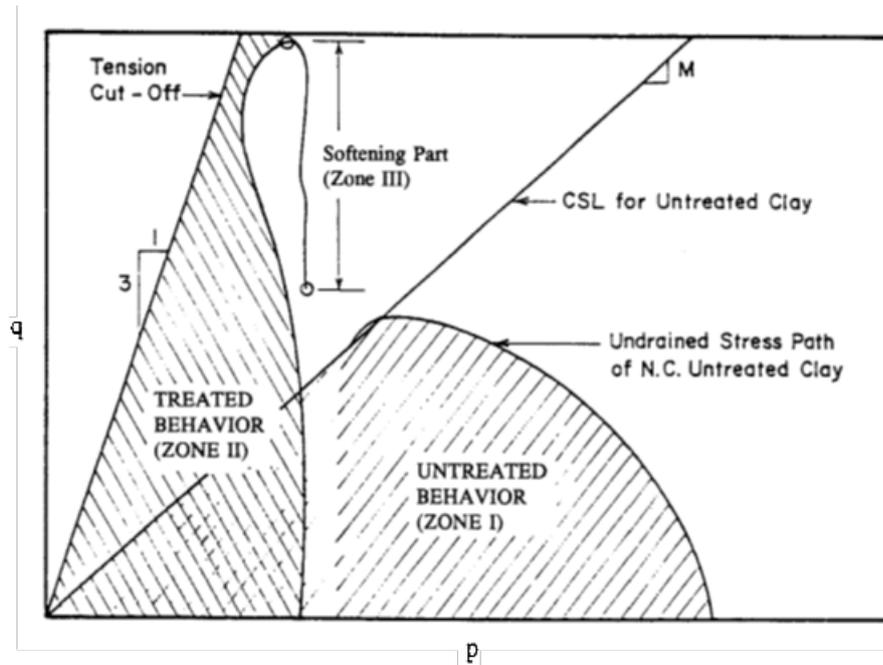


Figure 2.4: Undrained behaviour of cement treat clay in deviator stress (q) vs. mean effective stress (p) space (Bergado et al. 1996).

Zone-I postulates a behaviour for cemented soil similar to that of un-cemented soils at which the confining pressure is relatively high, which allow a complete de-structuration in the cemented soils; within this zone, cementitious bonding is destroyed and the behaviour is purely frictional as indicated by the q - p curve. In Zone-II where the confining pressure is low, the effect of cementitious bonds is most prominent. The left end of the zone is defined by a tension cut-off, which corresponds to a state of zero effective radial stress (i.e. excess pore water pressure = confining pressure). Within this zone, the q - p stress path rises almost vertically to achieve a peak strength before reducing to a strength envelope corresponding to large strain state, i.e. Zone-III identified as de-structured strength envelope.

Artificial cementation promotes a change in the gradient and q -axis intercept values of the failure envelope, implying improved effective shear strength parameters, i.e. effective cohesion (c') and effective angle of internal friction (ϕ') (Uddin et al. 1997; Kamruzzaman 2002; Horpibulsuk et al. 2004; Consoli et al. 2007). Uddin et al. (1997), Kamruzzaman (2002) and Horpibulsuk et al. (2004) indicate that the friction angle significantly increases with the increase of the cement content. While conversely, Consoli et al. (2007) reported that the friction angle is independent of the level of cementation, and the cohesion increases with an increase of cementation level. In all

cases, the increase in the effective cohesion of the treated clay can be attributed to the increase in the cementation offered by the binder (Horpibulsuk et al. 2004). The increase in the effective friction angle can be attributed to the effect of the large particles formed from the bonded clusters of clay after treatment (Lorenzo et al. (2006). However, all mentioned studies confirm the ability of the artificial bonding to move the state of stress of the bounded soil outside the failure envelope of the unbonded soil.

For artificially cemented soil, the volumetric changes in response to shearing are found to be dependent on confining pressure. At low confining pressures (up to 1000kPa as shown in Figure 2.5), cemented soil specimens undergoing undrained shearing, exhibit a noticeable reduction in excess pore water pressure after the initial positive pore pressure development (Kamruzzaman et al. 2009). The presence of cementitious bonding prevents the soil dilating before yield stress, resulting in positive excess pore water pressure (i.e. contraction) after which the gradual degradation of the bonds allows the voids between de-structured particles, within the failure planes, to expand causing a rapid decrease in pore water (i.e. dilation). When shearing from elevated confining pressure (Figure 2.5), the excess pore pressure shows an increase with the increase in shear strain and this increase is associated with more strain-softening behaviour. Under drained conditions, and due to the heavy de-structure during the consolidation phase, the dominant volumetric change upon shearing is dilation. This type of pore pressure response is typical to that of overconsolidated remoulded soils (Uddin et al. 2002).

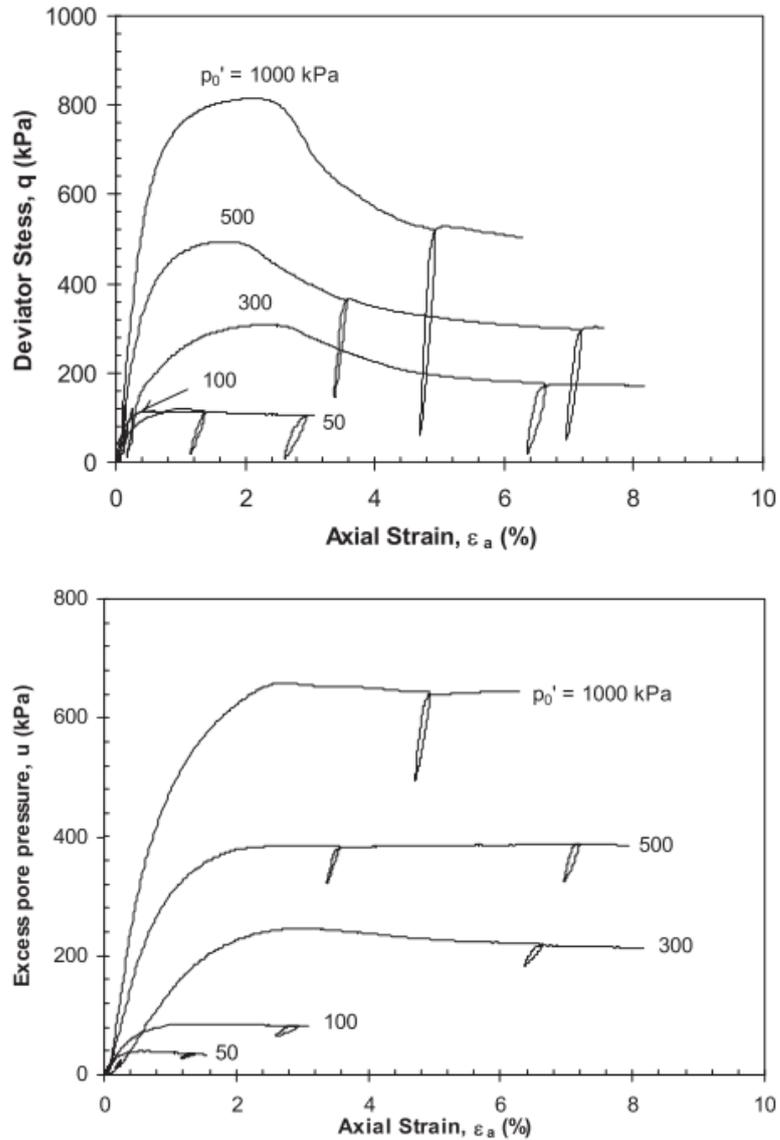


Figure 2.5: Effect of confining pressure on undrained excess pore water pressure behaviour of treated clay (10% cement, curing 28 days) (Kamruzzaman et al. 2009).

2.3.2.2 Stiffness behaviour

A consistent finding in the literature is that increasing artificial cementation level (linked to binder content) increases the peak strength of cemented soil and decreases the shear strain at failure, thereby increasing the overall stiffness of soil (Porbaha et al. 2000). Malandraki et al. (2001) developed a comparison of stiffness behaviour for three different soil and soft rock types, advantaging from the relative similarity in the mechanical performance between artificial and natural structured soils, as shown in Figure 2.6. The curve of unbonded soils shows low stiffness with first and final yield but no bond yield. The first yield represents the limit of linear elastic behaviour, and

the final yield represents the destruction of any structure within the soil, whereas the bond yield represents the limit or recoverable behaviour. The curve of bonded soils indicates a significant contribution of bonds towards an increase in initial stiffness. These soils show both first and bond yield similarly to the curve of soft rocks.

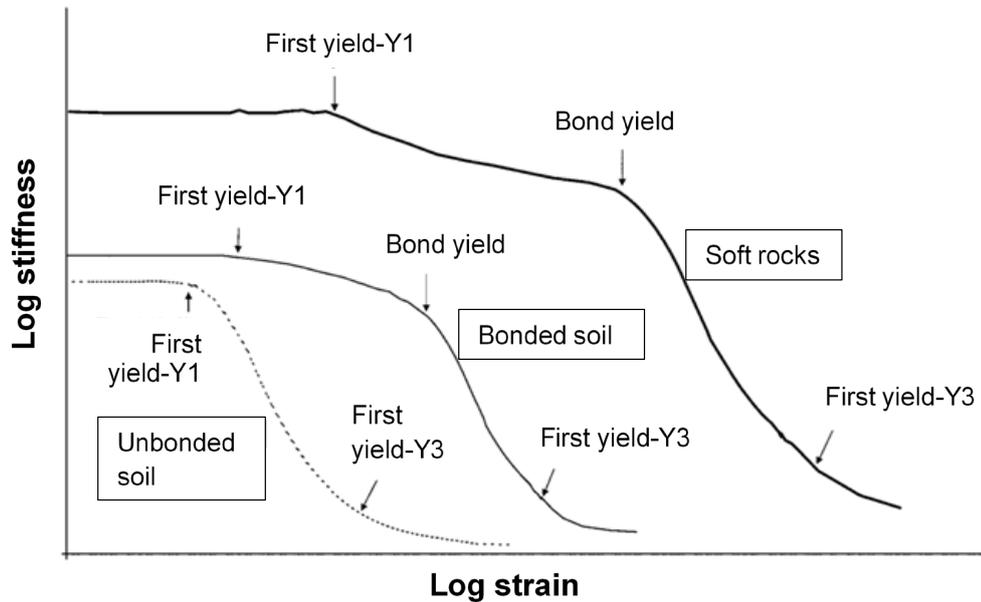


Figure 2.6: Stiffness variation against strain for unbonded and bonded soils and soft rocks (Toll et al. 2000) as cited in (Malandraki et al. 2001).

2.3.3 Environmental impacts of traditional binders

The production of lime and OPC involves thermal decomposition of calcium carbonate from limestone (Oates 1998; Provis et al. 2014). Such a process demands intensive energy to maintain the necessary high temperatures of 450-1500°C for OPC and 100-1000°C for lime. The chemical reaction involved during thermal decomposition process and the combustion of fuel for the required heat and power generation are responsible for considerable CO₂ emissions. Manufacturing of 1 tonne of OPC has been found to release about 0.8–1.0 tonne of carbon dioxide (CO₂) to the atmosphere (Garcia-Lodeiro et al. 2014a). Considering the heavy reliance of construction industry on OPC (as stated in the introduction chapter), manufacturing of OPC is responsible for about 8-10% of the global artificial CO₂ emissions per year (Tayibi et al. 2009; Garcia-Lodeiro et al. 2014a; Provis et al. 2014). In the case of lime, its global contribution to CO₂ emissions is near 1% with an average of 0.95 tonne of CO₂ released per 1 tonne of lime (Oates 1998). In addition to being CO₂ intensive, OPC and lime productions have been marked with other environmental limitations related to the

sourcing of the raw materials. According to Garcia-Lodeiro et al. (2014a), 2.0 billion tonnes of OPC production consumes over 3.0 billion tonnes of raw materials on average (70% of which are limestone). Such over exploration of the raw materials, with the overuse of coal quarries as a source of energy, contributes to severe degradation of landscapes (Tayibi et al. 2009; Sargent 2014). Therefore, in the context of global warming, utilisation of lime and cement in bulk applications such as soil stabilisation has an additional impact on the environment. This has encouraged the development of new alternative binders that have a smaller environmental footprint without compromising soil stabilisation capabilities.

2.4 GEOPOLYMER: NON-TRADITIONAL BINDERS FOR SOIL STABILISATION)

In recent years, an increasing number of non-traditional binders have been proposed as an alternative to traditional binders for use in clay soils to improve their mechanical behaviour (Brandon et al. 2009; Turkoz et al. 2014). Such binders are usually classified into major categories, including salts, acids, enzymes, emulsions and polymers (Das 2010; Murthy et al. 2016). The chemical formulas of such products are developed often based on the market tendency such as cost, particular performance and availability (Murthy et al. 2016). However, the interest in non-traditional binders is in part due to environmental/energy issues. To minimise these issues associated with traditional binders, research has suggested the use of aluminosilicate industrial by-products materials (e.g. fly-ash and granulated ground blast furnace slag, GGBFS) to partially eliminate the use of traditional binders. Such materials are rich in silica and alumina with little or no cementation value, and when mixed with OPC, the hydration reaction produces an additional amount of C–S–H and C–A–H gels, thereby stabilise the soil (Das 2010). Recently, the recycling of such aluminosilicate by-products materials through alkali-activated binders constitutes an attractive non-traditional option to fully eliminate the use of traditional binders (Roy 1999; Hardjito 2005; Van Deventer et al. 2006; Duxson et al. 2007b; Komnitsas et al. 2007; Davidovits 2008; Siddique et al. 2011; Garcia-Lodeiro et al. 2014a; Pacheco-Torgal 2014). The alkaline activation process of industrial aluminosilicate residues usually includes dissolution of silica and alumina by using a basic (high pH) liquid phase, and the formation of cementitious products. Considering the nature of the cementitious components of different

aluminosilicate materials used in alkaline binders, three alkaline activation models can be derived (Garcia-Lodeiro et al. 2014). These models are summarised in Table 2.1.

Table 2.1: Alkaline reaction models (Garcia-Lodeiro et al. 2014a).

<i>Model</i>	<i>Activated source material</i>	<i>Alkaline conditions</i>	<i>Reaction Output</i>
<i>I</i>	Low <i>C</i> content with High <i>A</i> & <i>S</i> content. (e.g. class F fly- ash and metakaolin)	Aggressive, at High temperature	N-A-S-H gel (alkaline aluminosilicate hydrate gel, i.e. geopolymer).
<i>II</i>	High <i>C</i> & <i>S</i> content (>70%) (e.g. GGBFS).	Moderate, at ambient temperature	C-A-S-H gel (calcium silicate hydrate gel).
<i>III</i>	High <i>C</i> , <i>A</i> and <i>S</i> content (>20%). (e.g. GGBFS + fly ash + OPC).	Diverse	C-A-S-H gel and, N-A-S-H gel

$C = CaO$, $S = SiO_2$, $A = Al_2O_3$, $N = Na_2O$, $H = H_2O$,

Model-I is usually derived from low calcium high aluminosilicate materials such as fly-ash or metakaolin which, when activated using an alkaline solution, forms a product of bonding characteristics of a three-dimensional framework (i.e. geopolymer) (Pacheco-Torgal et al. 2008; Shi et al. 2011). The geopolymer model is represented by the chemical structure Sodium Aluminate Silicate Hydrate (N-A-S-H), which differs than OPC and lime reactions described earlier (Section 2.3.1). This model requires aggressive synthesise conditions (i.e. high alkali media and elevated temperature) (Garcia-Lodeiro et al. 2014a), and the literature includes several studies that have used this model for soil stabilisation (e.g. Markou et al. 2002; Wilkinson et al. 2010b; Cristelo et al. 2011; Cristelo et al. 2013; Zhang et al. 2013; Liu et al. 2016).

Model-II produces a fundamental Calcium Aluminate Silicate Hydrate (C-A-S-H) product similar to that obtained during the OPC hydration. It is important to emphasise that the activation of by-product aluminosilicate materials of this model with high calcium content > 70% (e.g. GGBFS) usually leads to a reaction output different from that of geopolymer. Consequently, there is a debate in the literature of whether or not this model may be classified as geopolymer; therefore, it is excluded from discussion throughout this thesis. However, examples of previous studies carried out using this model for soil stabilisation include the work done by Hughes et al. (2004), Wilkinson et al. (2010a), Sargent et al. (2013) and Yi et al. (2014).

Model-III is comprised of the two preceding models (hybrid), which consists of activated fly-ash and GGBFS, where a combination of the N-A-S-H and C-A-S-H gels

is introduced in the reaction process. This model requires an ambient temperature and low alkali conditions, which may assist in bringing the strength of treated soils to the economical boundary since elevated temperature and high alkalinity are not practical conditions for soils. Limited studies are available in the literature on the use of this model for soil stabilisation (e.g., Sargent et al. 2013; Singhi et al. 2017; Arulrajah et al. 2018).

In Australia, the coal-fired power plants generate approximately 14 million tonnes of fly-ash per year; over 58% is dumped directly into the landfill (Sing et al. 2015). The use of such abandoned quantity of fly-ash for the production of geopolymer (*Model I*) may reduce the need for the thermal decomposition of calcium carbonate from limestone required for OPC manufacture. Consequently, it may contribute to 80% reduction in the CO₂ emission (Duxson et al. 2007b) and may consume 70% less energy compared to OPC manufacturing (Tempest et al. 2009). With the reduced emission and energy consumption, fly-ash based geopolymer has a good potential as a sustainable binder for soil stabilisation. In comparison, geopolymer through *Model-III* (see Table 2.1) seems to be most representative than *Model-I* for clay stabilisation at ambient temperature and thus appropriate to consider. However, it is necessary to understand the basic reaction mechanism of geopolymer through *Model I* for clay stabilisation before advancing the use of *Model III* for the same purpose. In the following sections, the state-of-the-art of geopolymer (i.e. *Model-I*) including involved material, reaction nature and engineering properties of treated soils is described. This is followed by a discussion on the suitability of fly-ash based geopolymer incorporating calcium-based components including GGBFS (i.e. *Model-III*) for clay stabilisation at ambient temperature, which will be the focus of this thesis.

2.4.1 Geopolymer treated soil - (*Model I*)

In the 1970s, the term “geopolymer” was coined for the first time to name the tri-dimensional aluminosilicates that are formed rapidly by naturally occurring low calcium aluminosilicate material (Shi et al. 2006; Komnitsas et al. 2007; Davidovits 2008; Provis et al. 2009). The term “geopolymer” has also been described in the academic literature by many different names; “mineral polymers”, “inorganic polymers”, “inorganic polymer glasses”, “alkali-bonded ceramics”, “alkali ash material”, “soil cement”, “hydroceramics”, “alkali-activated cement” and a variety of other names. While there is an ongoing debate over the correct terminology

(Komnitsas et al. 2007), for the purpose of consistency, the term “geopolymer” will be used throughout this thesis. Geopolymer is rapidly proving itself to be a suitable candidate for soil stabilisation overcoming the environmental issues faced by traditional binders. In the following sections, most significant features and the chemistry governing the activation reaction of geopolymer (*Model I*) for soil stabilisation is described

2.4.1.1 Geopolymer ingredients

Geopolymer usually comprises a mix of low calcium aluminosilicate material and an alkaline activator. In general, any reactive low-calcium aluminosilicate powder is a possible material for synthesising geopolymers. Aluminosilicate material can be classified into two main categories, calcined and uncalcined (Xu et al. 2003). Calcined aluminosilicate materials are, for instance, fly-ash, metakaolin, construction residues and pozzolanic wastes. Non-calcined aluminosilicate materials are feldspars and rock-type aluminosilicate minerals (Komnitsas et al. 2007; Garcia-Lodeiro et al. 2014a). However, the use of calcined aluminosilicate material promotes higher mechanical properties of geopolymer such as strength and stiffness. This is attributed to the thermal treatment history during formation which affects the structural coordination of aluminium and oxygen ions, and transfers any stable crystalline material into an amorphous one with high reactivity for alkaline activation (Davidovits 2008).

The most common calcined aluminosilicate materials used for geopolymer are metakaolin and coal fly-ash (Provis et al. 2009; Garcia-Lodeiro et al. 2014a). Metakaolin is kaolinite clay calcined at high temperature (500 to 750°C), whereas the fly-ash is an abundant waste material produced from coal-fired steam power plants. The facts that fly-ash has already undergone thermal treatment history and is widely available as a waste material promotes its use as a more economical and sustainable option than metakaolin geopolymer production (Hardjito 2005). Therefore, fly-ash has been a focus of many studies conducted on soil stabilisation (e.g. Cristelo et al. 2011; Cristelo et al. 2013; Phetchuay et al. 2016; Phummiphan et al. 2016; Rios et al. 2017). Along with various types of aluminosilicate materials, a wide range of alkaline activators can also be utilised for geopolymer production including sodium and potassium-based solutions (van Jaarsveld et al. 1999; Xu et al. 2000; Komnitsas et al. 2007). The use of such varied materials in geopolymer industry has led to a focus on understanding the chemistry of the different reactive components. In the following

sections, a detailed discussion on the ingredients of fly-ash based geopolymer is provided.

2.4.1.1.1 Fly-ash

Fly-ash is a fine-grained, powdery particulate, inorganic aluminosilicate material collected from the flue gases of furnaces at pulverised coal power plants (Siddique et al. 2011). During combustion, mineral impurities in the coal (clay, feldspar, quartz and shale) are thermally transformed into coal ash. 75–85% of the total coal ash raises spherical particles as fly-ash. The raised spherical particles removed from flue gases by dust collection system either mechanically or by using electrostatic precipitators. The remainder is collected as bottom ash or boiler slag (Siddique et al. 2011). The type of equipment for dust collection controls the particles size of fly-ash, which typically finer than OPC particles and ranges from less than 1-150 μm . In general, fly-ash comprises of oxides of silicon (SiO_2), aluminium (Al_2O_3) and calcium (CaO). Amongst these oxides, iron, magnesium, titanium, sulphur, potassium and sodium are also present in a lesser amount.

Fly-ash production forms the fifth largest material resources in the world (Gianoncelli et al. 2013). Due to the increase in power demand and coal consumption, the recent estimated annual production of fly-ash as a waste material is more than 750 million tonnes per year with an increase of 50% over last decade. However, the growing environmental awareness and increasing demand for energy and raw materials foster recycling of fly-ash in the construction industry.

In geotechnical applications, there are numerous possible ways in which fly-ash can be utilised; namely, backfill material, base material and soil stabilisation material (alone or mixed with lime or OPC) (Siddique et al. 2011). The use of fly-ash as an engineering material for soil stabilisation is a well-documented practice. ASTM C618 categorises fly-ashes into two categories as mineral admixtures with OPC; namely, Class C (self-cementing) and Class F (non self-cementing) (Little et al. 2009). Table 2.2 presents the chemical analysis of two different fly-ash types (Siddique et al. 2011).

Table 2.2: Chemical analysis of fly-ash, Class F and C (Siddique et al. 2011).

Parameter	Class F	Class C
Silicon dioxide (%)	45–64.4	23.1–50.5
Calcium oxide (%)	0.7–7.5	11.6–29.0
Aluminum oxide (%)	19.6–30.1	13.3–21.3
Iron oxide (%)	3.8–23.9	3.7–22.5
Sodium oxide (%)	0.3–2.8	0.5–7.3
Magnesium oxide (%)	0.7–1.7	1.5–7.5
Potassium oxide (%)	0.7–2.9	0.4–1.9
Loss on ignition (%)	0.4–7.2	0.3–1.9

In general, Class F fly-ash has less than 10% calcium oxide (CaO) and offer pozzolanic with rare self-hardening properties, whereas Class C fly-ash contains high value of calcium of more than 20% and incorporate pozzolanic and self-cementitious properties (Shi et al. 2006; Khale et al. 2007). However, calcium oxide amount in later class is insufficient to react with all the pozzolanic material of the fly-ash to form high-performance C-S-H cementation material for soil stabilisation and therefore it is utilised by mixing with binders rich with calcium (i.e. OPC or lime) (Das 2010).

Fly-ash low in calcium is of a higher content of silica and alumina, and may promote geopolymer production of high mechanical properties (Hardjito 2005). This puts low calcium (Class F) fly-ash in favour as a raw material for geopolymer production than high calcium (Class C) fly-ash (Hardjito 2005; Duxson 2009; Garcia-Lodeiro et al. 2014a). It is worthwhile noting that most of the fly-ash commercially available in Australia is deficient in calcium content, complying with the classification terms of ASTM Class F fly-ash (Hardjito 2005). However, the degree of coal pulverisation (fineness), type of furnace, oxidation conditions, collection method, along with power load demand in a 24 hr interval are factors that may contribute to varying the properties of fly-ash from various power plants and even in one plant itself (Siddique et al. 2011). Based on this, and according to Garcia-Lodeiro et al. (2014a), fly-ash (Class F) must meet specific requirements to be used as a source material in geopolymer, see Table 2.3. In terms of fineness, and according to Bignozzi et al. (2014), the Australian fly-ashes exhibited higher content of particles in the range of 2–100 μ m promoting geopolymers with high strength and low porosity.

Table 2.3: Requirements of fly-ash type F for geopolymer production (Garcia-Lodeiro et al. 2014a).

Parameter	
Fe ₂ O ₃	≤ 10%
CaO	≤ 10%
reactive SiO ₂	> 40%
80–90% particles	< 45 μm
vitreous phase content	> 50%
SiO ₂ reactive / Al ₂ O ₃ reactive	ratio > 1.5

2.4.1.1.2 Alkaline activator

Hydroxide and silicate solutions of great interest as alkali element in geopolymerisation are those based on potassium or sodium (van Jaarsveld et al. 1999; Xu et al. 2000; Komnitsas et al. 2007). Alkali hydroxides are generally produced from chloride salts, whereas melting carbonate salts with silica and warm water produce the alkali silicates in the form of a viscous, sticky solution, which also known as a water glass (Provis et al. 2014). Potassium based solutions have been found to have cost limitations especially for bulk applications (Provis 2009), therefore, sodium-based solutions are most commonly suggested as alkali activator for geopolymer in soil stabilisation (Cristelo et al. 2011; Cristelo et al. 2012; Cristelo et al. 2013; Zhang et al. 2013; Zhang et al. 2015; Phetchuay et al. 2016; Phummiphan et al. 2016; Rios et al. 2016a; Rios et al. 2016b; Rios et al. 2017). It should be noted that cheaper alternative solutions, for example, non-silicate solutions weak acid salts like (Na₂CO₃), strong acid salts (Na₂SO₄) and aluminates (M₂O.nAl₂O₃), have been found to give unfavourable phase behaviour with poorly reacted and porous geopolymer product (Provis 2009).

2.4.1.2 Reaction mechanism

The geopolymer formation process described above in *Model I* of alkaline activation is referred to (geopolymerisation) and involves multi-reaction steps including: (1) dissolution of solid aluminosilicate oxides, (i.e. fly-ash), by the high hydroxyl (OH⁻) concentration (high pH) provided by an alkaline medium; (2) diffusion (migration) of the dissolved Al and Si complexes; (3) condensation (or polycondensation) with residual alkali cation (e.g., sodium Na⁺); (4) gel formation; (5) reorganization; and (6) gel evolution with curing time and crystallization to hardening. This final geopolymer

product is represented by the chemical structure Sodium Aluminium Silicate Hydrate (N-A-S-H), which differs than C-A-H and C-S-H products from hydration and pozzolanic reactions presented earlier for OPC and lime-soil mixtures. Figure 2.7 presents the various stages comprising the transformation of the aluminosilicate material into geopolymer (Duxson et al. 2007a).

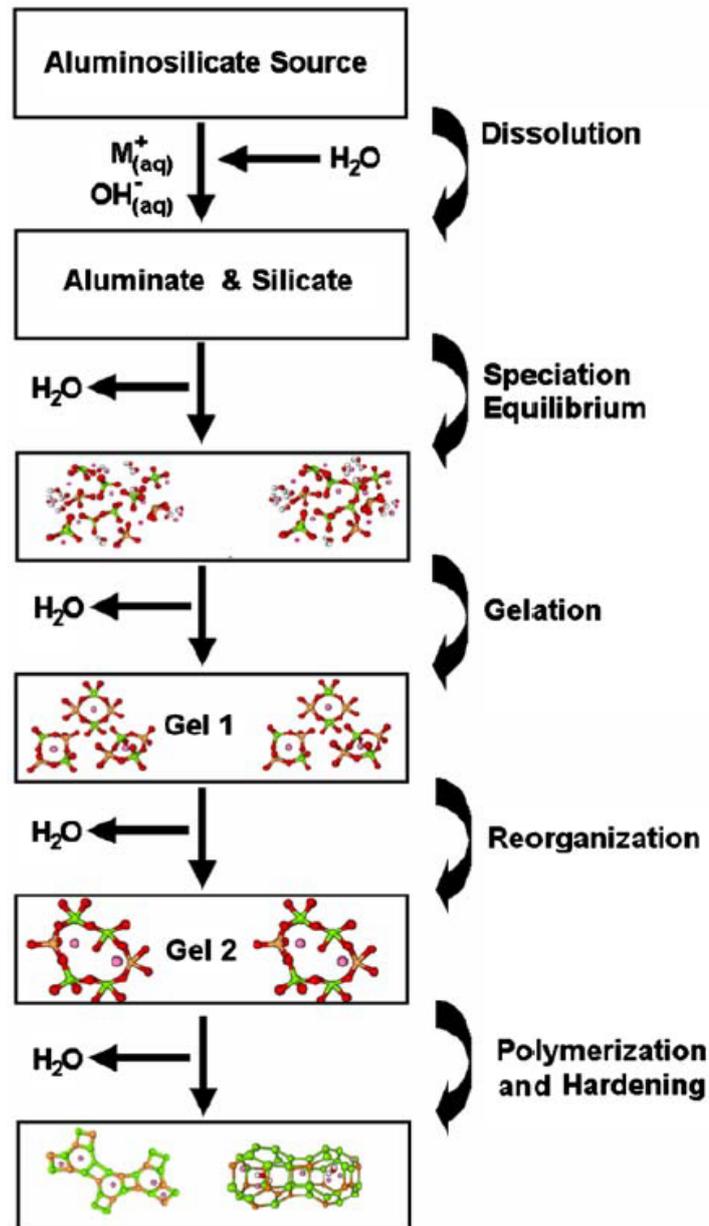
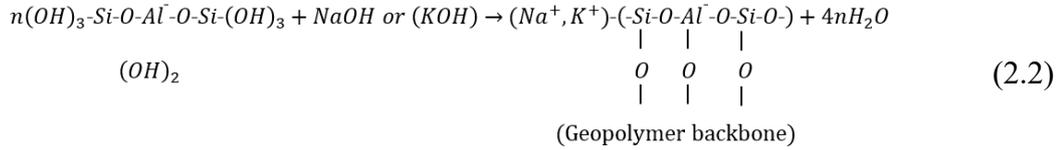
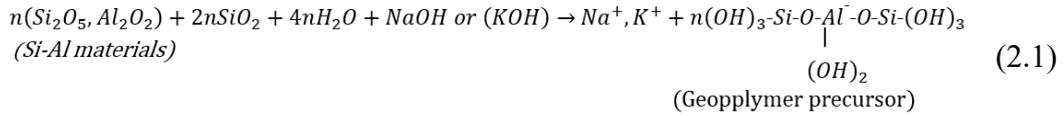


Figure 2.7: Conceptual model for geopolymerisation (Duxson et al. 2007a).

The reactions described in Figure 2.7 yielding polymeric Si-O-Al-O bonds, which can be schematically presented as (Xu et al. 2000):



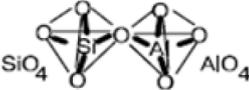
Based on the above equations, geopolymer products can be described as a highly connected three-dimensional chain network bond of sialite. Sialate network bond (Si-O-Al) is consists of tetrahedral silica (SiO₄) and alumina (AlO₄) joined by sharing all the oxygen (O₂).

The general empirical formula for geopolymer gel can be then displayed as (Komnitsas et al. 2007; Davidovits 2008; Garcia-Lodeiro et al. 2014a):



where *M* is an alkali cation such as potassium (K⁺) or sodium (Na⁺) that balances the negative charge that localises on one or more of the bridging oxygen in each aluminate tetrahedron, *n* is the degree of polymerisation and *z* is the Si: Al molar ratio, ranging from 1 to 15 or from 1 to 32 (Zhang et al. 2013). Depending on the value of *z*, the geopolymer can take one of several basic systems (Davidovits 2008) where *z* > 3 produces a rubbery geopolymer of a linear linked two-dimensional network, whereas *z* < 3 produces a brittle cementitious product of crossed linked three-dimensional network mainly suitable for soil stabilisation (Zhang et al. 2013). The different forms are summarised in Table 2.4.

Table 2.4: Different geopolymer systems (Davidovits 2008).

Si:Al	Geopolymer form	Chain-ring polymer bond
1	Poly(sialate) [-Si-O-Al-O-]	
2	Poly(sialate-siloxo) [-Si-O-Al-O-Si-O-]	
3	Poly(sialate-disiloxo) [-Si-O-Al-O-Si-O-Si-O-]	
> 3	Poly(sialate-multisiloxo)	(i.e. through linking Si-O-Al between two Poly(sialate))

2.4.1.3 Engineering properties of geopolymer-treated soil

When an aluminosilicate material (i.e. fly-ash) comes in contact with an alkaline activator added to the soil, geopolymerisation reaction (*Model I*) begins immediately. This reaction is responsible for the formation of an artificial cementitious product within soil particles, thereby stabilise the soil and enhances its properties. There is an increasing number of studies that seek to investigate the engineering benefits of the geopolymer-soil mixtures; the key research has been summarised in Table 2.5. Based on these studies, the improvement in the properties of soils mixed with geopolymer promotes engineering benefits that can be summarised in the four primary ways:

- **Improved soil structure** – enhances the uniformity and homogeneity of the soil fabric on a microstructural level and develops artificially cementation bonds between soil particles, both of which are known to have a significant impact on soil behaviour.
- **Improved strength** – enhances the ability to resist shear stresses developed as a result of applied service loading.
- **Improved stiffness** – minimises permeant deformation along with the service life of the structure.
- **Improved durability** – maintains stability and engineering properties when exposed to environmental conditions (i.e. moisture and temperature changes).

However, the studies summarised in Table 2.5 which highlight the engineering benefits of geopolymer for soil stabilisation are limited to specific soil types tested using certain types of additives and concentrations. Most importantly, the studies did not provide insights into the effects of soil mineralogy and plasticity on the treatment results, both of which are known to have a significant impact on the performance of clay soil stabilisation (Little et al. 2009). However, for a better understanding of the use of geopolymer, and to enhance its performance for clay soil stabilisation, considering mineralogy/plasticity effects of the clay on treatment, it is necessary first to evaluate and summarise the existing knowledge and engineering benefits/properties of the geopolymer-soil mixture.

In the following sections, further detail on the impact of geopolymer on microstructure, strength, stiffness and durability characteristics of treated soil and controlling factors will be discussed.

Table 2.5: Summary of research into the use of geopolymer for chemical stabilisation of soils.

Soil type	PI, %	SM type	Activator chemicals	SM/(SM + Soil) or SM/soil ^d or percentage ^e , %	A/(SM + Soil), %	A/SM, %	NaOH molarity or NaOH:H ₂ O ratio	Na ₂ SiO ₃ /NaOH (mass)	Curing Temp. (°C)	Tests conducted on stabilized soils					Reference
										Compaction	Peak UCS, 28 days, (MPa) ^c	Triaxial	Durability	Microstructure	
Silty sand	-	Same soil ^a	NaOH+NaAlO ₂	-	40	-	10	0.285 ^b	25	-	2.5	-	-	✓	[1]
Sandy Clay	21.9	FAF	NaOH+Na ₂ SiO ₃	20-40	40-50	-	10-15	2	amb.	-	11.4	-	-	-	[2]
Fat clays	32.2	FAC&FAF	NaOH+Na ₂ SiO ₃	10-20	40	-	10	2	19-23	-	1.2&1.8	-	-	✓	[3]
Sandy clay	21.9	FAF	NaOH+Na ₂ SiO ₃	20-40	40-50	-	10-15	2	19-23	-	10	-	-	✓	[4]
Silty sand	12	FA	NaOH+Na ₂ SiO ₃	10 ^d	-	50	-	2	20	-	0.6	-	✓	-	[5]
		FA&GGBS	NaOH+Na ₂ SiO ₃								3.5				
lean clay	15	MK	NaOH+Na ₂ SiO ₃	3-15	*	-	-	-	23	-	3.8	-	-	✓	[6]
Lean clay	15	MK	NaOH+Na ₂ SiO ₃	8,13	*	-	-	-	amb.	-	4.0	-	-	✓	[7]
Clay	14	FAC	NaOH+Na ₂ SiO ₃	4-20 ^d	-	45-85	12-14.5	varies	amb.	✓	0.2	-	-	-	[8]
		FAC&GGBS									10.5				
Silty clay	33	FAF&CCR	NaOH+Na ₂ SiO ₃	25-45 ^d		50-200	3-18	1.5-9	25-40	-	1.2	-	-	-	[9]

Table 2.5: Summary of research into the use of geopolymer for chemical stabilisation of soils (Cont.).

Soil type	PI, %	SM type	Activator chemicals	SM/(SM + Soil) or SM/soil ^d or percentage ^e , %	A/(SM + Soil), %	A/SM, %	NaOH molarity) or NaOH:H ₂ O ratio	Na ₂ SiO ₃ /NaOH (mass)	Curing Temp. (°C)	Tests conducted on stabilized soils					Reference
										Compaction	Peak UCS, 28 days, (MPa) ^c	Triaxial	Durability	Microstructure	
Loess	13.7	FAF	NaOH+Na ₂ SiO ₃	10-30	40	-	-	-	23	-	4.5	-	-	✓	[10]
			KOH+Na ₂ SiO ₃							7	-	-	-		
silty clayey sand	6	FAC	NaOH+Na ₂ SiO ₃	-	15.7-19.1	-	-	100:0-50:50	27-30	✓	11	-	-	-	[11]
Silty sand, N	NP	FAF	NaOH+Na ₂ SiO ₃	15-25	11-19	-	7.5	0.5	20	✓	2.3	✓	-	✓	[12], [13]
Silty sand, N	NP	FAF	NaOH+Na ₂ SiO ₃	10-20	8-8.8	-	5-12.5	0.5-1	20	✓	4.2	✓	✓	✓	[14]
Silty clayey sand	6	FAC	NaOH+Na ₂ SiO ₃	30	9.6-17.1	-	5	0.66-9	27-30	✓	9	-	-	✓	[15]
		FAC&GGBFS		40-60							16				
Clayey sand with gravel	14.9	FAF	NaOH+Na ₂ SiO ₃	2-10	-	35	6-18	1-4	27-30	✓	20.5 ^g	-	-	✓	[16]

PI = Plasticity index, FAF = Fly ash (type F), FAC = Fly ash (type C), MK = Metakaolin, GGBS = slag, N = natural deposit, S = synthesized, SM = source of aluminosilicate material, A = activator, T = Temperature, CCR = Calcium carbide residue, a = soil reactivity utilised, b = NaAlO₂ solids used instead Na₂SiO₃ solution, C = Approximated values, f = soaked, g = 14 days available, * set with Si/Al = (1.6-1.8) and Na/Al = 1

[1]. (Verdolotti et al. 2008); [2]. (Cristelo et al. 2011) ; [3]. (Cristelo et al. 2012); [4]. (Cristelo et al. 2013); [5]. (Sargent et al. 2013); [6]. (Zhang et al. 2013); [7]. (Zhang et al. 2015); [8] (Singhi et al. 2015); [9] (Phetchuay et al. 2016); [10]. (Liu et al. 2016); [11]. (Phummiphan et al. 2016); [12]. (Rios et al. 2016b); [13]. (Rios et al. 2016a); [14] (Rios et al. 2017); [15] (Phummiphan et al. 2018); and [16] (Teerawattanasuk et al. 2018)

2.4.1.3.1 Microstructural characteristics of geopolymer treated soils

Several studies have been performed in the literature using Mineralogical X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) techniques to provide insights into the microstructure of geopolymer-treated soils, see Table 2.5. This involves tracking cementitious growth induced by geopolymer to explain the mechanism of enhancement within treated soils on a micro-scale. In general, fly-ash based geopolymer addition is reported to densify the fabric of treated soil similar to that of lime and OPC-treated soils (Sargent 2014). It was found the homogeneity of the clay fabric was improved with the addition of fly-ash based geopolymer resulting in more closely linked clay particles and fewer voids. Such enhancement is mainly attributed to the precipitation of artificial cementation products and the development of bonds within soil particles during curing (Cristelo et al. 2012; Liu et al. 2016), see Figure 2.8. This finding was supported by Phummiphan et al. (2016), who detected through SEM analysis conducted on marginal lateritic soil, a formation of etches holes on the surface of reacted fly-ash particles within treated soil, see Figure 2.9. It was claimed that the etches holes were caused by leaching silica and alumina from the surface of the activated fly-ash. In the current thesis, limited SEM technique will be used to analyse a typically treated clay mixture to increase the understanding of how the structure of the geopolymer-clay mixture contributes to its enhanced engineering characteristics.

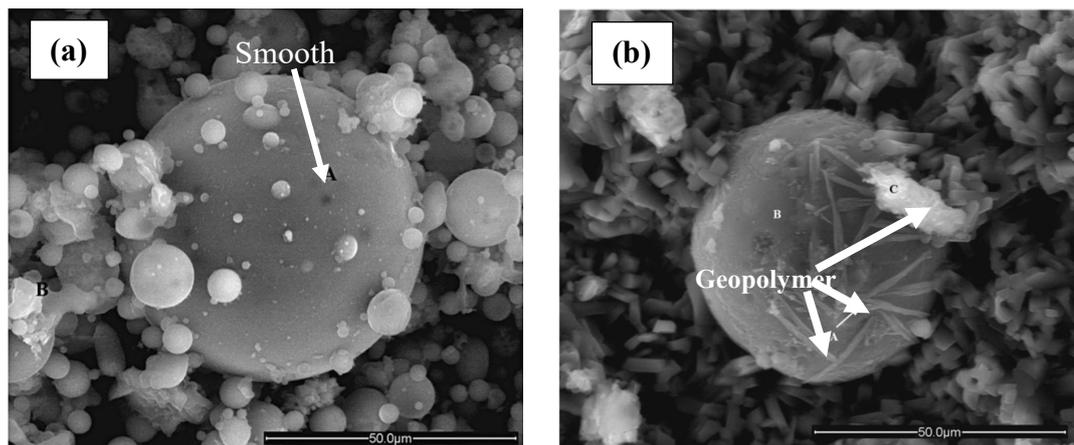


Figure 2.8: Scanning electron microscopy (SEM) showing a trace of geopolymer cementitious product: (a) typical unreacted fly-ash particle; and (b) activated fly-ash particle with treated soil (Cristelo et al. 2012).

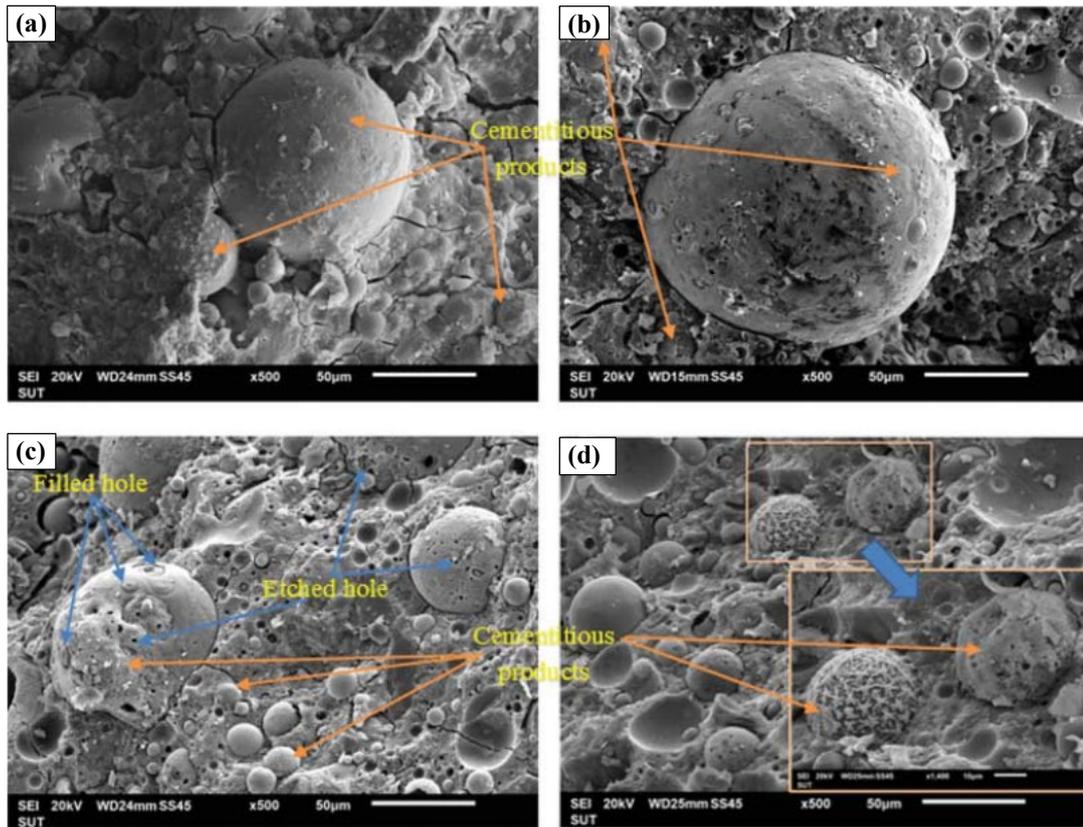


Figure 2.9: SEM images of geopolymer treated lateritic soil at various sodium silicate to sodium hydroxide ratios: (a) 100:0; (b) 90:10; (c) 80:20; and (d) 50:50 (Phummiphan et al. 2016).

2.4.1.3.2 Unconfined compressive strength characteristics

The unconfined compressive strength (UCS) is the most commonly used test to characterise the strength of geopolymer-treated soils, as reported by many researchers (see Table 2.5). In general, the mixing of geopolymer within the soil matrix was found to promote enhanced unconfined compressive strength (Cristelo et al. 2012; Liu et al. 2016; Phummiphan et al. 2016). The addition of geopolymer increases peak strength, and decreases the corresponding axial failure strain of the geopolymer-treated specimens, both of which contributes towards a stiff response similar to that of OPC-treated soils, Figure 2.10 (Rios et al. 2016b). The enhancement was attributed to the development of artificial bonding induced by the geopolymerisation within soil particles, as described previously in Section 2.4.1.3.1. However, considering the literature summary presented in Table 2.5, high variability was detected within UCS results of different research; while some geopolymer mixtures form products that may strengthen the treated soils significantly, others may form products associated with a

small strength increase. It seems that the effect of geopolimer on UCS is mixture dependent and varies according to the quantity and type of the reaction products through the treatment process. Curing conditions (i.e. temperature and time) also affect the amount of reaction products and the level of enhancement in the strength of treated soils (Cristelo et al. 2011). The factors controlling the strength enhancement of geopolimer-treated soils will be discussed in detail subsequently.

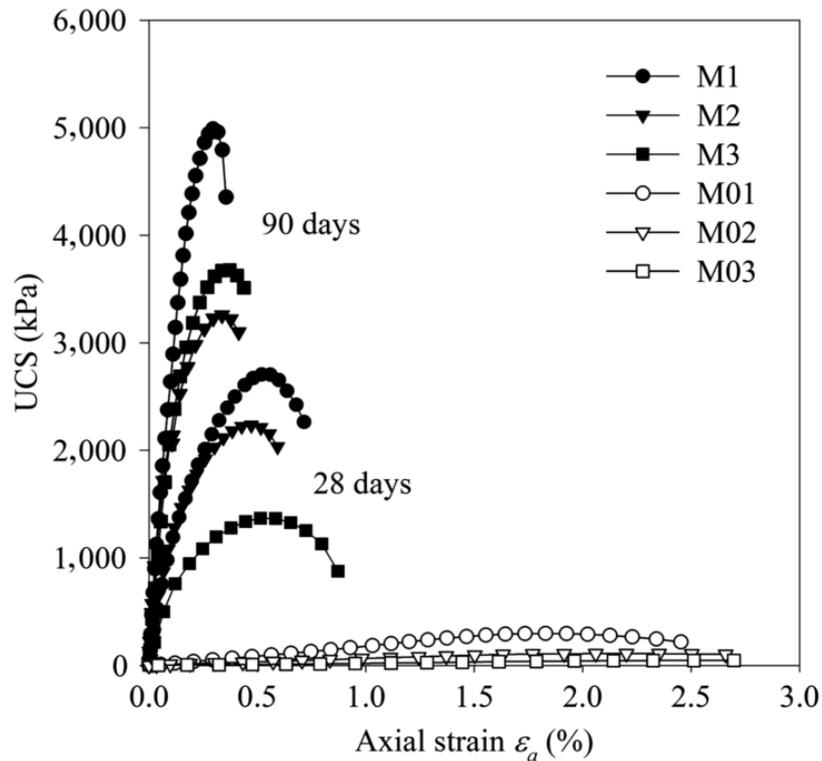


Figure 2.10: Effect of curing time on the stress-strain curve of geopolimer-treated soil (note: (M1-3) are denoted to activated fly ash-soil mixtures; (M01-03) denotes for unactivated mixtures) (Rios et al. 2016b).

2.4.1.3.3 Undrained/drained shear strength characteristics

Despite the fact that the UCS testing is commonly used to characterize the strength performance of stabilised soils, triaxial testing is recommended for further investigation of the response of geopolimer soils towards the monotonic loading as it simulates the effect of confining pressure and pore water pressure, which are considered critical in the evaluation of soil strength. Limited literature exists on the shearing behaviour of the geopolimer-soil mixtures considering the drained/undrained conditions. Triaxial studies focusing on the geopolimer use are scarce and mostly dedicated to treating sands rather than clays. Among limited research, Rios et al.

(2016b) carried out anisotropic triaxial drained tests on silty sand treated with geopolymer. The geopolymer binder comprised fly-ash (Class F) and chemical activator based on 50% weight ratio of sodium silicate to sodium hydroxide. Based on the triaxial test results, a range in deviator stress was documented for different stabilised mixtures (Figure 2.11). All tested mixtures showed a general stress-strain behaviour of a brittle response similar to those of OPC-treated soils by which high peak deviator stresses for the stabilised specimens were recorded at low strains followed by strain-softening. Volumetric strain change indicates an initial contraction tendency before a significant dilation.

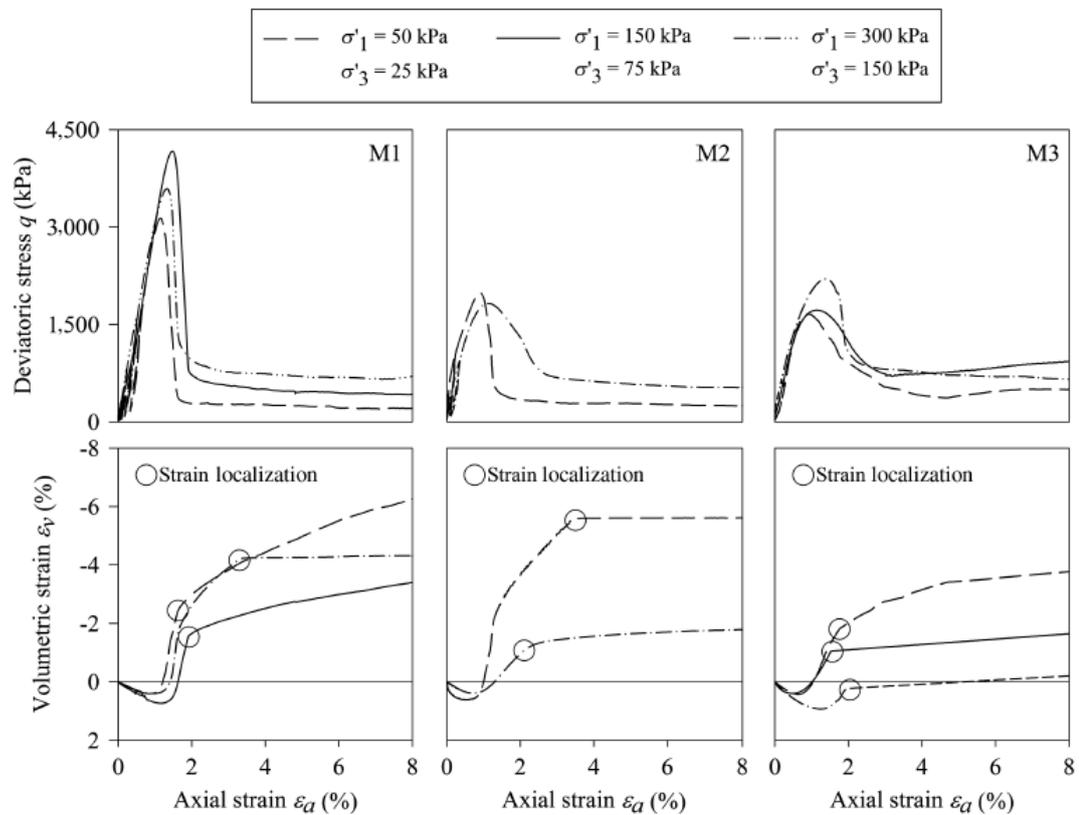


Figure 2.11: Triaxial CU test results of geopolymer stabilised silty sand (Rios et al. 2016b).

Rios et al. (2017) reported the behaviour of the strength failure envelope in the q - p' space (q = deviator stress and p' = mean effective stress) for the untreated and geopolymer-treated sand specimens using triaxial testing. The test results show a higher failure envelope compared to untreated soil, see Figure 2.12. It was also reported that treated soil showed higher strength parameters, i.e. angle of internal friction (ϕ) and cohesion (c) comparable to those in lime and OPC-treated soils. These

were related to the effect of cementation effects induced by geopolymer within treated soil.

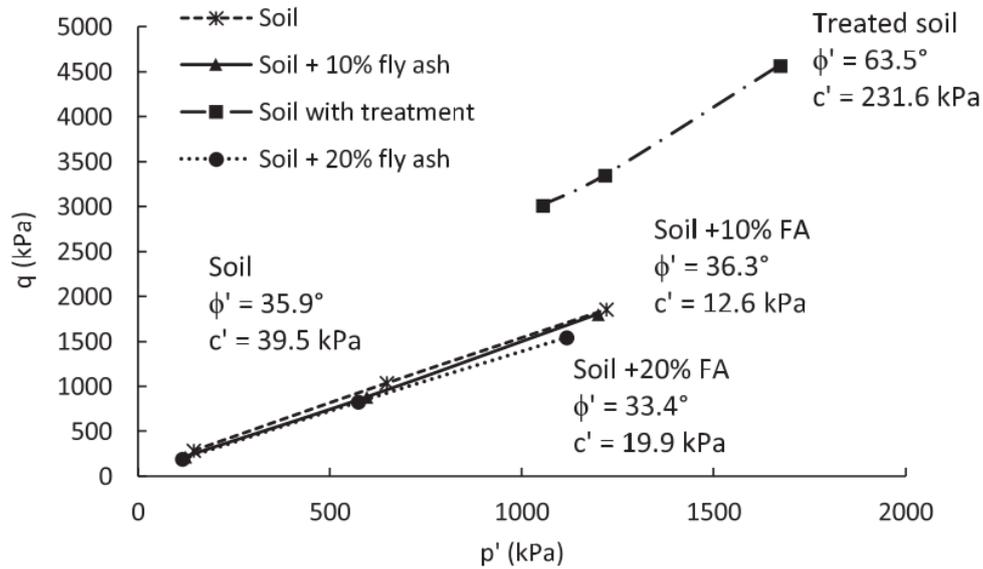
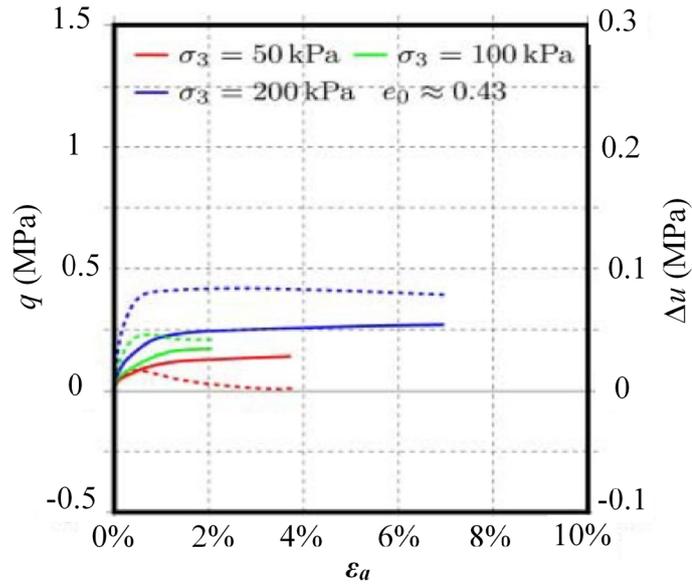
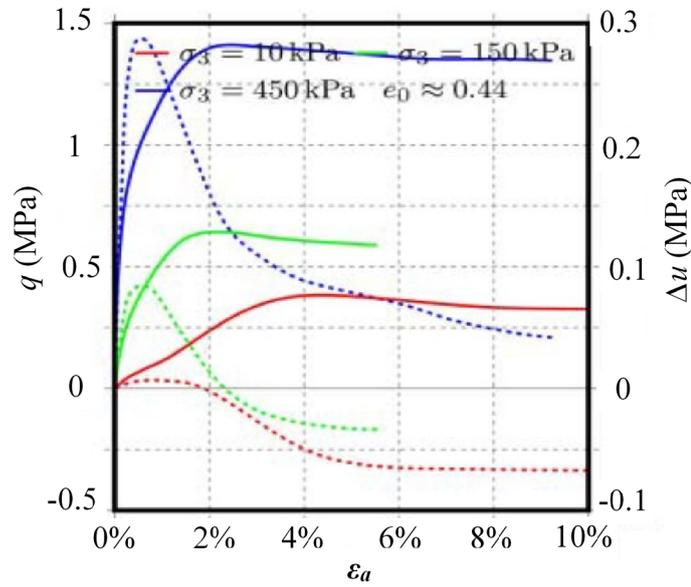


Figure 2.12: Strength envelope for tested mixtures and derived strength parameters (Rios et al. 2017).

Based on limited testing, Corrêa-Silva et al. (2018) reported the undrained shear behaviour of clay soil before and after treatment with fly-ash based geopolymer. According to the test results (see Figure 2.13), higher peak resistance and lower corresponding strain were detected for the treated clay compared to the untreated clay. In terms of pore water pressure, the stabilised specimens showed an increase in the pore water pressure at low strains before a reduction shortly before the peak strength. It was also reported that the significant influence of stabilisation was through the increment of the friction angle and cohesion values, see Table 2.6.



(a) original soil



(b) Mixture A15

Figure 2.13: Consolidation undrained triaxial tests performed after 28 curing days (q evolution indicated with a solid line and Δu with dash line): (a) original soil; and (b) traeted soil (Corrêa-Silva et al. 2018).

Table 2.6: Shear strength parameters from consolidated undrained triaxial tests (Corrêa-Silva et al. 2018).

Mixt.	Total stress				Effective stress			
	Peak		Critical state		Peak		Critical state	
	c_p	φ_p	c_{cs}	φ_{cs}	c_p	φ_p	c_{cs}	φ_{cs}
Soil	Not present		25 kPa	19°	Not present		17 kPa	28°
A15	88 kPa	33°	75 kPa	33°	27 kPa	47°	7 kPa	42°

2.4.1.3.4 Durability characteristics

Durability is a crucial concern as extreme weather conditions with variable moisture and temperature may result in degradation of the mechanical properties of treated soils (Nicholson 2014). Durability studies focusing on the use of geopolymer are scarce and mostly dedicated to wetting-drying tests. The literature concerning the effects of geopolymer on the freezing-thawing performance of treated clays and strength performance after leaching is not existed. The findings of some wetting-drying durability studies will be reviewed below as they of particular relevance to the present study.

Little literature exists on the wetting-drying durability performance of the geopolymer-soil mixture. Among limited research, Rios et al. (2017) performed wetting-drying durability study on silty sand specimens treated with fly-ash based geopolymer adopting OPC-Soil standards. For specimens subjected to wetting-drying durability cycles at seven days of curing, the volumetric change, water content and soil loss due to brushing were monitored and recorded for each 48 h cycle. Unconfined compressive strength was also tested at the end of specific cycles. The test results confirmed the stable performance (i.e. low volumetric change and reasonable residual strength) of fly-ash based in treated sand against wetting-drying durability cycles. The study promoted stabilisation of sand with geopolymer as a competitor to OPC. Sargent et al. (2013) reported similar high resistance for the volumetric changes in clay treated with fly-ash based geopolymer, see mixture PFAAA in Figure 2.14. However, although geopolymer-treated clay mixture survived the 12 durability cycles, low residual UCS performance was detected. According to Sargent et al. (2013), the low performance of the individual activated fly-ash binder was attributed to the lack of clay content in the stabilised soil, which limited the cation exchange capacity and the chemical reactions to occur. However, the lack of information on the chemical composition and the reactivity of the fly-ash used (high or low calcium content) at ambient temperature might interfere with later evaluation.

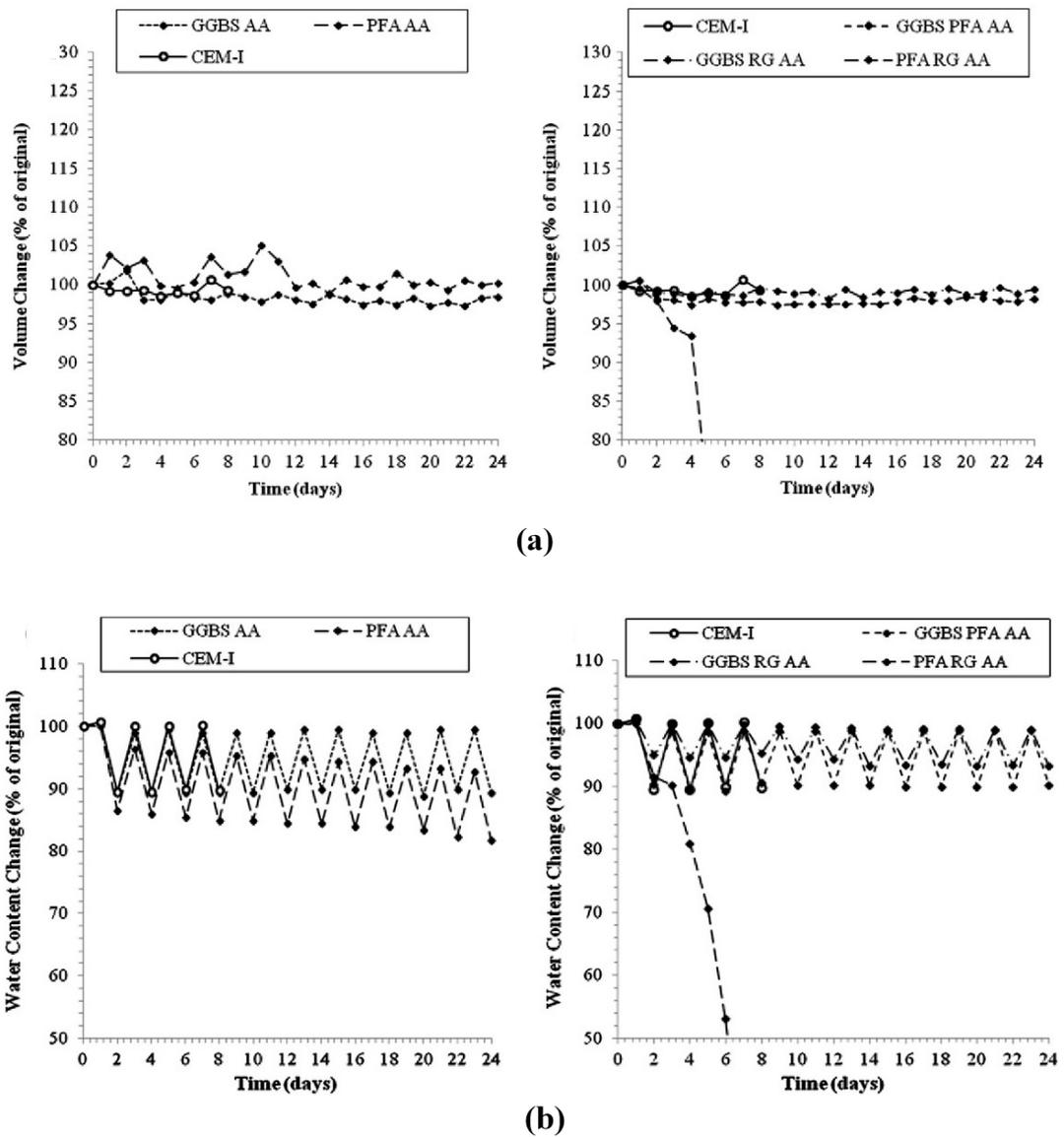


Figure 2.14: Wetting-drying durability cycles of soil treated with different alkaline binders: (a) volume changes; and (b) water content changes (Sargent et al. (2013)).

2.4.1.4 Factors affecting the level of cementation of geopolymer-treated soils

It is well defined in the literature that the level of cementation within treated soils and the corresponding enhancement of soil properties using traditional binders depends upon several factors such as the type of binder, binder content, type of soil and curing conditions (Little 1995; Bell 1996; Bergado et al. 1996; Little 1998; Ismail et al. 2002; Horpibulsuk et al. 2004; Lorenzo et al. 2006; Kavak et al. 2007; Khattab et al. 2007; Sakr et al. 2009; Sariosseiri et al. 2009; Rahimi et al. 2015; Nguyen et al. 2017). Therefore, it is critically important to determine and understand the factors that may affect the level of formation of the N-A-S-H product within geopolymer-treated soils.

Following are some literature details related to the controlling factors of geopolymer-treated soils.

2.4.1.4.1 Fly-ash and activator types

The type of fly-ash used in the geopolymer has an important effect on the strength enhancement of geopolymer-treated soils, as each type of fly-ash (i.e. Class C and Class F, see Section 2.4.1.1) has different chemical composition, which affects the reaction products. The alkaline activator attacks all types of fly-ash; however, the type denoted as Class F is more effective for strength improvement than Class C (Cristelo et al. 2012), Figure 2.15. This is attributed to the availability of a higher content of silica and alumina in Class F than that of Class C, which contributes in producing a higher amount of cementations products and thereby enhances the soil fabric. Similar to the effect of fly-ash type, the type of activator also has a significant impact on soil enhancement. This is evidenced in work carried out by Liu et al. (2016), who promoted the use of potassium hydroxide based activator than sodium hydroxide for a more compact and a higher artificial bonding on a micro-structural level within treated soils, resulting in a substantial strength improvement. Although potassium-based activator enhances the soil structure through geopolymer, it has been found to see some cost limitations (Provis 2009). Therefore, the sodium-based activator is commonly suggested for geopolymer in soil stabilisation (Cristelo et al. 2011; Cristelo et al. 2013; Zhang et al. 2015; Phetchuay et al. 2016; Phummiphan et al. 2016; Rios et al. 2016a; Rios et al. 2017).

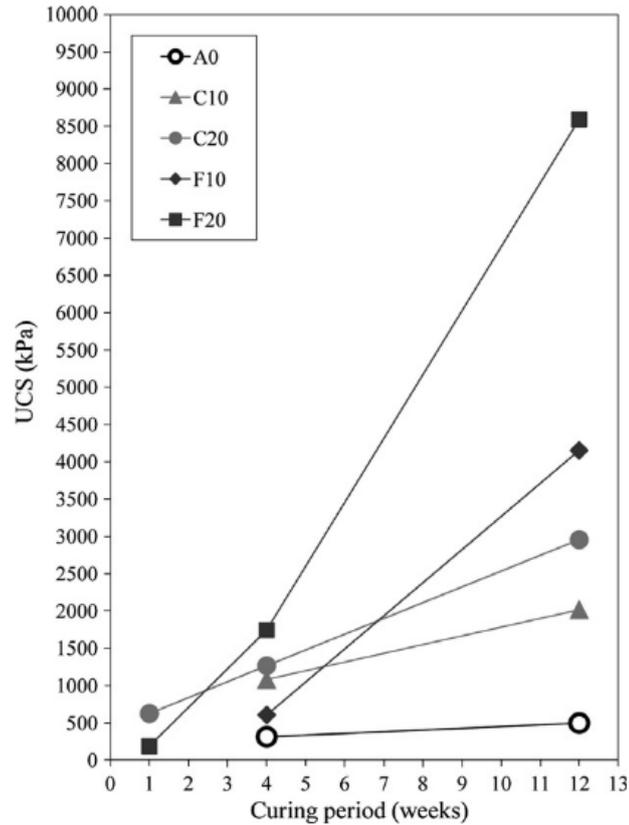


Figure 2.15: UCS values with curing time [note: (C) and (F) are denoted to fly-ash types used; (A) denotes for natural soil] (Cristelo et al. 2012).

2.4.1.4.2 Fly-ash content

The fly-ash content impacts the enhancement of geopolymer-treated soils significantly. Cristelo et al. (2011) reported an enhancement in strength of treated soils up to 120% to 2.5 MPa at 28 days when fly-ash/soil weight ratio is increased from 20% to 50%. Similarly, Phetchuay et al. (2016) reported an approximately linear increase in unconfined strength of geopolymer-treated specimens with the increase of activated fly-ash content, see Figure 2.16. In all cases, the increase in the mechanical properties of geopolymer-treated soils due to the increase in activated fly-ash content was attributed to the increased formation of the N-A-S-H cementitious product within stabilised soils due to the increase in silicate and aluminate minerals leached by the chemical activator (Phummiphan et al. 2016). The increase in the amount of cementitious products increased the level of bonding between soil particles, which is responsible for the enhancement of soil properties (Liu et al. 2016). However, no literature exists on a process to determine the optimum amount of fly-ash within geopolymer for soil stabilisation.

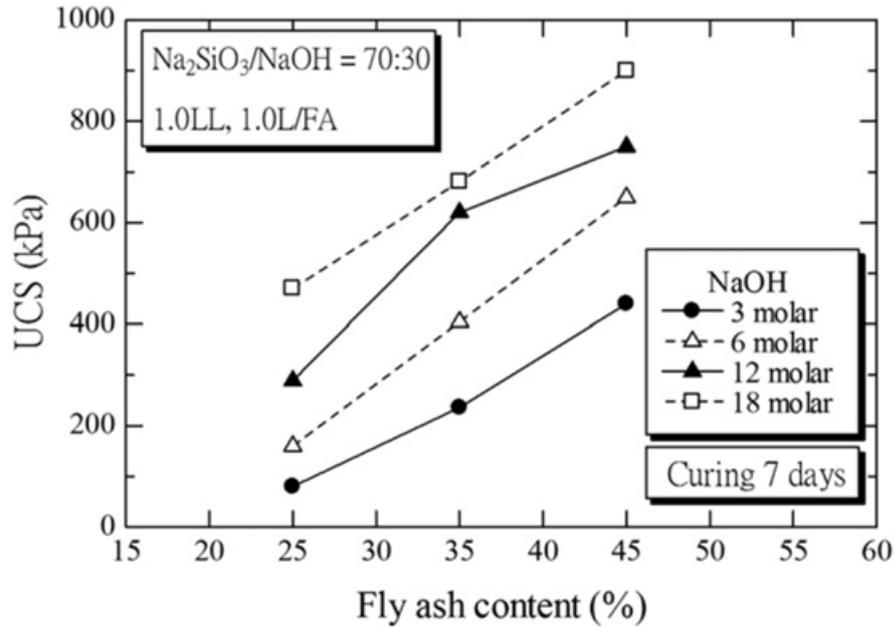


Figure 2.16: Effect of fly-ash concentration on strength performance of geopolymer-treated soil (Phetchuay et al. 2016).

2.4.1.4.3 Activator content

A strong dependency exists between the activator/fly-ash weight ratio and the level of enhancement in the mechanical properties of geopolymer-treated soils (Cristelo et al. 2013). The increase in the activator concentration may increase the alkalinity (i.e. pH) of the reaction environment, which in turn increases the dissolution of solid aluminosilicate oxides within treated soils and the amount of cementitious products developed. However, it has been found in the literature that the optimum activator concentration is not necessarily the maximum value. This conclusion was highlighted by the work done by Cristelo et al. (2011), who investigated the UCS performance of three mixtures activated with sodium silicate solution and different NaOH concentrations of 10, 12.5 and 15 molal. The test results revealed that specimens treated with 12.5 molal concentration gained high long-term strength than 15 molal mixture. This was attributed to the ratio of silica to sodium oxides within the activator, which was approximately one and causes the activator solution to be unstable (crystallises) and affected the geopolymer reaction negatively. However, for economic reasons, it is necessary to minimize the amount of activator to achieve the required enhancement; the activator contributes with the highest costs affecting considerably the total costs of geopolymer (BZE 2017). Similarly to the fly ash content, no literature exists on the

practical methods to quantify the optimum amount of activator within geopolymer-treated soils.

2.4.1.4.4 Curing time

The strength gain in geopolymer-treated soils was found to be time-dependent, and it was revealed that a significant increase in strength could be achieved up to one year curing time (Cristelo et al. 2011). The increase in curing period allows the formation of more cementation products within treated soils, thereby enhances the soil more. This was particularly supported by Phummiphan et al. (2016), who stated that the increase of curing time increases the leaching process and the number of the etched holes that developed, which in turn increases the amount of cementitious products developed (Phummiphan et al. 2018).

The rate of gain in the strength of geopolymer-treated soils is not constant and gradually decreases with the increase of curing time. Figure 2.17 shows the effect of curing time on the UCS of geopolymer-treated soils (Phetchuay et al. 2016), and it can be observed, as indicated by the logarithmic relationship, the progressively decreasing rate of strength improvement with time. The gradual decrease in the rate of increase of UCS can be attributed to the progressive slowing down of the geopolymerisation reaction due to the exhaustion of different components in the reaction environment.

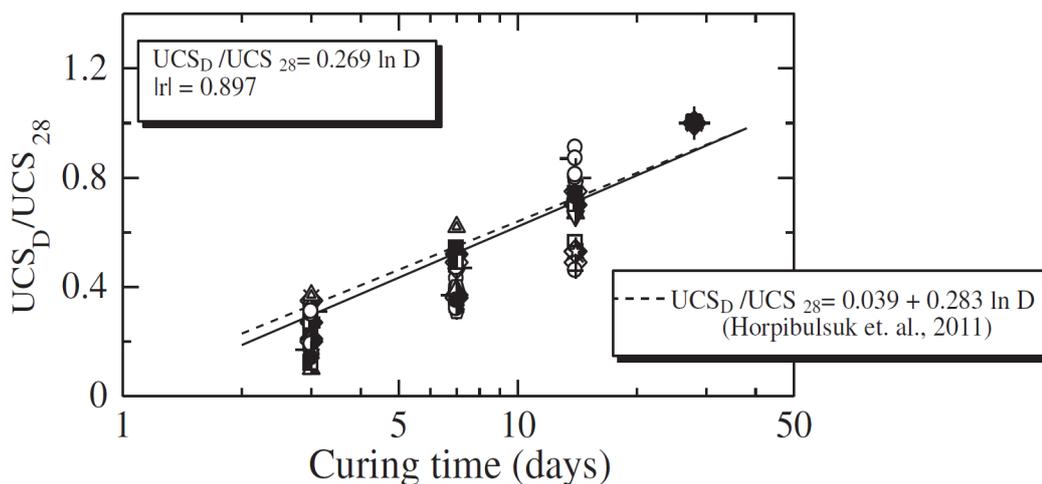


Figure 2.17: Strength development with curing time for Coode Island Silt treated with geopolymer (Phetchuay et al. 2016).

2.4.1.4.5 Curing temperature

It has been found in the literature that the geopolymerisation reaction described earlier in Equations 2.1 and 2.2 was favoured at elevated temperature (Palomo et al. 1999; Sindhunata et al. 2006; Davidovits 2008; Rovnaník 2010). Van Jaarsveld et al. (2002) reported that increasing curing temperature to 70°C causes an increase in compressive strength of fly-ash based geopolymers. Temuujin et al. (2009) recommended a range between 40°C and 100°C for 4 to 48 hours for high strength gain in geopolymers. Sindhunata et al. (2006) attributed the positive effect of elevated temperature to the increased solubility of the silicate and aluminate that increase the amounts of cementitious products produced, which enhance the rate of strength gain. However, curing temperature is the most challenging issue for geotechnical bulk applications such as in-situ ground improvement (Xu et al. 2000; Gianoncelli et al. 2013; Yazdani et al. 2013; Deb et al. 2014). This is because elevated temperatures are not possible for ground improvement and treatment must be conducted at ambient temperature. However, when cured at ambient temperature, geopolymer-treated soils typically take a considerably longer period for strength development compared to that of OPC-soil mixtures. According to Cristelo et al. (2011), UCS of geopolymer-treated soils reveal only 20-40% of their strength at 28 days of curing compared to OPC stabilised samples, whereas 40 to 60% of strength is gained after 3 months of curing. This forms a drawback in geopolymers utilisation for soil stabilisation. Therefore, to substitute the elevated temperature effects as a reaction accelerator, more aggressive alkaline media is usually required. This is a significant limitation in the practical utilisation of geopolymers for soil stabilisation, which leads to the development of an enhanced geopolymer mixtures.

2.4.2 Geopolymer treated soil - (*Model III*)

Utilising calcium-based components as a supplementary material to geopolymer mixtures through *Model III* of alkali-activation described earlier, to enhance the mechanical properties of treated soil, may involve more complex reactions than *Model I*. This is because the resulted products may consist of multi gels (i.e. C-A-S-H and N-A-S-H), which react at different rates (Granizo et al. 2002; Yip et al. 2005). García-Lodeiro et al. (2013) attempted to describe a conceptual model the complicated activation stages of *Model III* using a mixture of fly-ash and OPC as a source of calcium, as presented in Figure 2.18. The conceptual model involves: (1) dissolution of aluminosilicate and calcium components in the alkaline solution through rupturing the Si-O-Si and Al-O-Al bonds in the fly-ash, and the Ca-O and Si-O bonds in the calcium-based material (i.e. OPC); (2) formation of the N-A-S-H gel from Na⁺ and silicon and aluminium dissolved species and C-S-H gel from Ca⁺ and silicon dissolved species; (3) developing N-A-S-H and C-S-H gels through uptake more Si into the systems; (4) diffusion of the Al and Ca⁺ (not participating in the formation of C-S-H gel) across the cementitious matrix formed resulting to form (N,C)-A-S-H gel of three-dimensional structure and C-S-A-H gel of two-dimensional structure; and (5) distortion of the Si-O-Si bonds by the polarising effect of Ca⁺ and the formation of Si-O-Ca bonds which induces more stress and ultimately rupture. According to García-Lodeiro et al. (2013), Steps (2) and (3) may occur rapidly, whereas Step (4) occurs over 28 days of curing. The final stage of reaction (i.e. Step 5) may take up to 1 year of curing.

Yip et al. (2005) attributed the enhancement in the mechanical properties of geopolymer-treated soils with calcium-based components to the role of C-A-S-H in filling the voids within geopolymer binder which helps to bridge the gaps between alkaline cement products (i.e. N-A-S-H) and unreacted particles which in turn contributes to the development of enhanced mechanical properties.

Phetchuay et al. (2016) suggested the use of calcium carbide residue, referred to CCR, as a source of calcium oxide to enhance the performance of fly-ash based geopolymer in the stabilisation of soft marine clay, see Table 2.5. CCR is a waste material from acetylene gas factories, which has high calcium hydroxide Ca(OH)₂ content. The study revealed that the addition of CCR by 12% can enhance up to 1.5 times the strength of clay stabilised by fly-ash based geopolymer using the same activator content. The

significance of this calcium oxide addition for enhancement of the mechanical performance of soil treated with fly-ash based geopolymer can also be detected from the work done by Sargent et al. (2013), Table 2.5. An increase of approximately 600% in the 28 days UCS value was detected when GGBFS is blended, as a calcium oxide source, with fly-ash in 1:1 ratio to stabilise silty sand soil. Singhi et al. (2015) also indicated a similar increasing trend in UCS when partially replacing the fly-ash by GGBFS within treated clay; for 20% of activated aluminosilicate materials mixed with clay, a higher 28 days UCS value of 2.5 MPa was achieved when replacing only 20% of the fly-ash with GGBFS compared to approximately 0.2 MPa for activated fly-ash at zero GGBFS content. It is worthwhile mentioning that GGBFS is a calcium-rich aluminosilicate material of low cost as it is produced from steel industry waste and it is one of the most commonly used materials in alkali-activated binders (*Model II*, Section 2.4).

Despite the advantage, fly-ash based geopolymer incorporating calcium-based components for soil stabilization is still not widely used in the geotechnical industry. This underutilization may be attributed to the lack of systematic approach; current literature is limited in terms of testing and conducted for specific individual soil types. Therefore, extensive research is required to support the utilisation of any enhanced (cost-effective) geopolymer mixture for soil stabilisation. The current thesis will focus on utilising GGBFS, due to its wide availability as a waste material produced from the steel industry, to enhance the performance of fly-ash based geopolymer for clay stabilisation.

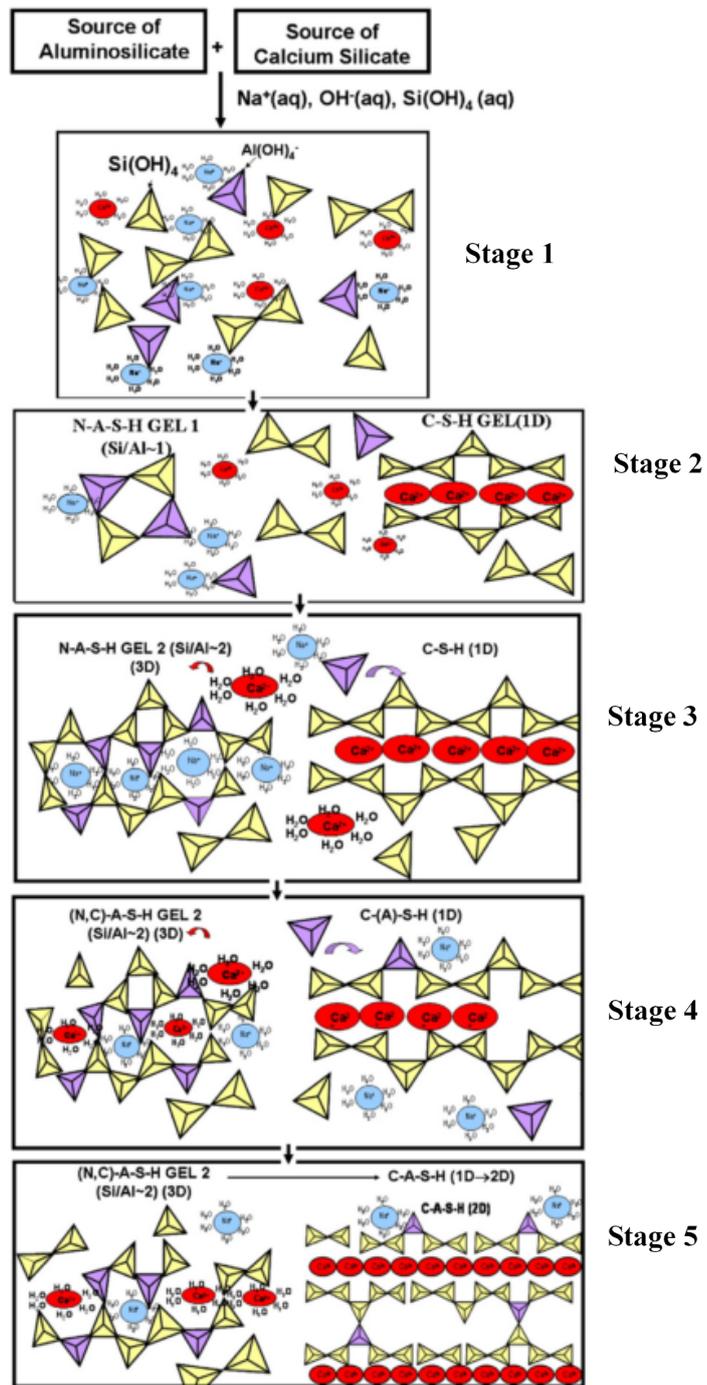


Figure 2.18: Conceptual alkaline activation model III for a mixture of fly-ash of high aluminosilicate and low calcium content blended with OPC of high-calcium content (García-Lodeiro et al. 2013).

2.5 LIMITATIONS AGAINST BROAD UTILISATION

As a new research trend, geopolymers are still unrecognised widely by the geotechnical construction industry for clay stabilisation. This is partly related to the position of OPC and lime as well-defined traditional binders and to the conservative attitudes of the geotechnical community towards replacing existing products (Komnitsas et al. 2007). However, the underutilisation of geopolymers for clay stabilisation is more related to the cost limitations caused by the need for the consumption of high activator contents to allow for an ambient curing. Also, due to the uncertainty that this binder is suitable to treat all clay types and the absence of practical design procedures. These limitations are presented in more detail in the following sections.

2.5.1 Curing at ambient temperature – cost limitation

Although the research presented in Sections 2.4.1.3 suggested that the soil treatment with fly-ash based geopolymers through *Model I* of alkaline activation was competitive, in terms of UCS compared to treatment with OPC, its research indicates a high consumption of activator contents. The use of high activator content in most geopolymer-soil studies was necessary to facilitate an initial dissolution and condensation reactions (i.e. geopolymerisation) within treated soils at ambient curing; otherwise, elevated temperature is required, which is considered non-applicable for soil treatment (Section 2.4.1.4.5). In Australia, fly-ash based geopolymers, as a binder used for concrete applications cured at elevated temperature, is currently 10-15% more expensive than OPC due to the cost of sodium silicate activator (BZE 2017); the fly-ash within geopolymers contributes to minimum cost as it is utilised from industrial waste. Increased activator content to facilitate ambient curing, as in soil stabilisation, may increase more the stabilisation costs, which in turn may restrict wider utilisation of geopolymers for soil stabilisation. This has led to the development of enhanced geopolymer mixtures for ambient curing (through *Model III*), as discussed previously in Section 2.4.2. However, the literature regarding enhanced geopolymer mixtures for clay treatment is still scarce. This study will address this knowledge gap.

2.5.2 Suitability of clay soils for geopolymer treatment

Adequate binders for soil stabilisation are generally required to cover a broad spectrum of soil types. However, not all soils are suitable for stabilisation using specific binders due to complexity in materials and relations (Sargent 2014). Over a wide variety of soil types and the effect of extreme field conditions (i.e. temperature and moisture

changes), a possibility exists for a premature failure within treated soils. This is due to both clay content and related mineralogy/plasticity within the soil that may affect the level of formation of artificial cementations bonds within the soil and the overall stabilisation effectiveness. For traditional binders, for instance, Woo (1971), as cited in Bishwajit (2013), reported that the effectiveness of OPC decreases with an increase of clay content and plasticity within the host soil due to mixing difficulties. In contrast, since the strength is developed through the participation of clay particles in the pozzolanic reaction described earlier in this chapter, the effectiveness of lime stabilisation is decreased with the decrease of clay content (Little 1995; Bergado et al. 1996).

Different studies have highlighted the feasibility of geopolymer to treat clayey soils (e.g. Cristelo et al. 2011; Cristelo et al. 2012; Cristelo et al. 2013; Zhang et al. 2013; Zhang et al. 2015; Liu et al. 2016). However, much of the geopolymer-soil research has so far been limited to a specific type of soil and the corresponding geopolymer mix, investigating the impact of the specific geopolymer mixture. To the knowledge of the author, the effects of such soil-related variables (i.e. mineralogy and plasticity) are still unknown with respect to geopolymer treatment of clay soils. It is anticipated that high plasticity of clays might influence the workability during mixing with geopolymer, as in the case of OPC, which in turn affects the distribution of the binder and the homogeneity of the mixture. On the other hand, the inherent nature of natural clay colloids (organic and inorganic) and their buffering capacity, i.e., absorbing, holding and releasing ions including the (OH-) ions, might affect the pH response and the reaction after treatment as in lime-soil treatment (Little 1995). Because the addition of geopolymer may alter the gradation and mineralogy of the host clay, the response for treatment may likely to be different from one clay to another. This thesis will address this gap among others to advance confidence in the use of geopolymer for clay stabilisation.

2.5.3 Availability of practical procedures

Practices vary among professional soil practitioners in the selection of traditional binders, testing procedures and in the criteria for evaluation of what constitutes an “effective” soil stabilisation. Consequently, several practical procedures have been established by several agencies like USACE (1984), CSIRO (1987), Portland Cement Association, National Lime Association and Texas Department of Transportation

(TxDOT) with the principal objective to facilitate appropriate selection of traditional binder type, effective dosage and recommended mixture evaluation tests. These diverse practical procedures for traditional binders are based on extensive research, each considering specific geological landscapes and certain weather conditions. At present, there are no practical procedures available to address such various aspects for clay stabilisation using geopolymer. However, the absence of clearer practical procedures is a significant barrier to the wider use of geopolymer for clay stabilisation; a procedure is needed that assesses the suitability of the clay for geopolymer treatment using criteria similar to those widely used for traditional binders. Also, there is a need for additional practical procedures to determine the initial geopolymer content using quick testing. This should reduce the need for extensive testing trials. This thesis will attempt to establish specific practical procedures to fill out this gap.

In order to establish a reliable practical procedure for geopolymer-treated clays, it is essential to understand the general criteria for evaluation of what constitutes an “effective” soil stabilisation using traditional binders. In the following, the general aspects related to binder selection, proper dosage and testing for soil stabilisation will be briefly examined, with discussion confined to the two most commonly traditional stabilisers, being lime and OPC.

2.5.3.1 Binder selection

Proper binder selection for soil stabilisation is related to several factors, namely; soil type, goal of treatment, reaction mechanism of the binder, desired properties, design life and cost (Bell 1987; Little 1995; Little et al. 2009; Nicholson 2014). In practice, soils favourable to specific chemical treatment are usually identified by correlating basic soil properties with the enhancement level (Nicholson 2014). This usually enables the suitability of a particular binder to be evaluated by soil gradation and plasticity index (PI). TxDOT (2005) practical procedures (Figure 2.19) and the USACE (1984) practical procedures (Figure 2.20 and Table 2.7) are examples for testing the suitability of OPC and lime for soil stabilisation using gradation and PI criteria. Both practical procedures recommend that high plasticity clay soils can be treated effectively with lime more efficiently than OPC to reduce plasticity and volume changes. On the other hand, low to medium plasticity ($PI < 30\%$) clay soils are recommended to be treated with OPC to produce higher strength gain in a short period of time. If the PI exceeds 30%, then OPC becomes challenging to mix with the clay

soil and the efficiency of treatment might be compromised. If OPC is to be used for the stabilisation of high plasticity soils, then lime may be mixed first to bring down the plasticity to an acceptable limit and to improve workability before adding OPC (CSIRO 1987). However, the reliance on the PI criterion only for the case of lime is not recommended as argued by CSIRO (1987), since the strength enhancement of lime stabilisation is mainly dependent on the type and amount of the clay minerals of soil, and there is a need for more sophisticated tests to determine whether lime should be used. Therefore, CSIRO (1987) practical procedures favour the use of OPC rather than lime and recommend only lime for heavy clays to reduce plasticity before adding OPC for strength improvement, as stated previously. This implies that there are no universal practical criteria for all binders to treat all clay types.

2.5.3.2 Binder content

After selecting the binder type, it is essential to establish an appropriate binder content to achieve the treatment goal. Design binder contents are generally determined based on an analysis of the effect of different binder contents on selected engineering properties of the binder-soil mixture. Depending on the treatment objective (i.e. modification or stabilisation), engineering properties of trial cured mixtures are first evaluated through proper testing before selecting the effective dosage that satisfies the construction requirements (Little et al. 2009). In practice, based on appropriate correlations with basic soil properties (i.e. gradation and plasticity index), several practical procedures have been developed which recommend initial binder contents for soil treatment. USACE (1984) practical procedures is an example of selecting the initial lime or OPC content for clay soil treatment using PI criteria, see Figure 2.21 and Table 2.8. Another example is the TxDOT (2005) practical procedures for lime treatment, which adopt the pH mixture design concept developed by Eades et al. (1966). The basic concept of the pH procedure is to add sufficient lime to the treated clay soil to ensure a pH of 12.4 in order to maintain the lime-soil pozzolanic reaction and the corresponding strength development. The effectiveness of the binder content determined for a specific soil using any method requires strict attention to controlling all the aspects of the construction process in the field, including soil pulverisation and mixing efficiency (Nicholson 2014). According to the Portland Cement Association (1995), clay soils are generally harder to pulverise, which may demand higher OPC content for effective stabilisation. Pulverisation of clay soils is important before

treatment to ensure intimate mixing of the soil with the OPC or lime (Felt 1955; Wohlgenuth 1987; Portland Cement Association 2001; Geiger et al. 2007; Bozbey et al. 2010; Bozbey et al. 2017). Pulverisation criterion usually requests a minimum of 80% of the soil-cement mixture to be passing sieve No. 4 (4.75 mm) and 100% of particles less than 25 mm (Portland Cement Association 2001).

2.5.3.3 Mixture evaluation

The engineering properties of cured mixtures that may be measured through tests to evaluate its suitability usually include plasticity, compressive strength and durability (CSIRO 1987; Little 1995). Plasticity of soil is an essential aspect of stabilisation. Plasticity describes the response of clay to moisture and can be measured using Atterberg limits tests, including Liquid limit (LL) and plastic limit (PL). For compressive strength, various measures are available in the literature and the most common is the unconfined compressive strength (UCS) since it can be easily obtained in the laboratory. The UCS may be measured at various curing periods, and the most common UCS measurements are those recorded after 7 and 28 days of curing. However, the UCS does not consider the undrained behaviour nor the effect of confining pressure, which are considered critical in the evaluation of strength. For advanced strength evaluation, the triaxial test is used, considering drained/undrained conditions.

Selected binder type and optimum content should also be validated based on actual field performance (Nicholson 2014). Moisture and temperature effects are examples of field related issues usually considered for a durable performance of binder-soil mixtures. Destructive techniques of wetting-drying and freezing-thawing have been usually employed to evaluate the durability of the properties gained in the field utilising a certain amount of OPC and lime binders in the stabilised soils (Little et al. 2009; Das 2010). Durability tests, i.e. wetting-drying and freezing-thawing, are typically conducted according to ASTM D559-03 (2003) and ASTM D560-15 (2015), respectively. Such specifications, for both tests, usually include test specimens with specific dimensions cured for 7 days and then subjected to 12 successive cycles, 48 h each, of temperature and moisture changes simulating potential extreme field conditions with a monitor to their volume changes and residual strength. Studies have shown that moisture and temperature cycles have less effect on lime and cement-treated soils compared with untreated soils. It has also been well recognised that the

resistance of treated soils against wetting-drying and freezing-thawing cycles is highly dependent on the binder type, dosage and immediate strength of the mixture as well as soil type and curing conditions (Rao et al. 2001; Bhattacharja et al. 2003; Marshall et al. 2007; Chittoori 2008).

Although leaching, as an area of research deals mainly with toxic or hazardous permeants such as leaching potential of fly-ash metals in fly-ash treated soils (Binshafique et al. 2002; Tiwari et al. 2015), it is also considered as an index for durability as it measures the permanency of the binder used in soil stabilisation (McCallister et al. 1992; Puppala 2016). Due to rainfall events and/or groundwater flow, the permeant water may react positively or negatively with the chemicals involved in the stabilising binder. Based on this, the modification of the clay-binder matrix and related properties are highly expected to be directly correlated to the water infiltrated. Barenberg (1970) conducted leach tests on lime and cement-treated soil mixtures. The treated mixtures were moulded in leach tubes of defined dimensions and then subjected to water leaching at a rate comparable to the estimated local rainfall, before conducting the chemical analysis. The analysis has proven that small amounts of chemical stabilizer leached out during these tests. McCallister et al. (1990; 1992) performed leachate tests for lime-treated soils by using a special leach fabricated cells which was designed based on column leach cylinder recommended by ASTM specifications (D34.02.02). Pressurised water was leachate through the stabilised specimens within the cells for 45-90 days. The change in variables including permeability, leach pH and leach cation changes were documented. Highest detrimental changes were detected when the lime dosage was less than the optimum amount required for modification purposes.

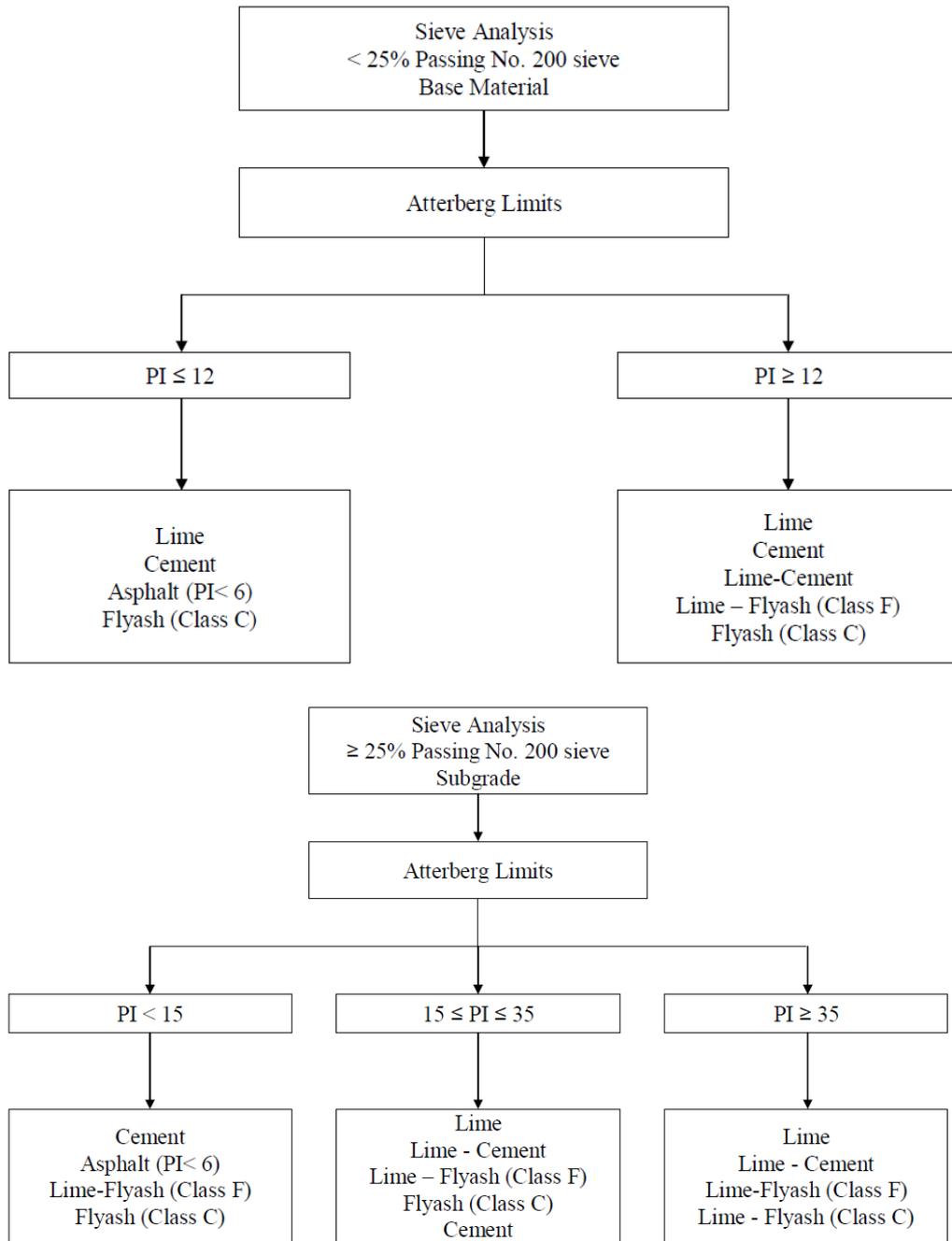


Figure 2.19: Decision tree for selecting traditional stabilisers for subgrade and base materials (Texas Department of Transportation 2005).

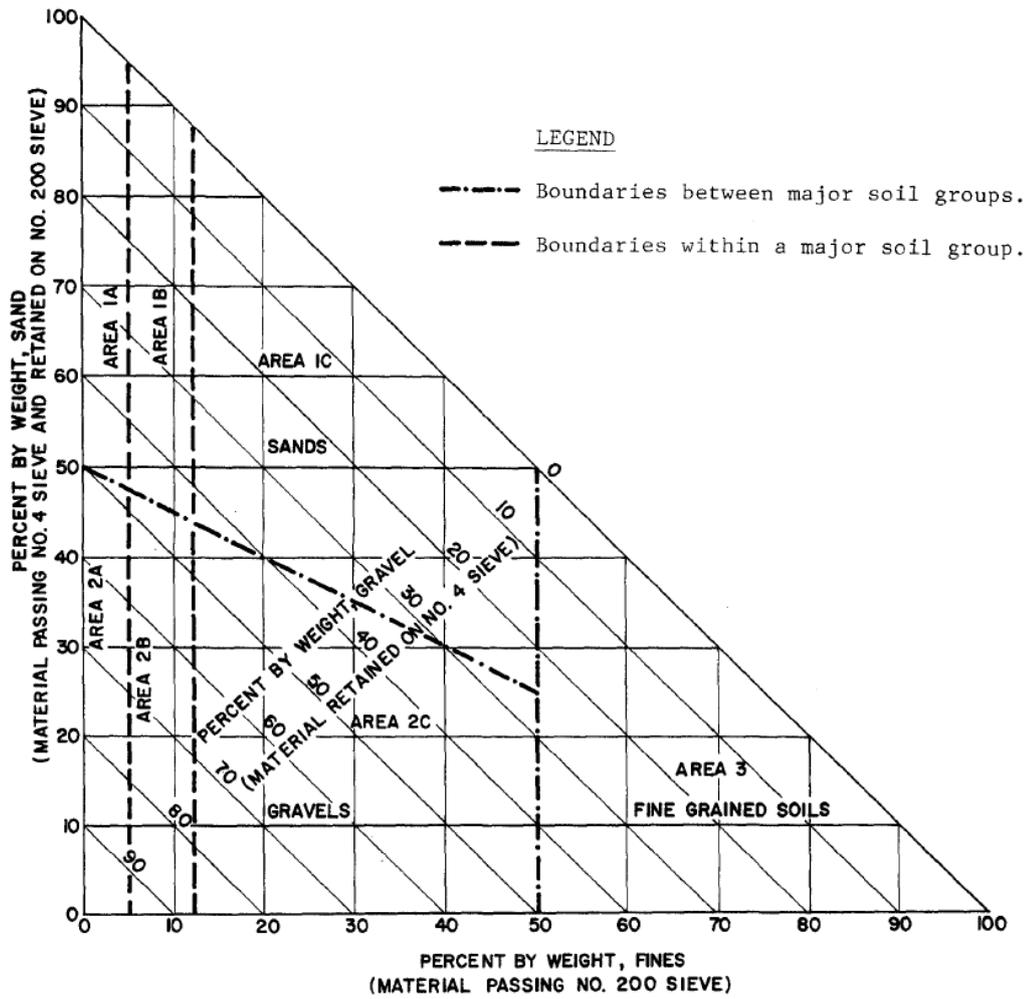


Figure 2.20: Gradation triangle for aid in selecting a commercial stabilising agent (USACE 1984).

Table 2.7: US army stabilisation practical procedures for selecting a stabilising agent, re-generated by Chittoori (2008).

Area	Soil Classification	Type of Stabilizing Additive Recommended	Restriction on LL and PI of Soil	Restriction of % Passing No. 200 sieve	Remarks
1A	SW or SP	(1) Bituminous			
		(2) Portland -Cement			
		(3) Lime-Cement-Fly Ash	PI not to exceed 25		
1B	SW-SM or SP-SM or SW-SC or SP-SC	(1) Bituminous	PI not to exceed 10		
		(2) Portland -Cement	PI not to exceed 30		
		(3) Lime	PI not to exceed 12		
		(4) Lime-Cement-Fly Ash	PI not to exceed 25		
1C	SM or SC or SM-SC	(1) Bituminous	PI not to exceed 10	Not to exceed 30% by weight	
		(2) Portland -Cement	*		
		(3) Lime	PI not less than 12		
		(4) Lime-Cement-Fly Ash	PI not to exceed 25		
2A	GW or GP	(1) Bituminous			Well-graded material only Material should contain at least 45% by weight of material passing No.4 sieve
		(2) Portland -Cement			
		(3) Lime-Cement-Fly Ash	PI not to exceed 25		
2B	GW-GM or GP-GM or GW-GC or GP-GC	(1) Bituminous	PI not to exceed 10		Well-graded material only Material should contain at least 45% by weight of material passing No.4 sieve
		(2) Portland -Cement	PI not to exceed 30		
		(3) Lime	PI not less than 12		
		(4) Lime-Cement-Fly Ash	PI not to exceed 25		
2C	GM or GC or GM-GC	(1) Bituminous	PI not to exceed 10	Not to exceed 30% by weight	Well-graded material only Material should contain at least 45% by weight of material passing No.4 sieve
		(2) Portland -Cement	*		
		(3) Lime	PI not less than 12		
		(4) Lime-Cement-Fly Ash	PI not to exceed 25		
3	GH or CL or MH or ML or OH or OL or ML-CL	(1) Portland	LL less than 40 and PI less than 20		Organic and strongly acid soils falling within this area are not susceptible to stabilization by ordinary means
		(2) Lime	PI not less than 12		

* $PI \leq 20 + [(50\text{-percent passing No. 200 sieve}) / 4]$

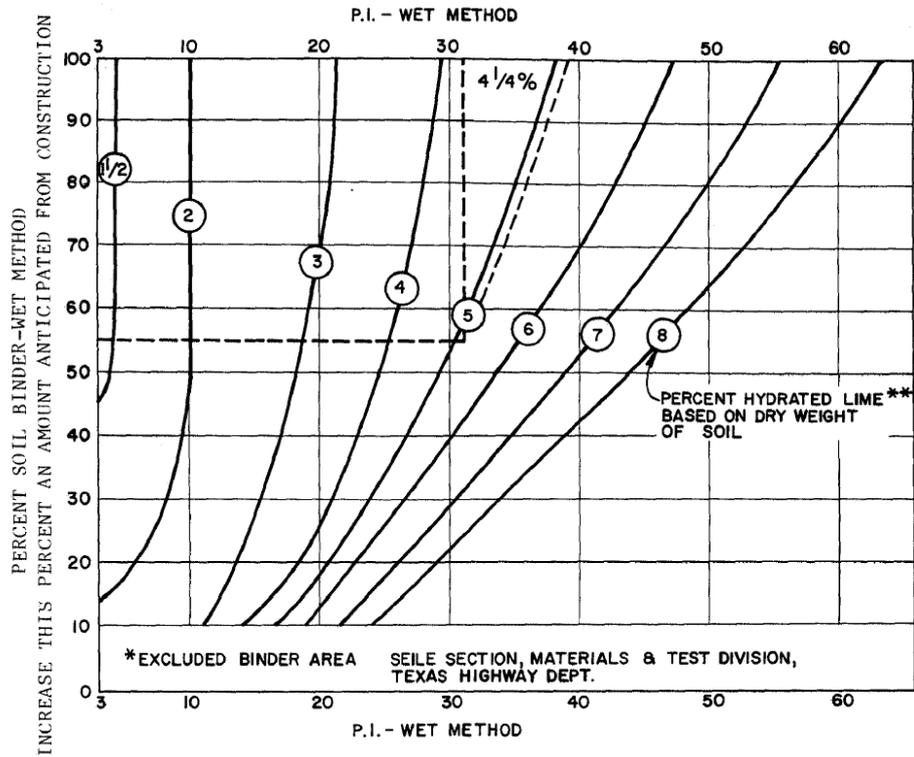


Figure 2.21: Initial design lime content (USACE 1984).

Table 2.8: Estimated cement percentages for various soil types (USACE 1984).

Soil Classification ^a	Initial Estimated Cement Requirement Percent Dry Weight
GW-SW	5
GP, SW-SM, SW-SC, SW-GM, SW-GC	6
GM, SM, GC, SC, SP-SM, SP-SC, GP-GM, GP-GC, SM-SC, GM-GC	7
SP, CL, ML, ML-CL	10
MH, OH	11
CH	10

^a soil classification corresponds to ML-STD-619.

2.6 SUMMARY

This chapter has provided a critical review of the available literature on soil stabilisation using lime and cement as popular binders and geopolymer as an eco-friendly alternative binder. Based on this review, the following conclusions can be drawn:

- Introducing fly-ash based geopolymer as a feasible binder for soil stabilisation may contribute to finding solutions to recycle the industrial by-products and to reduce the effect of the carbon footprint associated with the production of OPC and lime. Despite these advantages, the use of fly-ash based geopolymer for soil stabilisation is still not widely recognised within the geotechnical industry. This underutilisation is generally attributed to the entrenched position of OPC and lime as popular binders for soil stabilisation. Further research to confirm and demonstrate the engineering benefits of geopolymer for soil stabilisation are required from the geopolymer-soil research community to overcome this barrier.
- Due to the lack of knowledge and limited research, as well as the variability of the clay soil system, little progress has been made to adopt a standardised fly-ash based geopolymer mixtures for clay stabilisation. While various enhanced mixtures of geopolymer have been proposed (e.g. Singhi et al. 2015; Phetchuay et al. 2016), and specially designed for clay stabilisation at ambient temperature; these studies were based on limited testing and performed based on specific individual clay types, and do not provide insight into the effects of clay mineralogy and plasticity on treatment and resulting soil engineering properties, both of which are known to have significant impact in the performance of any stabiliser for clay stabilisation (Little et al. 2009).
- Although some similarity does exist between fly-ash based geopolymer and OPC in terms of soil strength enhancement, there are considerable differences that must be fully recognised. These differences are related to essential engineering properties such as strength gain rate and durability against extreme field conditions (i.e. wetting-drying, freezing-thawing and leaching); considering the effects of several factors including binder content, curing time and soil mineralogy. Moreover, the stress-strain mechanism of geopolymer-treated soil under drained/undrained conditions considering the effect of confining pressure is still not well understood.

- To gain commercial confidence and to support broader use of geopolymer for stabilisation of clay soils with varying plasticity, practical procedures are required; these should consider specimen preparation, curing conditions, testing procedures and specific mix design criteria. With the intention of providing adequate enhancement of soil properties, the practical procedures should enable the user to classify the clay soil with respect to its suitability for treatment by geopolymer and to determine appropriate binder contents for effective stabilisation. To date, no such practical procedures exist.

Considering the above conclusions, in-depth studies are required to investigate the use of fly-ash based geopolymer for stabilisation of clay soils. This thesis aims to narrow this gap. Thus, the following chapters present details of the experimental testing conducted to optimise an ambient cured fly-ash based geopolymer mixture for stabilisation of clay soils, and to further investigate and quantify the engineering properties of different treated clays. Practical procedures are also developed for geopolymer-treated clays.

CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

This research comprises a comprehensive testing program designed to investigate the effectiveness of geopolymer stabilisation as an eco-friendly alternative for traditional binders for improvement of the engineering properties of different clay soil types. This chapter provides details of the raw materials used, geopolymer admixture and preparation process, treated soil specimen preparation methods employed and details of the experimental program. Due to the wide range of experimental tests performed and the associated specifics regarding sample preparation, details of sample preparation have been included alongside details of each type of experimental testing followed by a summary of the entire testing program.

3.2 MATERIALS

3.2.1 Soils

Nine different clay soils were used in this research including seven engineered and two natural clays, each with different mineralogy. In the following sections, the properties and classifications of the soils used are presented.

3.2.1.1 Engineered clay soils

The engineered clay soils were prepared by mixing pure kaolin clay with bentonite or clean sand in different combinations of 0%, 10%, 20% and 40% (see Table 3.1); designed to represent the three most frequently occurring minerals in clay deposits (i.e. kaolinite, montmorillonite and quartz). The kaolin clay was commercially available in white-coloured powder obtained from Prestige NY, Western Australia, while the bentonite was sodium-based powder manufactured by Unimin Australia Limited, Queensland. The sand was obtained from the greater Perth region of Western Australia. Detailed characteristics of the engineered soils are listed in Table 3.2; further details regarding these clays will be presented subsequently.

Table 3.1: Engineered soil mixtures.

Mixture	Kaolin (%)	Bentonite (%)	Sand (%)
(100Kaolin)	100	0	0
(90Kaolin:10Bentonite)	90	10	0
(80Kaolin:20Bentonite)	80	20	0
(60Kaolin:40Bentonite)	60	40	0
(90Kaolin:10Sand)	90	0	10
(80Kaolin:20Sand)	80	0	20
(60Kaolin:40Sand)	60	0	40

3.2.1.2 Natural clay soils

Two natural clay soils were included in the testing program to provide a comparison with the engineered clay soils and to assess the effectiveness of the binder mix for stabilising naturally occurring soils. The natural clay soils were collected from two different locations in the greater Perth region, i.e. Baldivis and Armadale areas (see Figure 3.1). Bulk specimens for the natural clays were collected from a depth of approximately 1.5-2.0 m during excavation works. Specimens for each natural clay type were collected from one borehole to minimise the effect of variability in soil type. XRD analysis performed on the natural clays used showed different origins (see Figure 3.2). Kaolinite is the principal clay mineral for NatSoil (1) with traces of Muscovite, whereas Vermiculite (expansive mineral) is the dominant clay mineral for NatSoil (2). Detailed characteristics of the natural clay soils used are listed in Table 3.2; further details regarding these clays will be presented subsequently. To characterise the different clay soils used (engineered and natural), a range of laboratory tests were performed, following relevant Australian Standards. The test procedures and soil classification are illustrated in the following sections.

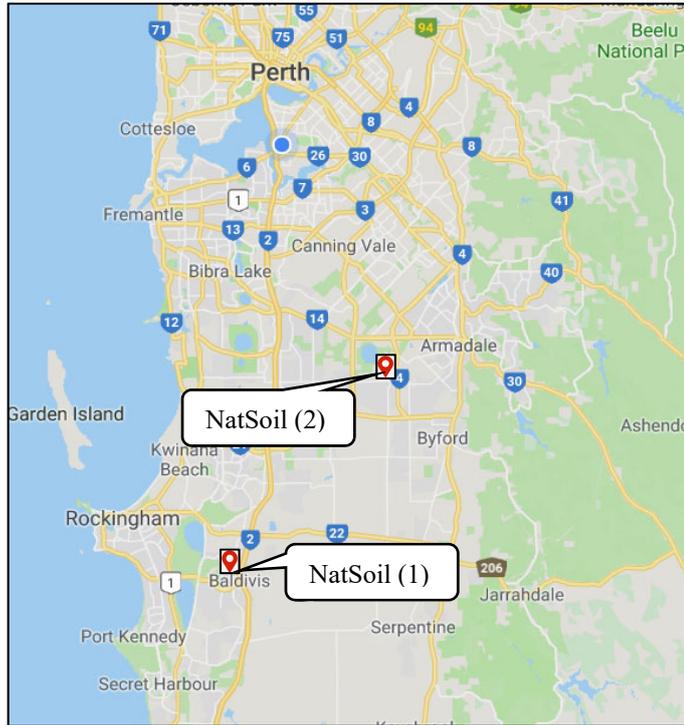


Figure 3.1: Locations of natural clays specimens: (a) NatSoil (1); and (b) NatSoil (2).

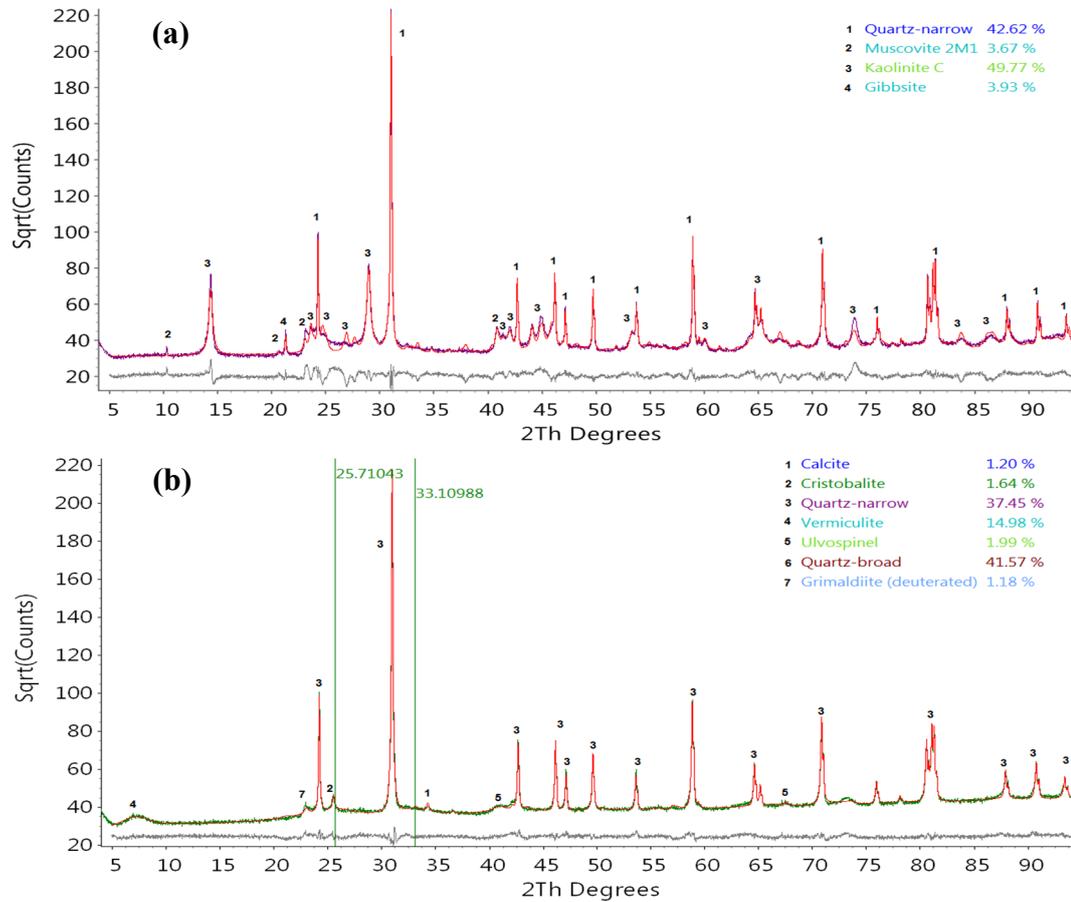


Figure 3.2: XRD analysis of natural clays: (a) NatSoil (1); and (b) NatSoil (2).

3.2.1.3 Soil properties

Representative soil specimens were prepared and tested to determine particle size distribution, Atterberg limits and soil pH for the purpose of soil classification. The compaction properties of the soils used were also determined through standard Procter compaction test.

3.2.1.3.1 Particle size distribution tests

Particle size distribution tests are performed for clay soils. The particle size distribution of the soil particles higher than 75 μm was determined using sieve analysis, while hydrometer analysis was conducted to determine the finer particle sizes less than 75 μm . Sieve and hydrometer tests were conducted as per the Australian Standards AS 1289.3.6.1 (2009b) and AS 1289.3.6.3 (2003b), respectively. Typical particle size distributions conducted for the engineered and the two natural soils are presented in Figure 3.3.

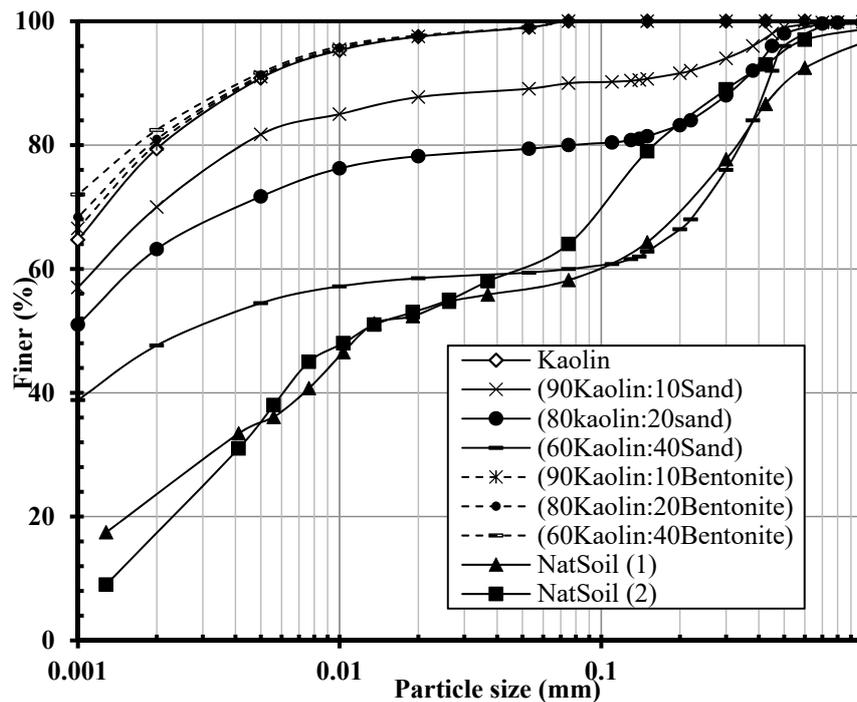


Figure 3.3: Particle size distribution of engineered and natural soils.

3.2.1.3.2 Atterberg limits

The Atterberg limits, including liquid limit (LL) and plastic limit (PL), are essential indices to quantify the response of clay to moisture. LL and PL were measured for the clay soils following the Australian Standards AS 1289.3.1.1 (2015) and AS 1289.3.2.1 (2009a), respectively. The plasticity index (PI), which is the numerical difference

between LL and PL, was determined to characterise the plasticity of the clay soils. The Atterberg limits and plasticity index of the engineered and natural clays selected for this study are listed in Table 3.2.

3.2.1.3.3 pH tests

The pH of the soils used was determined following the Australian Standards AS 1289.4.3.1 (1997) in which a 30 g of the untreated soil was sieved through 0.36 mm sieve and then stirred with 75 mL distilled water to form a suspension that was cured for 1 hr before the test. After 1 hour of curing, the pH was then determined using an electronic pH metre. The pH of engineered and natural clays selected for this study are listed in Table 3.2.

3.2.1.3.4 Standard Proctor compaction tests

The optimum moisture content (OMC) of soil is the moisture content at which the soil exhibits maximum dry unit weight (MDU), which is usually correlated with the maximum soil strength. Compaction tests were performed for all soil types used for the determination of OMC and MDU. All compaction tests were performed according to the Australian Standards AS 1289.5.1.1 (2003a). The compaction parameters of the engineered and natural clays selected for this study are listed in Table 3.2.

3.2.1.4 Soil classification

Using the results of some of the above tests (i.e. particle size distribution and Atterberg limits), the soils were classified using the Unified Soil Classification System (USCS). Table 3.2 summarises the classification of each soil used in this research. According to the Unified Soil Classification System (USCS), the kaolin and kaolin-bentonite samples are classified as high-plasticity (CH) clays, whereas kaolin-sand samples are classified as low plasticity clay. In terms of natural soils, NatSoil (1) and NatSoil (2) are classified as low and high-plasticity clays, (CL) and (CH), respectively (see Table 3.2). For further classification, the swelling potential of the soils used when interacting with water were investigated to determine the soil problematic nature (i.e. the degree of swelling). Several criteria and tests have been developed in the literature to measure the swelling potential of soil. Among the criteria found in the literature, Skempton (1953) proposed a correlation between the swelling and activity index; the amount of swelling in soils generally increases with an increase in its activity index. The activity index is the ratio of the plasticity index to the percentage of clay fraction

less than 2 μm . Based on Skempton's criteria, the proposed relation between the activity index and the degree of swelling (low or high) are: (0-0.75 = low), (0.75-1.25 = medium) and (1.25 or more = high). Free swell is defined as the increase in the volume of soil from a loose dry state when it is poured into water (Head et al. 2006). Among several laboratory test-based procedures proposed to estimate the potential swelling of the soil, the free swell test is usually conducted due to its simplicity. Based on the Indian Standards 2720 (1977), the test consists of pouring slowly 10 g of oven-dried soil passing 425 μm sieve into 100 cm^3 measuring jars containing distilled water and kerosene, respectively, for a minimum period of 24 hrs, then measuring the final volume of sediments. The free swell index is then determined using the following equation:

$$FSI (\%) = \frac{v_d - v_k}{v_k} \quad (3.1)$$

where v_d and v_k are the volumes of sediments in cylinders containing water and kerosene, respectively. Using Eq. (3.1), the swell potential is then classified as per the guidance proposed by Indian Standards 1498 (1970); (<50 = low), (50-100 = Medium), (100-200) = High) and (>200 = Very High). Indian standards 2720 (1977) was used to determine the swelling potential of the clays. Based on test results (Table 3.2), 60Kaolin:40bentonite and Natsoil (2) were classified as expansive clays, whereas kaolin, kaolin-sand group, 80Kaolin:20bentonite, 90Kaolin:100bentonite and NatSoil (1) were classified as non-expansive clays (see Table 3.2).

3.2.1.5 Soil pulverisation

Unlike engineered clays, which are originated in a powder form, the natural clays require pulverisation before treatment to allow for an adequate mixing with the binder, ensuring an effective treatment. According to Portland Cement Association (2001), when the soil is stabilised with OPC, it must be pulverised to 80% passing 4.75 mm and 100% passing 25 mm sieve. To the authors' best knowledge, no literature exists on the recommended pulverisation level for geopolymer-treated soils, and thus the effect of pulverisation level (i.e. high, medium and low) on geopolymer treatment of the two natural clays used in this research is considered. For high pulverisation, 100% of dried clay passing 4.75 mm sieve, whereas 80% and 60% of dried clay passing 4.75 mm sieve for medium and low pulverisation, respectively, with 100% each passing 9.51 mm sieve. In this study, the soil is pulverised by hand using a mortar and pestle.

Table 3.2: Characteristics and classification of soils.

Clay property	Engineered soil						Natural soil		
	100Kaolin	(90Kaolin:10Bentonite)	(80Kaolin:20Bentonite)	(60Kaolin:40Bentonite)	(90Kaolin:10Sand)	(80Kaolin:20Sand)	(60Kaolin:40Sand)	NatSoil (1)	NatSoil (2)
Liquid limit (%)	53	61	87.5	139	48	45	39	42	57
Plastic limit (%)	27	32	34.3	40.2	24	22.8	20	23.4	25
PI (%)	26	29	53.2	98.8	24	22.8	19	18.8	31
Maximum dry unit weight (kN/m ³)	14.95	13.85	13.65	13.19	15.09	15.67	17.3	16.28	14.88
Optimum moisture content (%)	25.2	25.3	25.7	26.0	24.0	22.0	16.0	19.7	22.3
Passing sieve No. 200 (75 μm) (%)	99.0	96.9	94.8	90.6	89.1	79.2	59.4	58.2	64.1
Clay fraction (%) < 2 μm	79.4	78.8	78.0	77.1	73	72	65	24	17
Activity index (A)	0.33	0.37	0.68	1.28	0.33	0.32	0.29	0.78	1.82
Free swell index, FSI (%)	2.9	31	73	165	2	0	0	45	110
Degree of swelling*	Low	Low	Low-	High	Low	Low	Low	Medium	High
Loss of ignition (%)	–	–	–	–	–	–	–	5.90	5.06
Soil pH	7.8	9.02	9.15	9.3	7.58	7.37	7.28	4.37	8.5
Soil classification (USCS)	CH	CH	CH	CH	CL	CL	CL	CL	CH

3.2.2 Geopolymer ingredients

The geopolymer used in this study comprises a mixture of fly-ash, ground granulated blast furnace slag (GGBFS) and sodium-based activator. The details of these materials are listed below.

3.2.2.1 Fly ash and GGBFS

Low calcium fly-ash (Class F, Australian Standard AS 3582.1, 1998), produced by *Cement Australian Limited* from Gladstone Power Station, was used. This type of fly-ash is rich in aluminosilicate material and is one of the most commonly used ingredients of geopolymers (Hardjito 2005; Garcia-Lodeiro et al. 2014a). GGBFS, supplied by Independent Cement & Lime Limited, was used to enhance the performance of the fly-ash based geopolymer after activation at ambient temperature (Yip et al. 2005; Nath et al. 2014). The particle size distribution and the chemical compositions of the fly-ash and GGBFS used are provided in Figure 3.4 and Table 3.3, respectively.

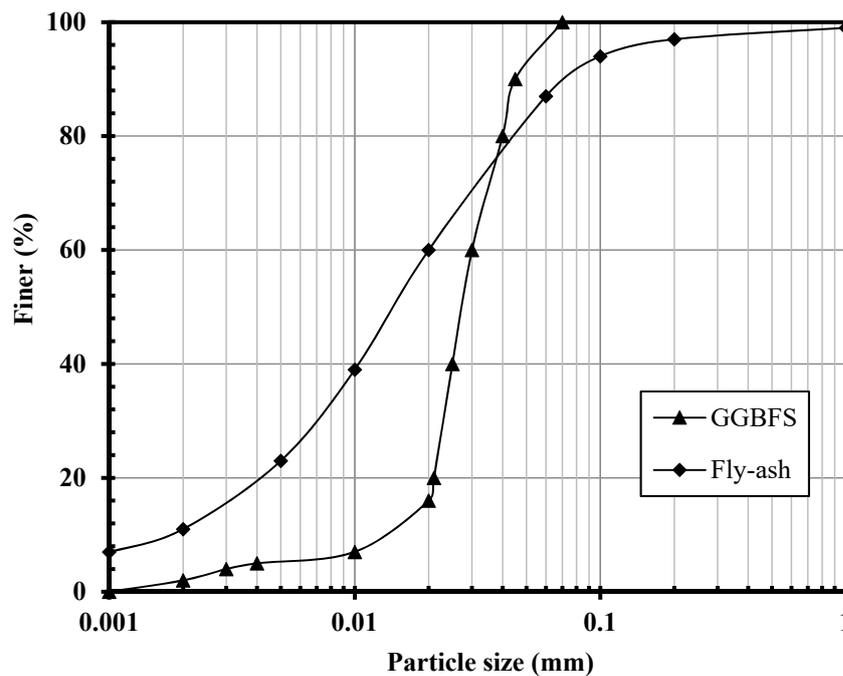


Figure 3.4: Particle size distribution of fly-ash and GGBFS.

Table 3.3: Chemical compositions of materials used, as provided by suppliers.

Material	Chemical composition (%)								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	LOI
Fly-ash	51.11	25.56	12.48	4.30	1.45	0.70	0.77	0.24	0.57
GGBFS	29.96	12.25	0.52	45.45	5.99	0.38	0.31	3.62	2.39

3.2.2.2 Alkaline activator

In this study, an alkaline activator comprising of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) was used, as suggested by many researchers (e.g. Verdolotti et al. 2008; Cristelo et al. 2013; Sargent et al. 2013; Phummiphan et al. 2016; Rios et al. 2016b). The sodium silicate was supplied by PQ Australia and contains 14.7% Na_2O and a mass ratio ($\text{SiO}_2/\text{Na}_2\text{O}$) of 2. The sodium hydroxide, originally in pellet form, was dissolved in deionised water to a concentration of 14 molar for at least 24 hours before mixing with the sodium silicate. The weight ratio of sodium silicate to sodium hydroxide was 2.33, which is required to maximise the reaction phase in the geopolymer, as recommended by Hardjito (2005).

3.3 SPECIMEN PREPARATION

A range of different specimen sizes and preparation methods are required for the various experimental testing conducted, but in all cases, the procedure of preparing geopolymer-soil mixture and compaction remains the same. This is detailed here, while additional details specific to particular specimen sizes, preparation methods and tests are detailed subsequently, with a final summary of the overall specimen details provided towards the end of the chapter.

3.3.1 Geopolymer admixtures

To prepare the geopolymer-soil mixtures, the fly-ash and the GGBFS were first added in different combinations as a partial replacement (by weight) of pulverised (natural) or powered (engineered) dry clay to form the total dry material in the mixture. Hobart mixer was then used to initially mix the dry material for three minutes at low speed. The pre-mixed activator solution and free water were then gradually added to the dry components and the mixing of different ingredients continued for an additional 3-5 minutes. In this investigation, the concentrations of different geopolymer components (i.e. fly-ash, GGBFS and activator) were varied within the mixture to allow the investigation for the effects of the different additives on the treatment and the determination of a practical combination. The volume of free water was also varied to allow for the investigation of optimum compaction parameters within the treated mixture. As mentioned above, the details of the mixtures used will be discussed in detail, subsequently.

3.3.2 Compaction and curing

To fabricate the specimens, the treated and pre-mixed clays are manually compacted in split cylindrical moulds in layers of controlled weight/thickness to replicate similar MDU obtained from the standard Proctor compaction tests. Compaction tests were performed for all treated clays for the determination of optimum compaction properties (i.e. MDU and OMC). All compaction tests were performed according to the Australian Standards AS 1289.5.1.1 (2003a). Care was taken to complete the sampling within 30 minutes after mixing to avoid any interaction due to stabiliser hardening. Specimens were then removed from split moulds, wrapped in cling film and left to cure at 60% humidity and 20-25°C temperature. In the following sections, details of different experimental tests, mixtures, specimen sizes and curing periods are presented.

3.4 EXPERIMENTAL METHODS

The experimental work of the current research was carried out using a soil testing facility in the Geomechanics and Pavement laboratory in the Department of Civil Engineering at Curtin University. The experimental testing regime used in this research includes unconfined compressive strength (UCS), durability, permeability and triaxial testing. Performing these tests in addition to Atterberg limits, pH tests and compaction test is crucial to evaluate the soil enhancement and to characterise the engineering properties of different clay soils treated with geopolymer. Atterberg limits and pH tests were determined for each treated soil prepared as in Section 3.3, following the methods described in Section 3.2.1.3. For Atterberg limits, the tests were conducted using materials collected from compaction specimens prepared at MDU and OMC properties. Individual pieces of each specimen were cured for 7 and 28 days before being ground to powder. All prepared specimens were then air-dried and tested to determine the liquid and plastic limits. For pH tests, materials collected from compaction specimens prepared to MDU and OMC cured for 1 hour, 7 days and 28 days, were used. At designated curing time, a 30 g sample of the compacted material (i.e. treated soil) was grounded to powder, sieved through 2.36 mm sieve and stirred with 75 mL distilled water to form a slurry. After 1 hour of curing, the pH was then determined for the slurry using an electronic pH. More details of the testing procedures, key features of testing machines, sample preparation and the prescribed testing program are presented in the following sections.

3.4.1 Unconfined Compressive Strength (UCS) tests

To investigate the strength development of geopolymer-treated clays, a series of unconfined compressive strength (UCS) tests were performed on treated specimens, prepared as in Section 3.3 and cured for 7, 28 and 90 days, as well as untreated specimens compacted at optimum moisture content. The specimens used for the UCS tests were prepared using cylindrical polyvinyl chloride (PVC) split tubes of 42 mm in diameter and 84 mm in height so that an aspect ratio of height-to-diameter of 2:1 was attained.

In this study, a universal testing machine (UTM-14P) manufactured by IPC was used to test all treated and untreated clay specimens for UCS. The UTM-14P is a servo-pneumatically testing machine with a loading capacity of 14 kN which is equipped with Control Data Acquisition System (CDAS) and IPC-UTS software. Utilising UTM-14P, the applied load and resulting displacements were measured using a load cell and a Linear Variable Displacement Transducer (LVDT), respectively. All UCS tests were performed according to the Australian Standards AS 5101.4 (2008) using a strain rate of 1% per minute. Two specimens for each curing period were tested with the average result considered. When the variation between the UCS results of the two specimens was significant ($> 20\%$), a third specimen was tested to establish the accuracy with the outlier discarded.

3.4.2 Durability tests

In tropical geographical zones where extreme weather conditions occur, soil durability is a key concern as wetting-drying or freezing-thawing damage may result in degradation of the engineering properties of the treated soils. Typically, a soil is assessed for its propensity to resist such damage through the use of wetting-drying, freezing-thawing and leachate testing (as discussed briefly in Chapter 2). Details of the testing procedures are described in the following sections.

3.4.2.1 Wetting-drying test

Extensive testing for the wetting-drying cycles was conducted for different treated clays. The wetting-drying durability tests were conducted on treated specimens, prepared as in Section 3.3, as well as untreated specimens, in accordance with ASTM D559-03 (2003). After 7 days of curing, specimens with dimensions of 105 mm in diameter and 115.5 mm in height were immersed in water for 5 hours to commence the first wetting phase and were then oven-dried at 70 °C for 43 hours. Specimen dimensions were determined after

each wetting-drying cycle to determine the corresponding volumetric change; a durable specimen is defined as one that experiences less than 10% volumetric change over the test period (Pedarla et al. 2011). This 48-hour process, representing one wetting-drying cycle, was repeated 12 times. To assess the residual strength of soil, additional UCS tests were then performed on soil specimens after 3, 6, 9 and 12 cycles. It should be noted that UCS testing is not a typical procedure for durability evaluation; however, it provides a parameter to quantify the degradation experienced by the treated specimens (Horpibulsuk et al. 2015). UCS testing directly on oven-dry specimens would give an unrealistic indication of specimen strength as such a moisture content is not representative of the in-situ conditions. Therefore, the specimens were submerged in water for 1 hour and were allowed to air dry for 15 minutes before UCS testing.

3.4.2.2 Freezing-thawing test

The freezing-thawing durability tests were conducted for treated and untreated clay in accordance with ASTM D560-15 (2015). Freezing was performed for the treated specimens (105 mm in diameter and 115.5 mm in height) at a temperature equal to -18°C for 24 hrs. Following the freezing stage, the specimens were placed in a moist room of a temperature of 21°C and relative humidity of 100% for 24 hrs. As per the ASTM standards, care was taken to provide water-saturated felt pads between the specimens and the container in the thawing stage. Similar to the wetting-drying durability test, the freeze-thaw procedure was designed to continue for 12 cycles. In this research, a successful durability performance in treated samples was characterised by attaining the 12 wetting-drying durability cycles with volume changes of less than 10%. To investigate the residual strength under the effect of freezing-thawing, a set of 2 specimens of 42 mm in diameter and 84 mm in height with an aspect ratio of height-to-diameter = 2:1 were examined for UCS tests after completing 3, 6, 9 and 12 durability cycles. For all freeze-thaw samples, the UCS tests were directly performed after thawing.

3.4.2.3 Leaching test

The leaching tests were carried out using a triaxial cell based on the procedure recommended by McCallister et al. (1992). The cell was assembled to hold a sample for height-to-diameter of 2:1. A rubber membrane was used to hydraulically isolate the specimen. Pressurised water was then leached into the sample through a porous stone from the bottom to ensure full sample saturation. Another porous stone was used on top of the sample to prevent solids from washing-out. Sufficient leachate for one cycle was

achieved in 24 hrs, which is equivalent to leaching out a collection of one pore volume change presented in the compacted soil samples. A number of 3, 6 and 9 cycles of leaching were conducted to monitor the strength performance and pH values of the geopolymer-stabilised soil. After the designated number of leaching cycles, samples were tested for the UCS residual strength determination and were then broken down and tested for the pH determination. The pH determination was carried out in accordance with the Australian Standards AS 1289.4.3.1 (1997) as outlined previously in Section 3.4. The pH value was measured for the soil suspension using an electronic pH metre, and a comparison between the pH values before and after leaching was then possible.

3.4.3 Permeability test

To investigate the permeability of geopolymer-treated clays, a series of permeability tests were performed on treated specimens, prepared as in Section 3.3 and cured for 28 days, as well as untreated specimens compacted at optimum compaction parameters. The permeability tests were based on the falling-head method following Australian Standards (2001). This method is recommended for fine-grained soils (e.g. clay) where the coefficient of permeability, k , is expected to be low (i.e. less than 10^{-4} m/sec) (Head et al. 1994); k is the measure of the capacity of soil with which the water can easily flow through it.

The permeability apparatus consists of two main components, namely, the cell and a permeability standpipe panel as per the Australian Standards (Standards Australia 2001). After designated curing time, the permeability cell including a compacted specimen of 110 mm diameter and 101 mm height was connected to the standpipe, and the valve at the top of the cell was opened. The outflow was then observed. After ensuring continuous flow, the value of the coefficient of permeability was determined as follows (Standards Australia 2001):

$$k_{\theta} = \frac{2.3 * a * L}{(6 * 10^4) * A * t} * \log_{10} \left[\frac{h_i}{h_f} \right] \quad (3.1)$$

where k_{θ} is the coefficient of permeability at test temperature θ ; a is the cross-sectional area of the standpipe; L is the length of the specimen; A is the cross-sectional area of the specimen; h_i is the initial height in the standpipe (in mm) (at $t = 0.0$); h_f is the final height in the standpipe after time interval t .

3.4.4 Microstructure analysis

The use of scanning electron microscopy (SEM) has been used widely in the soil stabilisation literature to provide insights into tracking cementitious growth induced by the stabiliser and explaining the mechanism of enhancement within treated soils. Such techniques were also employed in this study to examine the soil fabric before and after stabilisation to detect changes caused by the stabiliser after treatment. This was performed using a fully PC controlled MIRA3 XMU equipment at the John de Laeter Centre, Curtin University. The tests were conducted using fragment materials collected from UCS specimens after testing. It should be noted that the SEM study allows only a small, localised area of the untreated and treated specimen to be examined (unlike engineering laboratory specimens). However, it is assumed to be representative of the reaction process of treated specimens (Solanki et al. 2012).

3.4.5 Triaxial testing

One of the main aims of this study is to characterise the undrained shear behaviour of clay under different loading conditions. Accordingly, a series of static triaxial tests were conducted on untreated and geopolymer-treated clays. Details of the triaxial testing apparatus and procedures are given here.

3.4.5.1 GCTS STX-300 triaxial apparatus

All triaxial tests were conducted using the GCTS STX-300 stress path/static-cyclic triaxial apparatus available at Curtin University, Figure 3.5. The GCTS STX-300 is an electronic-hydraulic testing system with direct servo control of axial actuator, cell pressure and back pressure (GCTS 2007). The system consists of a loading frame, triaxial cell, PCP-2000 cabinet and GCTS CATS system. The loading frame has a capacity of ± 100 kN static loading and ± 80 kN dynamic loading of a maximum frequency of 50 Hertz. The triaxial cell capacity is 2000 kPa with a cell pressure applied through a computer-controlled pressure controlled panel PCP-2000. The PCP-2000 cabinet operates under pressure generated by a hydraulic pump. The PCP-2000 cabinet is also equipped with two actuators for supplying cell pressure and back pressure each with a pressure transducer, linear variable differential transducer and servo valve.

The triaxial cell is shown in Figure 3.5, which consists of three main components, namely the cell base, an acrylic plastic cylinder (2000 kPa capacity) and top head assembly. The cell base consists of a pedestal for the setup of the sample with multi drainage connections

(two for the back pressure and one for the cell pressure), and one electrical connection for the pore water pressure measurement. The acrylic plastic cylinder encloses the pressurised water. The cell and back pressures system consist of a digital system actuator with a pressure capacity of 2000 kPa. Pressures are measured using pressure transducers of high accuracy. Axial displacement is measured via a linear variable differential transformer (LVDT).

Test control within GCTS STX-300 was performed using GCTS CATS system, which is composed of a GCTS SCON-1500 Digital System Controller and a graphical user interface (i.e. GCTS CATS software) that runs on a computer (Figure 3.5). The GCTS SCON-1500 Digital System Controller consists of a digital servo controller and data acquisition system. The software handles the database aspects of the system to control soil saturation, consolidation and shearing. The program allows different stress paths to be applied with graphical output of simultaneous plots describing different stress-strain, volumetric and pore water pressure responses during tests.

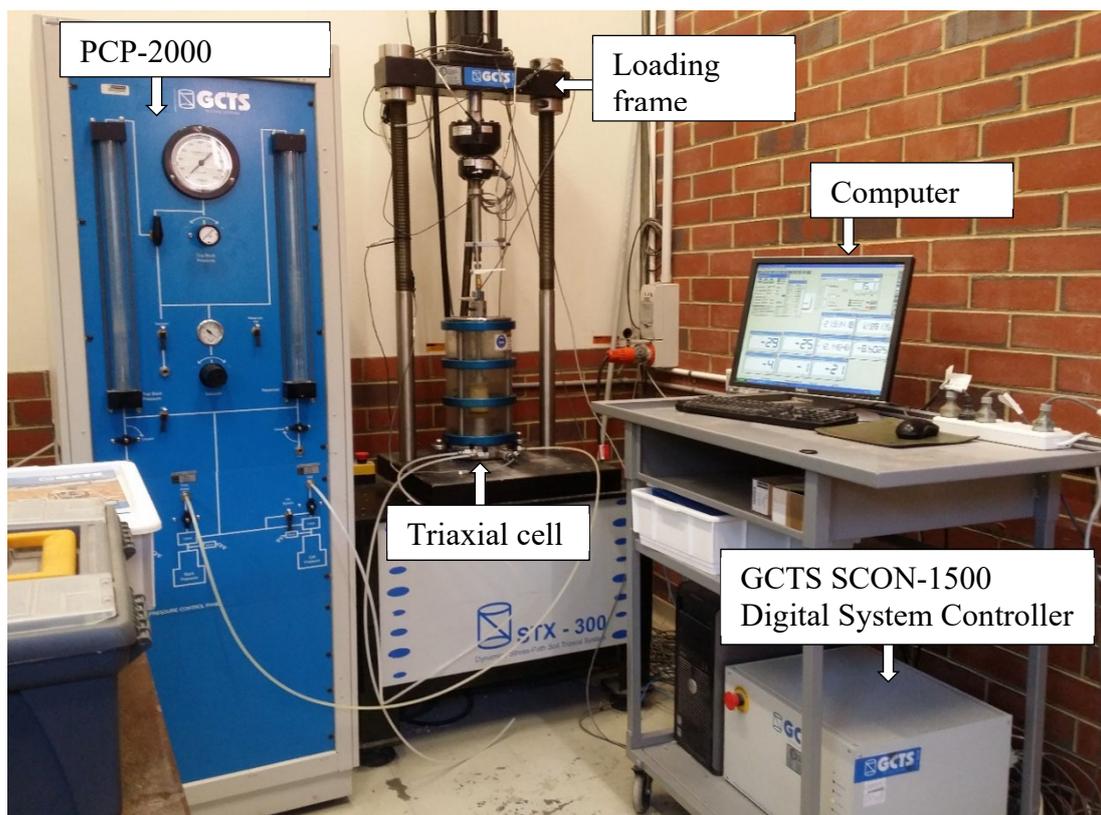


Figure 3.5: STX-300 triaxial setup

3.4.5.2 Specimen set-up

All triaxial tests were performed on 35 mm diameter, 2:1 aspect ratio specimens. Before starting the tests, all drainage lines were flushed with de-aired water to avoid any air inside the system during testing. The sample was set on the porous base of the triaxial apparatus and the top cap was then installed. Filter paper discs were placed between the build-in porous stones and specimen ends to avoid the inclusion of fine materials into the system. Filter paper strips were also used around the specimen to accelerate the saturation (Head et al. 2014). To this end, a rubber membrane was fixed to the pedestal and the top cap with the aid of two O-rings each side. The pressure cell of the apparatus was assembled and loading arm set to achieve full contact with the top cap of the specimen. The cell was then filled with tap water and a nominal cell pressure and deviator stress of 35 kPa and 5 kPa, respectively, were applied. A back pressure of 25 kPa was applied to the specimen.

3.4.5.3 Saturation

All untreated and treated soil specimens used for the triaxial tests were saturated via incremental backpressure up to 850 kPa, with the aid of side filter paper strips and de-aired water. The saturation technique was implemented by simultaneously increasing the cell pressure and the backpressure, while keeping the differential pressure constant at 10 kPa throughout the saturation process. The test was continued by increasing the cell pressure and the backpressure until the sample was fully saturated. The degree of saturation was estimated by computing Skempton's B -coefficient (Head et al. 2014). Usually, the specimen is considered saturated when the value of B is ≥ 0.95 . For geopolymer-treated specimens, achieving a B value higher than 0.91 was difficult even when higher backpressure was applied. However, such B value (i.e. 0.91) for cemented soil specimens indicates saturation, as mentioned by Head et al. (2014) at which the B value is more related to the rigidity of the specimen skeleton rather than the presence of air in the voids. The time for the saturation stage was monitored; on average, the total saturation for the treated specimens took place over a period of 12-24 hours. Once saturation was reached, the consolidation stage started.

3.4.5.4 Consolidation

In this study, the cell pressure was increased to increase the effective stress to the required level. Three isotropic consolidation confining pressures were used: 100 kPa, 200 kPa and 400 kPa. Assuming a coefficient of earth pressure at rest $K_o = 1$; these isotropic

consolidation pressures were selected to simulate expected average field effective stresses for soil elements located at 5, 10 and 20 m depth within a saturated soil mass of a unit weight of 20 kN/m³. The consolidation stage was terminated when the volume changes were constant. In general, a minimum period of 7 hours was necessary to allow for dissipation of the excess pore water pressure. By achieving the consolidation, the sample was then ready for the shearing stage.

3.4.5.5 Undrained Shearing

At the end of the consolidation phase, untreated and geopolymer-treated clay specimens underwent Consolidated Undrained (CU) triaxial compression tests according to Australian Standards AS 1289.6.4.2 (2016). The shear strain rate for all undrained axial compression tests was set to a constant rate of 0.0116 mm/min; the rate of the applied axial strain was sufficiently slow to allow for the pore water pressure to equalise for the entire specimen. To further allow for the evaluation of the effect of loading path on the undrained shearing behaviour of geopolymer-treated clay, a limited set of specimens were tested under constant- p loading stress path. For the constant- p loading stress path, the undrained tests were conducted under stress-controlled condition and the shear stress rates were set at +5 kPa/min (i.e. +300 kPa/hr) and -1.67 kPa/min (i.e. -100.2 kPa/hr) for the deviator stress and radial stress, respectively, so as to maintain constant total mean stress values of $p = 100, 200$ and 400 kPa. In all cases, undrained tests were allowed to continue until failure defined as axial strain $\geq 10\%$.

3.4.5.6 Drained shearing

To allow for the comparison of different drained/undrained conditions, a limited set of selected mixtures were tested under drained condition (CD). The CD tests were performed with a rate of loading of 0.007 mm/min. As in the undrained tests, the drained tests were allowed to continue until the failure criterion was reached. The failure criterion adopted for the drained tests was the same as the drained condition (i.e. axial strain $\geq 10\%$).

3.5 EXPERIMENTAL PROGRAM

The experimental investigation outlined in this thesis was conducted in three phases. Details of these phases are given in the following sections with an overview summary provided for each phase in a specific table.

3.5.1 Phase I – Optimum combination of geopolymer ingredients

The reactivity of geopolymer is governed largely by the concentration of different ingredients (i.e. fly-ash, GGBFS and activator). Therefore, initial testing (referred to as Phase I) was carried out to determine the optimum combination of geopolymer ingredients for the purpose of soil stabilisation at ambient temperature. In Phase I of the testing program, the UCS testing was selected as a suitability indicator of geopolymer-ingredients reactivity; higher UCS value indicates more effective geopolymer mixture. In this part of the investigation, kaolin clay was selected as a base clay and the effect of different additive contents (i.e. fly-ash, GGBFS and activator) was investigated for different specimens at different curing periods of 7, 28 and 90 days. The details of UCS tests and influenced variables are given in Table 3.4. Additional samples were prepared and stabilised by adding 3%, 6% and 9% OPC (denoted herein as OPC3, OPC6 and OPC9, respectively), as shown in Table 3.4. These samples were tested to provide a baseline and to compare with the other geopolymer-stabilised samples.

In this part of the study, SEM analysis for treated kaolin clay was conducted to investigate the general trends of the effects of geopolymer on the microstructure fabric of clay. The SEM was carried out on treated kaolin specimens having 20% geopolymer and cured for 7 and 28 days. SEM was also performed on samples in which only fly-ash and GGBFS were mixed with kaolin clay at the optimum moisture content without activator. Additional images were necessary to distinguish the different shapes of dry particles involved before mixing.

3.5.2 Phase II – Compaction, consistency, pH, UCS, durability and permeability tests

An extensive laboratory testing was undertaken in this phase of the testing program to evaluate the engineering properties over a range of clay soil types treated with geopolymer at the practical combination of geopolymer ingredients. Engineering properties determined throughout Phase II include compaction tests; Atterberge limits; pH testing; Unconfined Compressive Strength (UCS); durability through wetting-drying, freezing-thawing and leaching tests; and permeability tests. In this testing phase, the effect of geopolymer contents, curing periods and clay characteristics are investigated. The details of the tests variables are given in Table 3.5. It should be noted that the percentage of geopolymer in Table 3.5 represents the ratio of ash- GGBFS additive to the total weight of the dry clay and ash- GGBFS before activation.

3.5.3 Phase III – Triaxial tests

For a typical curing time of 28 days, 35 mm diameter specimens of selected treated clay types were tested for Consolidated Undrained (CU) compression triaxial tests (see Section 3.4.5). The tests were conducted according to Australian Standards AS 1289.6.4.2 (2016). To allow for the comparison of different drained/undrained conditions, a limited set of selected mixtures were tested under drained condition (CD). To allow for another comparison of different effective stress paths and loading types, another set of selected mixtures were tested at a constant- p loading condition. An outline of the CU, CD and constant- P tests is presented in Table 3.6.

Table 3.4: Details of Phase I of the testing program.

ash-GGBFS (%)	GGBFS/ ash (%)	activator/ ash-GGBFS (%)	OPC (%)	Curing periods (days)	
				UCS	SEM
0	0		–	0	1
10		0	–	7, 28	-
20	20		–	7, 28	1
30			–	7, 28	-
	0		–	7, 28, 90	-
10	10	40	–	7, 28, 90	-
	20		–	7, 28, 90	-
	0		–	7, 28, 90	-
20	10	40	–	7, 28, 90	-
	20		–	7, 28, 90	28
	0		–	7, 28, 90	-
30	10	40	–	7, 28, 90	-
	20		–	7, 28, 90	-
10			–	7, 28, 90	-
20	20	80	–	7, 28, 90	-
30			–	7, 28, 90	-
			3	7, 28, 90	-
0	0	0	6	7, 28, 90	-
			9	7, 28, 90	-

Table 3.5: Details of Phase II of the testing program.

Clay type	Geopolymer (%)	Curing periods (days or hrs)							
		Compaction tests	Atterberg limits	pH	durability			Permeability	UCS
					wetting-drying	Freezing-thawing	Leaching		
Kaolin	untreated	1 day	1 day	1hr	0 days	0 days	-	0 days	
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	7 days	-	28 days	0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	7 days	-	28 days	7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	7 days	-	28 days	7, 28, 90 days
(90Kaolin:10Bentonite)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
(80Kaolin:20Bentonite)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
(60Kaolin:40Bentonite)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
(90Kaolin:10Sand)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
(80Kaolin:20Sand)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
(60Kaolin:40Sand)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
NatSoil (1)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
NatsSoil (2)	untreated	1 day	1 day	1hr	0 days	-	-		7, 28, 90 days
	10	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		0 days
	15	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days
	20	Uncured	7, 28 days	1hr, (7&28 days)	7 days	-	-		7, 28, 90 days

Table 3.6: Details of Phase III of the testing program.

Clay type	Geopolymer (%)	Triaxial tests, 28 days	
		Pc' (kPa)	test type
Kaolin, NatSoil (1) and NatSoil (2)	Untreated	100	CU, Compression
		200	CU, Compression
		400	CU, Compression
Kaolin, NatSoil (1) and NatSoil (2)	10	100	CU, Compression
		200	CU, Compression
		400	CU, Compression
Kaolin, NatSoil (1) and NatSoil (2)	20	100	CU, Compression
		200	CU, Compression
		400	CU, Compression
Kaolin	20	100	CD, Compression
		200	CD, Compression
		400	CD, Compression
	20	100	CU, Constant- <i>p</i>
		200	CU, Constant- <i>p</i>
		400	CU, Constant- <i>p</i>

3.6 SUMMARY

This chapter has given details on sample preparation for clays treated with fly-ash based geopolymer. This chapter also has described in detail the test equipment and procedural stages followed in the testing program to determine the engineering properties for the geopolymer-clay mixtures. In the following chapters, the test results of different phases of the experimental program on geopolymer-treated clays are presented and discussed. The optimisation results through UCS and SEM testing, corresponding to Phase I of the testing program, are presented and discussed in Chapter 4. Chapter 5 presents the results and discussions regarding compaction, consistency, UCS, durability, permeability and pH testing on a variety of engineered and natural clays corresponding to Phase II of the testing program. Finally, Chapter 6 presents Phase III of the testing program with results and discussions related to the undrained shearing behaviour of treated clays.

CHAPTER 4

OPTIMISATION OF GEOPOLYMER MIXTURE- INCORPORATING GGBFS

4.1 INTRODUCTION

The effectiveness of fly-ash based geopolymer as a binder is affected by several factors, among which are the concentration of the activator and curing temperature. Soil stabilisation using geopolymers is only practical at ambient temperature, which requires higher alkaline conditions compared to curing at elevated temperature (as discussed in Chapter 2, Sections 2.4.1.4.5 and 2.5.1) and this increases the cost of stabilisation. This has led to the potential of incorporating calcium-based components within geopolymers, such as granulated ground blast furnace slag, GGBFS, a waste product from the steel industry, to enhance the effectiveness of geopolymers for soil stabilisation and to reduce the alkaline conditions required for the ambient curing. However, as mentioned in the literature review, limited relevant research exists on the use of GGBFS for geopolymer clay stabilisation. Therefore, the main aim of this chapter is to optimise the geopolymer mixture, including GGBFS content through an experimental laboratory testing program. Kaolin clay (i.e. rich with kaolinite minerals) was selected as a base clay for this investigation, due to its controlled characteristics with respect to the nature of minerals and organic content. According to Phase I of the experimental methodology described in Chapter 3, the Unconfined Compressive Strength (UCS) test was utilised as an indicator for investigating the reactivity of different geopolymer ingredients including GGBFS within treated clay at different curing periods of up to 90 days. Using UCS test results, the role of GGBFS in enhancing the strength performance of the geopolymer-clay mixture at ambient temperature was examined. The strength performance of the geopolymer-treated clay was then compared with the OPC-treated clay performance. Images captured using Scanning Electron Microscopy (SEM) technique have been analysed for a typically treated clay mixture to increase the understanding of how the structure of the geopolymer-clay mixture contributes to its enhanced strength characteristics.

4.2 TEST CONDITIONS AND VARIABLES

Specimens of kaolin clay were mixed with geopolymer components, i.e. fly-ash, GGBFS and activator, in different combinations to assess their respective impacts. The various total quantities of fly-ash and GGBFS mixtures (referred to as ash- GGBFS) were 0%, 10%, 20% and 30% by weight of the total weight of the dry clay and additive. The weight ratio of GGBFS for each quantity of ash-GGBFS content (referred to as GGBFS/ash-GGBFS) was varied as 0%, 10% and 20% in the mixtures. For each set of mixtures, two weight ratios of activator to ash-GGBFS additive (referred to as activator/ash-GGBFS), of 40% and 80% were considered, comprising a total of 12 mixtures tested. Additionally, untreated specimens prepared only with (clay and water), (clay, ash-GGBFS and water) or (clay, ordinary Portland cement OPC and water) were also adapted to act as controls, allowing comparison with the geopolymer-stabilised samples. The optimum moisture content (OMC) and the maximum dry unit weight (MDU) of kaolin clay specimen, stabilised with various additives and various quantities, are also presented in Table 4.1.

Table 4.1: Mix proportions and compaction properties of different geopolymer and OPC-soil mixtures.

ash-GGBFS (%)	GGBFS/(ash-GGBFS) (%)	activator/(ash-GGBFS) (%)	OPC (%)	MDU (kN/m ³)	OMC (%)
0	0		–	14.95	25.2
10		0	–	15.06	24.6
20	20		–	15.34	22.8
30			–	15.62	21.4
	0		–	15.14	23.4
10	10	40	–	15.07	23.7
	20		–	15.04	24.0
	0		–	15.61	20.3
20	10	40	–	15.47	20.5
	20		–	15.45	20.6
	0		–	16.67	17.3
30	10	40	–	16.64	17.2
	20		–	16.49	17.6
10			–	15.10	23.4
20	20	80	–	15.65	19.0
30			–	17.53	16.8
			3	14.60	25.1
0	0	0	6	14.54	25.8
			9	14.50	26.4

4.3 UNCONFINED COMPRESSIVE STRENGTH OF GEOPOLYMER-TREATED KAOLIN CLAY

To allow for direct comparison of the strength development between various mixtures, a strength development index (SDI) was used as a non-dimensional parameter to reflect the effect of the controlling factors, i.e. ash-GGBFS content, GGBFS/ash-GGBFS ratio and activator/ash-GGBFS ratio, on the unconfined compressive strength of geopolymer-treated clay. The SDI is defined as the unconfined strength difference between treated and untreated clay divided by the strength of untreated clay (Uddin et al. 1997). The findings from this investigation are presented in the following sections.

4.3.1 Effect of fly-ash (zero GGBFS content)

The effect of fly-ash on the strength improvement of geopolymer-treated clay was examined for mixtures of various fly-ash contents of 0%, 10%, 20% and 30% at zero GGBFS concentration, i.e. GGBFS/(ash-GGBFS) of 0% and fixed activator/(ash-GGBFS) ratio of 0.4, after 7 and 28 days of curing, as depicted in Figure 4.1.

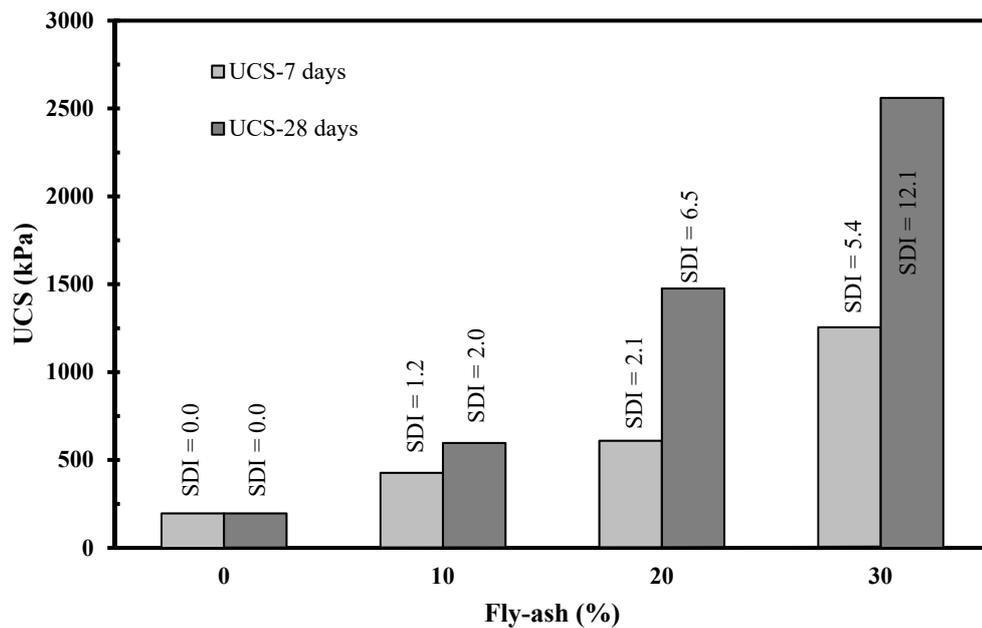


Figure 4.1: Unconfined strength (UCS) and strength development index (SDI) of fly-ash based geopolymer for different mixtures (ash-GGBFS = 0%, 10%, 20% and 30%, GGBFS/ash-GGBFS = 0 and activator/ash-GGBFS = 40%) and different curing time periods.

Irrespective of the curing time, it can be seen that the increase in fly-ash content (i.e. ash-GGBFS percentage at zero GGBFS content) enhances the UCS for the

geopolymer-treated clay. The strength, represented by the SDI value, was found to increase by 1.2 and 2.0 with only 10% fly-ash, for curing time of 7 and 28 days, respectively. When the fly-ash content was further increased to 20%, a higher increment in the SDI was detected up to 2.1 and 6.5 for curing time of 7 and 28 days, respectively. It is observed that the increase in the SDI is more pronounced at higher additive content and increased curing time. However, such findings were found to be similar to those reported in the geopolymer-soil literature (e.g. Cristelo et al. 2013; Sargent et al. 2013), (see Chapter 2, Section 2.4.1.4) which are mainly related to the formation of cementitious bonds through the formed N-A-S-H geopolymer product.

4.3.2 Effect of GGBFS

The effect of GGBFS addition on the strength of geopolymer-treated clay was examined for mixtures of various ash-GGBFS contents of 0%, 10%, 0% and 30%, and GGBFS/ash-GGBFS of 0%, 10% and 20% at fixed activator concentration, i.e. activator/ash-GGBFS = 40%, after 7 and 28 day curing, as depicted in Figure 4.2. Generally, partial replacement of fly-ash by GGBFS results in increased UCS and SDI values for the treated mixtures, and at both 7 and 28 curing days. For instance, with an ash-GGBFS content of 20% and 7 days of curing, the partial replacement of fly-ash by GGBFS by 10 % increases the SDI from 2.1 to 3.9 (Figure 4.2a), whereas a further increase of GGBFS replacement ratio from 10 % to 20 % causes an overall increase in the SDI of 6. For ash-GGBFS content of 20% at 28 days of curing, the SDI improvement was detected to be increased from 6.5 at zero GGBFS to 9.7 with GGBFS (Figure 4.2b). As discussed in the literature review, such an increase in the SDI response to the increase in GGBFS/ash-GGBFS replacement ratio is mainly related to the role of the C-A-S-H product resulted from the GGBFS activation within the fly-ash based geopolymer, which enhances the overall strength performance at ambient temperature (Granizo et al. 2002; Yip et al. 2005). It is evident from Figure 4.2(a and b) that the GGBFS works synergistically with the fly-ash, which suggests that when mixed together, an increase in the short-term strength performance of treated clay as well as the performance for the long-term occurs. This increases the feasibility of using geopolymer for soil stabilisation at ambient temperature curing. However, as the UCS of the mixtures increases with the GGBFS content, there is no evidence of an optimum ratio of GGBFS within the ash-GGBFS mixture; since the GGBFS is a waste material, GGBFS/ash-GGBFS maximum ratio of 20 % was selected for further investigation.

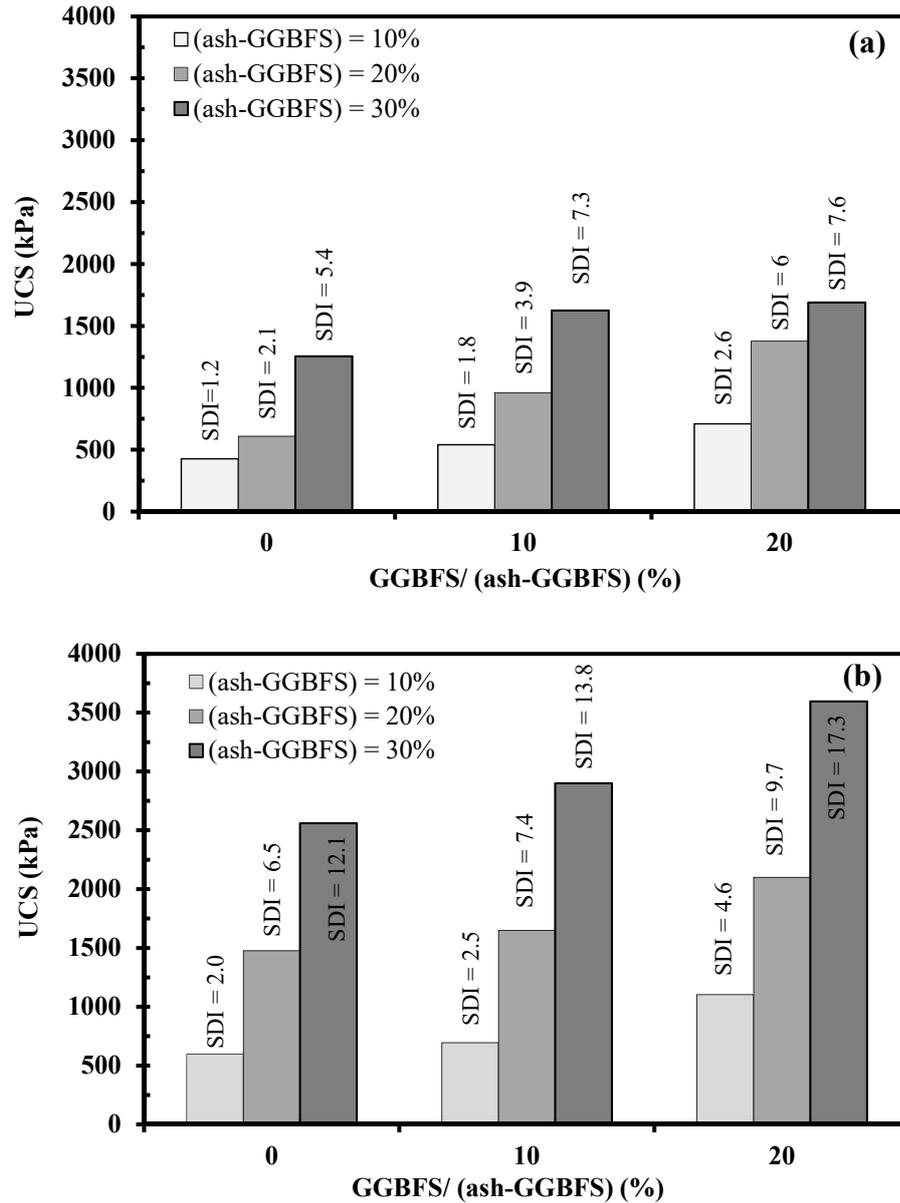


Figure 4.2: Effect of GGBFS addition on strength gain of fly-ash based geopolymer for different curing periods: (a) 7 days; and (b) 28 days.

4.3.3 Effect of activator

The effect of activator on the strength improvement of geopolymer-stabilised clay was investigated and the results presented in Figure 4.3. The results were for testing of different mixtures with a fixed GGBFS/ash-GGBFS ratio of 20% and various combinations of ash-GGBFS of 10%, 20% and 30%, activator/ash-GGBFS of 0%, 40% and 80% (selected based on trial mixtures). It can be seen that at zero activation (i.e. activator/ash-GGBFS = 0%), no UCS improvement was detected regardless of the ash-GGBFS content. This can be attributed to the weak pozzolanic reaction (i.e. self-

cementitious characteristics) of the ash-GGBFS additive. On the contrary, for the activator to ash-GGBFS proportion of 40 % and 80 %, a significant increase in the strength gain was detected for all mixtures and curing periods. As explained by Phummiphan et al. (2016), this can be attributed to the increase in leaching of the silica and alumina of the aluminosilicate material (i.e. fly-ash and GGBFS) due to the increase of pH, which in turn increases the formation of cementitious products, i.e. N-A-S-H and C-A-S-H between the clay particles, thereby strengthen the soil.

For the mixture with 10% ash-GGBFS, it can be seen that an increase in the activator/(ash-GGBFS) ratio above 40% did not enhance the UCS values at either 7 or 28 curing days, see Figure 4.3. This may be attributed to the fact that the fly-ash and GGBFS, which take part in the geopolymerisation reaction, are exhausted within 7 days of curing and this reduces the formation rate of cementitious products. It is worthwhile noting that a lack of strength enhancement at this stage may also confirm a low reactivity of kaolin minerals for any short-term reaction with the activator. However, with the increase of the total quantity of dry additives (i.e. ash-GGBFS) the strength at all ages increases with the increase of the activator content. For instance, at ash-GGBFS content of 20%, an increase of 12% in the UCS value was detected with the increase of activator ratio from 40% to 80% for 7 curing days (Figure 4.3a), whereas a higher increase of 47% in the UCS value was detected for the same increase of activator content at 28 curing days (Figure 4.3b). As stated earlier, this indicates an increase in the formation of cementitious products within the treated clay due to the increase of alkalinity (i.e. pH value) induced by the higher activator concentration in the mixture.

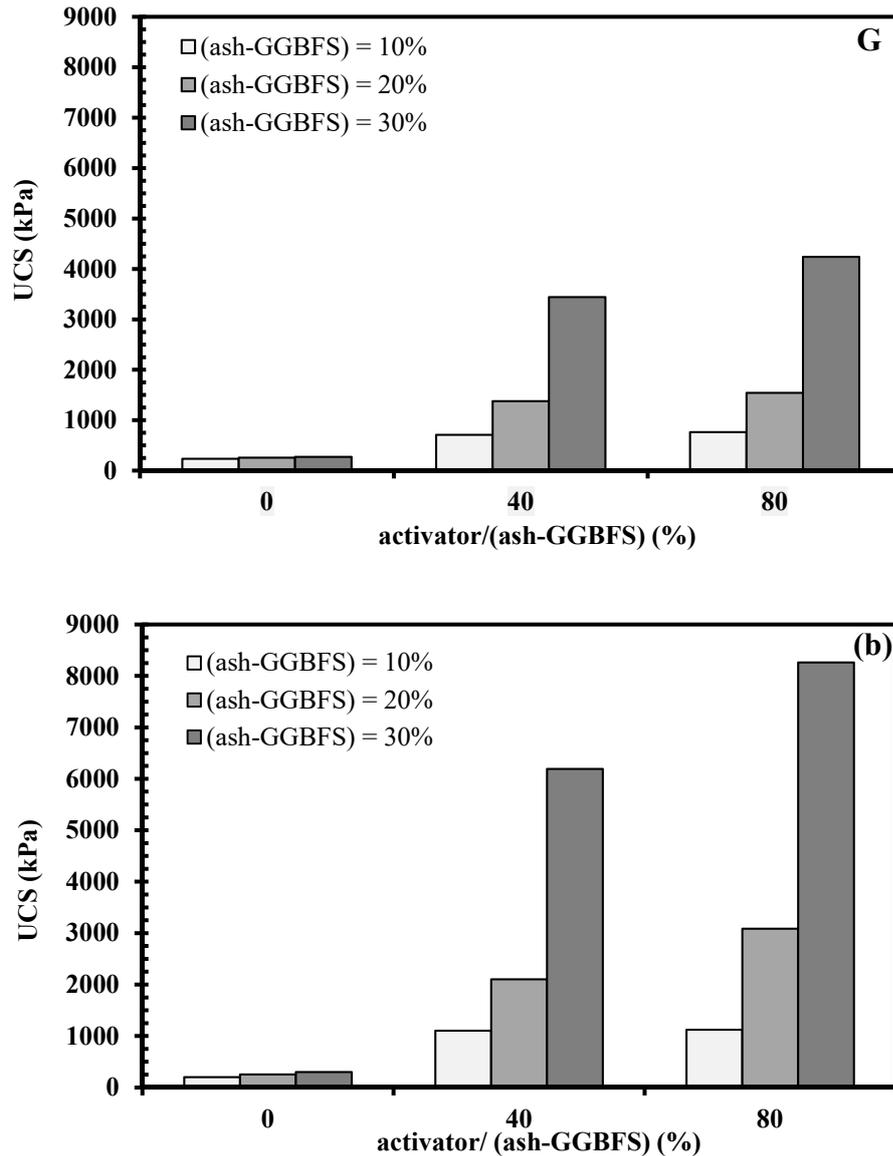


Figure 4.3: Effect of activator on strength performance of geopolymer-treated clay for different mixtures (ash-GGBFS = 10, 20 and 30%, and GGBFS/ash-GGBFS = 20%) and curing time: (a) 7 days; and (b) 28 days.

4.3.4 Strength comparison of geopolymer-treated clay with OPC treated clay

Two mixtures investigated in the current study containing of 10% and 20% of ash-GGBFS, GGBFS/ash-GGBFS of 20% and activator/(ash-GGBFS) of 40%, were found to give a strength range between 1000–2000 kPa at a typical curing period of 28 days. The strength performance of these two mixtures may fulfil the minimum requirements of most ground improvement applications, e.g. subbase or subgrade course in road construction (USACE 1984), and thus are compared with the strength performance of

the traditional OPC treated soil. Figure 4.4 shows the results of such a comparison in terms of the SDI and the corresponding curing time up to 90 days.

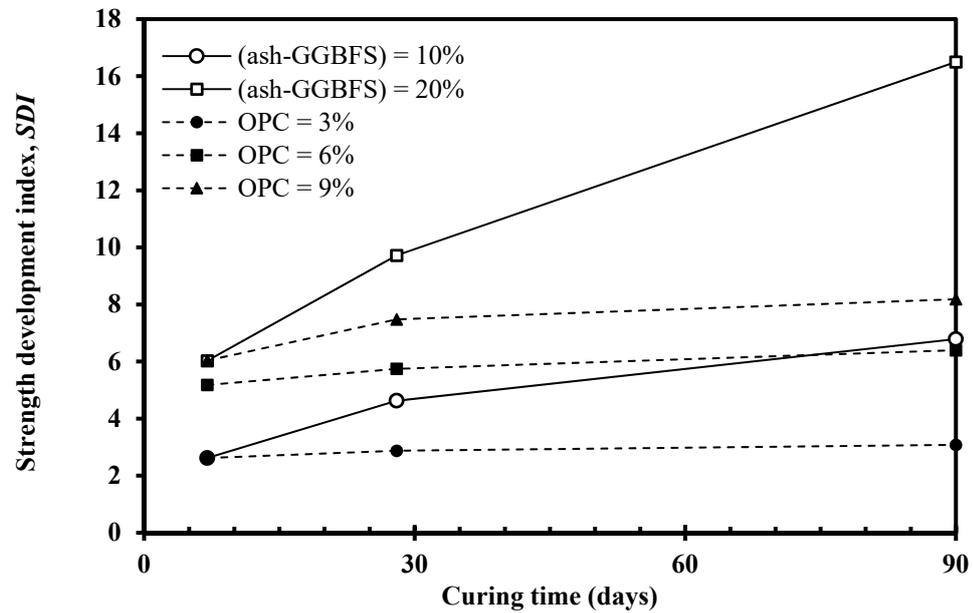


Figure 4.4: Strength development index of geopolymer-stabilised clay samples against OPC treated clay samples.

It can be seen that at 7 days curing, the strength of clay treated with 10% content of ash-GGBFS is equivalent to that of 3% OPC content with an SDI of 2.6, whereas the strength of the mixture treated with 20% content of additive is comparable to 9% OPC content with an SDI of 6.0. At 28 and 90 curing days, the strength of geopolymer-stabilised clay mixture at (ash-GGBFS) = 10% is almost equivalent to that of OPC = 6%. Interestingly, for the mixture with 20% ash-GGBFS additive, a superior SDI values were detected compared to all OPC treated mixtures, confirming a high rate of strength development in the geopolymer-stabilised clay mixture.

Figure 4.5(a and b) show a comparison between the UCS stress-strain curves for (ash-GGBFS) = 20% and OPC = 9% mixtures cured at 7 and 28 days, respectively. Both geopolymer and OPC treated clay mixtures show similar qualitative strength response (i.e. brittle stress-strain behaviour) at all curing periods. However, quantitative differences in their response (i.e. values peak strength and stiffness) were detected within the treated clay, especially at 28 days. Such quantitative differences may be attributed to the higher rate of geopolymerisation in the geopolymer-stabilised clay compared to the hydration reaction in OPC treated clay. In other words, the rate of

formation of cementitious products in the fly-ash based geopolymer-treated clay incorporating GGBFS is higher than that of the OPC-treated clay.

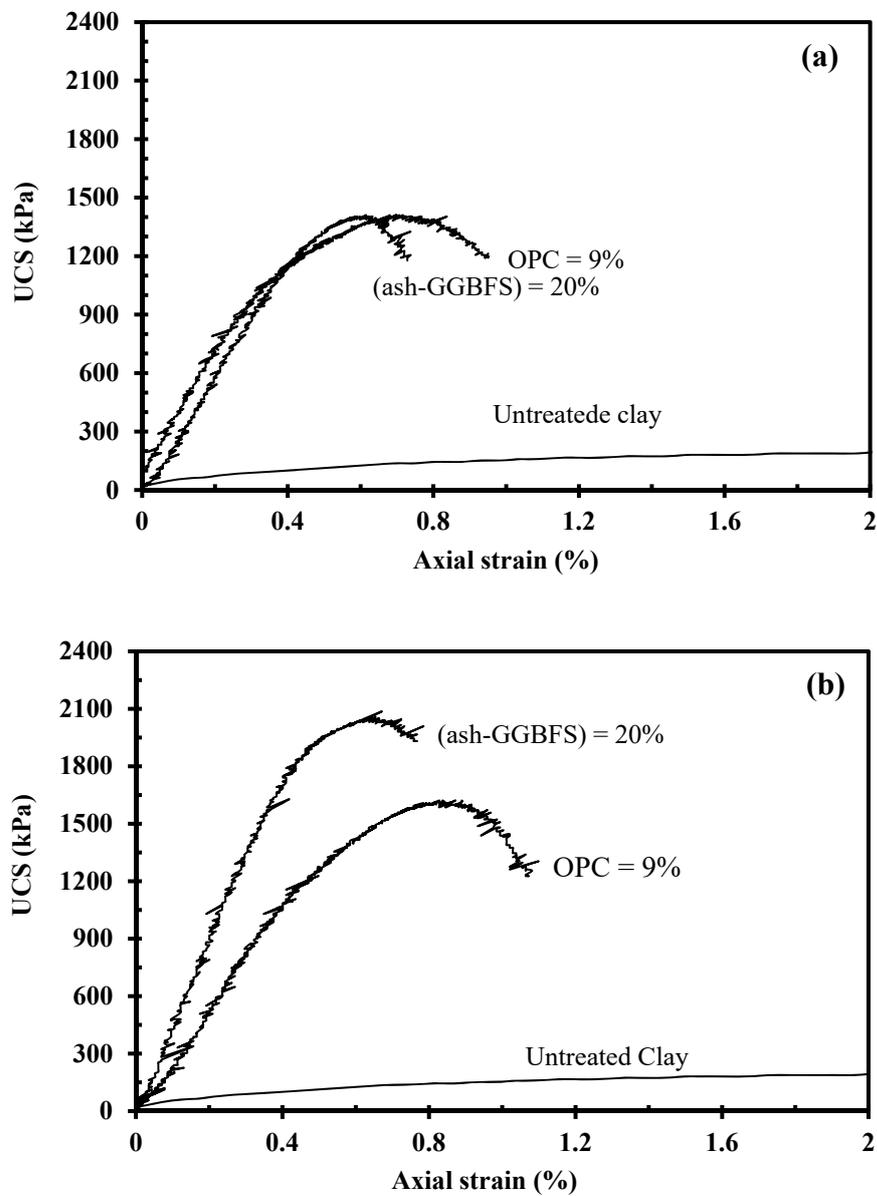


Figure 4.5: Typical stress-strain curves of geopolymer-stabilised clay and OPC treated clay: (a) 7 days; and (b) 28 days.

4.4 MICROSTRUCTURE CHARACTERISTICS OF GEOPOLYMER-STABILISED CLAY

To further understand the mechanism of the strength improvement of geopolymer, the microstructure fabric of untreated and geopolymer-treated clay was examined using scanning electron microscopy (SEM). Figures 4.6 and 4.7 show the microstructure of untreated and treated clay mixtures with (ash-GGBFS) additive at zero activation.

Figures 4.8 to 4.10 show the microstructure of kaolin clay treated with geopolymer synthesised at ash-GGBFS = 20%, GGBFS/ash-GGBFS = 20% and activator/ash-GGBFS = 40%, cured at 7 and 28 days, respectively, representing the early stage and advanced stage of geopolymerisation at ambient temperature.

The SEM image of the fracture surface of untreated kaolin clay, as shown in Figure 4.6, clearly indicates the plate-like shape of the clay particles and a considerable amount of micro-porosity. Figure 4.7 shows the distribution of the fly-ash and GGBFS additives within the clay matrix without activation. As can be seen, significant discontinuities and voids in the traded soil fabric are still indicated. The spherical nature of the fly-ash particles, as they exist between clay particles, is also evident. Due to its small quantity in the mixture, it was difficult to detect any traces of GGBFS particles. In comparison, the fragment-surface of geopolymer-treated clay, as shown in Figure 4.8, indicates an enhancement in the homogeneity of the clay fabric. It can be seen that the activation of fly-ash and GGBFS within the stabilised clay mixture result in more closely linked clay particles and fewer voids due to the initial compaction enhancement and the formation of cementitious products with the curing time. The enhancement in the clay fabric is believed to produce a higher strength clay structure as evident from the UCS tests (Section 4.3), and this finding is in agreement with those observed in the earlier geopolymer-soil studies (Cristelo et al. 2012; Zhang et al. 2013; Liu et al. 2016). However, Figure 4.8(a and b) shows the presence of spherical fly-ash particles that have not reacted. The silica and alumina of the fly-ash seem to be partially leached at 7 days (Figure 4.8a) of curing time under the current activator concentration. This is in agreement with the findings by Phummiphan et al. (2016) who confirmed the gradual reaction of fly-ash with the activator at specific concentrations within treated soil.

As discussed in the literature review, the silica and alumina oxides at high pH level get dissolved from the fly-ash and GGBFS particles within geopolymer and this forms substance classified as Sodium Aluminum Silicate Hydrate (N-A-S-H), which in turn harden with time and cement the soil particles (Cristelo et al. 2012; Liu et al. 2016; Phummiphan et al. 2016). This is also evident from Figure 4.9 as some substance can be detected on the smooth spherical surface of partially-reacted fly-ash particles as an indication of leaching the silica and alumina from the fly-ash particle and the formation

of cementitious products (i.e. N-A-S-H) within treated clay. It is believed that the partially-reacted fly-ash particles with cementitious products on the soil surface serve as nucleation sites that bond clay plates into clusters, see Figure 4.10. This process modifies the structure of the clay and enhances the strength response detected earlier in Figures 4.1 to 4.5.

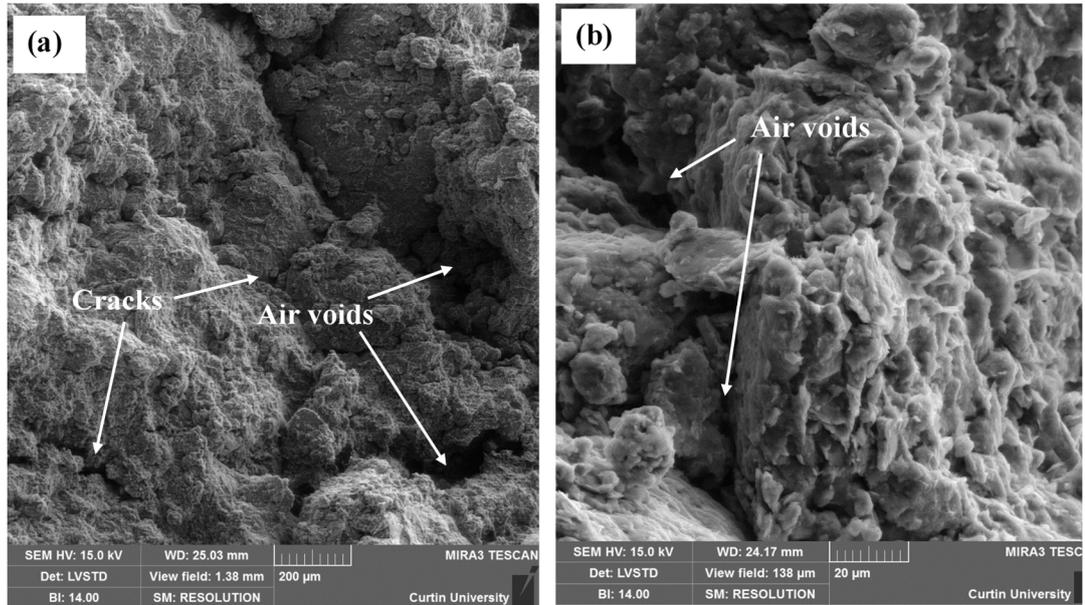


Figure 4.6: SEM of untreated kaolin clay: (a) 200 μm; and (b) 5 μm.

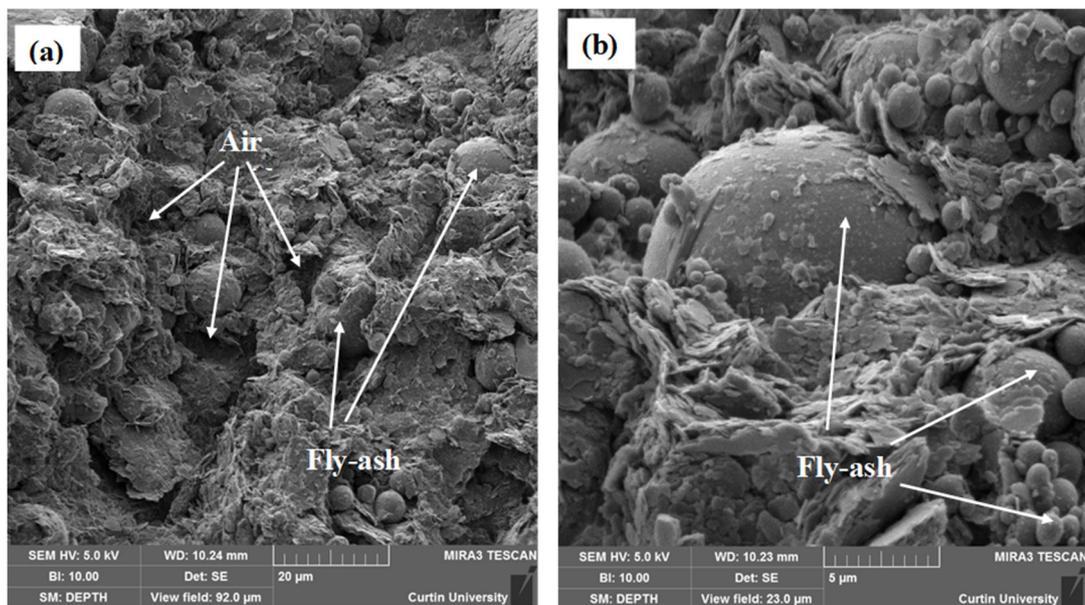


Figure 4.7: SEM of clay mixture treated with fly-ash and GGBFS (wet mixing without activation): (a) 20 μm; and (b) 5 μm.

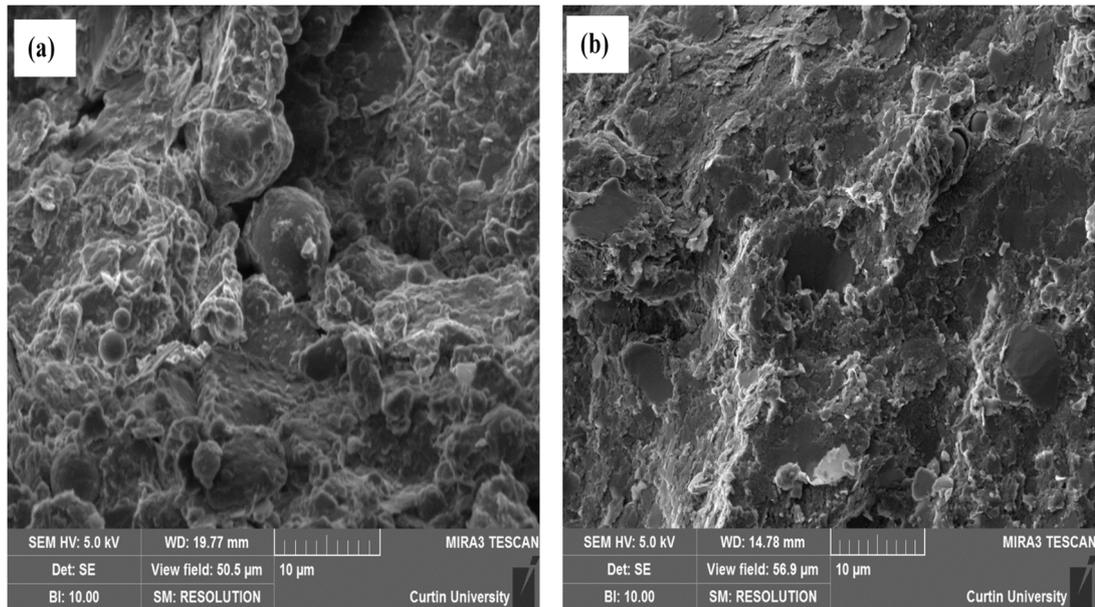


Figure 4.8: SEM of geopolymer treated clay – 10 μm : (a) 7days; and (b) 28 days.

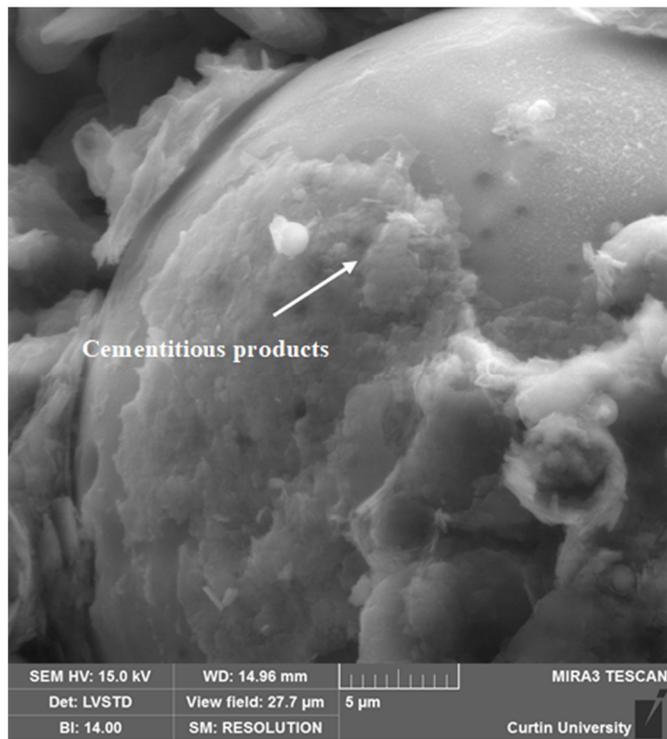


Figure 4.9: SEM of geopolymer treated clay - 28 days – 5 μm .

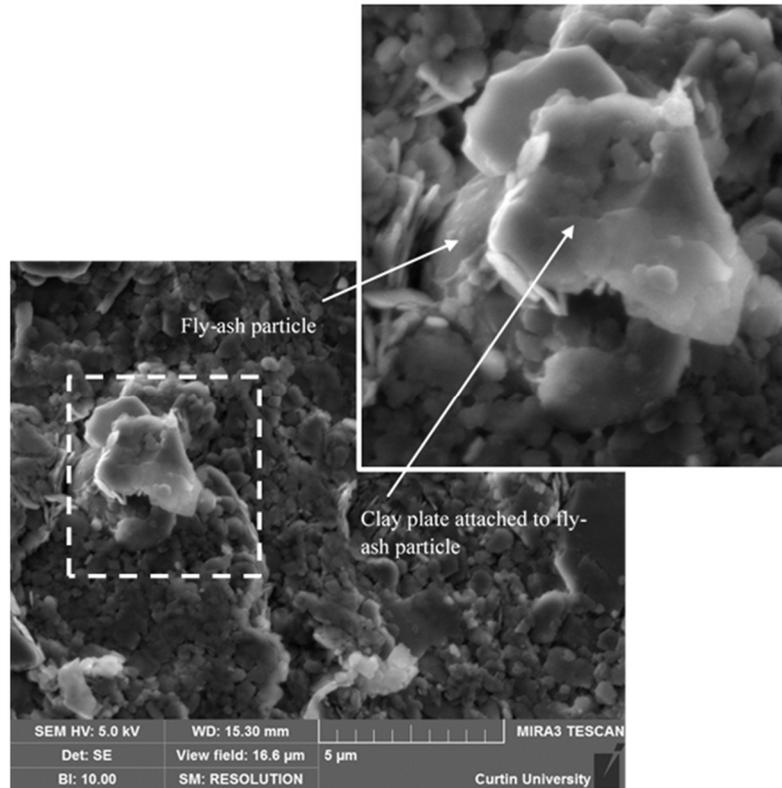


Figure 4.10: SEM of geopolymer treated clay - 28 days - 5 μ m: Clay plate-like grain attached to the surface of fly-ash particle by geopolymer-cementitious bond.

4.5 SUMMARY

In this chapter, the effects of fly-ash, ground granulated blast-furnace GGBFS and activator contents were investigated for the determination of a practical geopolymer mixture for clay stabilisation at ambient temperature. The Unconfined Compressive Strength (UCS) test was selected to investigate the degree of reactivity of different geopolymer components in treated kaolin clay and the results were evaluated for different curing periods of 7, 28 and 90 days. The microstructure of the optimum mixture was also investigated by Scanning Electron Microscopy (SEM) analysis. The following conclusions are drawn from this chapter.

- Fly-ash based geopolymer increases the UCS of treated clay. This is more pronounced as either the quantity of fly-ash, the quantity of activator or the curing time increased.
- The addition of GGBFS as a partial replacement of fly-ash proved to significantly enhance the UCS of geopolymer-treated clay at ambient temperature. It is evident that GGBFS works synergistically with fly-ash within

geopolymer which suggests an increase in the short-term strength performance of treated clay as well as long-term performance.

- There is no optimum ratios for GGBFS/ash-GGBFS or activator/ash-GGBFS in geopolymer-treated clay mixtures. The UCS of the mixtures increases with the increase of the amount of GGBFS and activator contents. Since the GGBFS is a waste material, GGBFS/ash-GGBFS maximum ratio of 20% was recommended as a practical percentage to synthesise geopolymer for clay stabilisation. In terms of activator content, activator/ash-GGBFS of 40% can be recommended to promote strength level in the geopolymer-treated mixtures which fulfils the requirements of most geotechnical applications. At 7 days of curing, the concentrations mentioned above were found to give soil strength equivalent to that obtained from soil stabilised with OPC. At curing times of 28 and 90 days, the geopolymer-treated mixtures provided superior strength improvement over soil stabilised with OPC.
- The evolution of the microstructure, responsible for the stress-strain response observed in the UCS tests, was detected by scanning electron microscopy (SEM) technique. It is believed that the reacted fly-ash particles with cementitious products on the soil surface serve as nucleation sites that bond clay plates into a form of clay clusters, which modifies/strengthens the structure of the clay and enhances the unconfined strength response.

The results obtained in this chapter clearly promote the potential use of fly-ash geopolymer-incorporating GGBFS as an effective alternative binder to OPC in the stabilisation of clay soils. However, it should be noted that the results presented in the current chapter focussed mainly on the strength performance of kaolin clay, and further studies are needed to be conducted on this clay as well as other types of clay, this will be the primary research focus in the next chapter. The next chapter presents particular compaction, consistency, strength, durability, permeability and pH studies for engineered and natural clays utilising the recommended geopolymer combination obtained from this chapter. The purpose of using different types of clay is to investigate the mineralogy and plasticity effects of clay treatment performance, both of which are known in the literature to have a significant influence on the stabilisation of clays using traditional binders.

CHAPTER 5

ENGINEERING PROPERTIES OF GEOPOLYMER-TREATED CLAYS

5.1 INTRODUCTION

To advance the use of geopolymer for clay stabilisation, it is necessary to understand the engineering properties of the geopolymer-clay mixtures. As discussed in the literature review, limited information is currently available in this regard, particularly around the impact of soil mineralogy and plasticity on the effectiveness of treatment, as these factors are known to have a significant influence on the behaviour of soil in general (Mitchell et al. 2005). This chapter presents and discusses the results of a laboratory testing program aimed at providing an understanding of the engineering properties of geopolymer treated clays. Considering the experimental methodology described earlier in Phase II of the testing program (Chapter 3), nine types of clay soils (i.e. engineered and natural) were investigated for geopolymer treatment. The investigation were conducted using different concentrations of geopolymer, synthesised at selected optimum combinations of GGBFS/ash-GGBFS ratio of 20% and activator/ash-GGBFS ratio of 40% as concluded earlier in Chapter 4. The influence of geopolymer addition at 0%, 10%, 15% and 20% by weight, considering the effect of clay mineralogy on the characteristics of treated clays were investigated and discussed in the following sections of this chapter:

Compaction

Section 5.2 presents the results of the compaction tests for all clay types. The standard compaction tests were carried out immediately after mixing the geopolymer with clays.

Atterberg limits

Section 5.3 presents and discuss the results of the plasticity studies for all clay types. Plasticity characteristic tests were carried out for specimens cured for 7 and 28 days.

Unconfined compressive strength

Section 5.4 details the undertaking of Unconfined Compressive Strength (UCS) testing for the treated clays over different curing periods of 7, 28 and 90 days.

Durability

Section 5.5 presents and discusses the results of the durability study conducted on the treated clays. This study investigated the behaviour of 7 days cured treated specimens exposed to a range of simulated weathering conditions, including the wetting-drying, freezing-thawing and leaching tests.

Permeability

Section 5.6 details the undertaking permeability test for the untreated and geopolymer-treated kaolin clay over curing periods of 7 and 28 days.

Impact of pulverisation

Section 5.7 assessed the impact of pulverisation of natural clays.

pH

Section 5.8 concludes the chapter, where a detailed study on the pH performance of different geopolymer-treated clays with curing time is presented.

5.2 COMPACTION PROPERTIES OF GEOPOLYMER-TREATED CLAYS

The optimum compaction properties (i.e. maximum dry unit weight, MDU, and optimum moisture content, OMC) of treated clay soils are important parameters given their use of in-field quality assurance and control. Accordingly, a series of standard compaction tests were conducted in this section on different treated clays to understand their compaction properties. The results of compaction tests for all treated clay soils (both engineered and natural) are given in Table 5.1. The data show that geopolymer-clay soil mixtures have a higher MDU than untreated clay soils, with a reduction in the corresponding OMC as geopolymer content increases. This may be attributed to the chemical activators used in the geopolymer mixtures, which act as lubricants that reduce the repulsion forces and increase the sliding between the clay particles (Elleboudy 1977). The lubrication induced by the activator may also reduce the volume of the free water required to achieve the optimum compaction, thus reducing the OMC. Additionally, due to the partial replacement of clay by additives, the silty characteristics of the fly-ash and GGBFS particles in the geopolymer mixture may reduce the overall surface area of the clay particles and thus the water demand (Santos et al. 2011). However, it is apparent that both the engineering and natural clays tested here differ markedly in terms of the magnitude of change in MDU and OMC with geopolymer treatment.

Table 5.1: Compaction characteristics of geopolymer-treated clays.

Clay	Geopolymer (%)	MDU (kN/m ³)	OMC (%)
100Kaolin	untreated	14.95	25.2
	10	15.04	24.0
	15	15.20	23.0
	20	15.45	20.6
(90Kaolin-10Bentonite)	untreated	13.85	25.3
	10	13.94	23.0
	15	14.33	23.0
	20	14.88	22.0
(80Kaolin-20Bentonite)	untreated	13.65	25.7
	10	13.86	24.0
	15	14.12	23.5
	20	14.70	22.5
(60Kaolin-40Bentonite)	untreated	13.19	26.0
	10	13.40	24.2
	15	13.72	23.2
	20	14.24	21.0
(90Kaolin-10Sand)	untreated	15.09	24.0
	10	15.25	22.0
	15	15.40	21.0
	20	15.85	19.0
(80Kaolin-20Sand)	untreated	15.67	22.0
	10	15.75	21.0
	15	15.90	19.7
	20	16.40	17.7
(60Kaolin-40Sand)	untreated	17.30	16.0
	10	17.38	15.4
	15	17.48	14.6
	20	17.70	13.0
NatSoil (1)	untreated	16.28	19.7
	10	16.33	19.0
	15	16.40	18.1
	20	16.45	16.9
NatSoil (2)	untreated	14.88	22.3
	10	15.00	19.2
	15	15.28	17.0
	20	15.80	15.9

To allow for comparison of compaction behaviour between different treated specimens, the treated clay compaction parameters, MDU and OMC, were normalised by the corresponding values of untreated clay, as shown in Figure 5.1(a and b). For all soil types, the change in the normalized MDU ratio is dependent on the geopolymer content as well as the clay type. The normalised OMC was observed to be more closely related to the geopolymer content than the soil type for the engineered clay soils (Figure 5.1a), while for the natural clays especially for NatSoil (2) of high plasticity (Figure 5.1b), the addition of geopolymer resulted in a notable decrease in the normalized OMC ratio. These changes within natural clays were driven significantly by the clay type (i.e. mineralogy/plasticity); the higher the plasticity of the clay, the higher the change in OMC ratio. The difference in behaviour between the treated

engineered and natural clay soils may relate to the initial interaction between the geopolymer and the individual clay minerals, which impacts the lubrication process and moisture demand described earlier in this section. In all cases, since an initial strength gain is usually expected due to the initial MDU increase, the changes in compaction properties of geopolymer-treated clays can be regarded as an advantage and should be featured in any systematic approach on geopolymer-based stabilisation.

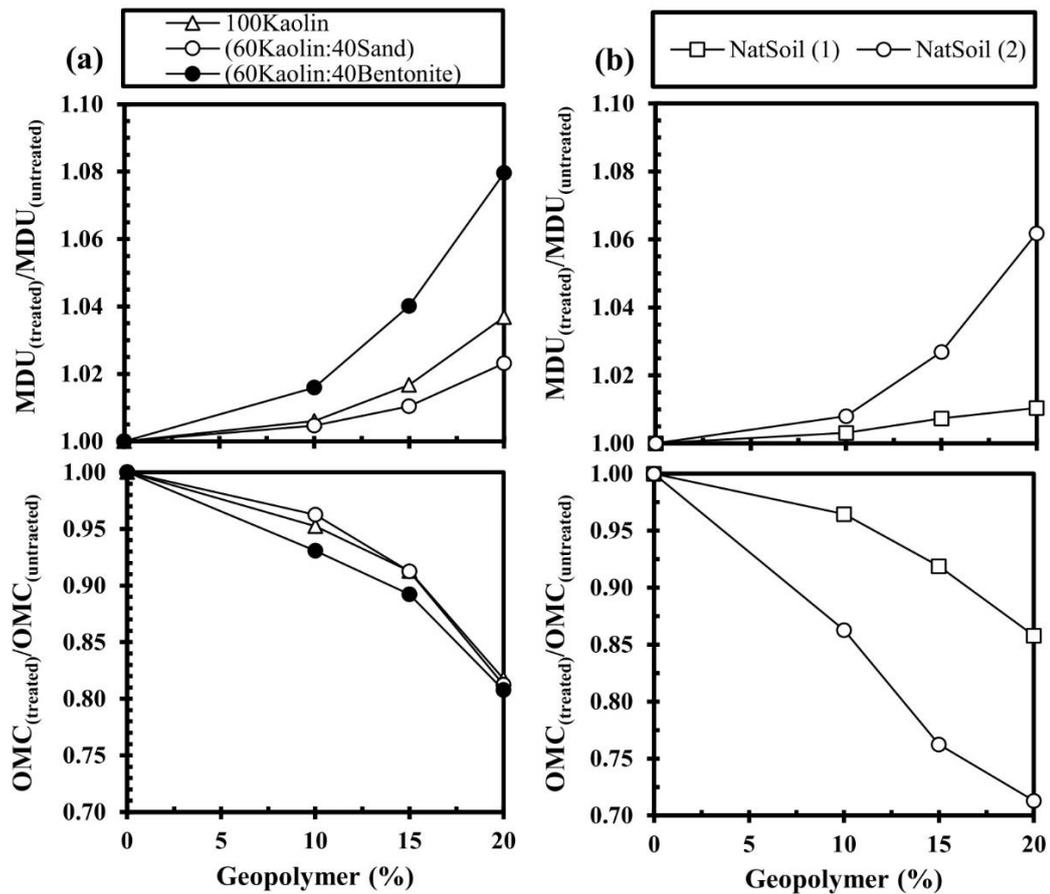


Figure 5.1: Effects of geopolymer addition on maximum dry unit weight (MDU) and optimum moisture content (OMC) of clays: (a) engineered clays; and (b) natural clay.

5.3 PLASTICITY INDICES OF GEOPOLYMER-CLAY MIXTURES

Plasticity of treated clays is an important aspect of stabilisation as it describes the response to moisture. The plasticity index was determined for each treated clay, i.e. Plasticity Index (PI) = Liquid Limit (LL) - Plastic Limit (PL), to determine the impact of geopolymer content, curing time and clay type on geopolymer treatment. The plasticity indices of the various treated mixtures are presented in Table 5.2, for 7 and 28 days of curing.

Table 5.2: Plasticity characteristics of geopolymer-treated clays at 7 and 28 days.

Clay	Geopolymer (%)	7 days curing time			28 days curing time		
		LL(%)	PL(%)	PI(%)	LL(%)	PL(%)	PI(%)
100Kaolin	untreated	53.00	27.00	26.00	53.00	27.00	26.00
	10	52.80	32.80	20.00	49.80	31.00	18.80
	15	50.00	33.90	16.10	50.20	36.10	14.10
	20	46.20	34.00	12.20	45.20	36.30	8.90
(90Kaolin:10Bentonite)	untreated	61.00	32.00	29.00	61.00	32.00	29.00
	10	54.00	34.00	20.00	57.00	38.10	18.90
	15	52.00	35.00	17.00	55.00	41.00	14.00
	20	49.00	36.30	12.70	52.00	42.10	9.90
(80Kaolin:20Bentonite)	untreated	87.50	34.30	53.20	87.50	34.30	53.20
	10	63.20	38.90	24.30	60.00	39.00	21.00
	15	56.60	38.90	17.70	55.00	39.30	15.70
	20	53.80	41.00	12.80	50.20	40.00	10.20
(60Kaolin:40Bentonite)	untreated	139.00	40.20	98.80	139.00	40.20	98.80
	10	95.00	41.00	54.00	70.60	44.20	26.40
	15	67.20	43.00	24.20	60.00	44.80	15.20
	20	59.00	46.00	13.00	55.00	46.00	9.00
(90Kaolin:10Sand)	untreated	48.00	24.00	24.00	48.00	24.00	24.00
	10	47.00	29.80	17.20	48.50	33.9	14.60
	15	45.00	33.50	11.50	47.20	36.3	10.90
	20	42.00	34.00	8.00	44.00	39.7	4.30
(80Kaolin:20Sand)	untreated	45.00	22.80	22.20	45.00	22.80	22.20
	10	44.00	28.80	15.20	47.50	34.33	13.17
	15	43.00	32.10	10.90	44.40	35.10	9.30
	20	41.40	33.00	8.40	38.00	36.00	2.00
(60Kaolin:40Sand)	untreated	39.00	20.00	19.0	39.00	20.00	19.00
	10	38.50	25.20	13.30	38.00	29.70	8.30
	15	37.50	28.00	9.50	38.00	-	non-
	20	36.80	30.50	6.30	36.00	-	non-
NatSoil (1)	untreated	46.00	26.00	20.00	46.00	28.40	17.60
	10	38.77	27.50	11.30	38.40	-	non-
	15	35.00	28.50	6.50	36.00	-	non-
	20	34.00	29.60	4.40	37.00	-	non-
NatSoil (2)	untreated	57.00	25.80	31.20	57.00	25.80	31.2
	10	54.00	27.40	26.60	52.20	27.60	24.6
	15	49.00	28.90	20.10	44.80	27.80	17.0
	20	44.20	31.00	13.20	41.80	28.70	13.1

5.3.1 Impact of geopolymer content

Figure 5.2 illustrates typical LL and PL values of engineered and natural treated clay soils cured at 7 days. As can be seen in Figure 5.2(a), all treated engineered clays exhibited a reduction in LL with increasing geopolymer content.

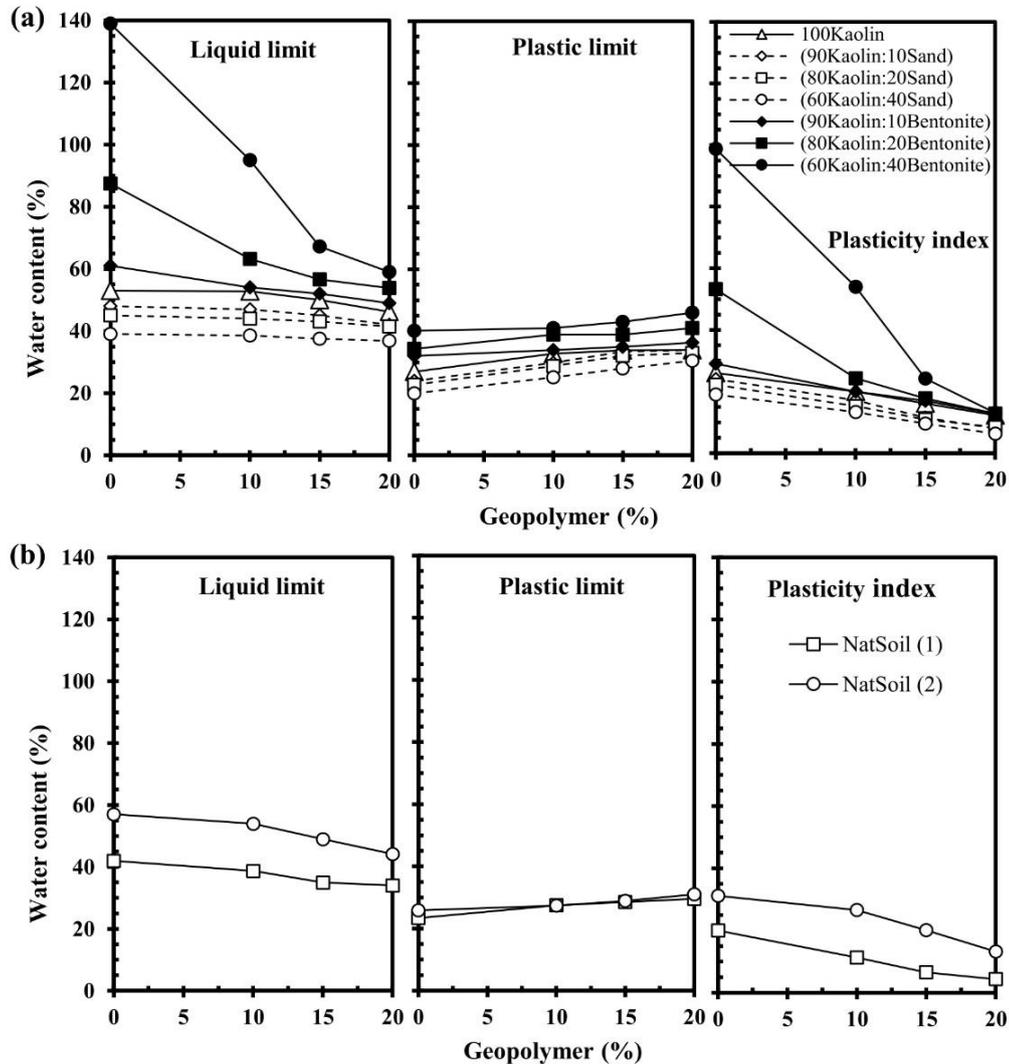


Figure 5.2: Effect of geopolymer addition on consistency limits of treated clay soils at 7 days curing: (a) engineered clay soils; and (b) natural clay soils.

As the geopolymer dosage increases, the kaolin-bentonite clay group shows a greater decrease in LL than pure kaolin clay and the kaolin-sand clay group. Pore fluid chemistry such as ionic concentration induced by geopolymer activator has a possible impact on the LL of geopolymer treated clays. High concentrations of ions in the pore water at high geopolymer content may decrease the double layer diffusion of clay particles, resulting in a decrease in the LL especially for the smectite contained clay, i.e. bentonite (Tiwari et al. 2005). However, the results suggest a high influence of clay

mineralogy on LL of geopolymer-treated clays, given the problematic mineral composition of montmorillonite (Chapter 2, Section 2.2.2) within the bentonite group compared to other soil types. Conversely, the PL exhibited a comparatively minor increase with the addition of geopolymer, irrespective of clay type. Similar behaviour was also observed for treated natural clays (Figure 5.2b) albeit to a slightly lesser degree than for the engineered soils, particular with regard to the PL. Figure 5.3(a and b) illustrates the typical LL and PL values of engineered and natural treated clay soils cured at 28 days and shows similar trends to those of 7 days curing (Figure 5.2).

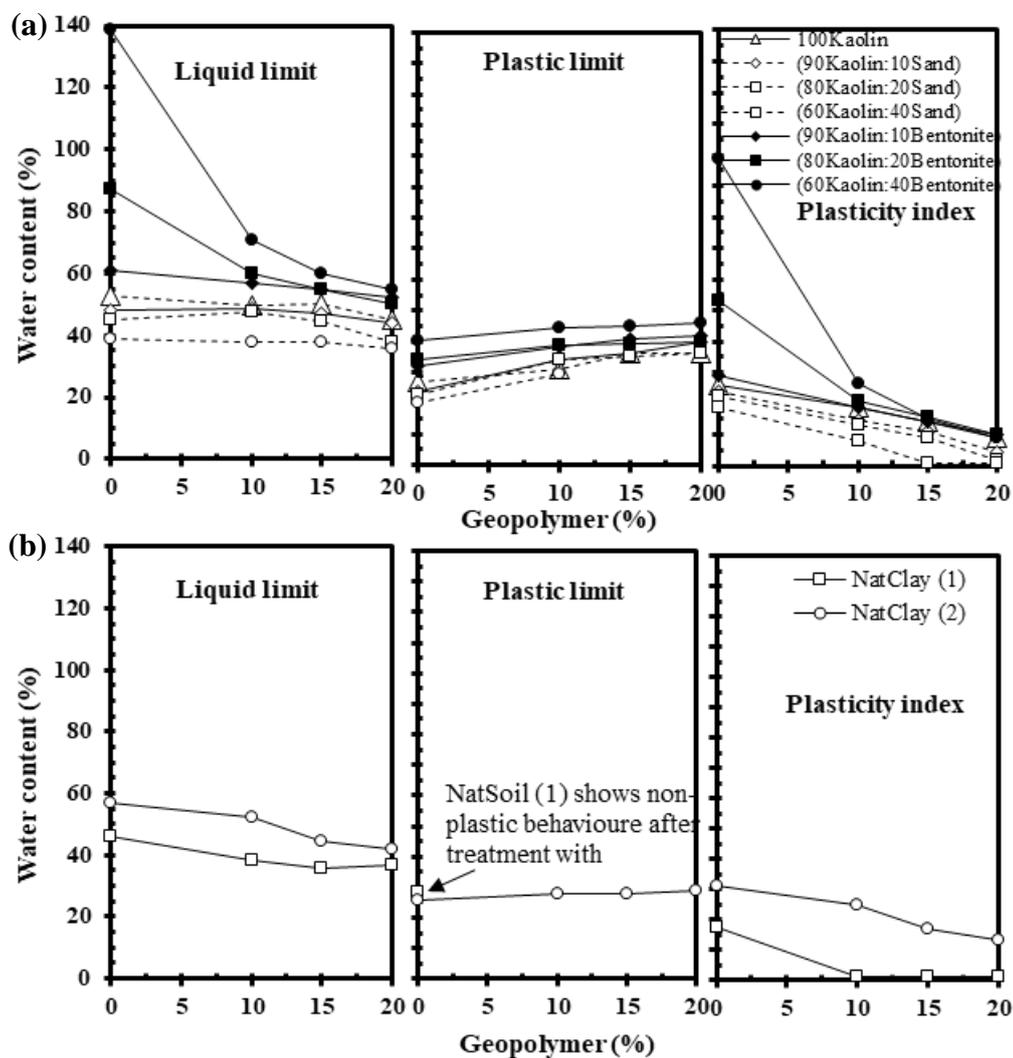


Figure 5.3: Effect of geopolymer addition on consistency limits of treated clay soils at 28 days curing: (a) engineered clay soils; and (b) natural clay soils.

The overall change in LL and PL for the engineered and natural clays may be attributed to the combined effects of the non-plastic silty fly-ash and GGBFS particles, the lubricant effects due to the geopolymer chemicals (as discussed previously with

respect to compaction behaviour) and the formation of cementitious products between the clay particles. It is believed that the geopolymer-cementitious bonds developed within the mixture bind the soil particles together in ‘micro-aggregates’, which then behave like silt particles, leading to reducing LL and increasing PL. This explanation is also supported by the silty texture visually observed in the cured mixtures during testing. The resultant PI for all engineered and natural clay soils showed an overall reduction with increasing geopolymer dosage, particularly for engineered clays of high bentonite content. This indicates a significant influence of clay mineralogy on plasticity characteristics of geopolymer-treated clay.

5.3.2 Impact of curing time

To assess the impact of curing time on the plasticity of the treated clay soils, the PI values for all specimens treated with 10% and 20% geopolymer contents and different curing periods of 7 days and 28 days were considered, as shown in Figure 5.4. Generally, it can be seen that the PI decreases with increasing the curing time for both 10% and 20% geopolymer content. The 10% geopolymer-treated (60Kaolin:40Sand) engineered clay, for instance, observed a 30% and 38% reductions in PI compared with untreated clay for 7 days and 28 days curing respectively, as shown in Figure 5.4(a). At 20% geopolymer, these reductions further increased to 67% and 85% at 7 days and 28 curing days, respectively, compared with the untreated clay, as depicted in Figure 5.4(b). The progressive development of geopolymer cementation with the increase of curing time may explain the corresponding reductions in PI, as discussed above. The increase in curing time also resulted in a notable reduction in the PI of treated natural clays. This is most extreme for NatSoil (1) curing at 28 days the specimen is rendered non-plastic. The test results for all investigated clay soils suggest that, although the ultimate achievable PI reduction is influenced by the curing period as well as the geopolymer content, the rate effects can be characterised principally by the type of clay soil, as reflected by the differences in plasticity and activity indices (see Table 3.2, Chapter 3). Overall, for all clay types, the reduction in plasticity suggests an improvement in the workability and potential suppression of soil high shrink-swell tendency, especially in high-plasticity clays, i.e. kaolin-bentonite group mixtures and NatSoil (2).

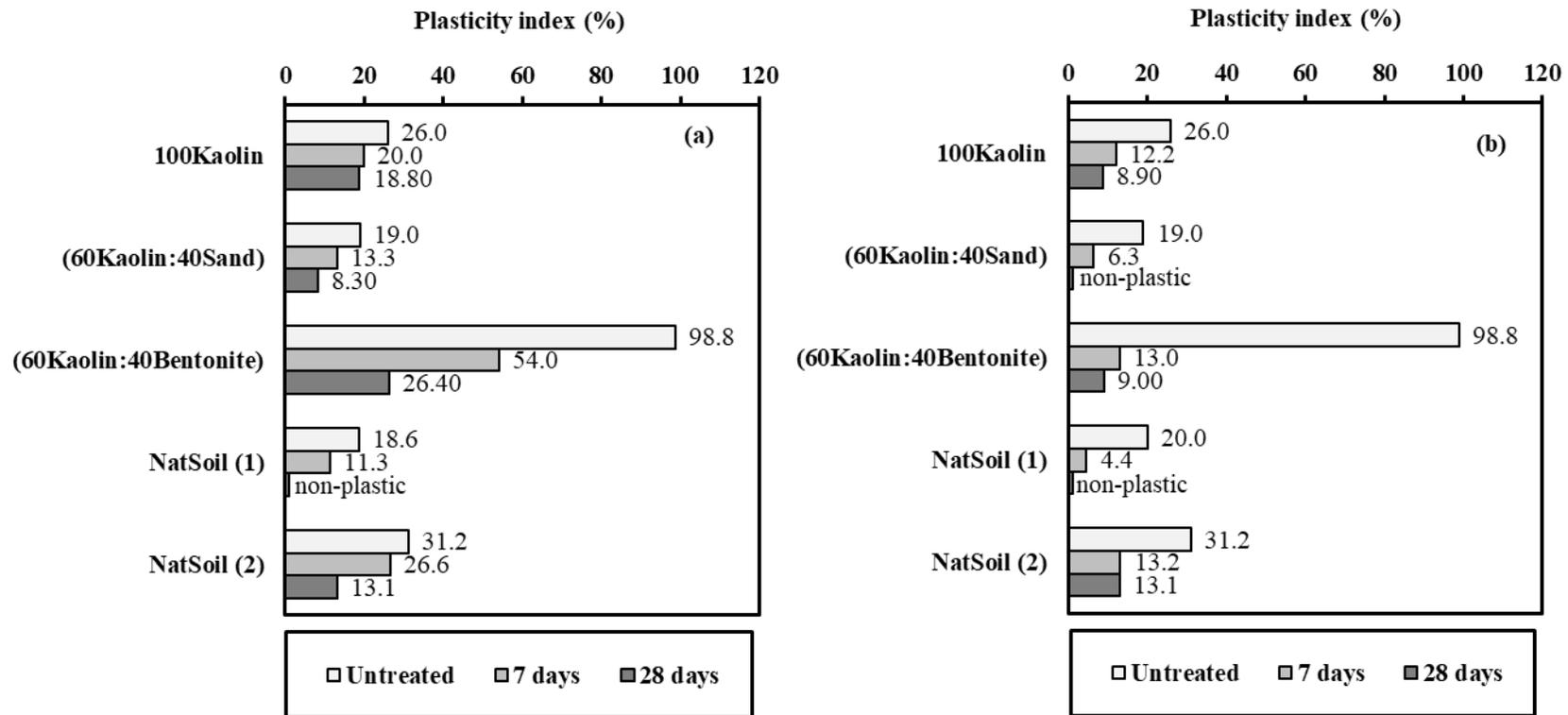


Figure 5.4: Effect of curing time on the plasticity index of engineered and natural clays treated with various geopolymer contents: (a) 10% geopolymer; and (b) 20% geopolymer

5.4 UNCONFINED COMPRESSIVE STRENGTH PERFORMANCE OF GEOPOLYMER-TREATED CLAYS

In this thesis, the test was used previously in the optimisation phase (Chapter 4), as an indication of the reactivity of different geopolymer ingredients within treated kaolin clay. Here, UCS testing was used to examine the effect of clay type on the compressive strength of geopolymer-treated mixtures as outlined in Chapter 3, Phase II.

5.4.1 Unconfined stress-strain behaviour of geopolymer-treated clays

The effect of geopolymer addition on the stress-strain behaviour of engineered and natural clay soils, as determined by Unconfined Compressive Strength (UCS) was examined as shown in Figures 5.5 to 5.8. The UCS stress-strain behaviour of each soil mixture, was considered for different geopolymer contents (i.e. 0%, 10%, 15% and 20%) and curing periods (i.e. 7, 28 and 90 days). Generally, with the addition of geopolymer, the dominate stress-strain response of the geopolymer-treated clays at 7 days of curing was found to be brittle yielding, by which the stress reached a peak before an abrupt failure. As the geopolymer content increases from 10% to 20%, yielding was associated with a stiffer response (i.e. higher unconfined strength and low axial strain). An exception to this behaviour was observed in the testing of the 7 days curing of the 10% geopolymer Kaolin and Kaolin-Bentonite specimens (Figures 5.5a and 5.7a), which exhibited a ductile response similar to that of untreated clay. However, the addition of geopolymer increased both the strength and initial stiffness beyond that of the untreated clay.

Similar to the observation made at 7 curing days, the stress-strain response of geopolymer-treated clays at 28 and 90 days is also found to be more prominent with increasing the geopolymer content (Figures 5.5 to 5.8b and c). It is worthwhile noting that at 90 days of curing, the Kaolin and Kaolin-Sand specimens treated with 20% geopolymer content (Figures 5.5 and 5.6) showed peak stress without post-peak response indicating an extreme brittle response at advanced curing periods. In all cases, the qualitative change in the stress-strain response of the treated clays can be attributed to the formation of artificial cementitious products. At high pH levels induced by the diluted activator (i.e. $\text{pH} > 11$), the silica and alumina oxides are dissolved from the fly-ash particles within the geopolymer, forming the N-A-S-H geopolymer product, which hardens with time and cements the soil particles (Cristelo et al. 2012; Liu et al. 2016; Phummiphan et al. 2016; Arulrajah et al. 2018). Given that the stress-strain

response is related to the formation of the geopolymer gel within the treated clays, the formation appears to vary with the change in soil mineralogical components.

The impact of clay mineralogy on the stress-strain responses of clays treated with 10% and 20% geopolymer cured for 28 days are shown in Figures 5.9(a and b) for engineered and natural clays, respectively. When comparing with treated kaolin clay, the engineered Kaolin-Sand and Kaolin-Bentonite clay groups show a typical brittle stress-strain response, as seen in Figure 5.9(a). However, it is apparent that these engineered clays differ markedly in the peak stress and the corresponding axial strain, particularly for 20% geopolymer. Figure 5.9(a) indicate that the Kaolin-Bentonite clay group provides the lowest response to geopolymer treatment compared with Kaolin and Kaolin-Sand clay groups. This also seems to be true for natural clays as they show different stress-strain responses with the increase of geopolymer content, as depicted in Figure 5.9(b).

The significant differences between the treated clay types are also evident in the respective plasticity and the activity indices (see Table 3.1, Chapter 3), with Kaolin-Bentonite clay group and NatSoil (2) having significantly higher values. The high indices associated with these clay soils appear to affect the ability to intimately mix the geopolymer with the soil and thus the overall production of a homogenous matrix of stabilised agglomerates; this, in turn, affects the strength performance of the cemented soil (Little 1995). Another possible reason for the variable stress-strain performance between the differently treated clay soils is the inherited nature of soil colloids (organic and inorganic) and their variable capacities to absorb, hold and release ions including the (OH⁻) ions which can cause a significant influence on the level of pH required for quality treatment. This indicates a high influence of clay mineralogy on the unconfined stress-strain response of geopolymer-treated clays.

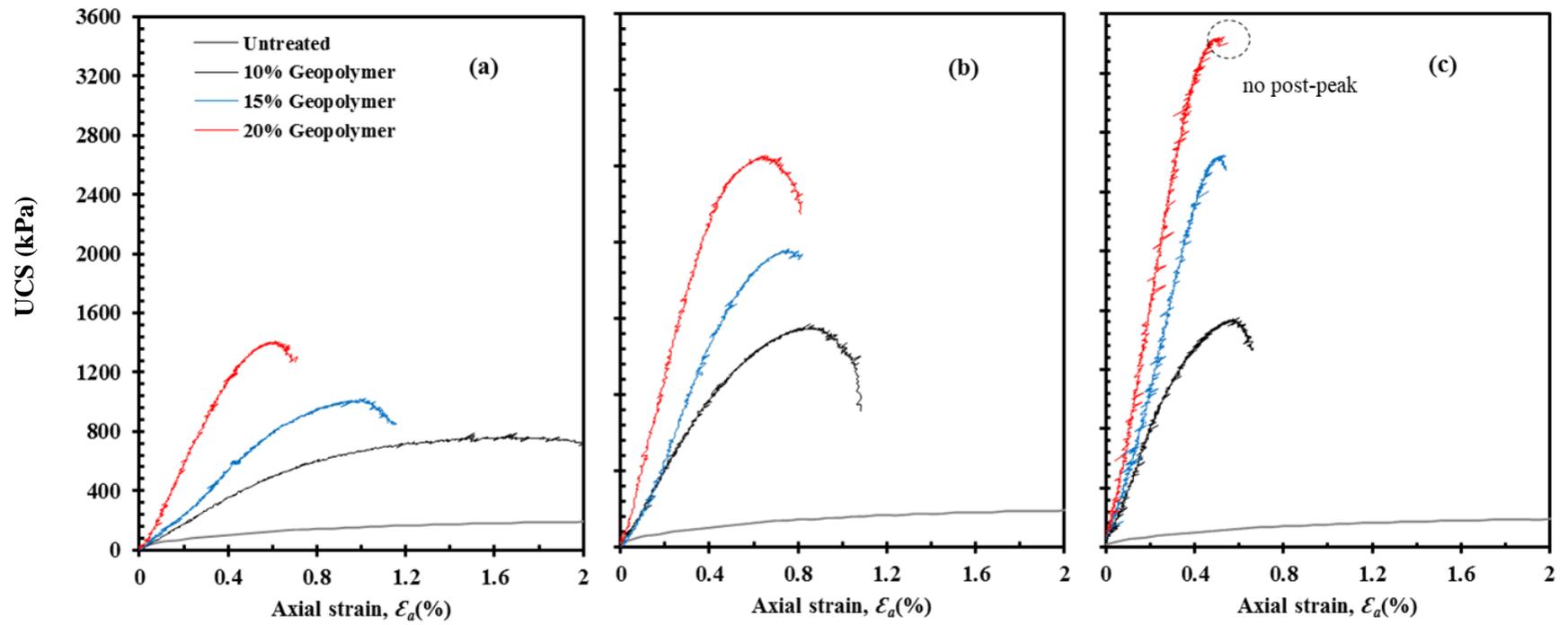


Figure 5.5: Unconfined stress-strain behaviour of kaolin clay treated with various geopolymer content and at curing periods: (a) 7 days; (b) 28 days; and (c) 90 days.

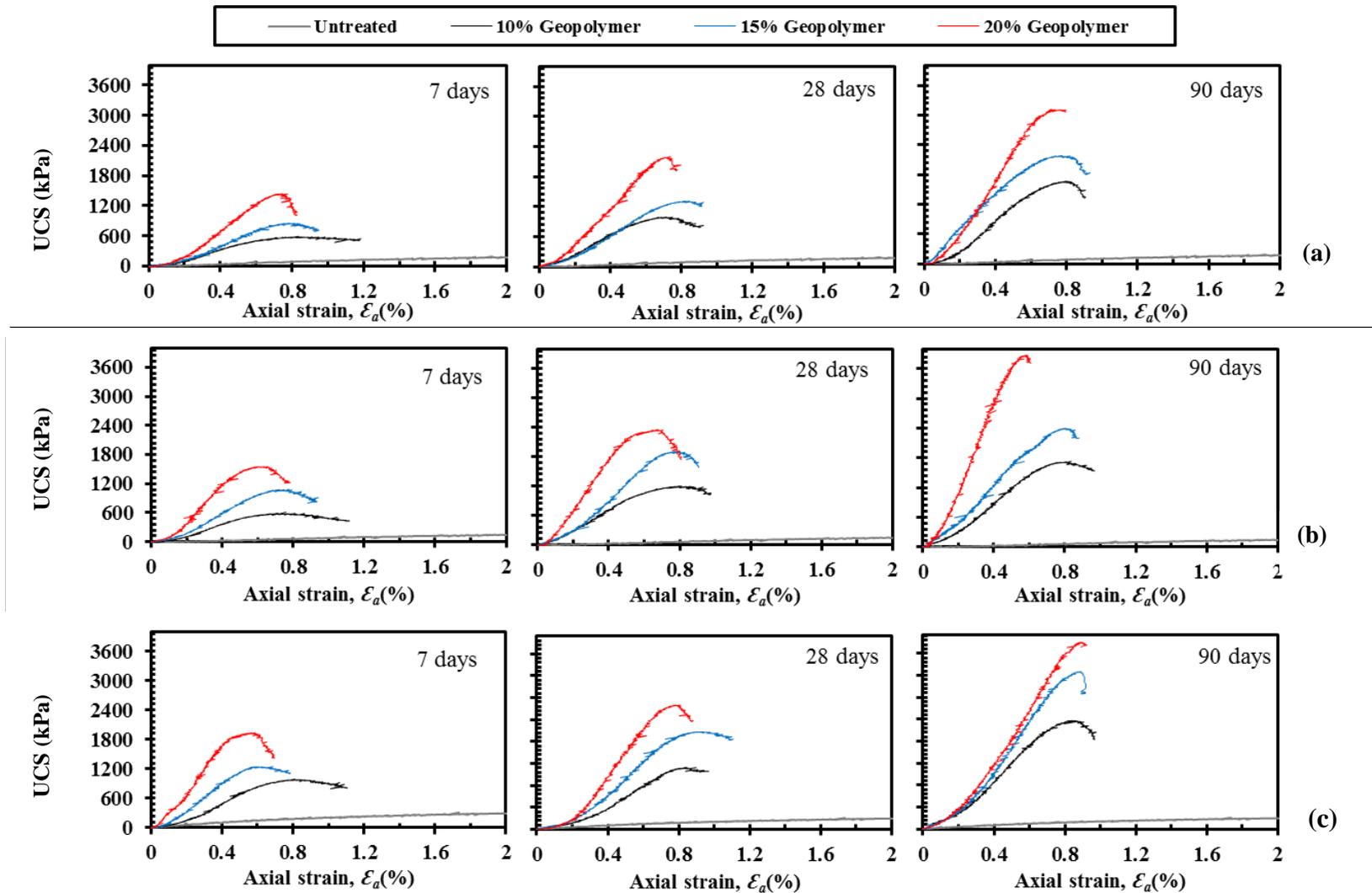


Figure 5.6: Unconfined stress-strain behaviour of Kaolin-Sand clay group treated with various geopolymer contents and at curing periods up to 90 days: (a) 90Kaolin:10Sand; (b) 80Kaolin:20Sand; and (c) 60Kaolin:40Sand.

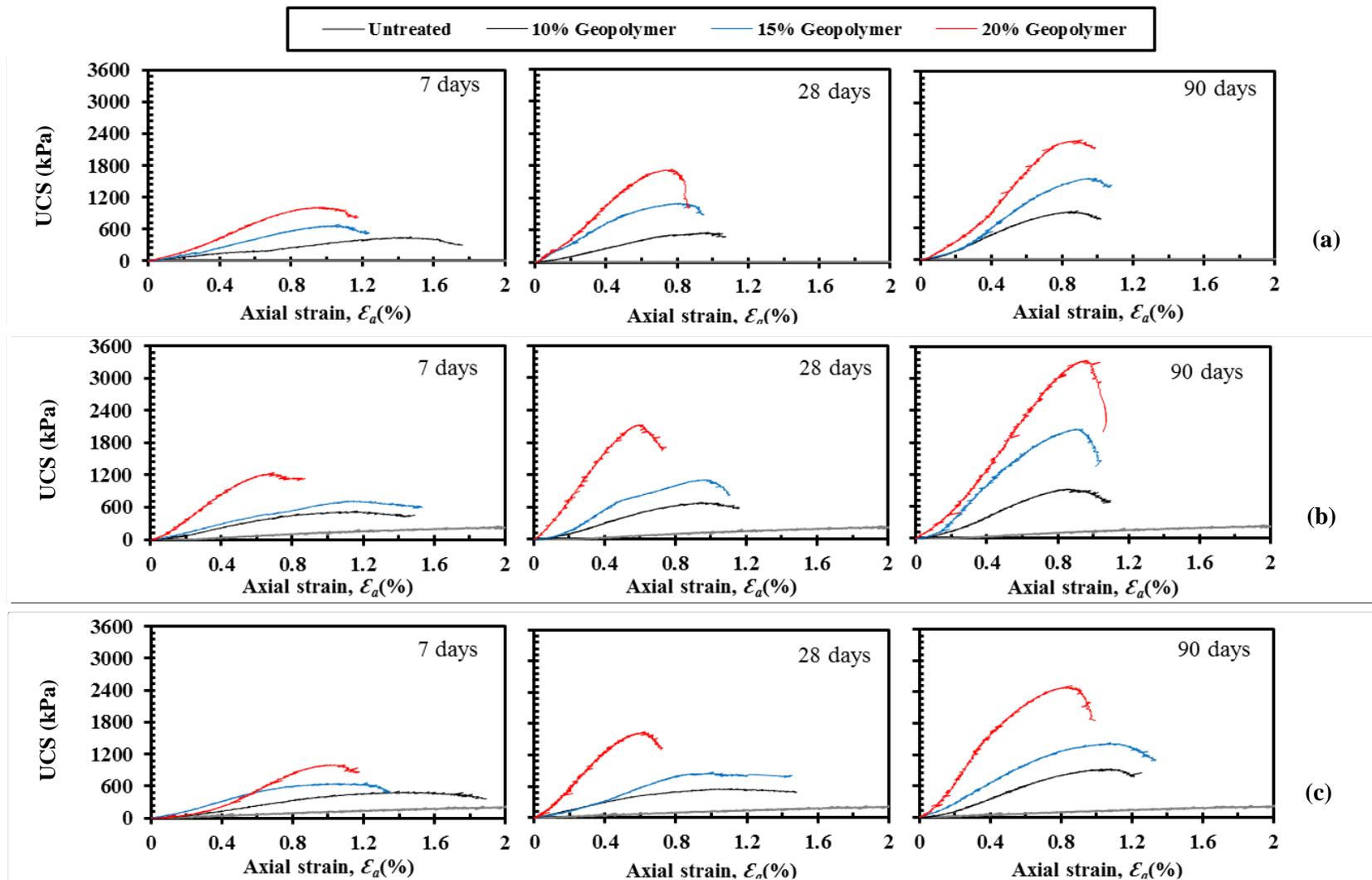


Figure 5.7: Unconfined stress-strain behaviour of Kaolin-Bentonite clay group treated with various geopolymer contents and at curing periods up to 90 days: (a) 90Kaolin:10Bentonite; (b) 80Kaolin:20Bentonite; and (c) 60Kaolin:40Bentonite.

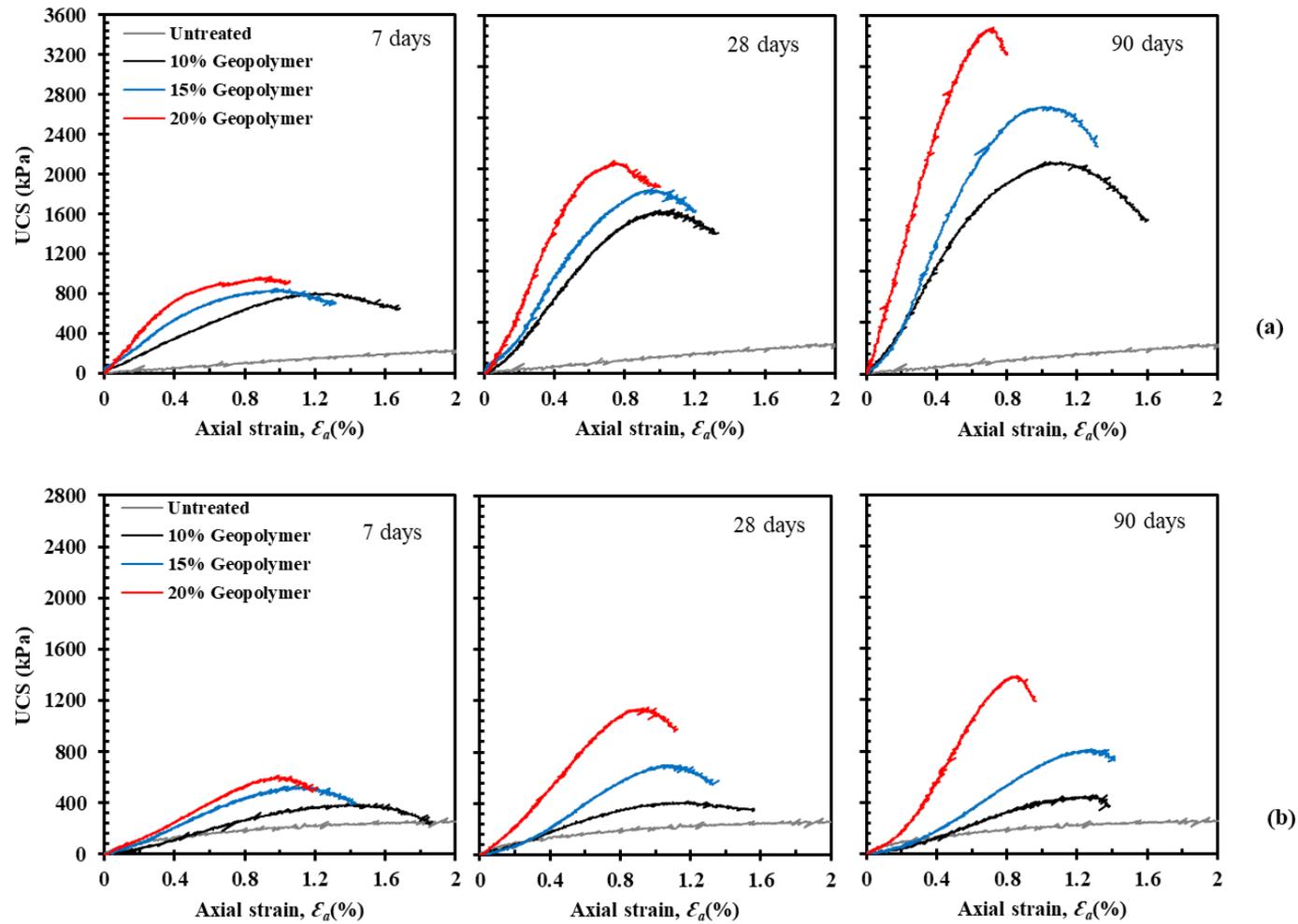


Figure 5.8: Unconfined stress-strain behaviour of natural clays treated with various geopolymer contents and at curing periods up to 90 days: (a) NatSoil (1); (b) NatSoil (2).

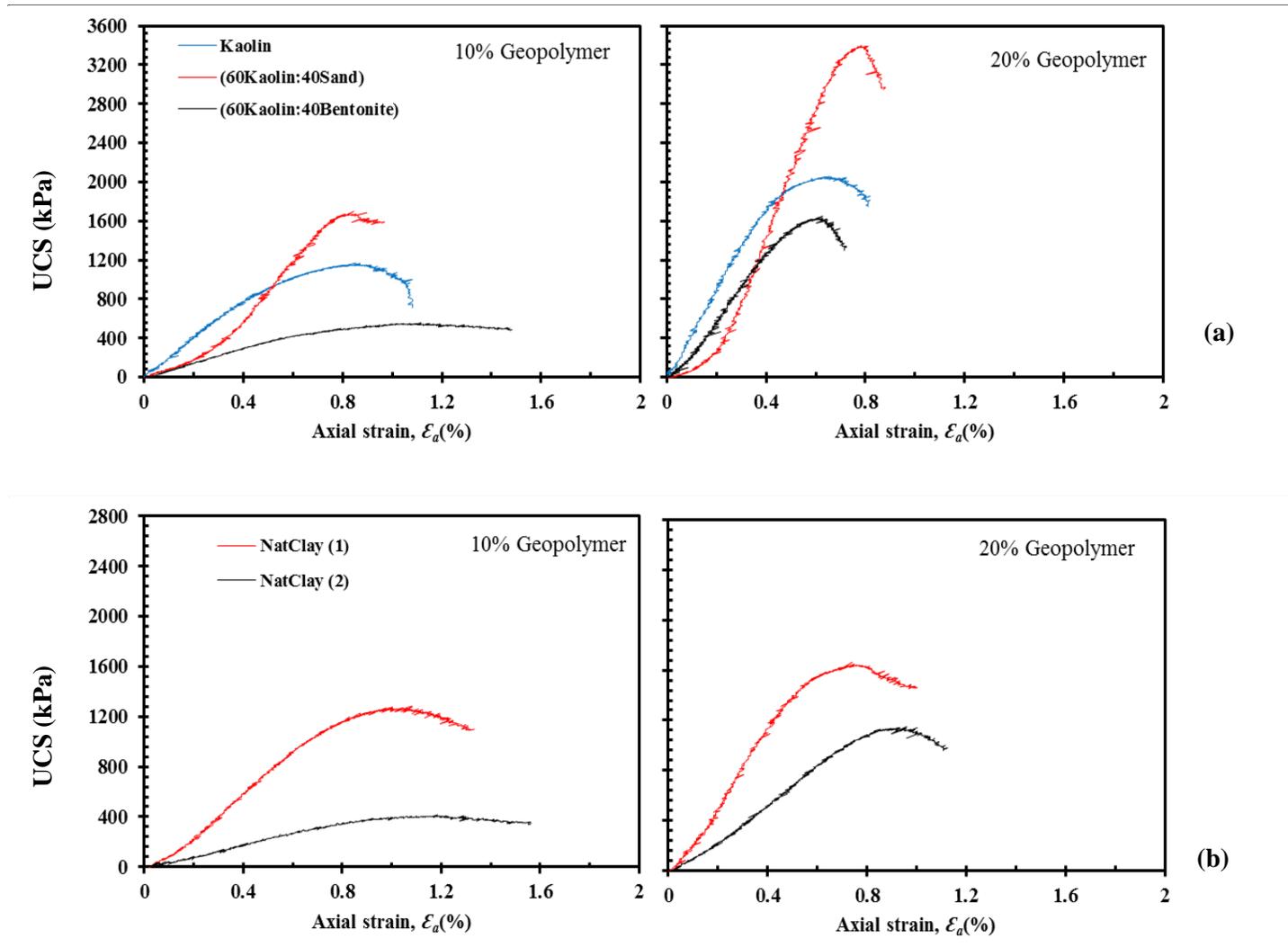


Figure 5.9: Effect of clay type on unconfined stress-strain behaviour of treated clay at 28 days of curing time: (a) engineered clays; and (b) natural clays.

5.4.2 Unconfined peak behaviour of geopolymer-treated clays

The variation of UCS peak values with geopolymer content and curing time for geopolymer-treated clays are presented numerically in Table 5.3 and graphically in Figures 5.10 to 5.13. Generally, the peak UCS values for all treated soils indicate high dependence on geopolymer content and curing period. Considering the 100Kaolin treated sample (Figure 5.10) at 7 curing days, an increase of ~260% in the peak UCS compared to untreated for 10% geopolymer was observed. Further increase of geopolymer content to 15% and 20% resulted in total strength increases of 458% and 630%, respectively, compared with the untreated clay. Such dependence of the peak UCS on geopolymer content can be attributed to the increased formation of cementitious products with increased geopolymer content (Phummiphon et al. 2016).

Overall, the strength enhancement of geopolymer-treated clays is impacted by the variation in the clay type, as evident from Figure 5.10. It can be seen that the addition of 10% geopolymer increased the peak UCS of mixtures (60Kaolin:40Sand) and (60Kaolin:40Bentonite) by approximately 175% and 60%, respectively, whereas the additional increase of geopolymer from 10% to 15% generated additional strength increases of 100% and 77%, respectively. Such variable influence of the geopolymer content on different treated clays was persistent over the selected range and curing time up to 90 days, as depicted in Figure 5.10. This also appears to be true for the natural clays, given the varied strength response with the increase of geopolymer content, as depicted in Figure 5.11. It can be seen in Figure 5.10 that the Kaolin-Bentonite clay group exhibits the lowest response to geopolymer treatment compared to the Kaolin and Kaolin-Sand groups. Similarly, Figure 5.11 suggests preference of NatSoil (1) for geopolymer treatment over NatSoil (2). The significant differences between treated clays are evident in the respective plasticity and activity indices (i.e. $A > 1$ and $PI > 30$, see Table 3.1, Chapter 3), with mixtures (60Kaolin:40Bentonite) and NatSoil (2) having significantly higher values than other soil groups. This confirms the role of soil mineralogy on the efficiency of treatment with geopolymer.

The results presented in Figures 5.10 and 5.11 also indicate that the curing time is a key factor in the enhanced strength response of the treated clay soil. To investigate this matter further, the strength development index (SDI) is utilised (as defined in Chapter 4, Section 4.3). The SDI is plotted versus the curing time for clay soils treated with 20% geopolymer, as shown in Figure 5.12. All treated clays were found to exhibit

largely similar behaviour, so only the results for three of the engineered and two of the natural clays are presented herein. For the engineered clay specimens (Figure 5.12a), a sharp increase in SDI, ranging from 2.2 – 6.0, is observed over the first 7 day of curing. Increasing the curing time to 28 and 90 days further increases the SDI values to ranges of 3.54-9.72 and 5.49-14.42, respectively, indicating a progressive development of the cementation bonds within the treated clays with the increase of curing period (Cristelo et al. 2012; Liu et al. 2016; Phummiphan et al. 2016). The slightly reduced rate of SDI increase, which was observed after the curing time was extended to 28 and 90 days, indicate a slower rate of cementitious bond formation within treated soil with the increased curing period. This may be attributed to a potential reduction in the pH value than the originally induced by the geopolymer activator and will be considered subsequently. By comparison, the natural clay soils showed similar increasing trends in the SDI but less pronounced, as depicted in Figure 5.12(b). It is obvious that the strength development with curing time is unique for each clay type.

As evident from the above results, geopolymer content and curing time both influence the unconfined peak strength of geopolymer-treated clays. As a result, an empirical relationship likely exists between strength and stiffness. However, such correlation, would likely vary depending on the clay type due to the significant effect of clay characteristics on treatment, see Figure 5.12. Therefore, for design purposes, it is not practical to adopt a single stress relationship without considering clay type as the main variable. However, under a wide variability of clay characteristics in the field, promoting such a relationship is a difficult task to achieve, as so with this in mind, an empirical relationship between stiffness and strength for geopolymer-treated clays was not investigated.

5.4.3 Unconfined stiffness behaviour of geopolymer-treated clays

For a realistic estimation of soil stiffness, the effect of several factors needs to be considered including confinement effects, strain level and stress path. Therefore, for stiffness estimated from the unconfined condition, the value may not represent the actual case in the field. However, a parametric study on the factors affecting unconfined stiffness may assist for a general understanding on the impact of different experimental variables (i.e. geopolymer content, curing time and clay type) on the stiffness of geopolymer treated clay soils. Figure 5.13 shows the measured stiffness of

geopolymer treated clays. The stiffness is reported herein as E_{50} , i.e. secant modulus at 50% of peak strength. Generally, the stiffness of treated specimens was found to be enhanced with the addition of geopolymer and curing time in all clay soil types. The observed increase in E_{50} is primarily related to the geopolymer ability to bind the clay particles after hardening, ultimately enhancing the soil fabric stiffness, as explained by many researchers (e.g. Cristelo et al. 2011; Zhang et al. 2015).

Table 5.3: UCS characteristics of geopolymer-treated clays at various curing periods.

Clay	Geopolymer (%)	UCS (kPa)		
		7 days	28 days	90 days
100Kaolin	Untreated	196	196	196
	10	709	1102	1476
	15	1098	1800	2117
	20	1377	2101	3023
(90Kaolin-10Bentonite)	Untreated	260	260	260
	10	549	820	1150
	15	770	1307	1723
	20	1300	1935	2524
(80Kaolin-20Bentonite)	Untreated	270	270	270
	10	450	691	944
	15	712	1059	1547
	20	1106	1625	2129
(60Kaolin-40Bentonite)	Untreated	290	290	290
	10	465	627	916
	15	688	909	1452
	20	927	1316	1882
(90Kaolin-10Sand)	Untreated	250	196	250
	10	719	1072	1491
	15	1100	1824	2199
	20	1376	2213	3165
(80Kaolin-20Sand)	Untreated	270	270	270
	10	750	1219	1823
	15	1210	1957	2425
	20	1670	2484	3877
(60Kaolin-40Sand)	Untreated	370	370	370
	10	1022	1688	3063
	15	1392	2615	4573
	20	2051	3228	5125
(60Kaolin-40Sand)	Untreated	370	370	370
	10	731	1223	1541
	15	867	1511	1990
	20	895	1720	2573
	Untreated	275	275	275
	10	423	430	426
	15	571	706	747
	20	620	1187	1487

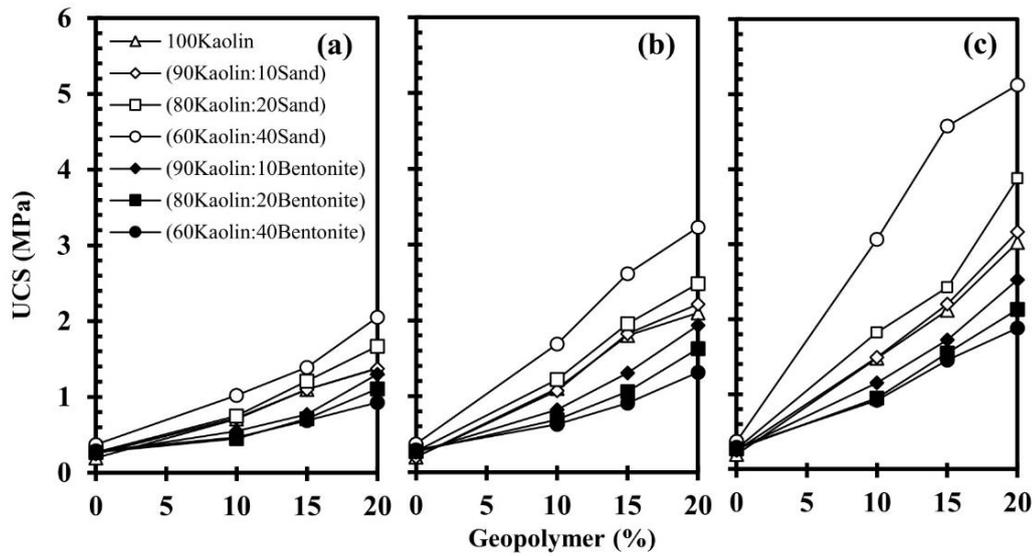


Figure 5.10: Effect of geopolymer content on strength gain of engineering clays at different curing periods: (a) 7 days; (b) 28 days; and (c) 90 days

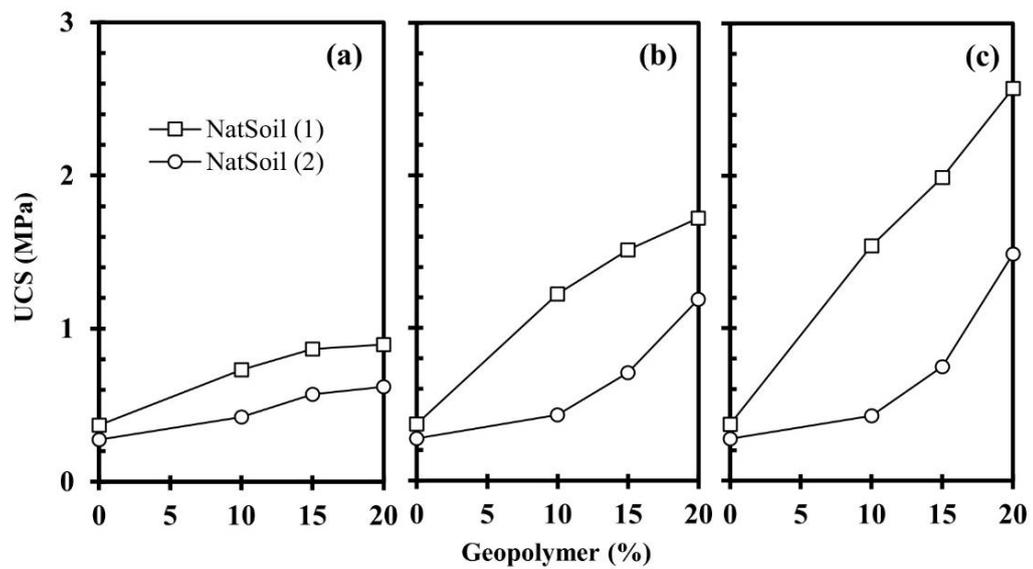


Figure 5.11: Effect of geopolymer content on strength gain of natural clays at different curing periods: (a) 7 days; (b) 28 days; and (c) 90 days.

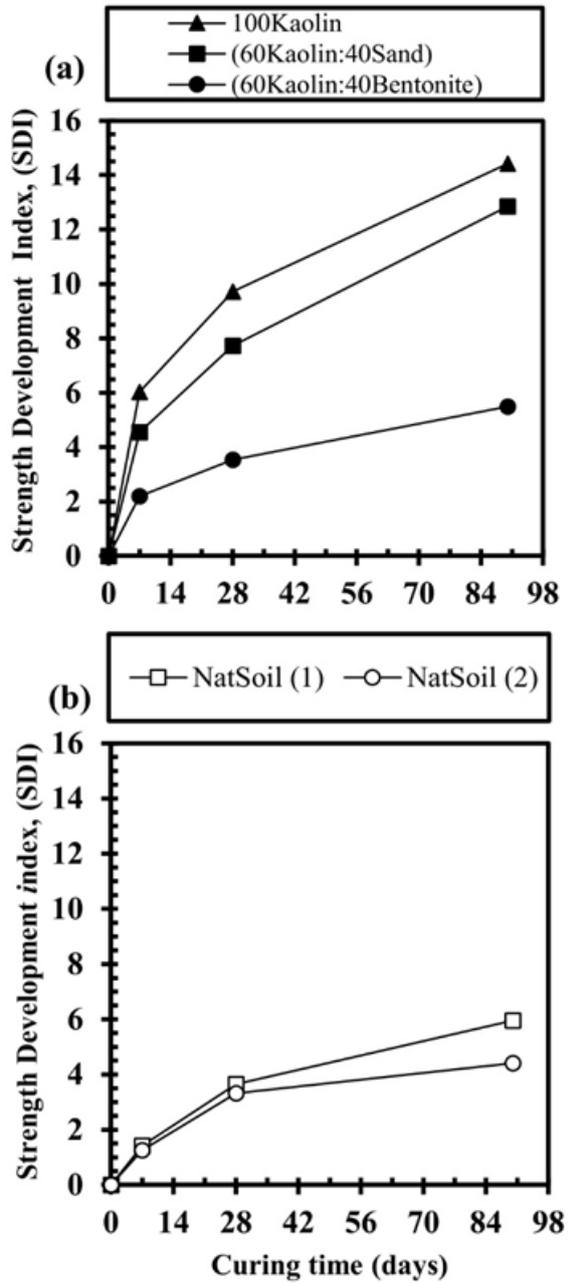


Figure 5.12: Relationship between Strength Development Index (SDI) and curing time for clays treated by 20% geopolimer: (a) engineered clays, (b) natural clays.

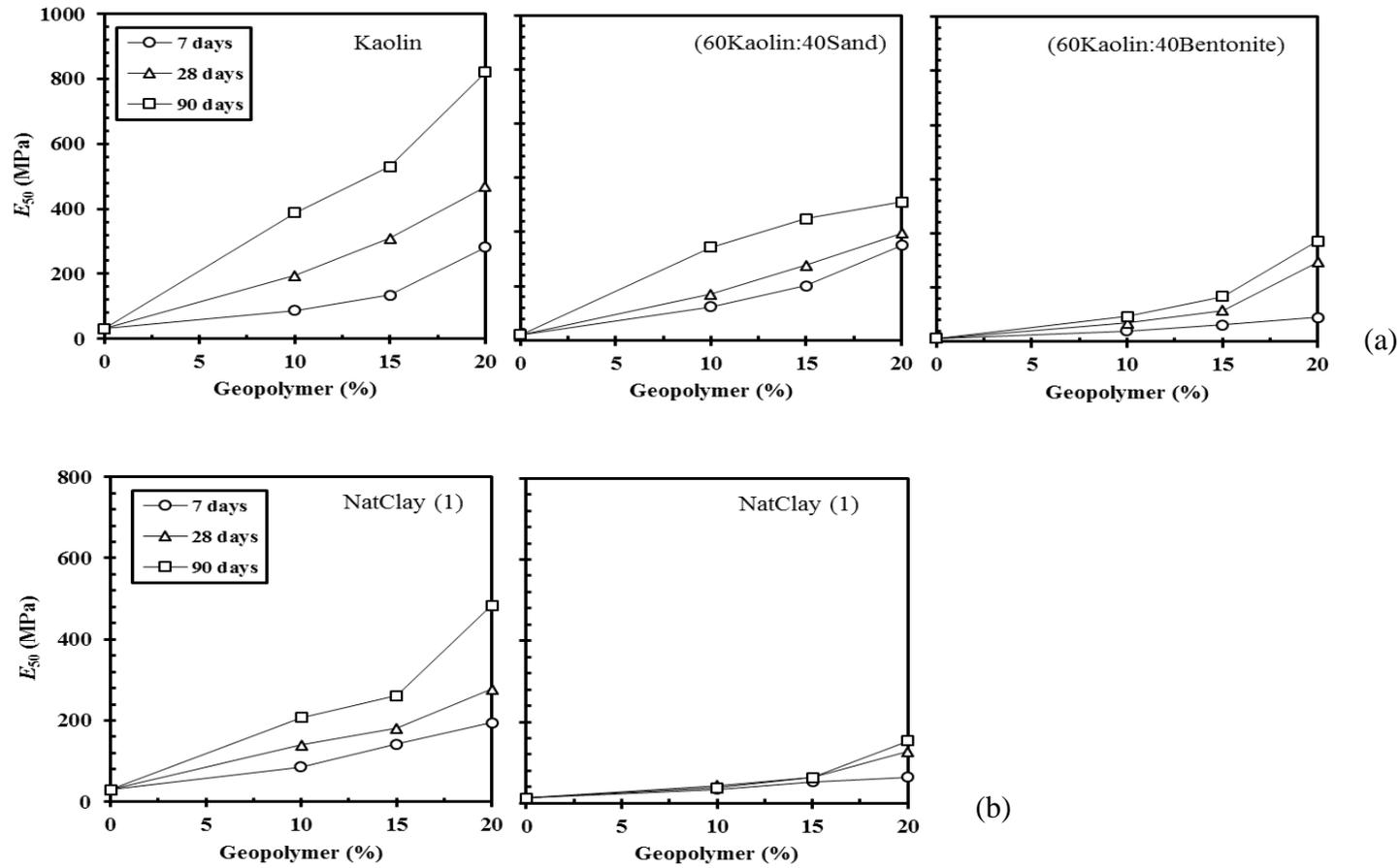


Figure 5.13: Typical values of E_{50} (secant modulus at 50% of peak strength) for some clays treated with various geopolymer contents cured up to 90 days: (a) engineered clays; and (b) natural clays.

5.5 DURABILITY PERFORMANCE OF GEOPOLYMER-TREATED CLAYS

Moisture and temperature effects are field-related issues that must be considered to ensure durable performance of a binder-soil mixture. As discussed in the literature review (Chapter 2, Section 2.4.1.3.4), limited information exist to evaluate the durability performance of geopolymer-treated clay soils. This section provides data and presents associated discussion concerning the effects of simulated weathering conditions (i.e. wetting-drying, freezing-thawing and leachate) on the performance of geopolymer-treated clays. Different clays were treated with 0%, 10%, 15% and 20% geopolymer content cured for 7 days. At the conclusion of the curing period, the various mixtures were tested for successive wetting-drying cycles. In this section, it was decided that limited testing on treated kaolin clay would be sufficient for determining the general trends of the effects of freezing-thawing and leachate on the geopolymer-treated mixture. For both wetting-drying and freezing-thawing tests, successfully treated samples were generally characterised by attaining the 12 wetting-drying cycles, and volume changes < 10% (Pedarla et al. 2011) with some residual strength. For leachate tests, residual strength was used as the governing criterion.

5.5.1 Wetting-drying tests

In tropical zones where extreme weather conditions occur, wetting-drying damage may result for treated soils. The wetting-drying test results for geopolymer-treated synthetic and natural clays are presented and evaluated in the following sections.

5.5.1.1 Number of cycles

Figure 5.14 shows the wetting-drying durability cycles achieved for each of the geopolymer-treated clay. It can be seen that at least 20% geopolymer dosage was necessary for the (100Kaolin) clay to attain the targeted 12 durability cycles. Such performance of this clay can be attributed to the formation of the stabilised matrix around the soil particles after hardening which resists the stresses generated during the successive wetting-drying cycles. However, for lower geopolymer contents (i.e. 10% and 15%), such level of treatment is shown to be inadequate with premature failure at earlier cycles. In contrast, the engineered specimens (80Kaolin:20Sand) and (60Kaolin:40Sand) showed an adequate response to treatment with low geopolymer contents, requiring only 15% and 10%, respectively, to resist the 12 durability cycles. Such enhancement for the kaolin-sand group appears to be associated with the

reduction in the clay minerals (i.e. kaoline) due to partial replacement by quartz. Additionally, the incorporation of sand particles in the engineering clay may decrease the overall surface area of clay and in turn the binder demand. Conversely, the partial replacement of kaoline minerals by montmorillonite of the kaolin-bentonite group showed an adverse effect, with no specimen sustaining more than three cycles. This suggests that the integrity and durability of the geopolymer matrix surrounding the agglomerates of kaolin-bentonite clay plates are compromised, resulting in significant matrix degradation. This confirms the impact of clay minerals on the treatment effectiveness of the engineered clay specimens, in which low plasticity/activity of clays show increased durability. This is also true for natural clays, where NatSoil (1) achieved stable durability performance with the addition of 15% and 20% of geopolymer, whereas NatSoil (2) exhibited significant material loss at the same concentrations during the first cycles indicating a premature durability failure and poor stabilisation.

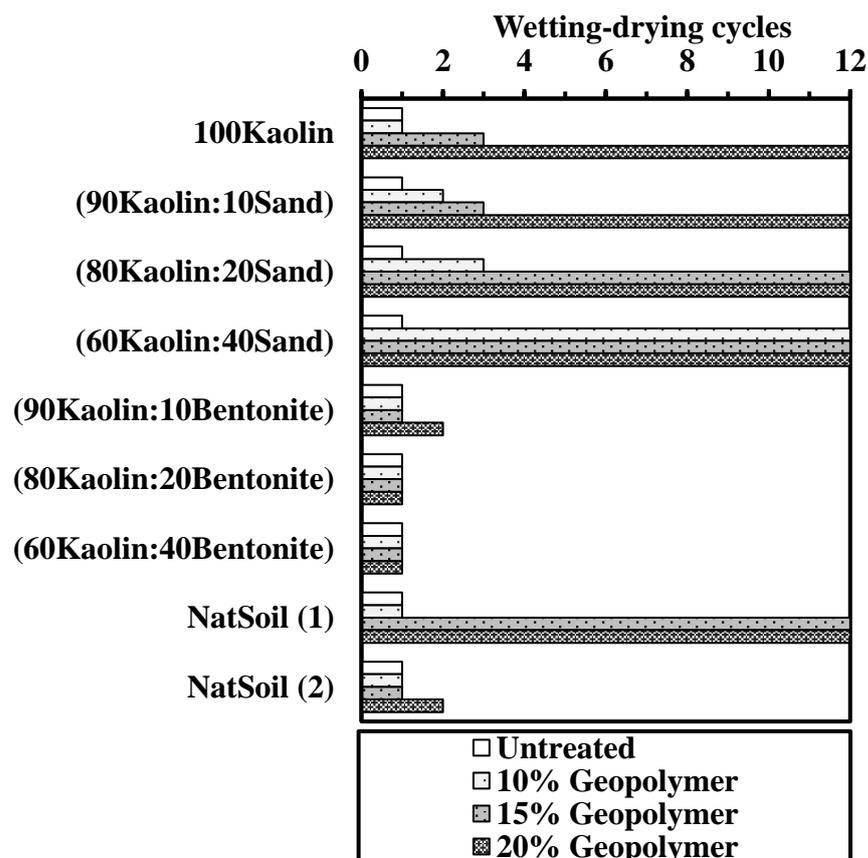


Figure 5.14: Wetting-drying durability cycles of different geopolymer-stabilised clay mixtures.

Figures 5.15 to 5.23 demonstrate a pictorial representation of the durability performance of the treated engineered and natural clays. The disintegration detected at low geopolymer content (i.e. 10%) in these clays (an example, kaolin clay Figure 5.15 b) can be explained by the limited formation of the cementitious products and clay clusters within the mass of treated clay as discussed above. At higher geopolymer content (i.e. 15% and 20%), the weak durability performance of some clays, i.e. Kaolin-Bentonite clay group (Figures 5.19 to 5.21) and NatSoil (2) (Figure 5.23) may be attributed to the highly problematic (expansive) nature of involved clay minerals. Such minerals seem to influence negatively the bonding between the clay particles, which in turn fail to resist the developed stresses within the specimen due to successive wetting-drying effects.



Figure 5.15: Changes of untreated and treated kaolin clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% geopolymer – 1st cycle; (c) 15% geopolymer – 3rd cycle; and (d) 20% geopolymer – 12th cycle.

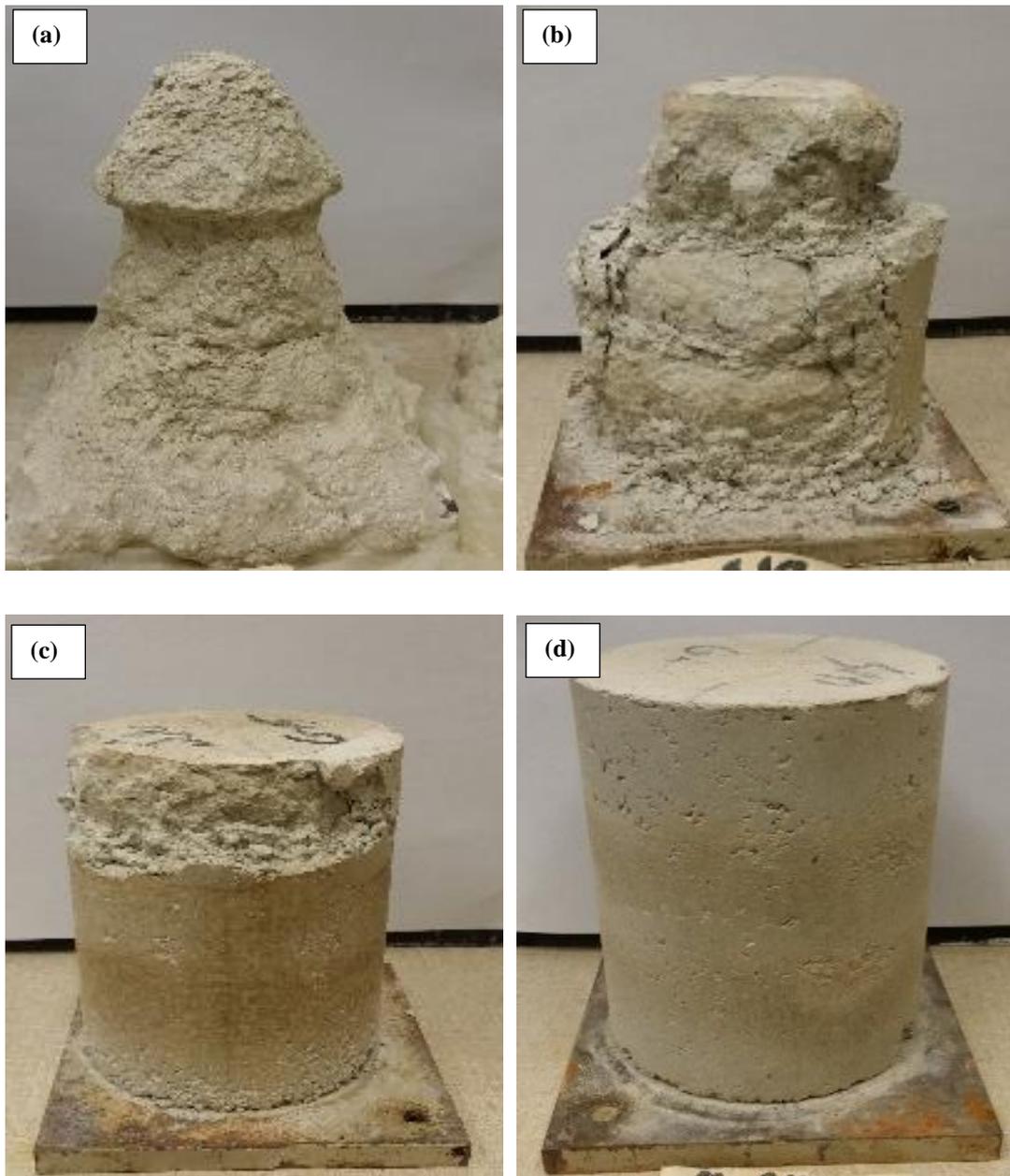


Figure 5.16: Changes of untreated and treated (90Kaolin-10Sand) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer – 2nd cycle; (c) 15% Geopolymer – 3rd cycle; and (d) 20% Geopolymer - 12 cycles.

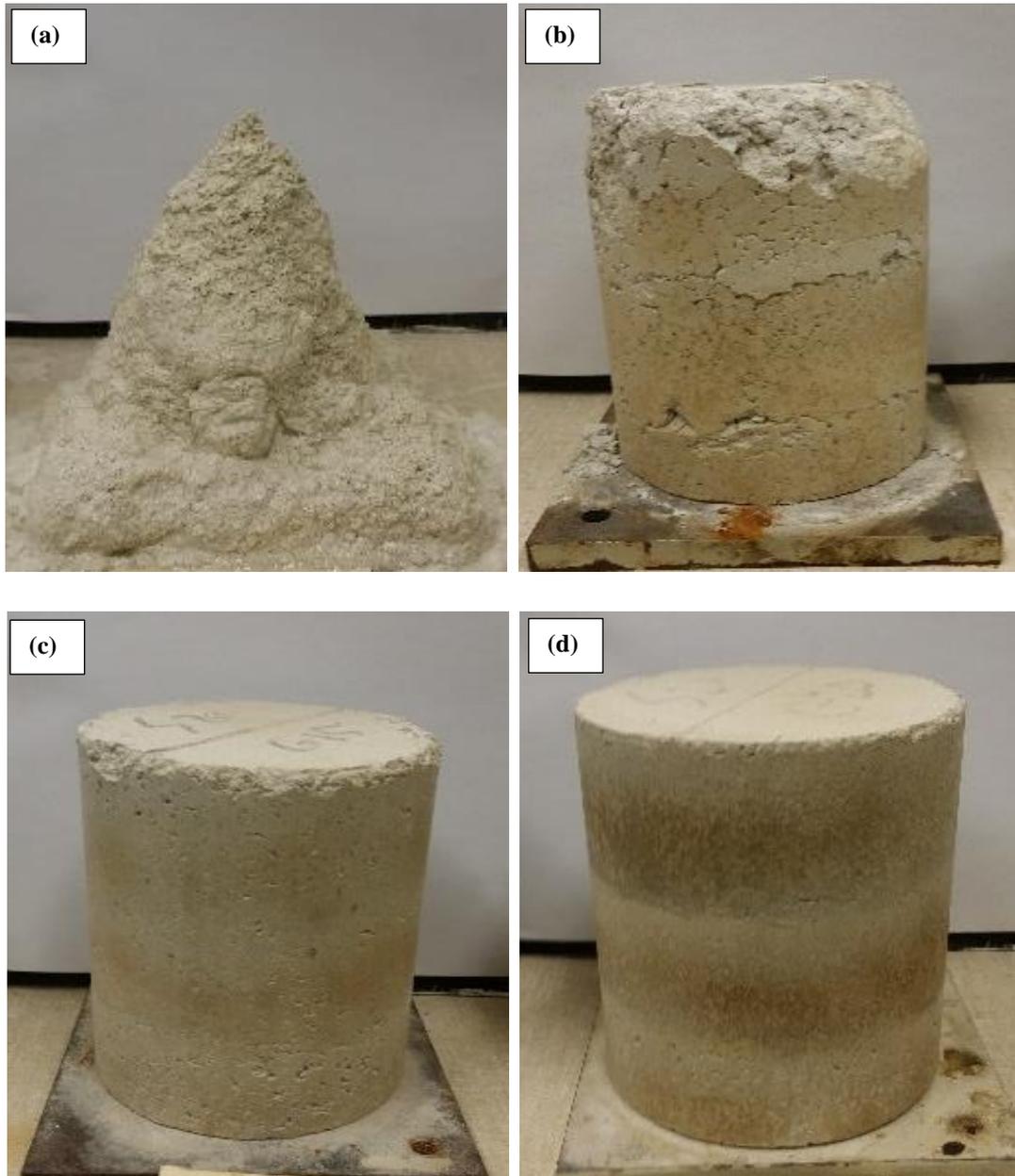


Figure 5.17: Changes of untreated and treated (80Kaolin-20Sand) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer – 3rd cycle; (c) 15% Geopolymer – 12th cycle; and (d) 20% Geopolymer – 12th cycle.

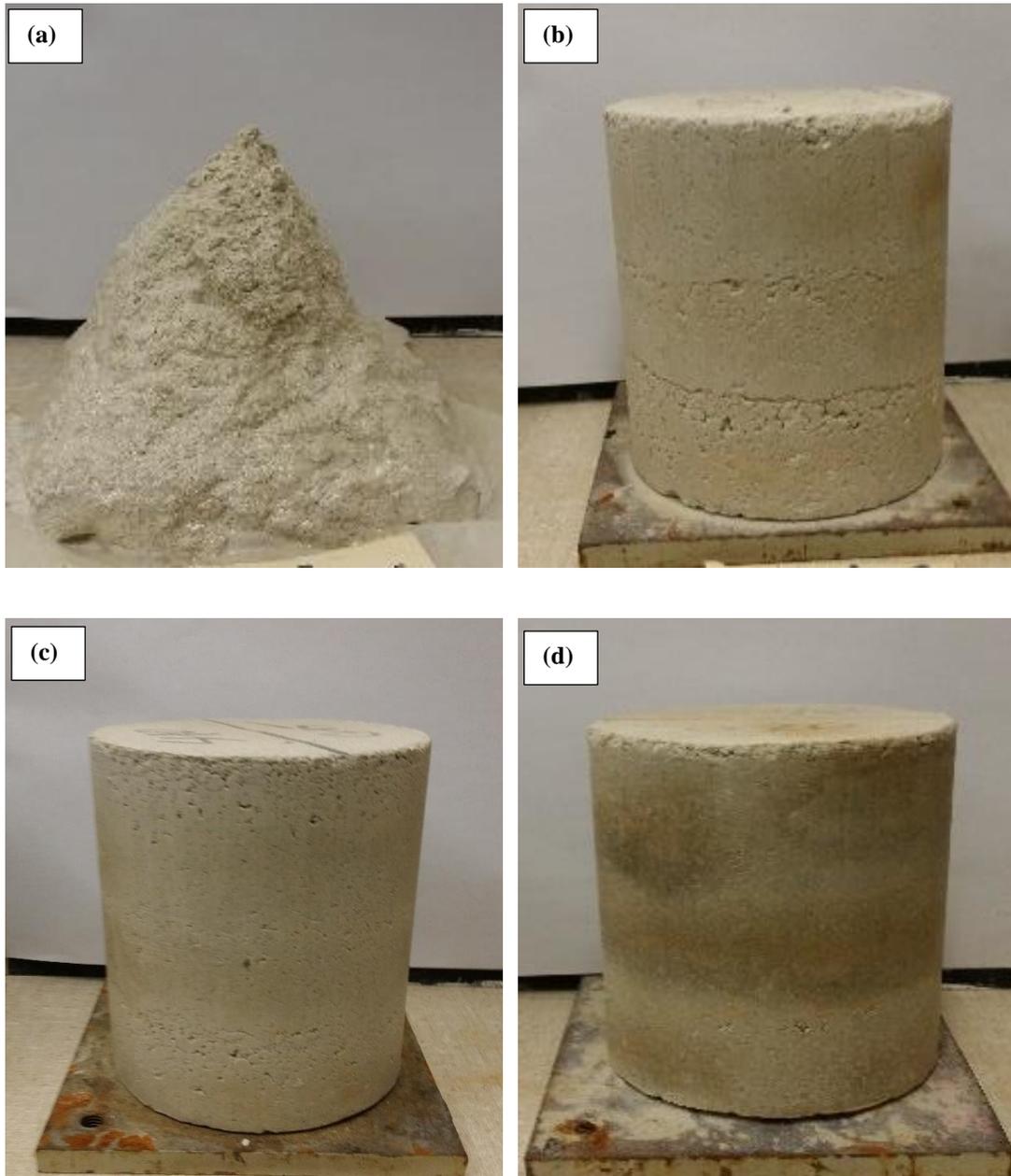


Figure 5.18: Changes of untreated and treated (60Kaolin-40Sand) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer – 12th cycle; (c) 15% Geopolymer – 12th cycle; and (d) 20% Geopolymer – 12th cycle.

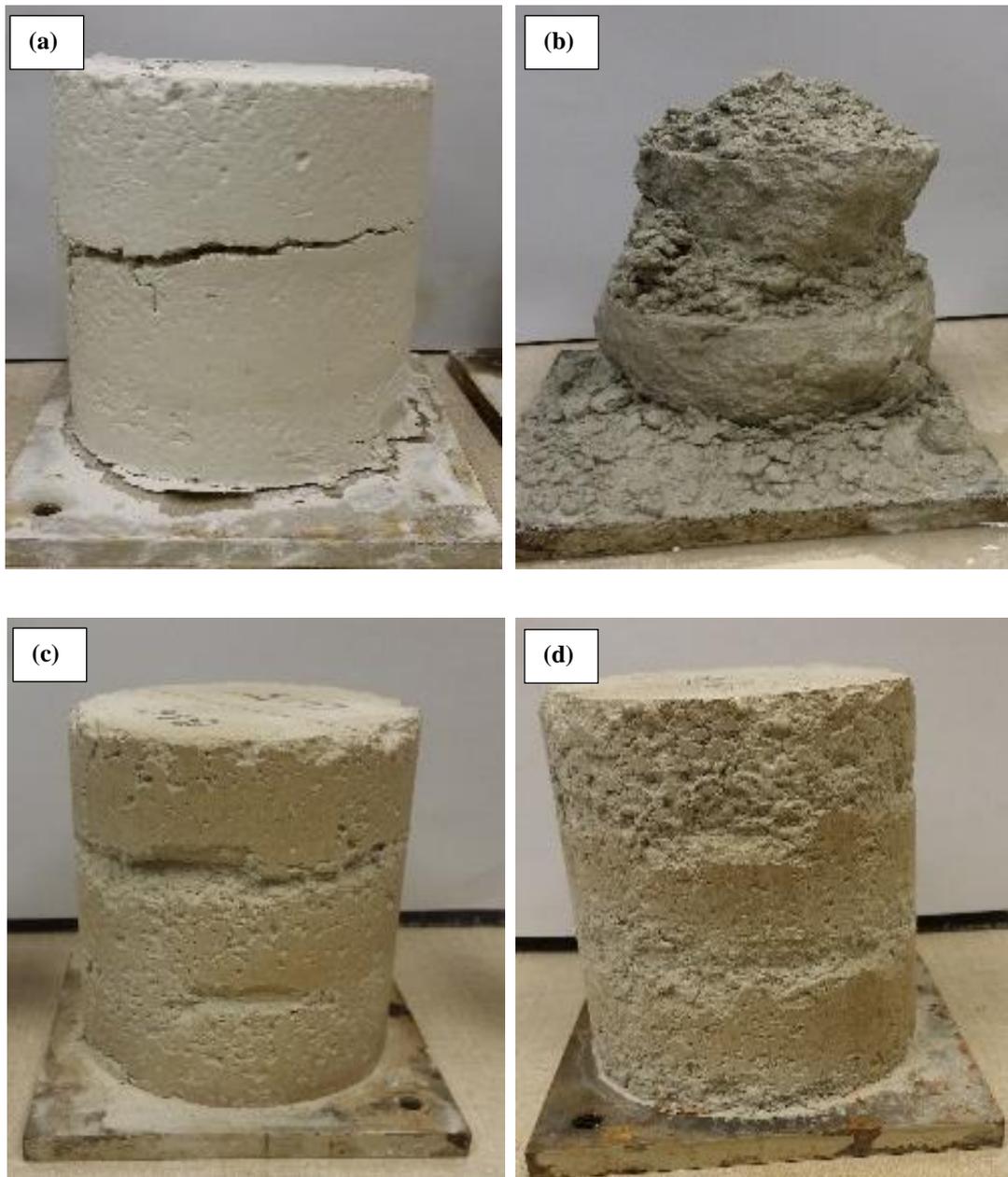


Figure 5.19: Changes of untreated and treated (90Kaolin-10Bentonite) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer – 1st cycle; (c) 15% Geopolymer – 1st cycle; and (d) 20% Geopolymer – 2nd cycle.

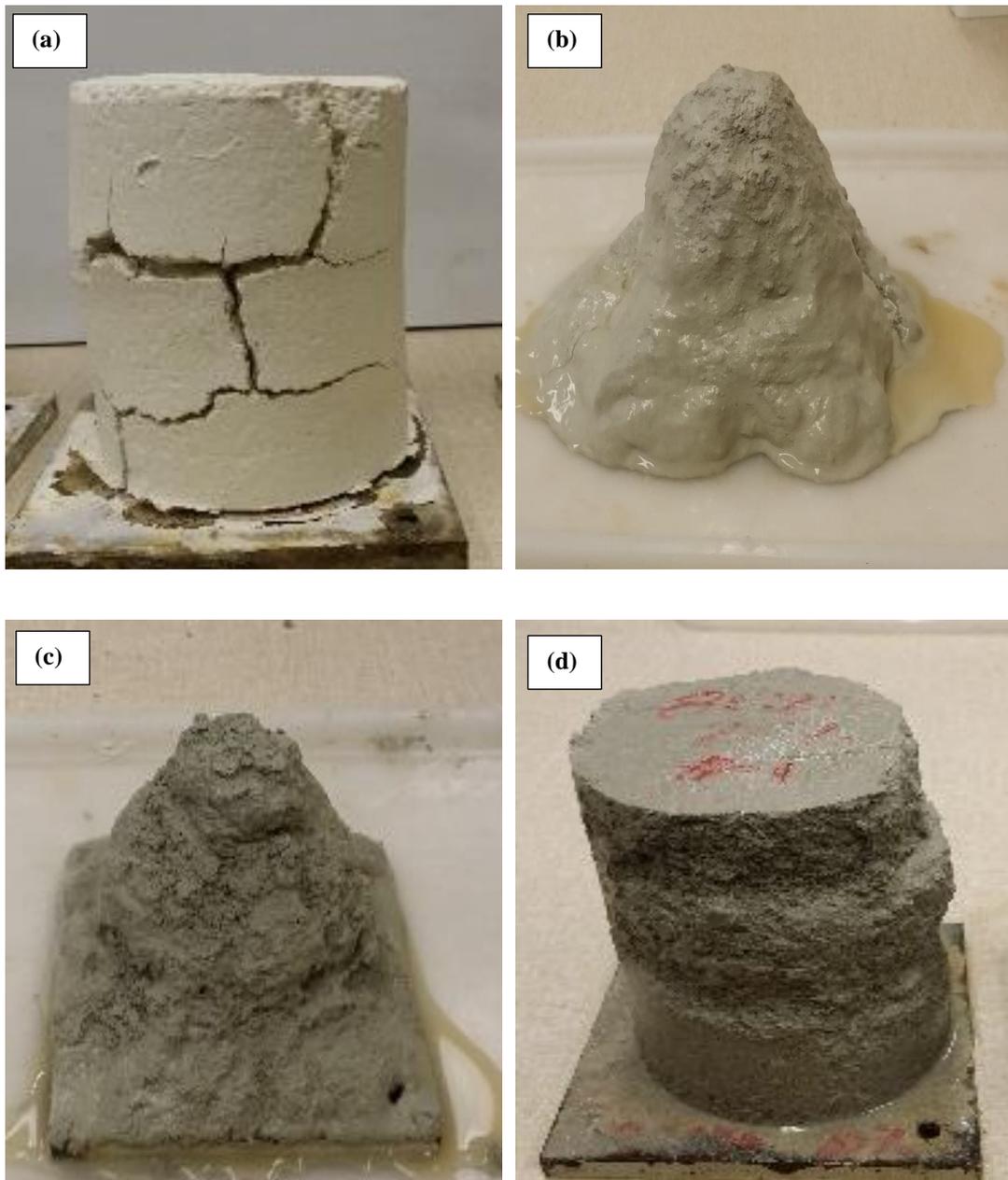


Figure 5.20: Changes of untreated and treated (80Kaolin-20Bentonite) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer – 1st cycle; (c) 15% Geopolymer – 1st cycle; and (d) 20% Geopolymer – 1st cycle.

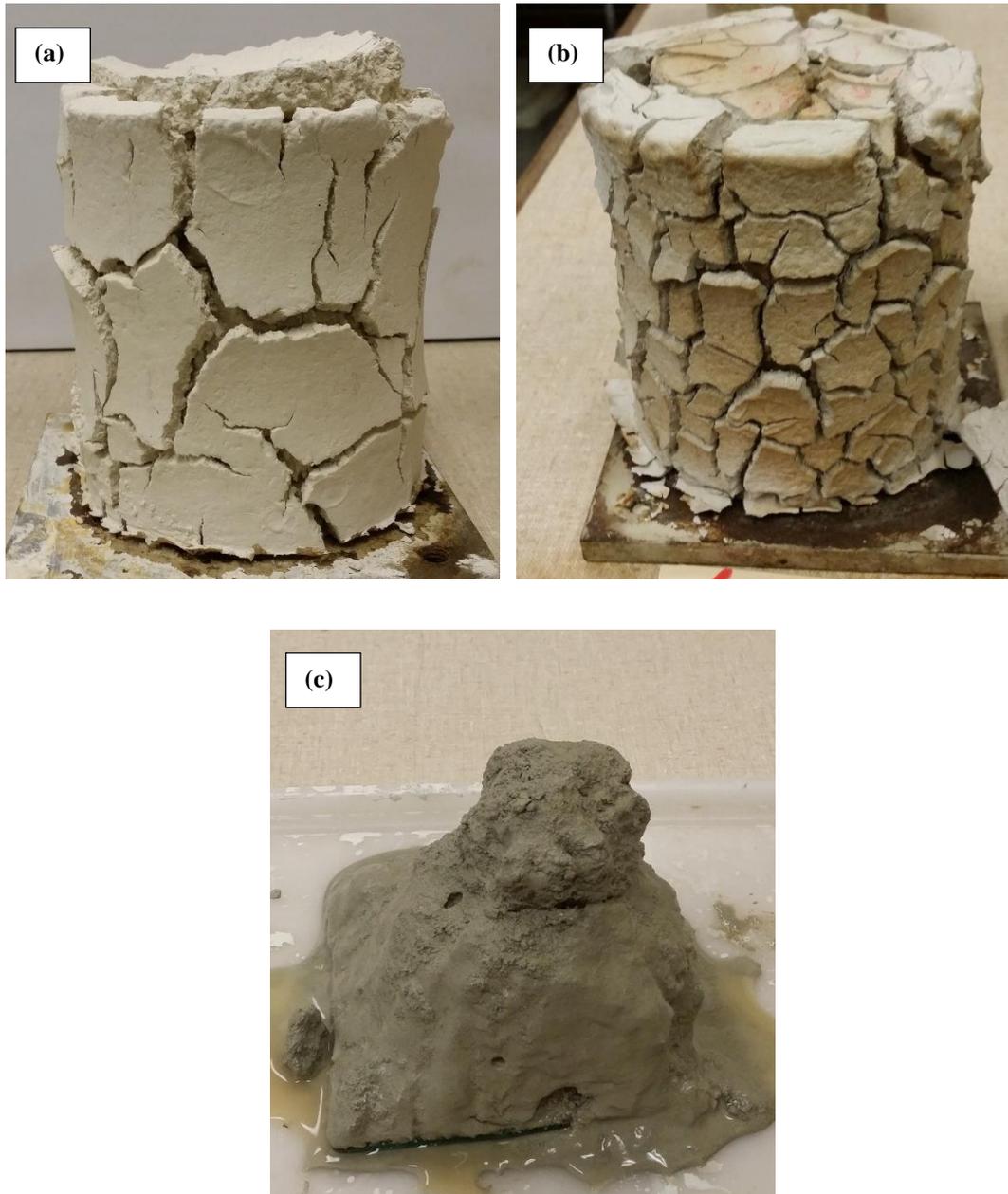


Figure 5.21: Changes of untreated and treated (60Kaolin-40Bentonite) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer - 1st cycle; and (c) 15&20% Geopolymer - 2nd cycle.

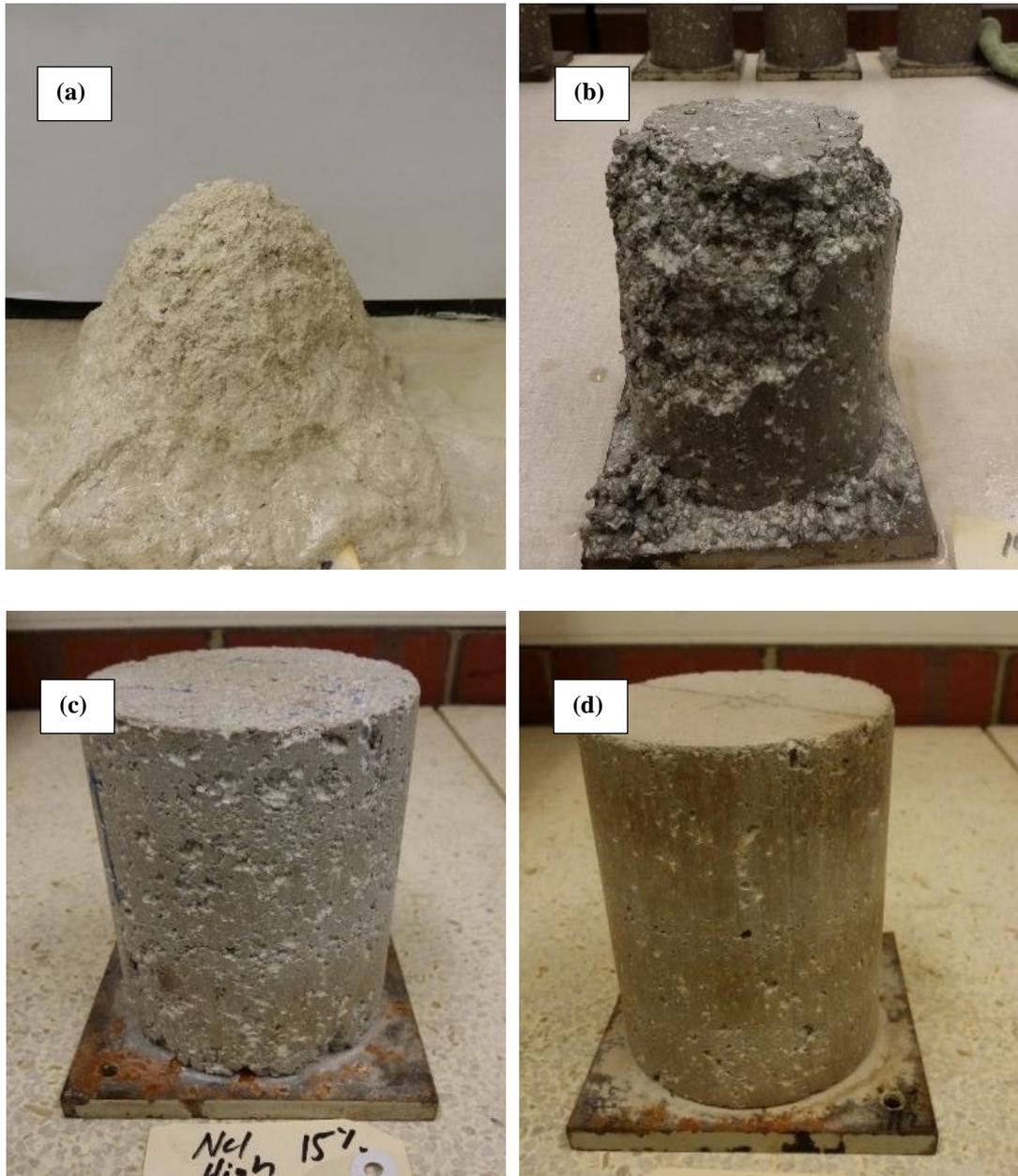


Figure 5.22: Changes of untreated and treated NatSoil (1) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% geopolymer – 1st cycle; (c) 15% Geopolymer – 12th cycle; and (d) 20% Geopolymer – 12th cycle.



Figure 5.23: Changes of untreated and treated NatSoil (2) clay samples subjected to different wetting-drying cycles: (a) Untreated - 1st cycle; (b) 10% Geopolymer – 1st cycle; (c) 15% Geopolymer – 1st cycle; and (d) 20% Geopolymer – 1st cycle.

5.5.1.2 Volumetric changes

Durable engineered and natural treated clays, i.e. those survived the 12 wetting-drying, were examined for the volumetric changes and the results are shown in Table 5.4. Generally, the surviving samples showed very stable performance throughout the 12 wetting-drying durability cycles with an insignificant volumetric change of less than 0.8%, which is considerably less than the 10% threshold considered to distinguish the volumetric stability limit in chemically treated soils, as mentioned by Pedarla et al.

(2011). As can be seen in Table 5.4, Kaolin and (90Kaolin:10Sand) displayed very low volumetric changes of 0.4% at 20% geopolymer content. At 10% and 15% treated kaolin mixtures, volumetric measurements were discontinued due to the excessive loss of material at earlier cycles. The small percentage of volumetric changes observed for (80Kaolin:20Sand) and (60Kaolin:40Sand) clays after 12 cycles of wetting-drying indicated that these mixtures were very durable at 15% and 10%, respectively. NatSoil (1) also displayed substantial resistance to volumetric changes; with 15% geopolymer content < 10% volume change occurred after 12 cycles.

Table 5.4: Wetting-drying volumetric changes after 12 cycles for engineered and natural treated clays.

Clay	Geopolymer (%)	Volumetric change (%)	
		wetting phase	drying phase
(100Kaolin)	10	-	-
	15	-	-
	20	0.4	0.12
(90Kaolin-10Sand)	10	-	-
	15	-	-
	20	0.5	0.11
(80Kaolin-20Sand)	10	-	-
	15	0.42	0.15
	20	0.33	0.1
(60Kaolin-40Sand)	10	0.71	0.19
	15	0.51	0.11
	20	0.42	0.05
NatSoil (1)	10	-	-
	15	0.40	0.07
	20	0.64	0.05

5.5.1.3 Residual strength

To examine strength degradation for wetting-drying cycles of survived treated clays, UCS testing was carried out on the 20% geopolymer-treated clay specimens after 3, 6, 9 and 12 durability cycles (Figure 5.24). For (100Kaolin) clay, a degradation trend was detected with an overall value of 33% after 12 cycles, whereas for the kaolin-sand group and NatSoil (1), the strength showed an increasing trend between the 3rd and 6th cycles, after which a slight degradation occurred. This may indicate a possible

continuation of the geopolymerisation process and the corresponding attributed gel formation/hardening for the low plasticity clays due to the heat effect of the durability-drying phase, followed by degradation in the stabilised soil structure due to the accumulative effects of the durability cycles. It should be noted that, as explained by Palomo et al. (1999), heat curing accelerates the geopolymerisation and the subsequent strength gain. This suggests a potential advantage of the geopolymer use for surface-soil mixing where a significant fluctuation in the sub-surface moisture levels exists.

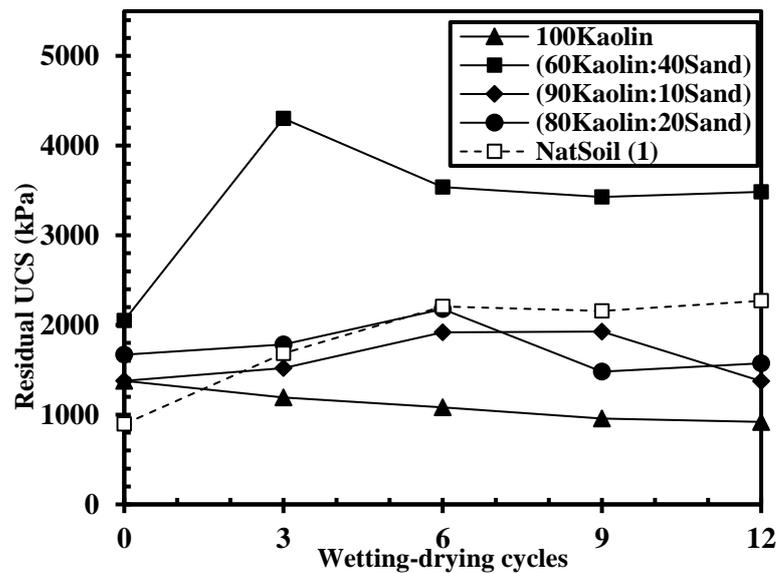


Figure 5.24: Influence of wetting-drying durability cycles on UCS values of clays treated with 20% geopolymer content.

5.5.2 Freezing-thawing tests

Based on trial testing, it was decided that testing of kaolin clay only would be sufficient to determine the general trends of freezing-thawing impact on the geopolymer-treated mixtures. Therefore, three kaolin samples with 10%, 15% and 20% of geopolymer content were considered for the freezing-thawing tests. Similarly to the wetting-drying tests, the number of cycles, volumetric change and residual strength were the criteria used for evaluating the freezing-thawing durability performance of geopolymer-clay mixtures. In general, all treated mixtures exhibited premature failure and were not able to sustain all 12 cycles of freezing-thawing. Treated mixtures displayed high volumetric changes after each cycle: for 20% geopolymer content mixture, high volumetric change of up to 20% was detected at the 6th cycle of the test, which is significantly higher than that of the 10% threshold considered to distinguish stability

limit in treated soils, as mentioned by Pedarla et al. (2011). The high volumetric change in the treated mixtures can be related to the expansion of pore water due to the freezing effect. Such expansion affects the artificial cementitious bonds negatively within the clay matrix resulting in a significant distortion and cracking in the structure of the treated clay, as shown in Figure 5.25. With successive freezing-thawing cycles, all specimens exhibited weaker structure and strength. This is clearly evident from the strength performance depicted in Figure 5.26.

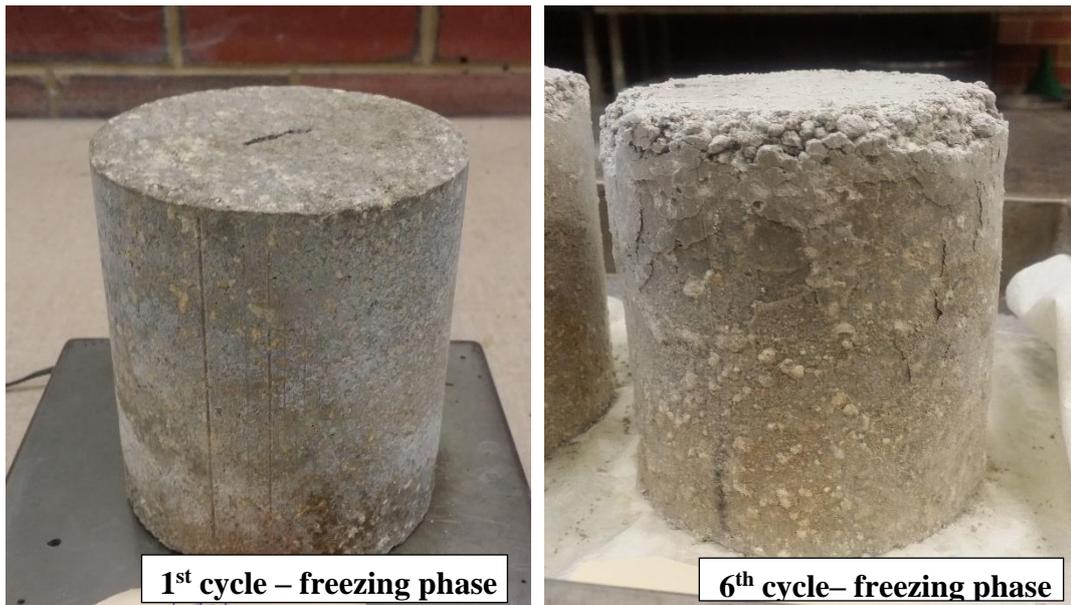


Figure 5.25: Volumetric changes of treated clay subjected to 6 successive cycles of freezing-thawing (20% geopolymer content).

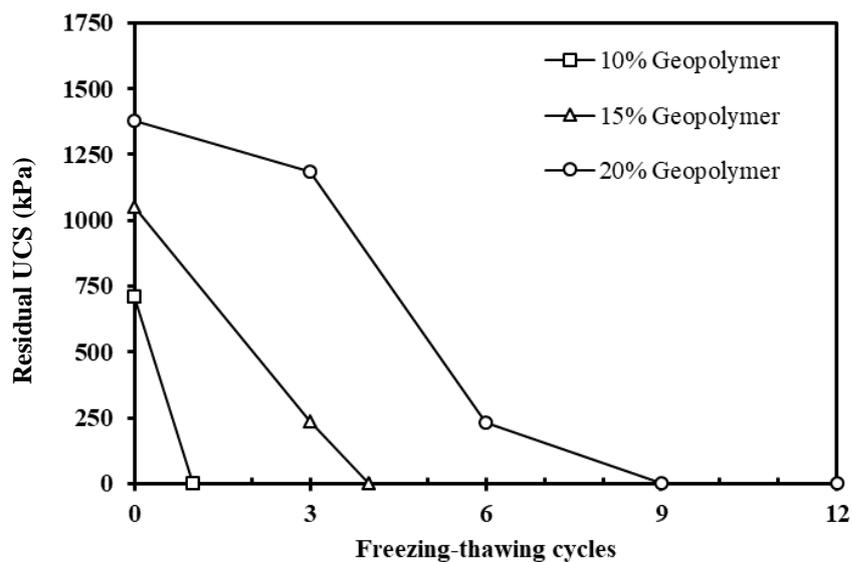


Figure 5.26: Influence of freezing-thawing durability cycles on UCS values for geopolymer-stabilised clay

It can be seen from Figure 5.26 that the unconfined strength of all treated mixtures is sharply affected by the increase in the freezing-thawing cycles. At 20% geopolymers content, a significant degradation in soil strength was observed only after the 3rd cycle. For lower geopolymers contents, no appreciable strength was observed after the 1st cycle of the test. Unlike the wetting-drying performance, treated specimens did not show progressive strength increase during the test cycles. This may indicate retardation in the geopolymers reaction at very low temperature. The overall degradation in the strength performance indicates a destructive impact of the freezing-thawing conditions.

5.5.3 Leaching tests

Compacted specimens treated with 20% geopolymers cured for 7 days were leached with pressurised water in the special leaching cell described earlier (Chapter 3, Section 3.4.2.3) for a number of cycles between 1–9. After a designated number of cycles, the corresponding residual pH values and the residual unconfined compressive strength were determined for the stabilised clay. Figure 5.27 shows the soil pH and UCS values compared against leaching. It can be seen that the pH value of the treated clay is decreased from 12.95 to 12.03, representing a total reduction of 7%, through leaching up to 3 cycles before stabilising at higher cycles. This may imply a reduction in the rate of the geopolymers reaction and the corresponding strength gain. However, as can be seen in Figure 5.27 (left axis), the overall strength of geopolymers-stabilised clay against the number of leaching cycles is not significantly affected by the leaching cycles, and the residual strength after 9 cycles was found to be 90% of the initial strength at zero cycle. This is in agreement with the findings obtained by Chittoori and Puppala (2013) for different leached clay types treated with lime and cement. Interestingly, a continuous development does exist up to the 3rd cycles and the degradation of strength occurs between the 3rd and 6th cycles before remaining approximately constant at further cycles. This can be related to the combined effects of water infiltration on the clay structure and the change of alkalinity concentration which affecting the solubility rate of the amorphous silica and alumina of the fly-ash particles. In light of this, the activator leaching may be a minor concern in geopolymers clay stabilisation during the initial curing period where it may disturb gel formation, and in turn, the targeted strength gain rate. To avoid any activator loss, field protection

from water infiltration (e.g. rainfall or groundwater) may be needed, for curing time up to 28 days.

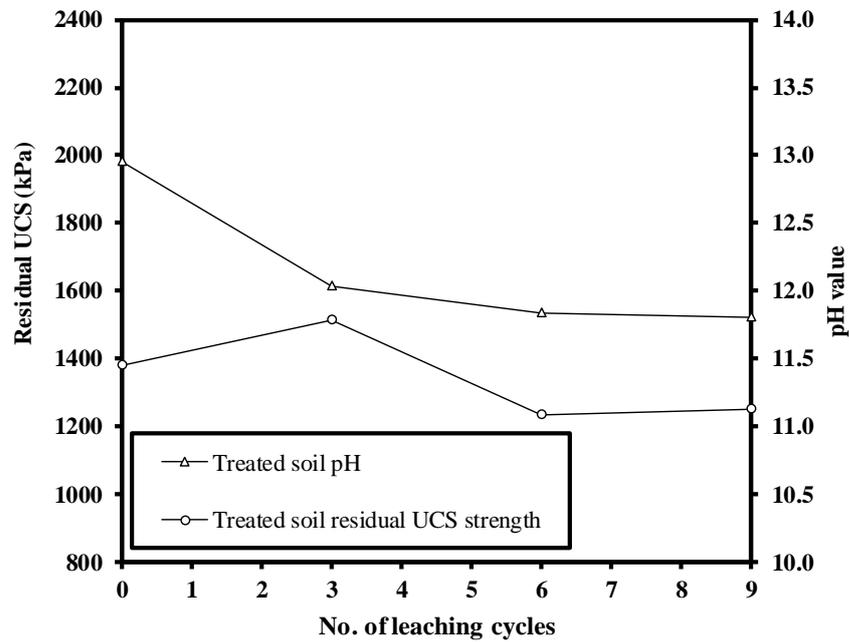


Figure 5.27: Residual strength and pH against leaching cycles of geopolymer-stabilised clay.

5.6 PERMEABILITY OF GEOPOLYMER-TREATED CLAYS

This section explores the ability of geopolymer-treated clay soils to transmit water through conducting a series of permeability tests. It was decided that testing on kaolin clay would be sufficient to determine the general trends. Compacted specimens of untreated and treated kaolin clay with 10%, 15% and 20 % geopolymer contents cured for 28 days were subjected to water flow in the permeameter cell described earlier in Chapter 3, Section 3.4.5. Figure 5.28 shows the coefficient of permeability (k) of different mixtures against curing time.

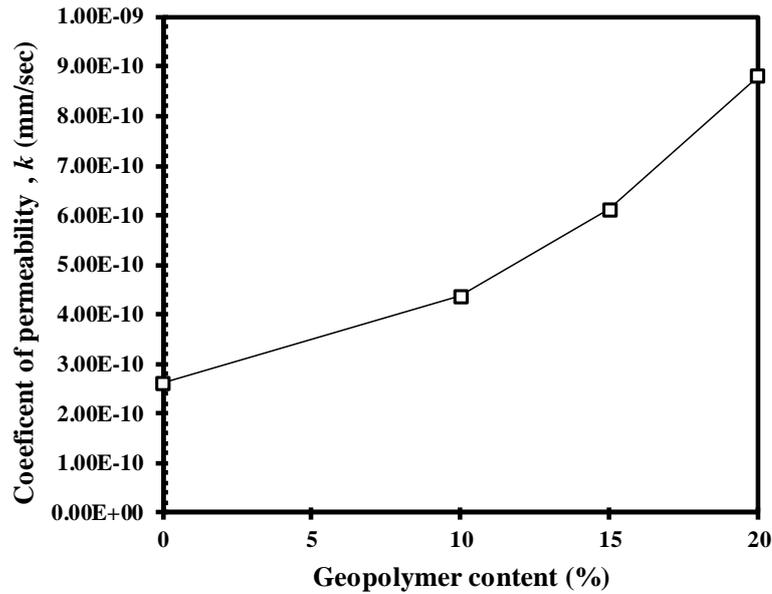


Figure 5.28: Coefficient of permeability of kaolin clay treated with various geopolymer contents.

It can be seen from Figure 5.28 that the initial k value of untreated clay increases with the increasing geopolymer content. Untreated kaolin clay shows the lowest k value. At 10% geopolymer content, the k value increases 1.7 fold. As the geopolymer content increases to 15% and 20%, the k value is associated with more increase of 2.3 and 3.3 fold, successively. Such an increase in k value with the increase of geopolymer content for treated clay soil contradicts, to some extent, the effect of ordinary Portland cement (OPC) and lime treated soils. It has been reported in the literature that the permeability of cement and lime-treated clay is almost identical to that of untreated clay but decreases significantly when the binder content exceeds a certain threshold i.e. 8% for OPC and 4% for lime (Quang et al. 2015). This is attributed to the fact that the OPC and lime-cementitious bonds developed in the mixture at high binder concentration, which bind the soil particles together into clusters with small intra-aggregate pore size reducing the permeability within the clusters and the overall treated soil. However, this might also be true for geopolymer-treated soil but adversely influenced more by the effect of the non-plastic silty fly-ash and GGBFS particles. Due to the partial replacement of clay by additives during treatment, the silty characteristics of the fly-ash and GGBFS particles in the geopolymer may reduce the overall surface area of the particles within the mixture (as discussed previously in Sections 5.2 and 5.3) and thus increase the pore sizes and eventually the permeability of the treated clay. It should be

noted that the presence of activator chemicals within the soil voids likely contributes also to the properties of the permeating water producing higher permeability. Major influencing properties of the permeating liquid affecting (increasing) permeability are the increase of ionic strength of the solutes (Madsen et al. 1989) and its dielectric constant (Fernandez et al. 1985), which is the case for geopolymer treatment.

5.7 IMPACT OF SOIL PULVERISATION ON STRENGTH AND DURABILITY

To assess appropriate pulverisation criteria for geopolymer-treated clays, the performance of the two natural clays of different pulverisation levels was investigated. Figures 5.29 to 5.31 show typical stress-strain curves, SDI values and residual UCS values, respectively, for 20% geopolymer treated NatSoil (1) and NatSoil (2) at different pulverisation levels (i.e. high, medium and low) at selected wetting-drying cycles.

Overall, the enhancement of treated clay is significantly impacted by the variation in pulverisation level. Figure 5.29(a) shows that a change in the pulverisation from low (i.e. 60% < 4.75 mm sieve) to medium (i.e. 80% < 4.75 mm sieve) increases the peak strength of NatSoil (1) at 28 days by about 9 %, whereas for higher pulverisation level (i.e. 100% < 4.74 mm sieve) a total increase up to 21% was detected. Similar results were detected for NatSoil (2), as shown in Figure 5.29(b). It is worthwhile noting that the reduction in the UCS peak values due to changes in the pulverisation level is also associated with a reduction in stiffness, as seen by the variations in the initial gradients of the UCS curves in Figure 5.29. However, the effect of pulverisation on strength seems to be more pronounced at the long-term curing periods (i.e. ≥ 28 days), as depicted in Figure 5.30, where the SDI values show a relatively higher variation at such advanced curing period than for 7 days.

With the successful wetting-drying durability performance for NatSoil (1) treated with 20% geopolymer content, the effect of pulverisation of clay was evaluated. In terms of the durability performance, the pulverisation level showed no pronounced influence on the ability of the treated clay to withstand wetting-drying cycles; all specimens were able to complete 12 cycles irrespective of the pulverisation level, as shown in Figure 5.31. However, a slight loss of material was detected throughout the successive wetting-drying cycles for mixtures of medium and low pulverisation; thus a variation

in the UCS values was expected for the durability cycles. The high-pulverised geopolymer-treated clays showed a significant increase in the UCS values up to 147% within the first six durability cycles, before UCS values stabilising for the remaining six cycles. However, for medium and low pulverisation levels, the residual UCS showed less pronounced increased before a significant reduction in strength after six cycles. Based on these findings, the pulverisation level of clay, as a construction factor, should be regarded as a limitation for an effective geopolymer treatment and thus must be included in any geopolymer stabilisation guidelines. Considering both the practicality and difficulties in achieving 100% pulverisation of heavy clay soils in the field, medium pulverisation quality may be considered a reasonable limit.

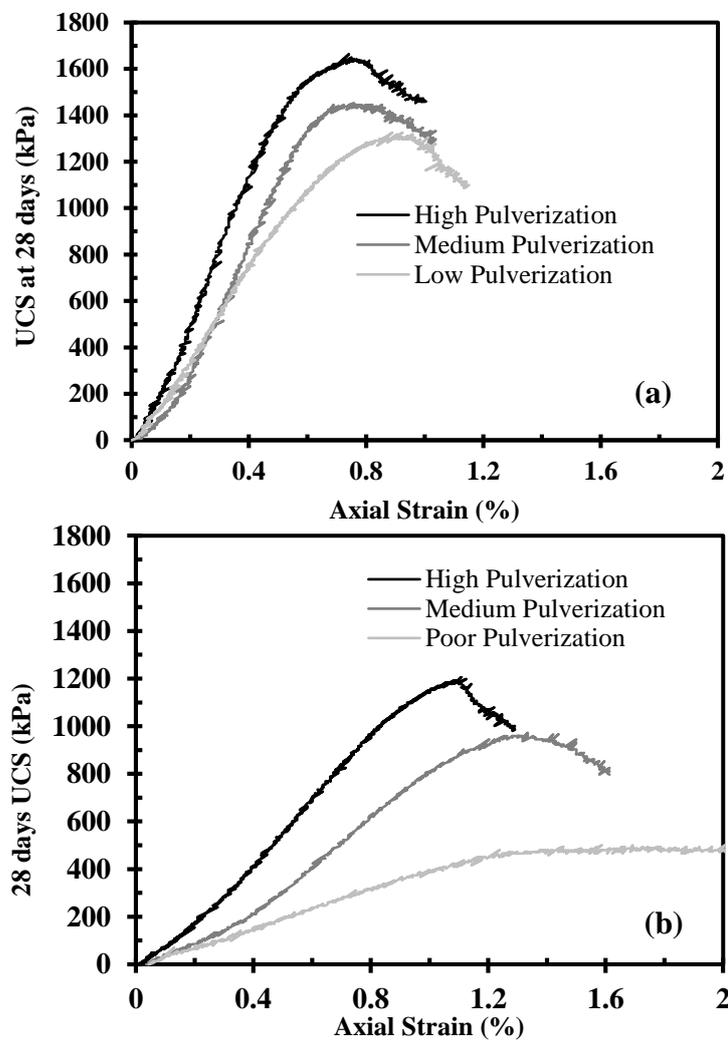


Figure 5.29: Pulverisation effects on the stress-strain response of natural clays treated with 20% typical geopolymer content and cured for 28 days: (a) NatSoil (1); and (b) NatSoil (2).

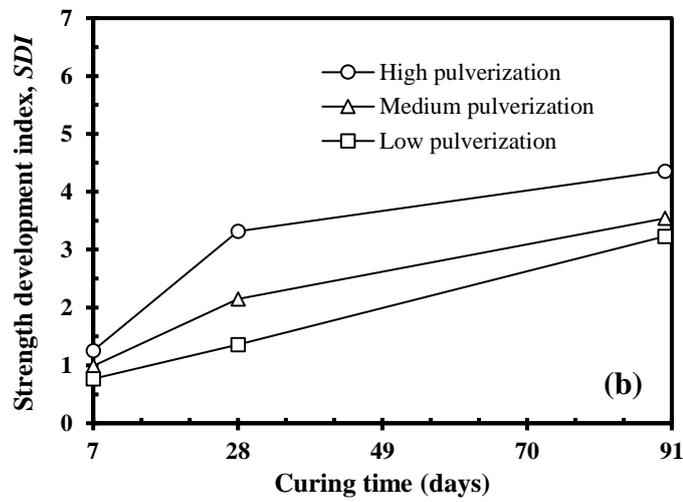
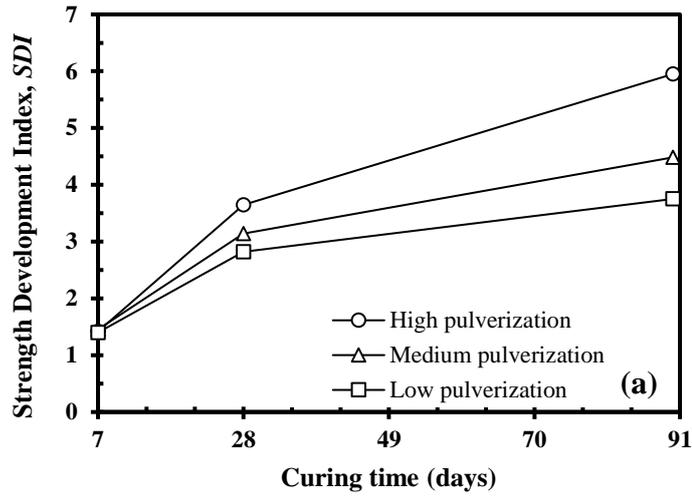


Figure 5.30: Pulverisation effects on SDI of natural clays treated with typical geopolymer content (20%): (a) NatSoil (1); and (b) NatSoil (2).

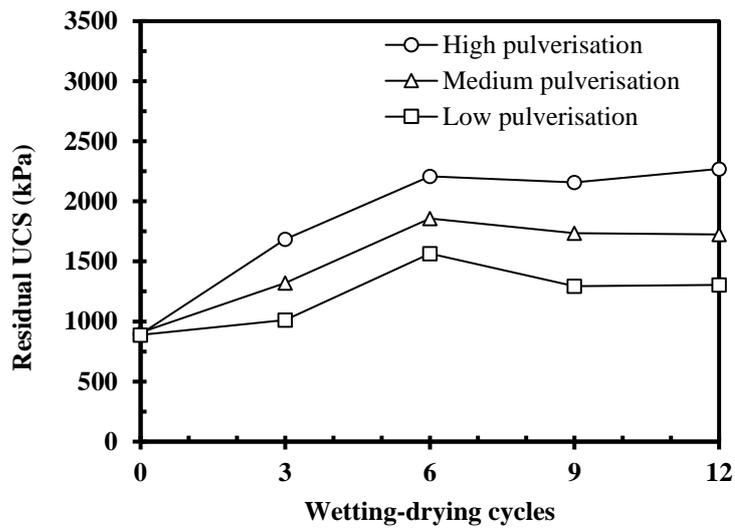


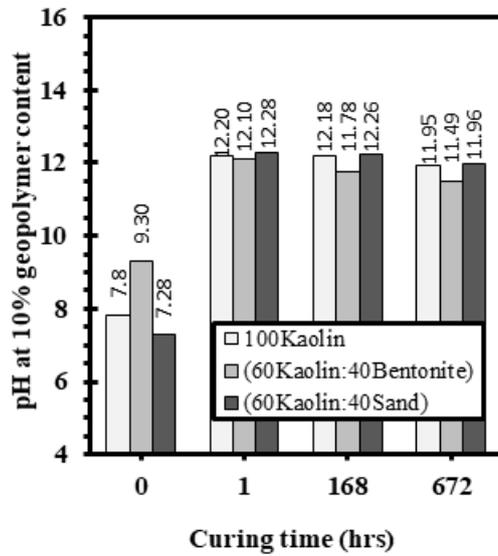
Figure 5.31: Pulverisation effects on wetting-drying durability tests of NatSoil (1) treated with typical geopolymer content (20%).

5.8 RESPONSE OF STABILISED SOIL TO pH

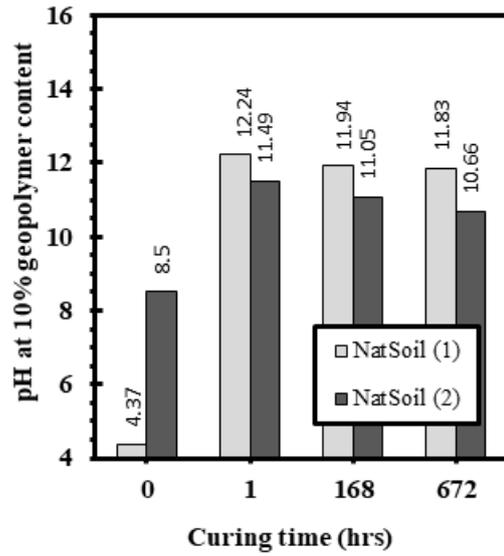
Hydroxide ions (OH^-) in the activation solution induce hydrolysis of silicate and aluminate bonds in the geopolymer (specifically the fly-ash and GGBFS components), by increasing the pH level to facilitate an initial dissolution and condensation reactions. A pH of 11 is the minimum level required to maintain the geopolymerisation reaction, as reported by Garcia-Lodeiro et al. (2014). As such, the pH level plays an essential role in the stabilisation of soils with geopolymer, similar to the effect of pH described widely in the lime/fly-ash treated clay literature (Little 1995).

In addition to determining the pH of treated soils during leaching test (Section 5.5.3), a more detailed investigation of the pH performance was conducted on the geopolymer-treated clays, specifically to investigate the compliance of geopolymer-clay treatment with the requirement of $\text{pH} > 11$. For this investigation, the pH level of three representative engineered clays and two natural clays were examined for 10% and 20% geopolymer, over curing periods of 1 hour, 168 hours (7 days) and 678 hours (28 days), as shown in Figure 5.32. After 1 hour of curing, it can be seen that the addition of geopolymer tends to increase the clay pH considerably. Specifically, an addition of 10% geopolymer increases the pH of the soil (100Kaolin) after 1 hour of curing from 7.8 to 12.2. Comparable increases in the pH level was observed for the other engineered clay specimens and NatSoil(1). However, NatSoil(2) showed a significant increase after 1 hour beyond that demonstrated by the other specimens. Such variation in the pH level may be related to the inherent nature of natural clay colloids (organic and inorganic) and their buffering capacity, i.e. absorbing, holding and releasing ions including the (OH^-) ions. Despite having the highest pH level in its untreated form, NatSoil (2) exhibited the lowest treated pH compared to NatSoil (1), which reflects a high buffering capacity (i.e. high resistance for pH change) for NatSoil(2). For all treated clays with 10% geopolymer, the increase in the pH level after 1 hour of curing seemed to comply with the minimum pH level required to trigger geopolymerisation, which agrees with Garcia-Lodeiro et al. (2014). It should be noted that further addition of geopolymer to 20% increased the pH level after 1 hour of curing to > 12.3 (see Figure 5.32c and d), allowing for continued geopolymerisation and higher enhancement of treated clay soils. As curing continues, the pH level decreased slightly; Figure 5.32 shows the rate of reduction is dependent on soil type. The overall drop in the pH may be justified by the consumption of (OH^-) ions during

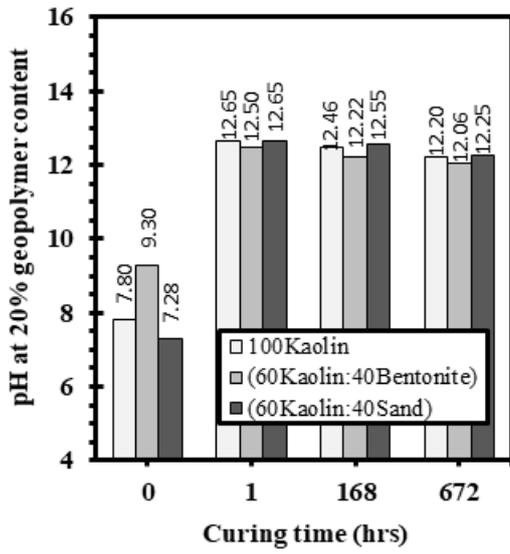
geopolymerisation, as explained by Duxson et al. (2007a), in addition to other reactions that may occur due to the complex clay mineralogy (Little 1995). It is noteworthy that the typical trends in the pH reduction observed in Figure 5.32 coincide with the increase in the UCS values observed in Section 5.4. Figure 5.33 highlights this correlation for the three engineered and two natural treated clays used at 10% geopolymer content. However, this is not the case for other treated clays, as the drop in the pH level for NatSoil (2) was not associated with any additional strength increase after 7 curing days, due to the reduction in the pH to less than 11, indicating retardation of geopolymerisation. Considering such a pH performance, it is necessary to increase the geopolymer content to maintain higher pH level during curing to ensure continuity of dissolution in the aluminosilicate materials and cementation reactions (Eades et al. 1966). This observation is a key point in the development of geopolymer stabilisation guidelines to ensure a minimum pH requirement for proper treatment of clay soils; this will be discussed subsequently.



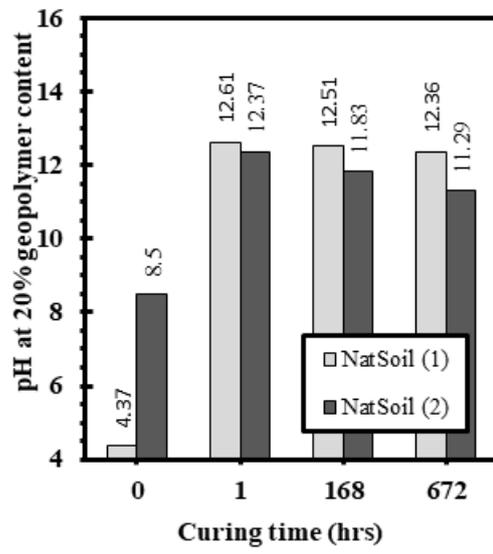
(a)



(b)



(c)



(d)

Figure 5.32: pH performance of various clays treated with 10% and 20% geopolymer contents: (a) engineered clays; and (b) natural clays

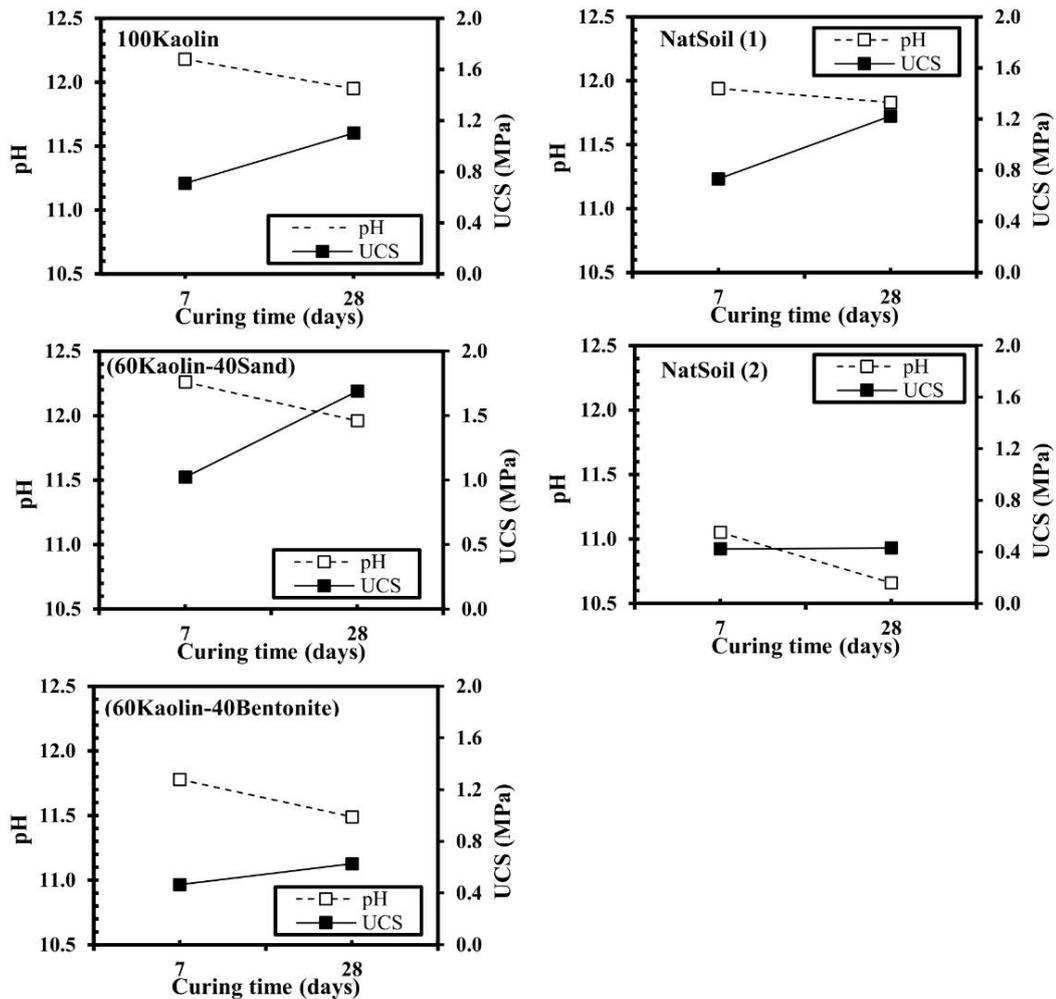


Figure 5.33: Typical relationship between residual pH and developed UCS for selected engineered and naturally treated clays at 10% geopolymer content

5.9 SUMMARY

A comprehensive testing program to investigate the influences of geopolymer addition on the physical and mechanical characteristics of geopolymer-treated clays was detailed in this chapter. The effects of geopolymer content were investigated for compaction parameters, plasticity index and strength performance of several clay types. Further testing was conducted to address the durability performance, pH performance and effect of pulverisation on geopolymer treatment for clays. The major findings from this investigation can be summarised as follows:

- Reduction in plasticity and increase in strength and durability can be attained for clay with a broad range of plasticities when treated with a proper dosage of geopolymer and properly cured.

- Insufficient geopolymer content may result in problems due to instability against field conditions (i.e. wetting-drying). However, despite the use of high geopolymer dosage, poor durability performance was detected in some clays. This is mainly related to the effect of mineralogy and the plasticity/activity on geopolymer treatment clays. Based on the initial plasticity of clays, the most ideal clay soils for stabilisation with geopolymer are those of low_to_moderate plasticity/activity, in which highly plastic/reactive clays are only suited for purposes where a temporary stabilisation is required (temporary projects), due to inadequate durability performance.
- During curing and under an extreme field wetting condition, leaching of the activator was found to be a minor concern for geopolymer clay stabilisation in terms of the residual strength. The field protection against water infiltration may be recommended during the initial curing time to maintain a long-term reaction and to attain good strength and durability performance.
- The addition of geopolymer produces an increase in the coefficient of permeability of clay.
- The pH performance of different treated clays shows variable degradation trends with the increase of curing time; however, the reduction in pH of treated clays coincided with an increase in strength, indicating continuity of geopolymer reactions and hence cementation.
- Pulverisation level of clay also shows a significant positive influence on treatment. As clay soils are more difficult to break down, multi-stage pulverisation before mixing with geopolymer may be required to achieve effective treatment.

In general, the results of this chapter have highlighted the significant impact of clay mineralogy and plasticity have on geopolymer treatment and dosage required for adequate strength and durability performance, implying that geopolymer is not a universal binder for stabilisation of all clay types. In the next chapter, the advanced shearing performance of geopolymer-treated clays will be investigated through undrained/drained triaxial testing, considering the impact of clay mineralogy, among other factors.

CHAPTER 6

UNDRAINED SHEAR BEHAVIOUR OF GEOPOLYMER-TREATED CLAYS

6.1 INTRODUCTION

Short-term stability in undrained conditions is most frequently the governing criteria for any geo-infrastructure supported on saturated clay soils. Therefore, it is critically important to understand the undrained shear behaviour of both the stabilised and host clays. As discussed in the literature review (Chapter 3, Section 2.4.1.3), limited work is available in this regard, considering the effect of mineralogy and plasticity characteristics of clays. In order to comprehend the undrained shear response of untreated and geopolymer-treated clay, a comprehensive isotopically Consolidated Undrained (CU) static triaxial testing program was undertaken as outlined in Chapter 3, Phase III. In this chapter, the salient features of the undrained shearing responses of the geopolymer-treated clays obtained from the results of the experimental investigation are presented and the possible mechanism behind the detected observations are explained and discussed.

There are several factors that may affect the undrained shearing behaviour of artificially cemented soils. Some of these factors are: (1) level of cementation induced by binder content; (2) confining pressure; (3) characteristic of the host soil; and (4) curing time. The effect of the first three factors on geopolymer-treated mixtures will be covered in this chapter. Due to time constraints the impact of curing time was not explicitly investigated here; however, by studying the influence of different levels of cementation on the shear behaviour of treated specimens cured for 28 days, it is possible to draw qualitative inferences about the impact of curing time. The effect of cementation level on the shearing behaviour under static loading was investigated for clay treated with 10% and 20% geopolymer at confining pressures of 100, 200 and 400 kPa. Three types of clay; engineered kaolin and two natural clays of different mineralogy, i.e. NatSoil (1) and NatSoil (2), were employed to explore the effect of clay characteristics on the undrained shear behaviour. While the primary goal of this investigation is to understand the undrained shearing behaviour of geopolymer-treated clay under undrained axial compression loading, additional test conditions (undrained constant- p and drained axial compression loading) were also considered to enhance

the general understanding of the shearing behaviour of geopolymer-treated clay. In the following sections, the undrained shearing behaviour of untreated and geopolymer-treated clays is reported and discussed for selected loading conditions in terms of stress-strain, excess pore pressure and stress path responses.

6.2 AXIAL COMPRESSION SHEARING BEHAVIOUR

In this section, the stress-strain behaviour, excess pore water pressure and stress path response of untreated and geopolymer-treated clays subjected to CU compression axial loading are presented and discussed. Different responses were reported in terms of deviator stress (q) and mean effective stress (p), in which:

$$q = \sigma'_1 - \sigma'_3 \quad (6.1)$$

$$p' = (\sigma'_1 + 2\sigma'_3)/3 \quad (6.2)$$

where σ'_1 and σ'_3 are the major and minor principal stresses, respectively.

6.2.1 Stress-strain response

The effects of level of cementation, confining pressure and clay characteristics on the stress-strain behaviour of geopolymer treated clays are investigated and discussed here with references to Figures 6.1 to 6.6.

6.2.1.1 Effect of level of cementation

Figure 6.1(a to c) shows the stress-strain responses obtained from CU compression triaxial tests in terms of deviator stress (q) versus axial strain (ϵ_a) for untreated and treated kaolin, with varying geopolymer contents of 10% and 20%, under confining pressures of 100, 200 and 400 kPa. Irrespective of the confining pressure and clay type used, Figure 6.1(a to c) shows that the untreated clay possesses a ductile stress-strain behaviour. It can be seen that with the addition of geopolymer, the post-yield strain-hardening response observed for any untreated clay disappears in favour of a brittle strain-softening response. At 10% geopolymer content, the deviator stress, q , increases monotonically with the axial strain until it reaches the peak value at low axial strain (0.4-0.6%), which is then followed by a post-peak strain-softening (brittle) response. Only the stress-strain response of treated NatSoil (2), Figure 6.1(c), was comparable with the untreated kaolin behaviour; the effect of clay type on the undrained stress-strain response of each of these mixes will be discussed in detail later. However, for

any clay type, as the geopolymer content increases to 20%, yielding was associated with a more pronounced stiffer response (i.e. higher q and low axial strain). This observation is consistent with the effect of geopolymer concentration on the UCS stress-strain response and may be explained with reference to the failure mechanism of cemented soils described by Coop et al. (1993), Ismail et al. (2002) and Nagaraj et al. (2001). Clay specimens treated with a high geopolymer content likely possess an increased amount of cementitious bonds, which can be held responsible for the high undrained shear performance at the initial stages of loading (i.e. before yield). Upon further shearing, the cementitious bonds break, resulting in a yield of cemented-soil, after which the stresses seem to be carried by a frictional response between the weak de-structured matrix of soil particles and broken stabiliser bonds (i.e. the residual strength post-yield). This particular failure mechanism occurs in cemented-soils when the consolidation stress is lower than the yield stress, such that the effect of the consolidation phase on the soil structure is insignificant (Coop et al. 1993).

6.2.1.2 Effect of confining pressure

The influence of the confining pressure on the behaviour of kaolin and natural clay soils can be seen from the results reproduced in Figure 6.2(a to c), for 10% and 20% geopolymer content and over a range of confining pressure of 100-400 kPa. The results show that the pre-shear consolidation pressure (p'_c) has a significant influence on the quantitative stress-strain response of geopolymer-treated clay. The yield stress of the treated specimens increases significantly with the increase in confining pressure. This may cause a slight de-structure in the cemented soil from the consolidation phase, resulting in a behaviour controlled by friction as well as cementitious bonds (Coop et al. 1993). At 100 kPa, the de-structuring of the stabilised soil seems to be insignificant, and the cementation bonds between the clay particles mainly govern the yield stress. With the increase of confining pressure up to 200 kPa and 400 kPa, the assumed potential damage of the cementitious structure of the specimen from the consolidation phase appears to be insignificant and the strength is still governed by the cementitious bonds with very little contribution coming from the mobilised friction associated with change in the specimen volume due to consolidation. The frictional response then governs the residual post-peak strength.

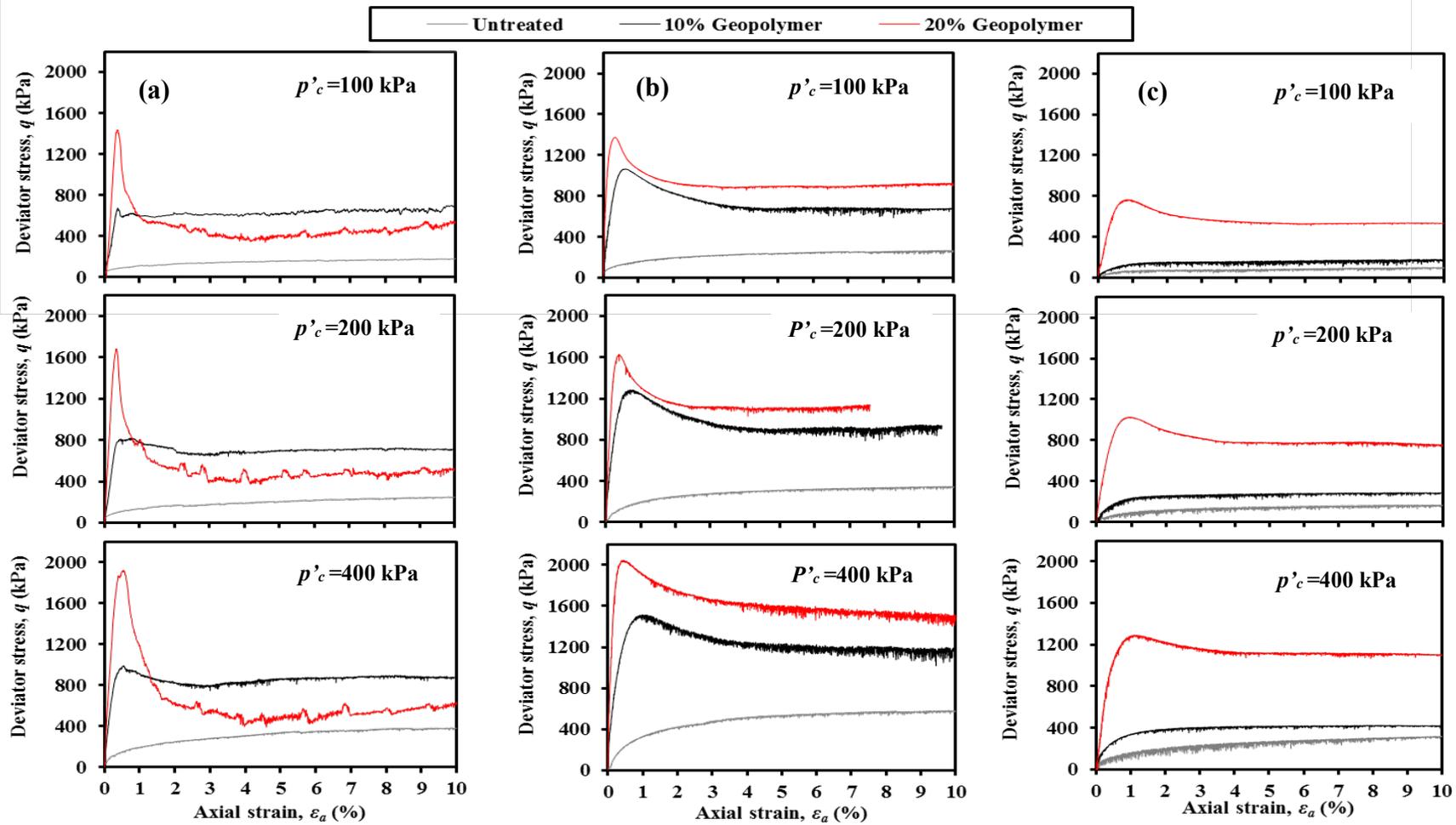


Figure 6.1: Effect of geopolymer content on the undrained stress-strain behaviour of treated clay: (a) Kaolin clays; (b) NatSoil (1); and (c) NatSoil(2).

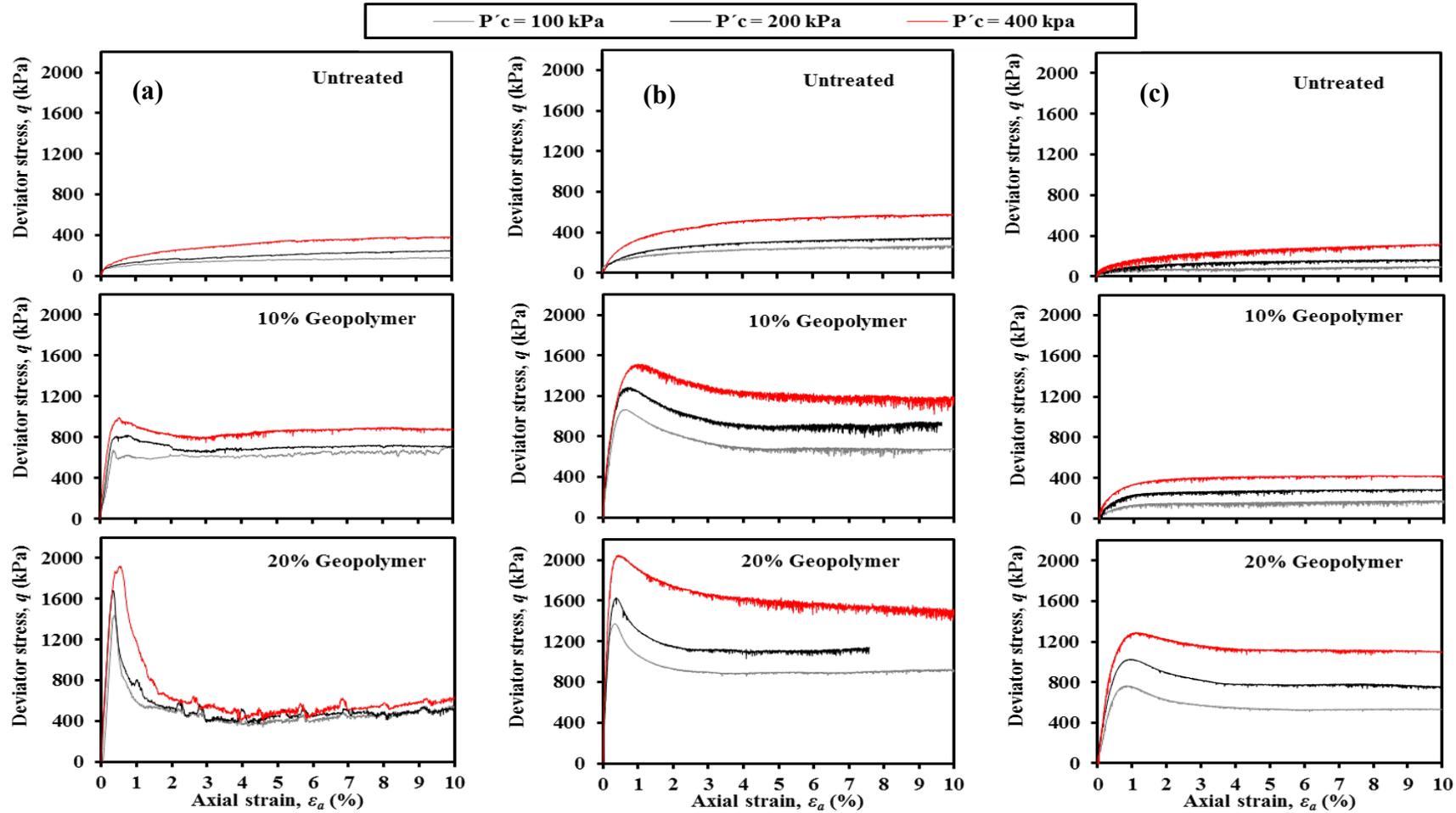


Figure 6.2: Effect of confining pressure on the undrained stress-strain behaviour of untreated and treated clays: (a) Kaolin clay; (b) NatSoil (1); and (c) NatSoil (2).

6.2.1.3 Effect of clay characteristics

The results presented in Figures 6.1 and 6.2 indicate the role of geopolymer and confining pressure in the undrained shearing response of engineered and natural treated clays, yet the type of clay also impacts the undrained shearing response. Figure 6.3 shows markedly different initial stiffness and stress-strain response for Kaolin, NatSoil (1) and NatSoil (2) respective. At 10% geopolymer content (Figure 6.3a), the difference between kaolin and NatSoil (1) is purely quantitative as evident from the similar strain-softening behaviour but different peak yield stresses. By comparison, NatSoil (2) shows only a qualitative difference in the stress-strain response, as evidenced from the fully-ductile response. At 20% geopolymer content (Figure 6.3b), the stress-strain response was mainly strain-softening for all treated clays and the difference is only quantitative, with NatSoil (2) showing a significantly lower peak-strength. This finding supports the preference for geopolymer to stabilise low plasticity/activity soils, as discussed previously with respect to the UCS testing (Chapter 5).

Figure 6.4 shows the relationship between the initial stiffness for each treated specimen and the type of clay. The initial stiffness is represented quantitatively as the secant modulus of elasticity, E_{50} , for each specimen at any confining pressure. In general, the low-plasticity treated NatSoil (1) exhibits the highest stiffness, with this trend persistent over the selected range of confining pressure and geopolymer contents. The high-plasticity kaolin also exhibited progressive stiffness increases for all geopolymer contents, yet to a lesser extent than NatSoil (1). However, NatSoil (2), with the highest plasticity, did not show an appreciable increase in stiffness except at higher geopolymer content (i.e. 20%). This observation is further supported by examining the failure modes (Figure 6.5), where only the 20% geopolymer NatSoil (2) specimens developed a distinct failure plane (Figure 6.5c). While both kaolin and NatSoil (2) specimens exhibited similarly high plasticity characteristics, there is a significant difference in their corresponding activity indices (Table 3.2, Chapter 3), which clearly demonstrates the impact of the clay fraction and the associated mineralogy on the success of geopolymer treatment. The difference in the initial stiffness between the different geopolymer-treated specimens (i.e. natural soils and kaolin) may relate to the interactions between the geopolymer and individual clay mineralogy (and the associated contents, Chapter 2, Section 2.2.2). These factors seem

to influence the geopolymerisation process and the particular characteristics of bonded clusters that formed along the shear plane. Such effect of soil mineralogy on the undrained strength performance of geopolymer-treated clay was found to be similar to that reported in the cement and lime-soil literature (Wissa et al. 1964).

All geopolymer-treated clays, under 100 kPa confining pressure, except for NatSoil(2) with 10% geopolymer, as discussed previously, experience a drop in the deviator stress from peak to residual, which suggests an overall strain-softening behaviour (Figure 6.1). This post-yield stress reduction is due to the bonding degradation of the geopolymer-clay material within the shear failure zone. The addition of geopolymer may alter the gradation and mineralogy of the host clay; thus, the residual strength may be different for each geopolymer content and clay type. Figure 6.6 shows the brittleness index, I_B , which relates to the strength difference between the peak and residual stresses divided by the peak strength from Figure 6.1. At low geopolymer content (i.e. 10%), the increase in I_B is restricted to kaolin and NatSoil (1) only. With the increase in geopolymer content to 20%, all treated clays show an additional increase in I_B values. For all cases, the three tested clays were found to be markedly different in I_B , indicating that the geopolymer has more influence on the fabric of some clays than the other factors, and this suggesting the need to treat high plasticity/activity clay with an increased geopolymer content (i.e. 20%) to reach similar post-yield behaviour to that of low plasticity/activity clay.

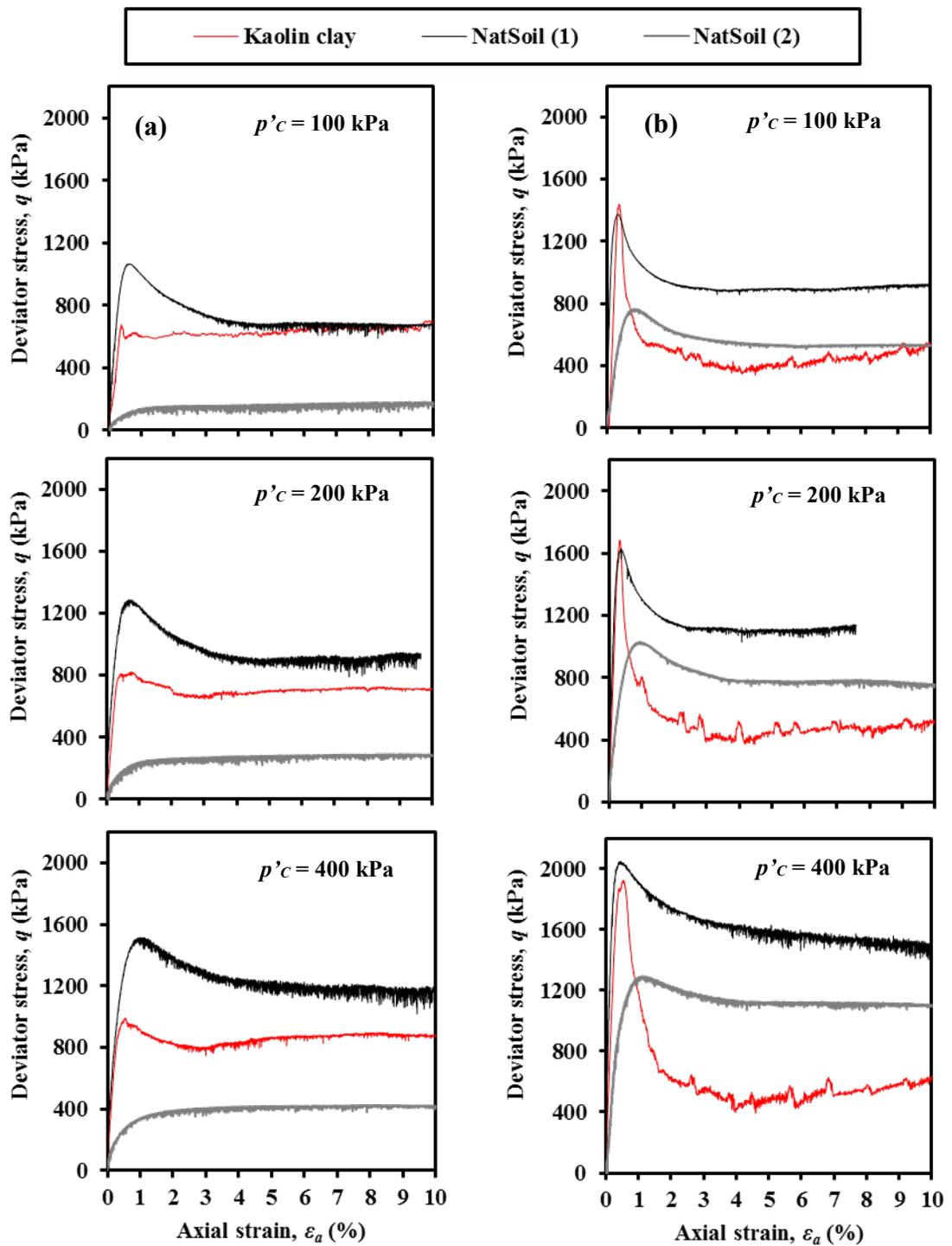


Figure 6.3: Effect of clay mineralogy on undrained stress-strain behaviour of geopolymer-treated clays: (a) 10% geopolymer; and (b) 20% geopolymer.

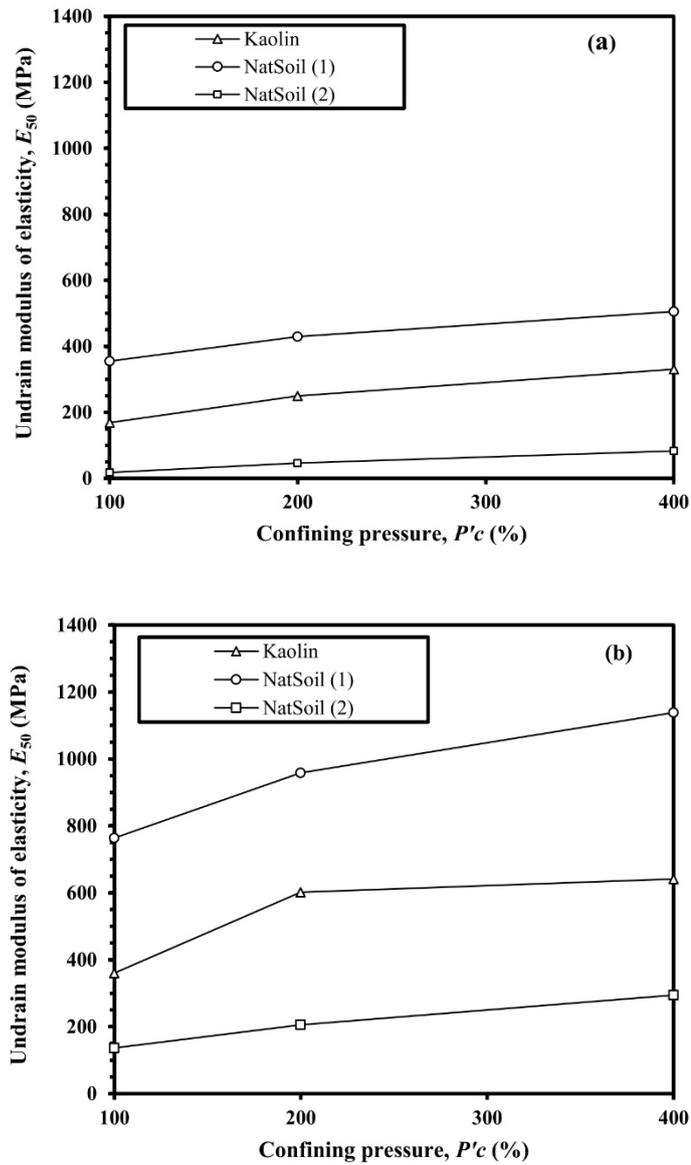


Figure 6.4: Effects of confining pressure on the modulus of elasticity (E_{50}) for different clays treated with various geopolymer contents in CU test: (a) 10% geopolymer; and (b) 20% geopolymer.

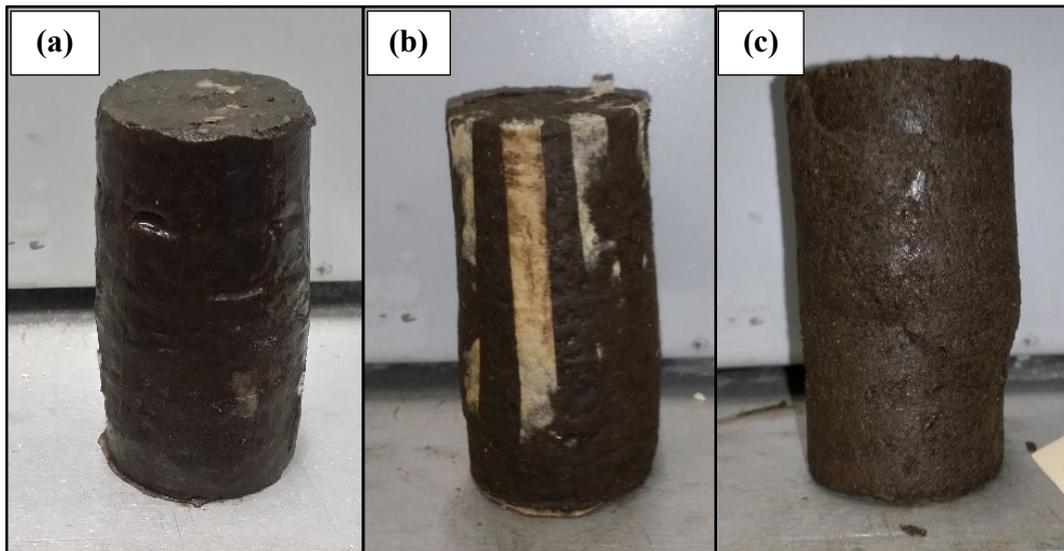


Figure 6.5: Influence of geopolymer addition on CU triaxial failure mode of NatSoil (2) (100 kPa confining pressure): (a) untreated; (b) 10% geopolymer; and (c) 20% geopolymer.

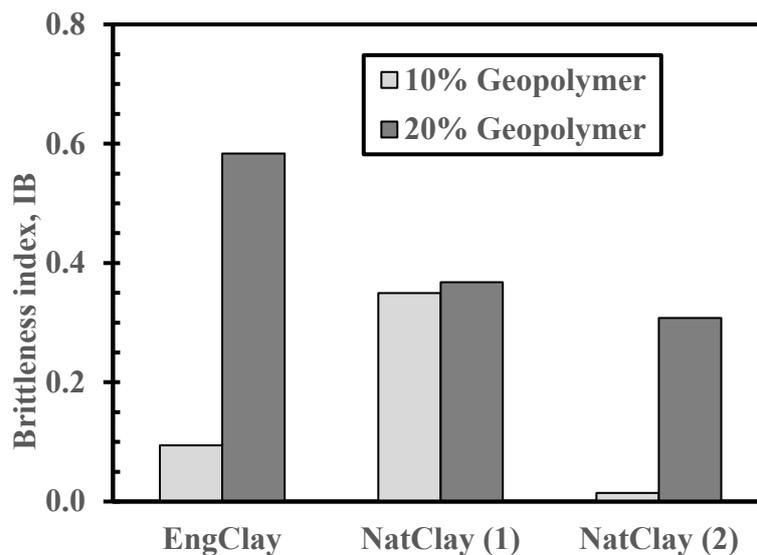


Figure 6.6: Variation of brittleness index for geopolymer treated clays (100 kPa confining pressure).

6.2.2 Pore pressure response

The development of the excess pore water pressure (Δu) during undrained shearing of cemented soil is due to the change in the mean total stress and the corresponding volume change tendency of the soil specimen (i.e. contraction or dilation). In the following sections, the effects of level of cementation, confining pressure and clay type on excess pore pressure development are presented and discussed.

6.2.2.1 Effect of level of cementation

Figure 6.7 shows the typical response of excess pore water pressure from CU compression tests at 100, 200 and 400 kPa confining pressures for different treated clays with 10% and 20% geopolymer content. The corresponding response of untreated clays is also shown in Figure 6.7 for comparison. As shown in Figure 6.7(a), for the untreated clays, positive pore water pressure is generally developed at strain levels of 0-2% before a slight reduction at higher strains (no peak stress evident), regardless of the applied confining pressure. This observation implies an initial specimen contraction at low strain followed by slight dilation at higher strain up until failure; failure was not observed within these tests due to the extreme ductile behaviour of the specimens. However, the transition from contractive to slightly dilative behaviour for the untreated clay implies a quasi-over-consolidated behaviour, i.e. normal consolidation behaviour with a tendency towards over-consolidation at higher strains (Head et al. 2014).

For treated specimens with 10% geopolymer content (Figure 6.7a, b and c), an initial contraction was achieved at a strain level corresponding to the peak stress before dilation began. At the transition zone from the contractive to dilative behaviour near the peak deviator stress, Δu indicated higher positive values followed by a reduction during strain-softening before levelling off, leading to an overall behaviour of over-consolidation. With an increase geopolymer content to 20%, higher dilation tendency can be detected during shearing as indicated by the increased negative pore pressure values observed (Figure 6.7a, b and c). The increasing dilation with increasing geopolymer content may be related to the effect of geopolymer content on the size of broken clusters formed and the size of voids generated upon shearing, which ultimately impact the residual frictional response.

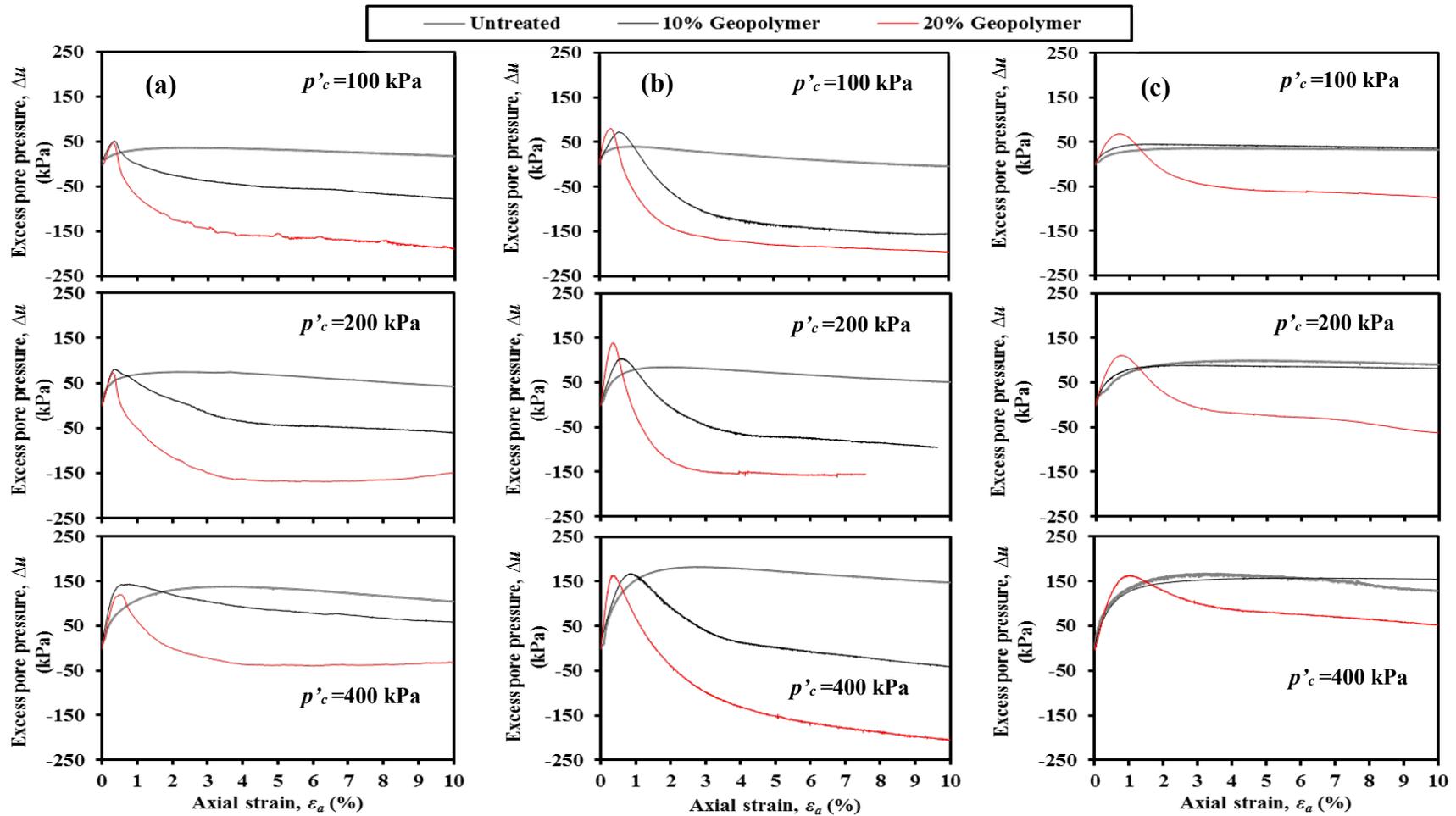


Figure 6.7: Effect of geopolymer content on undrained excess pore water pressure of geopolymer-treated clay: (a) kaolin clay; (b) NatSoil (1); and (c) NatSoil (2).

6.2.2.2 Effect of confining pressure

Figure 6.8 shows the relationship between the excess pore water pressure of untreated and geopolymer-treated clays and confining pressure. In general, the excess pore water pressure in untreated clays increases with the increase of confining pressure. This is also true for excess pore water pressure of geopolymer-treated specimens at the peak, for 10% and 20% geopolymer contents. At residual conditions, and with the increase of confining pressure, the tendency to develop negative Δu values for treated specimens is significantly reduced. This indicates a transition from strong dilation at low confining pressure to weak dilation at high confining pressures. This transition implies uniformity in the breakdown of cementation bonds throughout the sheared specimen, that is the bonded clusters may face difficulties to survive at high confining pressure resulting in suppressed dilation. However, the test results suggested that the increase of confining pressure does not influence the degree of dilation of treated clays, i.e. difference between the positive and negative values at peak and residual state, respectively.

6.2.2.3 Effect of clay characteristics

Figure 6.9 shows markedly different excess pore water pressure response and the degree of dilation for the different clay types tested. At a low level of cementation induced by 10% geopolymer content, both Kaolin and NatSoil (1) show significant post-peak dilation trends, for all confining pressures. In contrast, the excess pore water pressure response for treated NatSoil (2) was more congruent to the untreated one (i.e. contractive behaviour). However, as the geopolymer content increases to 20%, the pore water pressure response of NatSoil (2) was associated with a pronounced post-peak dilation. At this stage, although the overall qualitative excess pore water pressure behaviour of NatSoil (2) is similar to that of Kaolin and NatSoil (1), significant quantitative differences are observed between the two natural clays and also between natural clays and kaolin clay. The quantitative differences, especially in the degree of dilation, might be attributed to the response of the host clay to geopolymer treatment and its effect on the particular characteristics of bonded clusters formed along the shear plane.

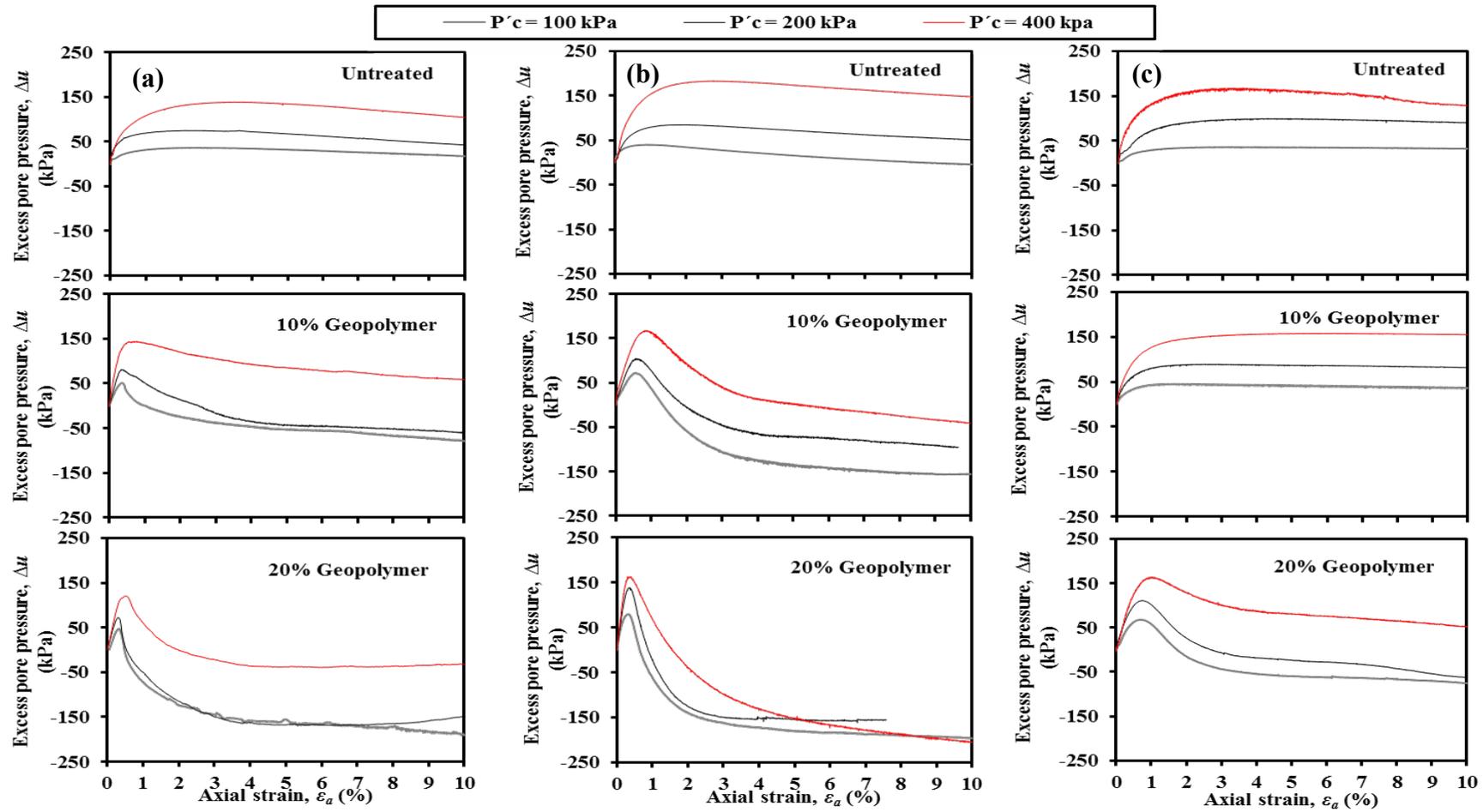


Figure 6.8: Effect of confining pressure on excess pore water pressure-shear strain behaviour of geopolymer-treated clay: (a) kaolin clay; (b) NatSoil (1); and (c) NatSoil (2).

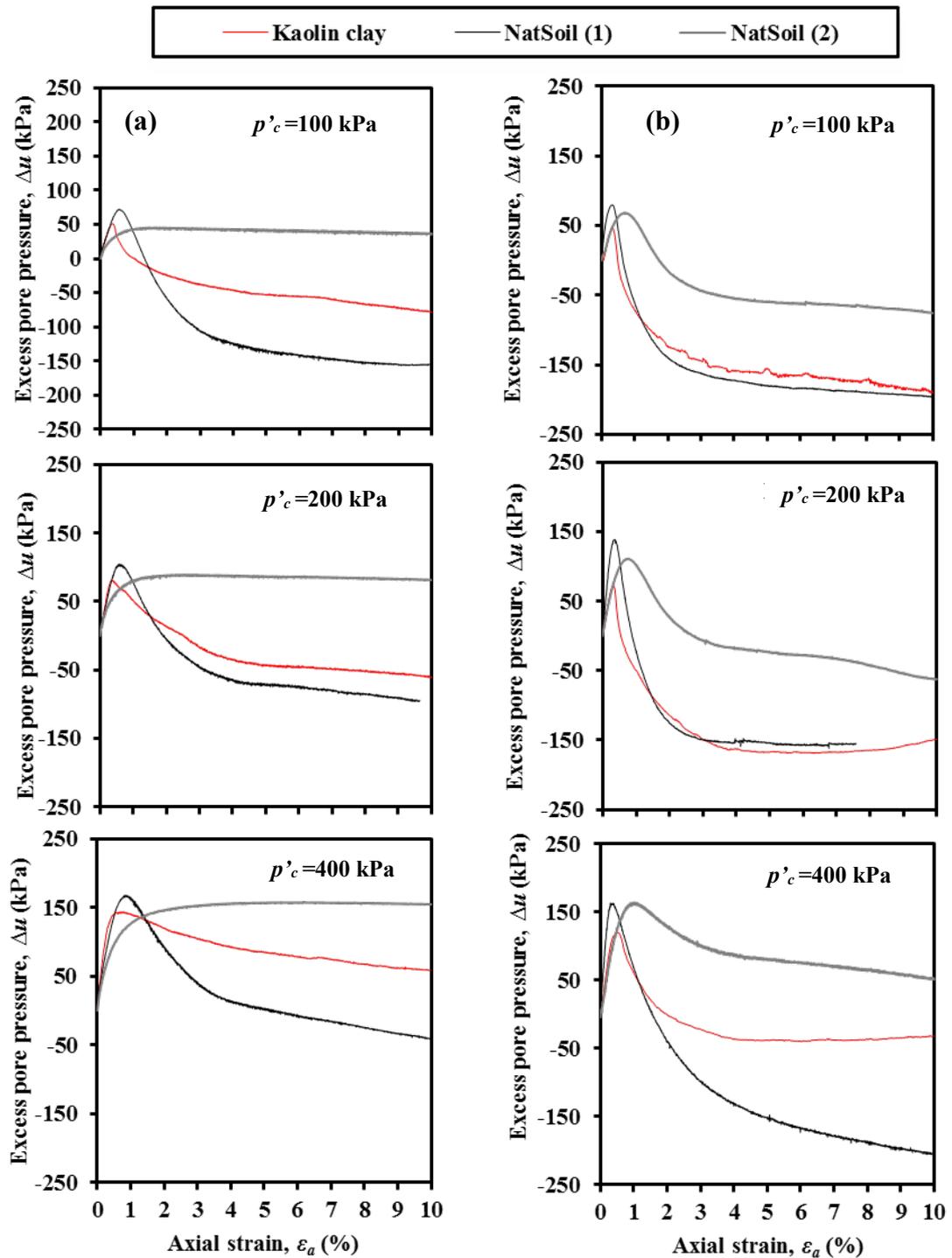


Figure 6.9: Effect of clay mineralogy on excess pore water pressure-shear strain behaviour of geopolymer-treated clay: (a) 10% geopolymer; and (b) 20% geopolymer.

6.2.3 Stress path response

The investigation of the q - p' stress path response of geopolymer treated clay can be useful for the evaluation of the factors affecting the mobilisation of undrained shear

resistance at different stages of loading. The effects of the cementation level, confining pressure, and clay characteristics on the q - p' stress path response of geopolymer-treated clays are investigated in this section with the help of Figures 6.10 to 6.12.

6.2.3.1 Effect of cementation level

The influence of the level of cementation, induced by the geopolymer addition, on the stress path response of kaolin and natural clay soils can be seen from the results presented in Figure 6.10, over a range of confining pressure of 100-400 kPa. It can be seen that the 10% geopolymer addition changed the initial quasi-over-consolidated characteristics of the remoulded clays to that of heavily over-consolidated. The heavily over-consolidated behaviour observed in the treated specimens agrees with the behaviour of OPC stabilised clays (e.g. Uddin et al. 1997; Lorenzo et al. 2006). Moreover, the length of the initial section of the stress-path for treated specimen increases in response to the geopolymer content, increasing from 10% to 20%, which is likely attributed to the increased amount of cementation bonds within the treated clay. Another important observation is that, regardless of the soil types, the gradient of the initial section of the stress path decreases significantly with increasing geopolymer content, which may be due to the increased stiffness of soil structure.

Up to 400 kPa, the stress path of geopolymer-treated clays can be typically represented by almost an initial straight line with a gradient towards the right, see Figure 6.10. Once it is reached the peak failure, the stress path is seen to move down the peak before levelling off as evidenced from the horizontal progression of the stress path towards the increasing mean effective stress value (p'). The sharp fall of the stress path beyond the peak might indicate the existence of micro-cracks within the treated specimens, whereas the horizontal progression may indicate a high tendency for dilation caused by the high de-structure state of the geopolymer-clay mixtures. The shape of the stress path of NatSoil (2), with 10% geopolymer, was congruent to that of untreated one and this might be attributed to the weak formation of cementitious bonds within the clay. However, at a low level of cementation, the effect of geopolymer addition on the stress path still evident for NatSoil (2), as indicated by the slightly over-consolidated behaviour. The vertical rise of the stress path with almost constant effective mean stress implies an elastic behaviour, whereas the bend towards the right shows a slight dilation.

6.2.3.2 Effect of confining pressure

As can be seen from Figure 6.11, a quasi-over-consolidated behaviour is the general behaviour for untreated clays, and this trend is persistent over confining pressures of 100, 200 and 400 kPa. The increase of the confining pressure in untreated specimens was associated with an increase in the stiffness, as indicated by the increase in the deviator stress at peak. For geopolymer-treated clays at different levels of cementation, the stress paths of all treated clays did not show a marked change in behaviour when the level of pre-consolidation pressure was increased from 100 to 400 kPa. It is seen that the length of the initial section of the stress-path increases with increasing confining pressure, which may be attributed to the reduction of the specimen volume during consolidation and the mobilised friction before shearing, contributing with higher stiffness.

6.2.3.3 Effect of clay characteristics

Figure 6.12(a and b) show the impact of soil type on the stress path performance determined from the undrained shearing tests conducted at 10% and 20% geopolymer contents, under 100, 200 and 400 kPa confining pressures. At 10% geopolymer content (Figure 6.12a), kaolin and NatSoil (1) give a similar qualitative undrained response (i.e. heavily over-consolidated); however, NatSoil (1) shows higher bulk stiffness as evidenced from the increased length and reduced gradient of the initial section of the stress path. When comparing NatSoil (2) with kaolin and NatSoil (1), quasi-over-consolidated behaviour is still observed although to a smaller extent, indicating that NatSoil (2) is less prone to dilation upon shearing than other treated clays at low degrees of cementation (i.e. 10% geopolymer). However, with the increase in cementation towards the higher level (i.e. 20% geopolymer), NatSoil (2) gave a typical heavily over-consolidated response (Figure 6.12b).

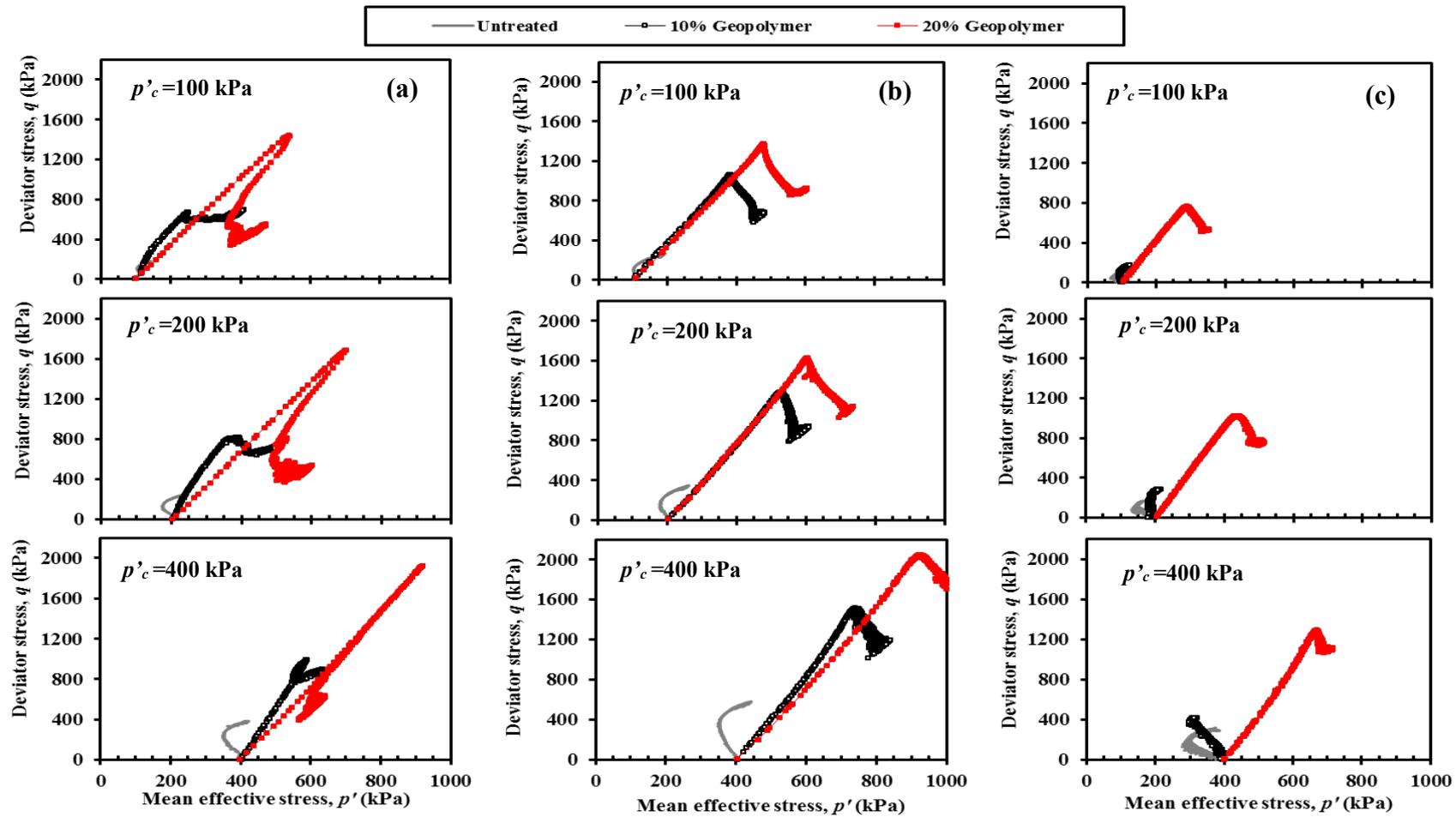


Figure 6.10: Effect of geopolymer content on the undrained stress path of geopolymer-treated clay: (a) kaolin clay; (b) NatSoil (1); and (c) NatSoil (2).

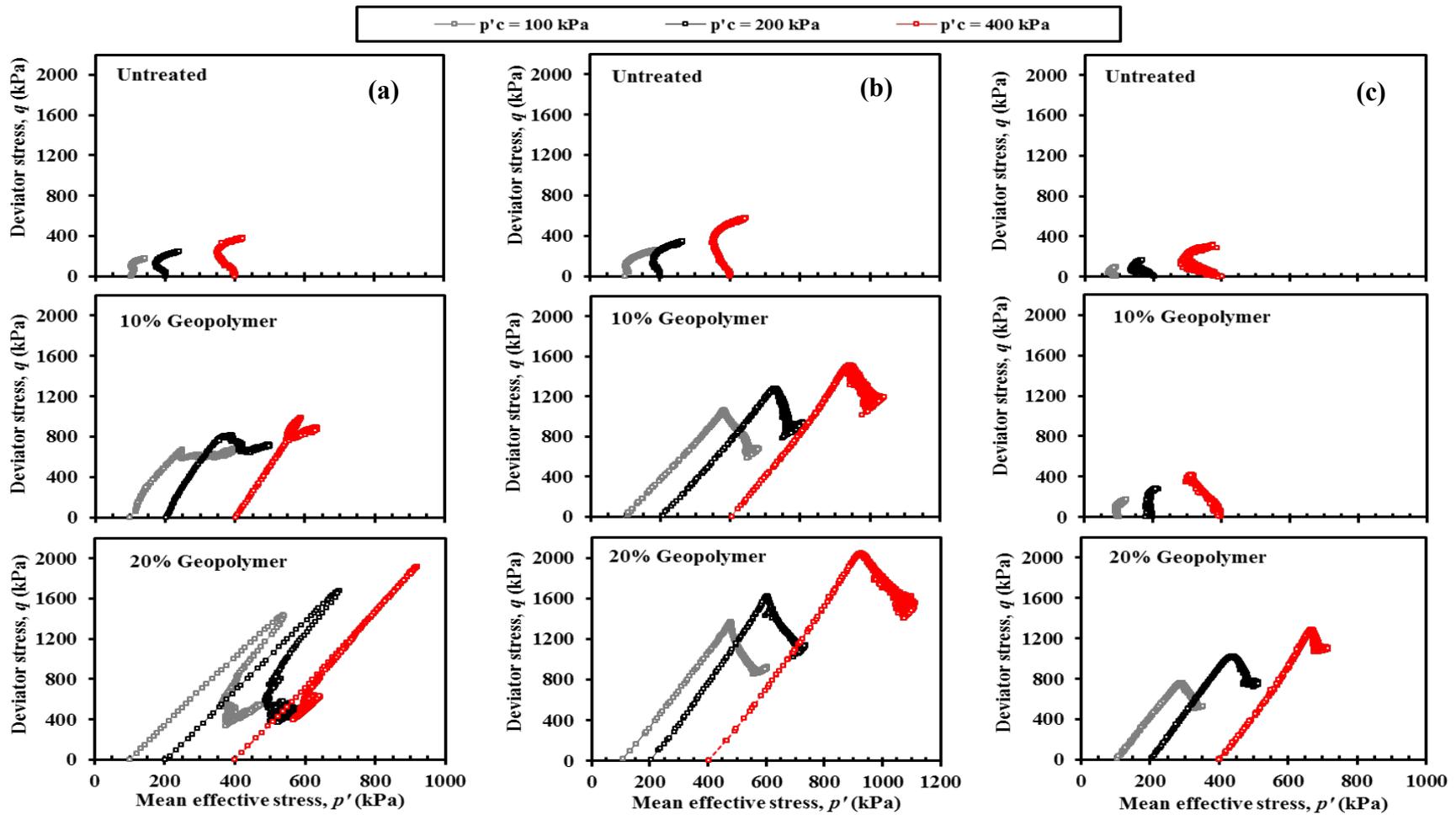


Figure 6.11: Effect of confining pressure on the undrained stress path of geopolymer-treated clay: (a) kaolin clay; (b) NatSoil (1); and (c) NatSoil(2).

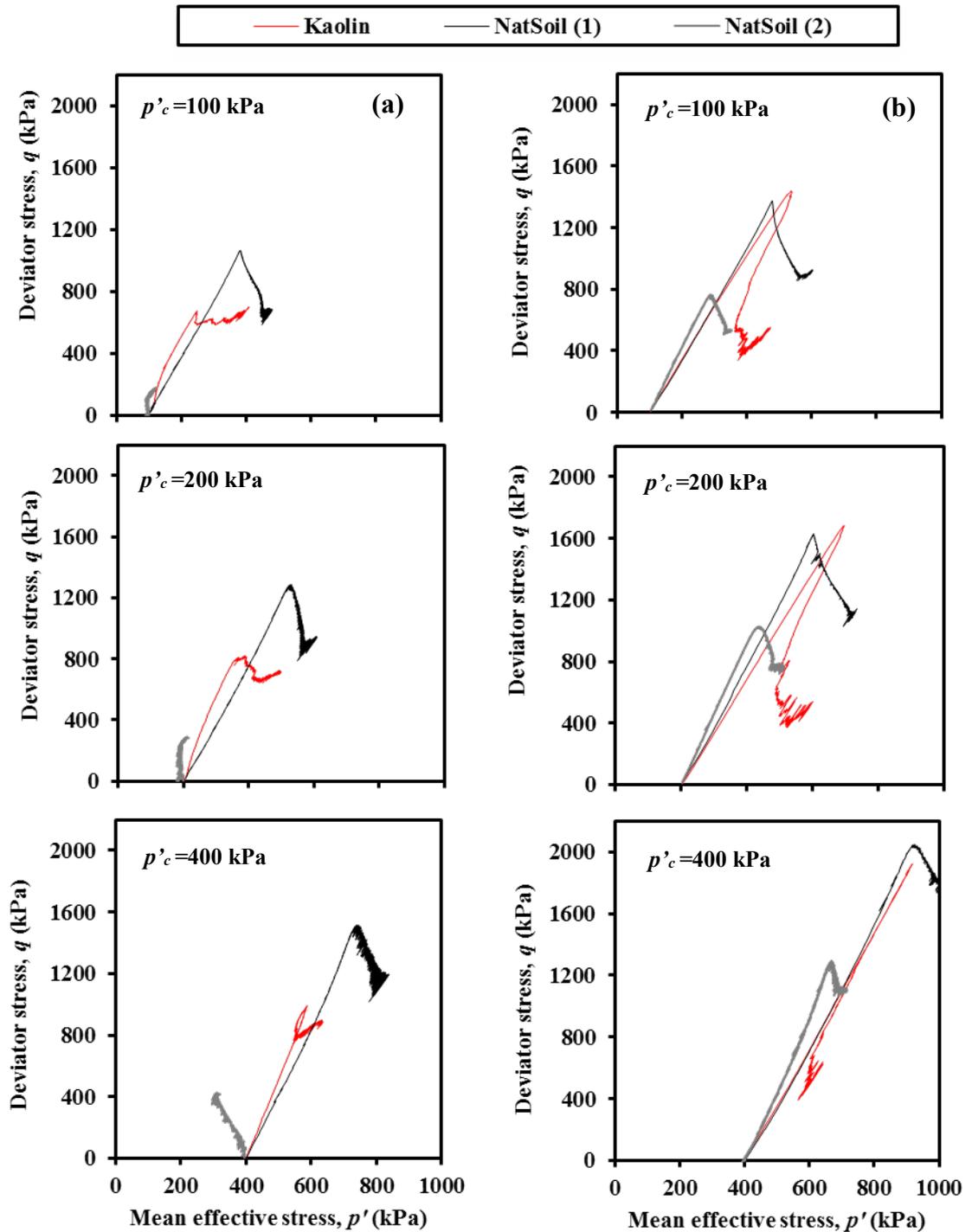


Figure 6.12: Effect of clay mineralogy on undrained stress path of geopolymer-treated clay: (a) 10% geopolymer; and (b) 20% geopolymer.

6.2.3.4 Undrained failure envelope

Corresponding to the different confining pressures used in the CU tests (i.e. 100, 200 and 400 kPa), the q - p' stress paths and peak failure envelopes are presented in Figures 6.13 to 6.15, for 10% and 20% geopolymer contents. All specimens tested under low

effective confining pressure (i.e. 100 kPa) showed peak stress near to the tension cut-off line ($q = 3p'$), which corresponds to a state of zero effective radial stress (excess pore water pressure = confining pressure). As the confining pressure increases, the CU specimens failed at higher effective stress levels, further away from the tension cut-off line. This phenomenon may be attributed to the volume reduction of the specimen during consolidation, which increases the frictional resistance and therefore the total shear capacity.

Linear failure envelopes can be used for an approximate representation of the undrained peak shear strength of treated clays in the $q-p'$ space, as shown in Figures 6.13 to 6.15. For all clay types, the ability of geopolymer treatment to move the stress states beyond the untreated clay failure envelope is evident, consistently generating a geopolymer treatment failure envelope greater than that of the untreated clay. For each clay type, the addition of geopolymer promoted the increase in the gradient and q -axis intercept values of the envelope, implying higher effective shear strength parameters, i.e. effective cohesion (c') and effective angle of internal friction (ϕ'), for the treated clays, as summarised in Table 6.1. The increase in effective cohesion of the treated clay can be attributed to the increase in the cementation offered by the geopolymerisation process, similarly to the effect of cementation bonds described widely in the OPC-treated clays (Horpibulsuk et al. 2004). The increase in the effective friction angle within these same specimens can be attributed to the effect of the large particles formed from the bonded clusters of clay after stabilisation (Lorenzo et al. (2006). However, the comparison between $q-p'$ slopes superimposed on Figures 6.13 to 6.15, as well as the corresponding effective shear strength parameters (Table 6.1), indicates a considerable influence of clay mineralogy on the stress path response of geopolymer-treated clays. Particularly for natural soils, NatSoil (1) showed higher strength improvement in response to geopolymer treatment, where the increases between untreated and 20% geopolymer specimens were $c' = 335.7$ kPa and $\phi' = 7.3^\circ$ compared with $c' = 175$ kPa and $\phi' = 14^\circ$ for NatSoil (2).

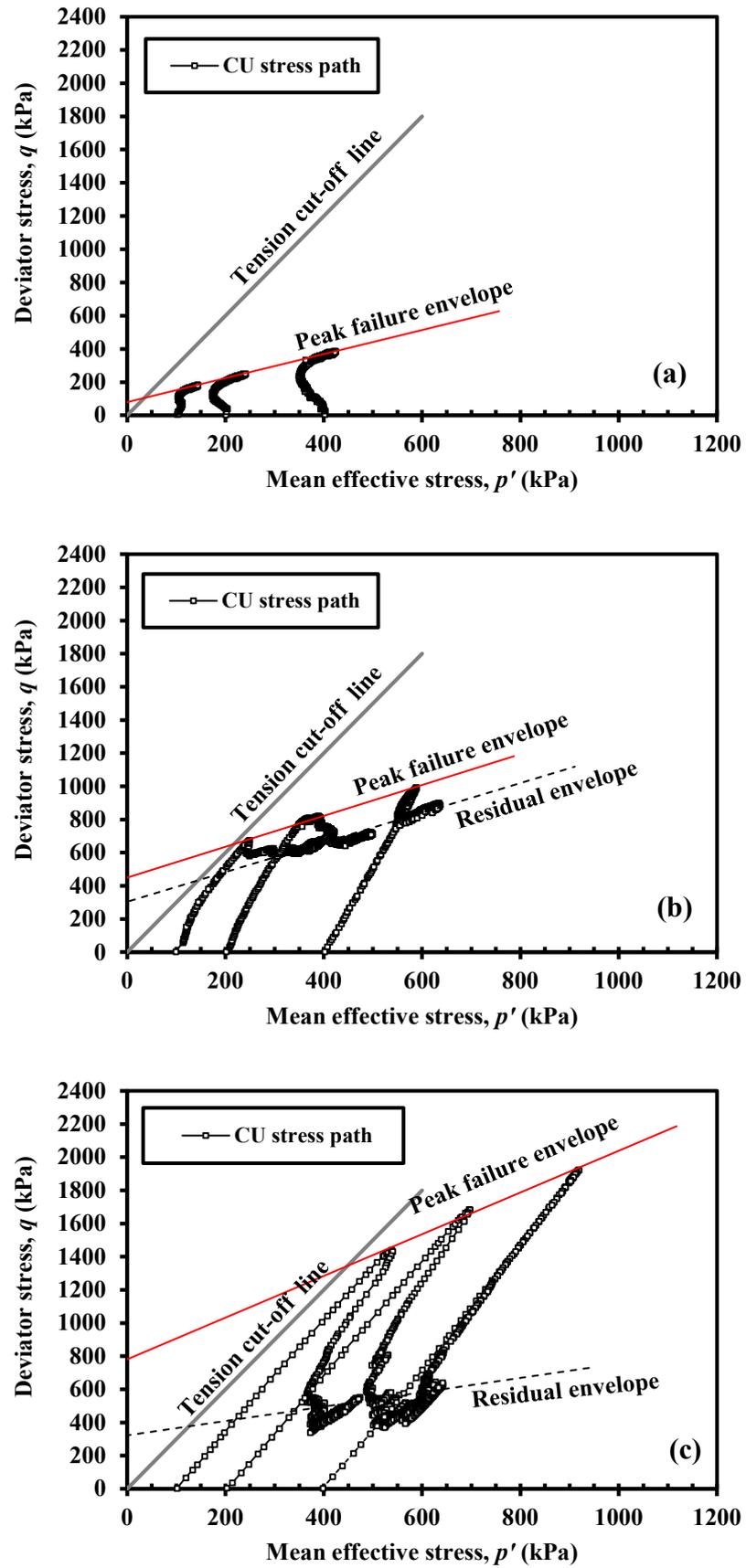


Figure 6.13: Failure envelope of kaolin clay treated with various amounts of geopolymer: (a) untreated; (b) 10% geopolymer; and (c) 20% geopolymer.

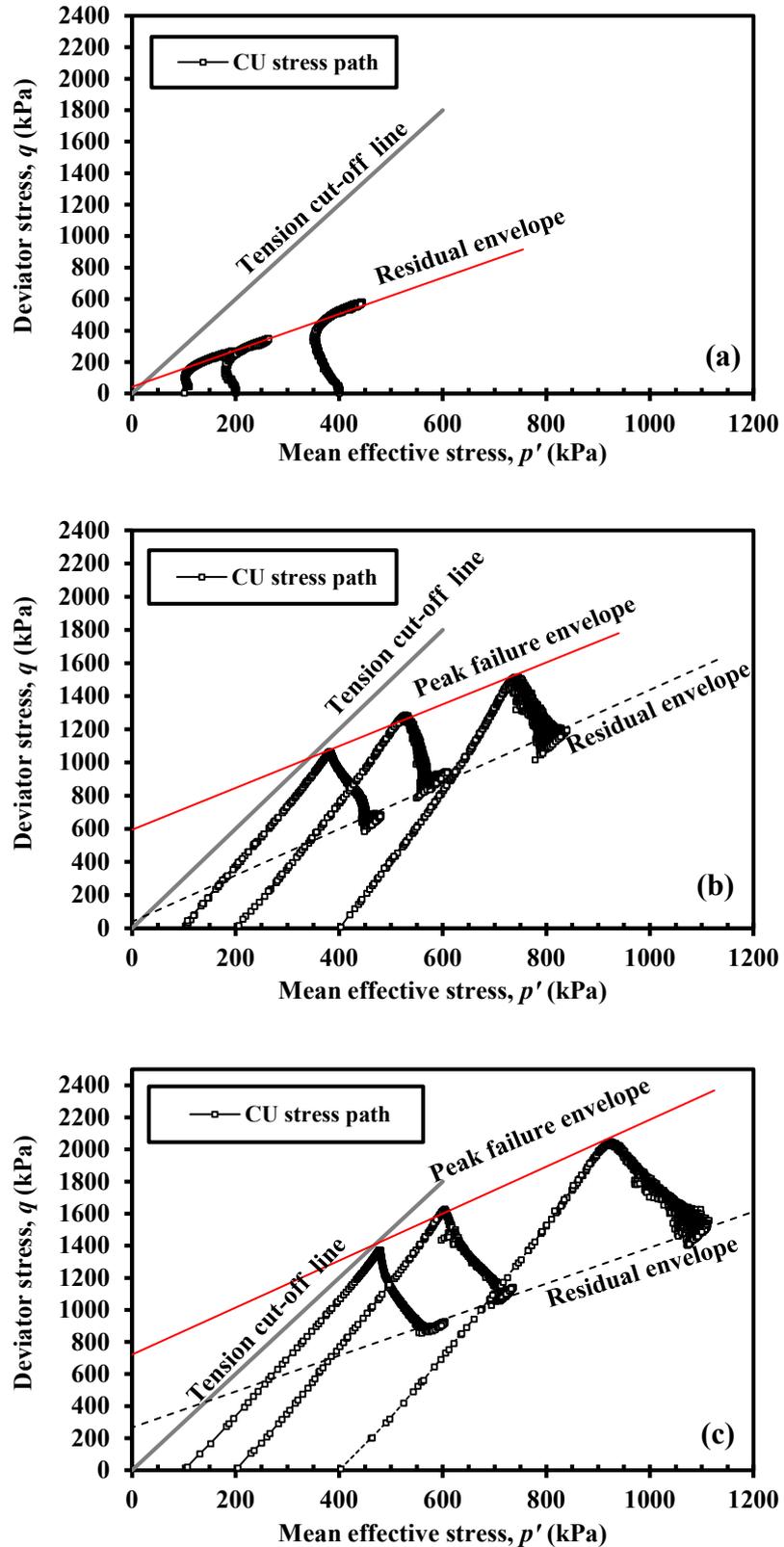


Figure 6.14: Failure envelope of NatSoil (1) clay treated with various amounts of geopolymer: (a) untreated; (b) 10% geopolymer; and (c) 20% geopolymer.

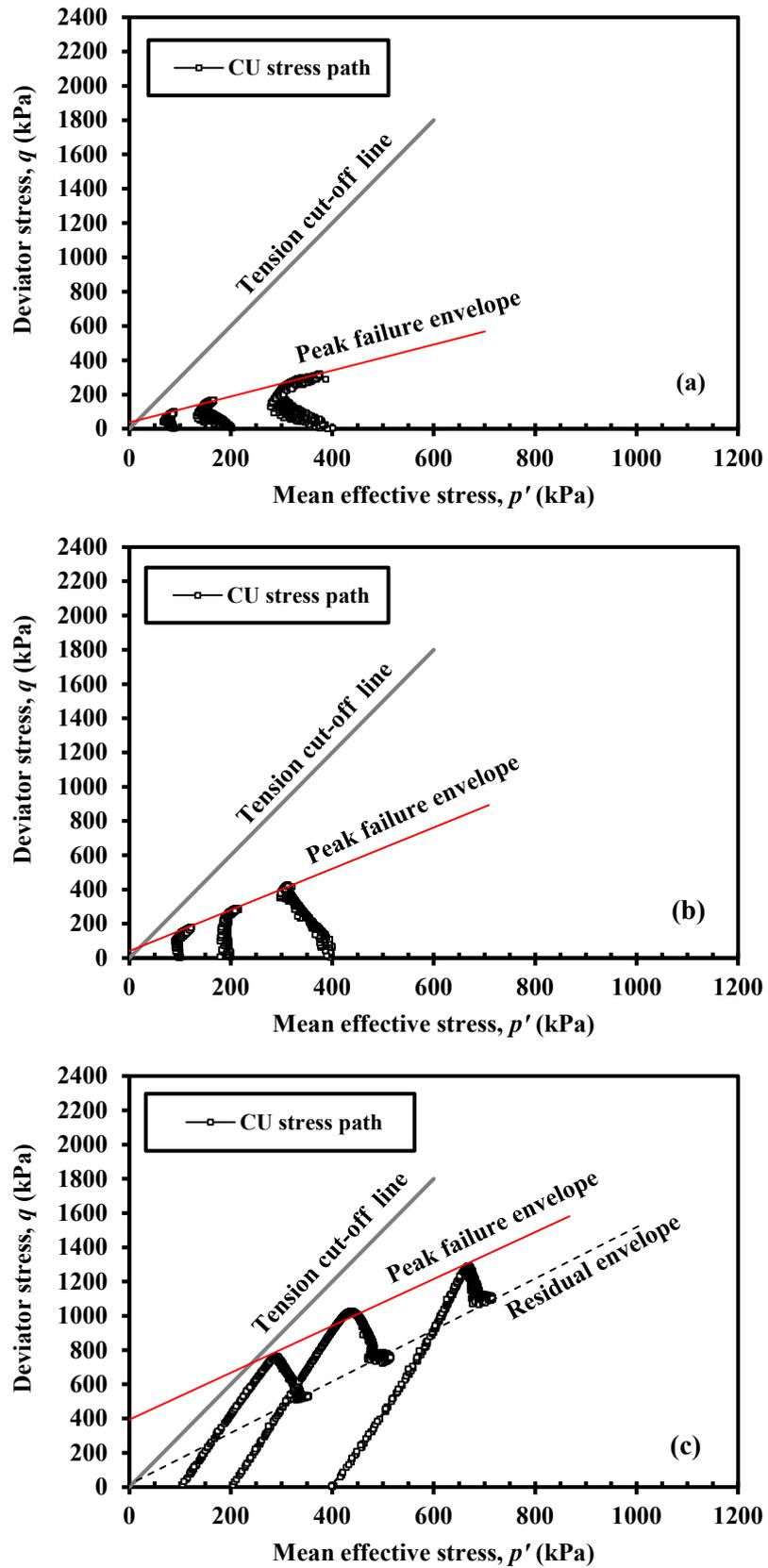


Figure 6.15: Failure envelope of NatSoil (2) clay treated with various amounts of geopolymer: (a) untreated; (b) 10% geopolymer; and (c) 20% geopolymer.

Table 6.1: Undrained effective stress parameters for untreated and geopolymer-treated clays.

Clay type	Geopolymer (%)	\acute{c} (kPa)	ϕ' (deg)
Kaolin	untreated	38	18.8
	10	212	23.7
	20	378	31.0
NatSoil (1)	untreated	22.3	28.6
	10	287	31.3
	20	358	35.9
NatSoil (2)	untreated	17.5	19.6
	10	19.2	30
	20	193	33.6

6.3 CONSTANT- p SHEARING BEHAVIOUR

It is also of interest to consider the response of geopolymer-treated clays in the constant- p test. Figure 6.16 shows the undrained stress behaviour for the kaolin clay treated with 20% geopolymer content under the constant- p stress path (i.e. applied σ_1 is increased while σ_3 is decreased so as to maintain constant total mean stress values of $p = 100, 200$ and 400 kPa, where σ_1 and σ_3 are the major and minor stresses, respectively). As can be seen in Figure 6.16(a), the peak deviator stress increases at very low corresponding strains with the increase in the confining pressures from 100 kPa to 400 kPa. It can also be seen from Figure 6.16(b) that the excess pore water pressure indicates only negative values (high dilation) at the shearing stage, leading to a deviation from its behaviour under the axial compression stress path.

To compare the behaviour of undrained stress-strain of geopolymer-treated kaolin clay under the two different stress paths used, Figures 6.17 and 6.18 show the results of such a comparison for treated kaolin mixtures at confining pressures of 100 kPa and 400 kPa. Generally speaking, the undrained peak deviator stress and the corresponding strain of the geopolymer-treated kaolin clay were found to be highly influenced by the stress path, especially at high confining pressures. As can be seen in Figure 6.17(a), at a confining pressure of 100 kPa, the stabilised specimen under the constant- p stress path attains lower peak q value at much lower strain compared to the axial compression

stress path. The same can be said for specimens subjected to a confining pressure of 400 kPa (Figure 6.17b) but with a more significant impact on the peak stress value. More interestingly, it can be seen from Figure 6.17(a and b) that, for the constant- p stress path, the geopolymer-treated clay soil shows a slight post-peak strain softening in which the post-peak soil strength was almost maintained with the increase of the axial strain. On the contrary, the axial compression stress path shows a sudden post-peak strain softening in which the post-peak soil strength suddenly decreases and a residual value is then attained. Figure 6.17(c and d) indicates that the excess pore-water pressure generated during shearing shows a tendency for the constant- p stress path to develop strong negative pore-water pressure values (high dilation) in comparison to a transition from positive to negative pore-water pressures for the axial compression stress path. This difference in the stress-strain behaviour can be attributed to the reduction in the confining pressure during the constant- p stress path route, which allows for a more specimen dilation during shearing.

In terms of the q - p' space, Figure 6.18 shows a slight deviation in the yield stress path which was found to be highly driven by the increase in the confining pressure at which the constant- p stress path specimens show a tendency towards being lightly over-consolidated at high confining pressure. Such a trend in the constant- p stress path behaviour is mainly due to the reduction in the effective mean stress caused by the strong impact of the negative pore-water pressure developed at high confining pressure.

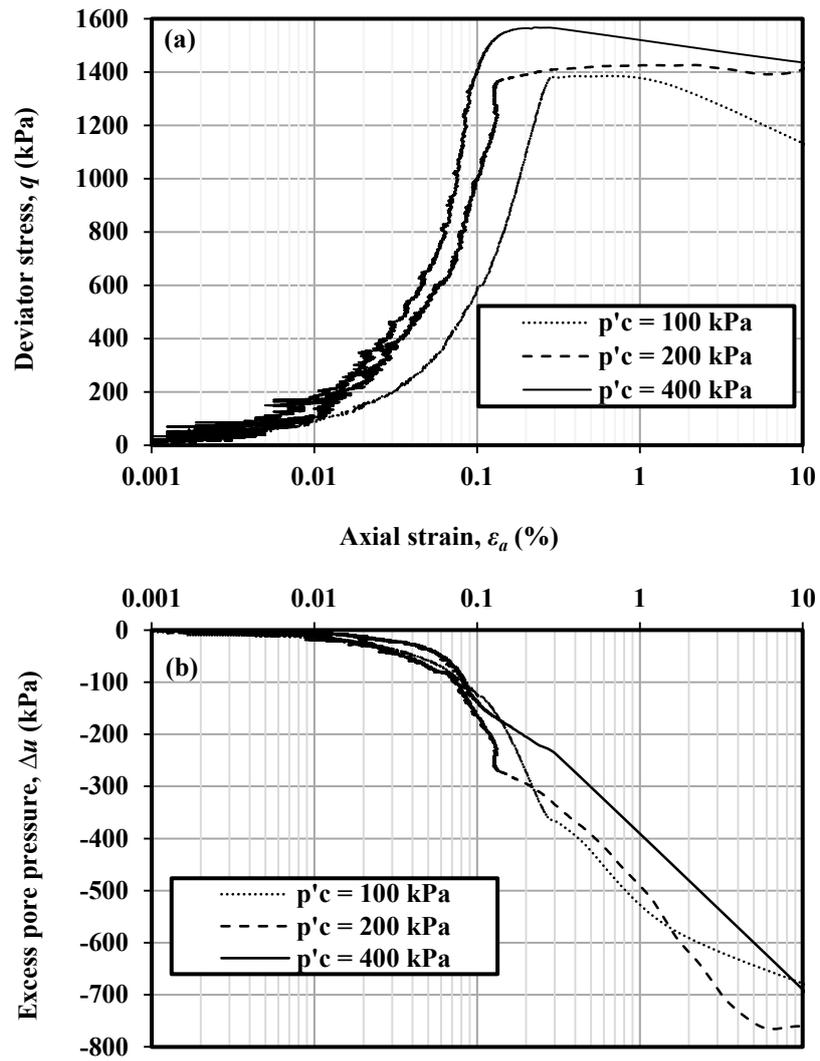


Figure 6.16: Deviator stress and excess pore-water pressure from CU test for treated kaolin clay at 20% geopolymer content under constant- p loading type.

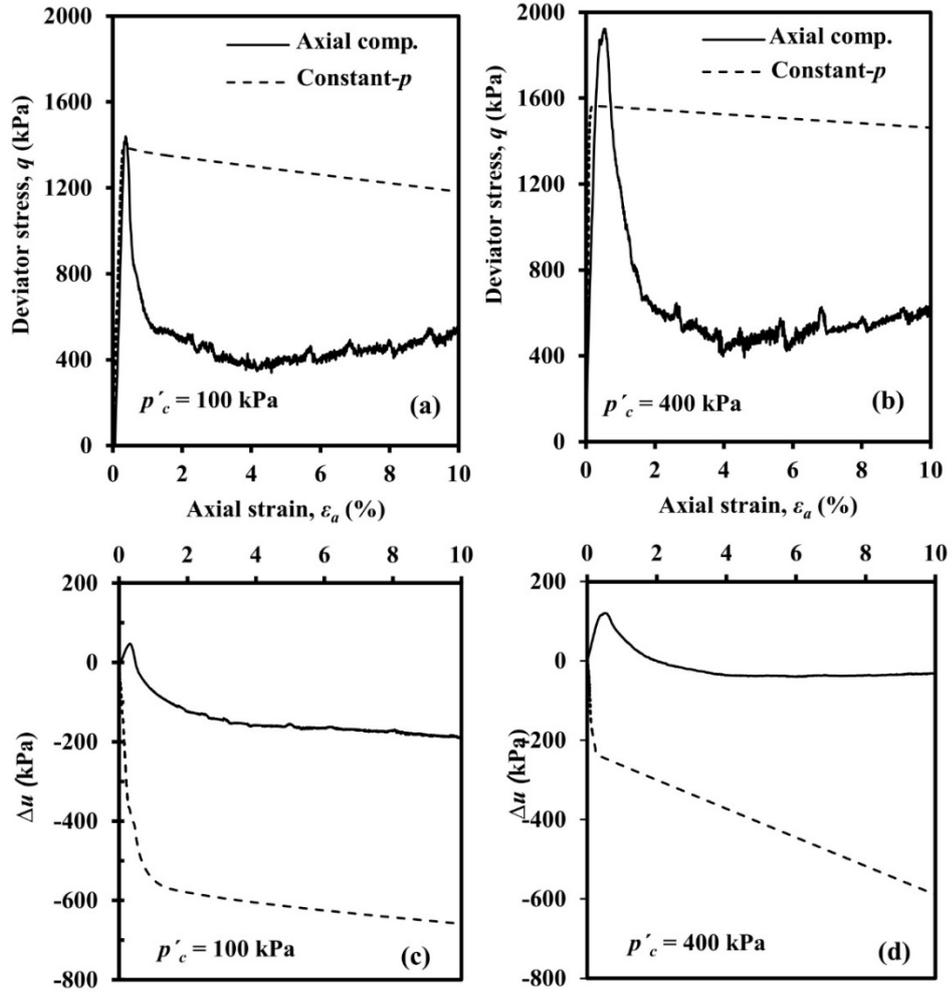


Figure 6.17: Variation of deviator stress and pore-water pressure for treated kaolin clay with 20% geopolymer content under different stress paths.

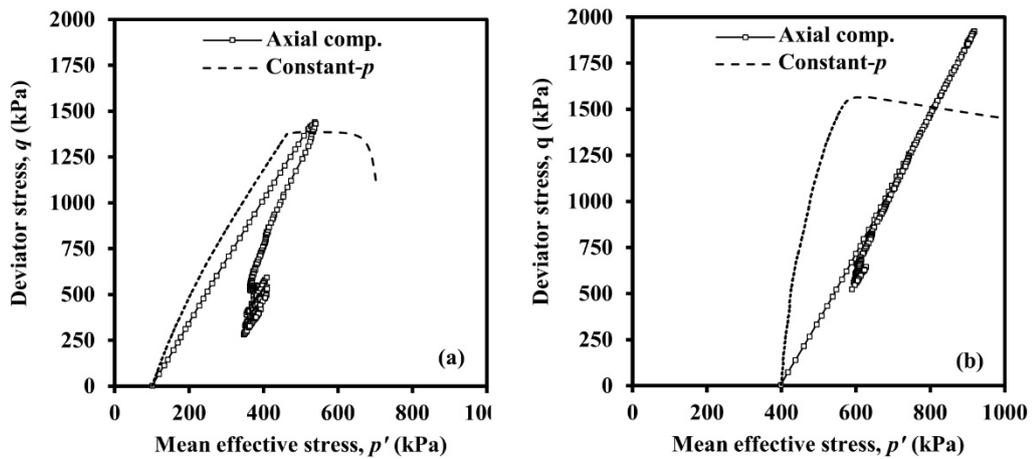


Figure 6.18: Variation of deviator stress vs mean effective stress for treated kaolin clay with 20% geopolymer content under different stress paths and confining pressures: (a) $p'_c = 100$ kPa; and (b) $p'_c = 400$ kPa.

6.4 UNDRAINED VERSUS DRAINED CONDITIONS

To provide a comparison between the undrained and drained behaviour of geopolymers treated clays, Consolidated-Drained (CD) triaxial compression tests, axially sheared to failure at different confining pressures of 100, 200 and 400 kPa, were performed on kaolin specimens treated with 20% geopolymers, Figures 6.19(a-c). The test results, shown in the q - ε_a space, indicated that the confining pressure influences the drained stress-strain behaviour significantly, similarly to the undrained behaviour, where an increase in the confining pressure produces an increase in the peak deviator stress (Figure 6.19a). Furthermore, the volumetric strain showed initial contractive behaviour for all confining pressures until the peak stress was achieved, followed by a highly dilative behaviour that decreased with increasing confining pressure (Figure 6.19b). While the drained stress-strain behaviour of the treated kaolin (Figure 6.19a) may indicate an over-consolidated trend similar to those of the CU conditions, the q - p' curves indicated higher peak deviator stress mobilisation under the CD conditions (Figure 6.19c). This was also observed in the OPC cemented soils and may be related to the higher contractive tendency of treated specimens under undrained shearing and development of the positive excess pore water pressure (Lade 2016). This promotes a lower shear strength for the undrained conditions compared with the corresponding drained conditions; the increase between the undrained and drained specimens were $c' = 53$ kPa and $\phi' = 11^\circ$. This resulted in a slightly steeper peak strength failure envelope for the drained conditions in the q - p' space, yet with almost identical intercept values with the q axis (Figure 6.19c), which suggests the undrained conditions to be the most critical for geopolymers-treated clays.

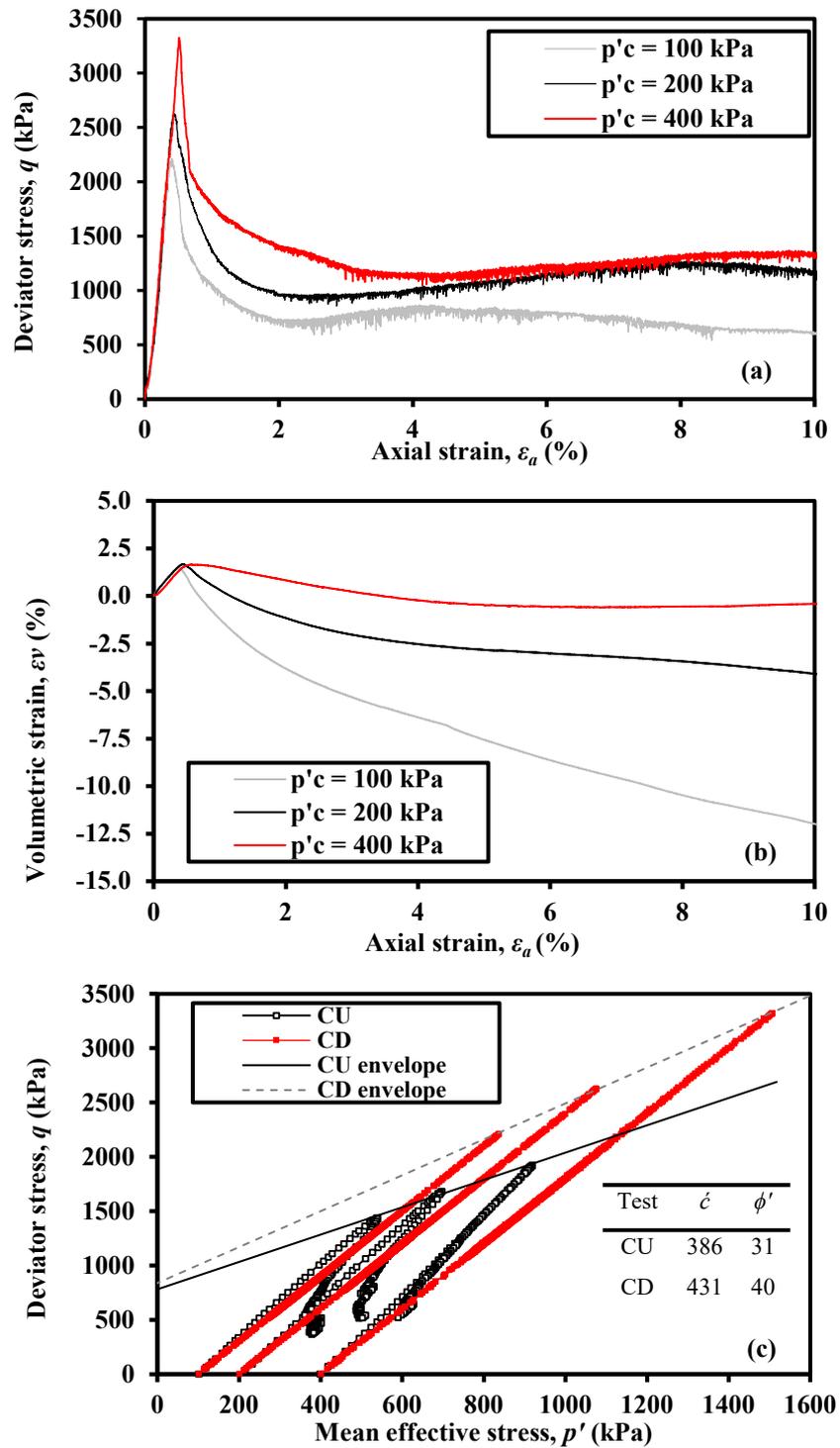


Figure 6.19: Drained shear response of kaolin clay treated with typical 20% geopolymer content.

6.5 SUMMARY

This chapter investigates the undrained shearing behaviour of geopolymer-treated clays incorporating granulated ground blast furnace slag, GGBFS. The following conclusions are drawn from this investigation:

- Geopolymer concentration plays a significant role in the uniaxial and triaxial stress-strain behaviour of geopolymer-treated clay soils. With the increase of geopolymer content, the stress-strain response of treated clays was found to develop progressively from the ductile response into a post-peak brittle response. The increase in geopolymer content mainly increased the yield strength of the clay, contributing to a higher stiffness.
- Irrespective of the clay soil type, the undrained shearing behaviour was found to be strongly correlated to confining pressure. The increase in confining pressure mainly increased the peak stress value of the clay.
- The addition of geopolymer modified the initial quasi-over-consolidated characteristics of the remoulded clays to that of heavily over-consolidated behaviour.
- A linear peak failure envelope in the $q-p'$ space was found to be representative of the test data under undrained conditions. The failure envelope for geopolymer-treated clays was greater than that of the untreated clay. A higher geopolymer concentration resulted in greater effective cohesion and friction angle.
- All the clay soil types gained significant improvements from the geopolymer treatment. However, the tested clays differed markedly in terms of the peak stress, stiffness and contraction/dilation tendencies upon shearing. The primary difference between the three treated clay types was related to the clay mineralogy reflected by the differences in the plasticity and activity indices. Given that the strength gain is related to the formation of the geopolymer gel of treated clays, the formation varied depending on the soil mineralogical components.
- The undrained deviator stress and stiffness, as well as the excess pore-water pressure of geopolymer-stabilised clay, were all found to be dependent on the loading route type (i.e. stress path) as a possible control factor.

- Typical comparison with the peak failure envelope under drained/undrained conditions confirmed the suitability of the undrained conditions for their short-term stability as the most critical case for geopolymer-treated kaolin clay.

The results obtained in this chapter suggest that fly-ash-based geopolymer can be an effective stabiliser for clay soils. However, the geo-mechanical behaviour of geopolymer-treated clays is related to clay mineralogy, implying that geopolymer might not be suitable for all clay types. In the next chapter, rational practical procedures will be presented to check the suitability and to quantify the use of geopolymer for clay treatment.

CHAPTER 7

PRACTICAL PROCEDURES FOR GEOPOLYMER STABILISATION OF CLAY SOILS

7.1 INTRODUCTION

To support broader use of geopolymer for stabilisation of clay soils, rational practical procedures for geopolymer treatment are required. The practical procedures are necessary to:

1. Determine suitability of clay for geopolymer treatment;
2. Select geopolymer content for effective treatment;
3. Determine mixture preparation and evaluation criteria for the enhancement of treated clay; and
4. Apply practical construction considerations.

As discussed in the literature review (Section 2.5.3), such approaches are currently inexistent for geopolymer-treated soils. In this chapter, criteria related to the above points will be discussed in light of plasticity, strength, durability and pH behaviours as discussed in previous chapters. The outcomes will then be synthesised into practical procedures for the use of geopolymer for clay stabilisation through surface mixing applications.

7.2 SUITABILITY OF CLAYS FOR GEOPOLYMER TREATMENT

Several agencies often adopt soil plasticity as an index to identify the suitability of different soil types for treatment by traditional binders (as discussed previously in Section 2.3.3, Chapter 2). To assess specific clay suitability for geopolymer treatment, the results presented in this thesis (Chapter 5) suggest that soil plasticity may provide a suitable indication. This is clearly indicated in Figure 7.1, which shows the relationship between the plasticity index of untreated clay against strength, durability and plasticity of treated clays cured at 28 days; the most common strength measurements in the literature are those recorded after 28 days of curing, thus was adopted.

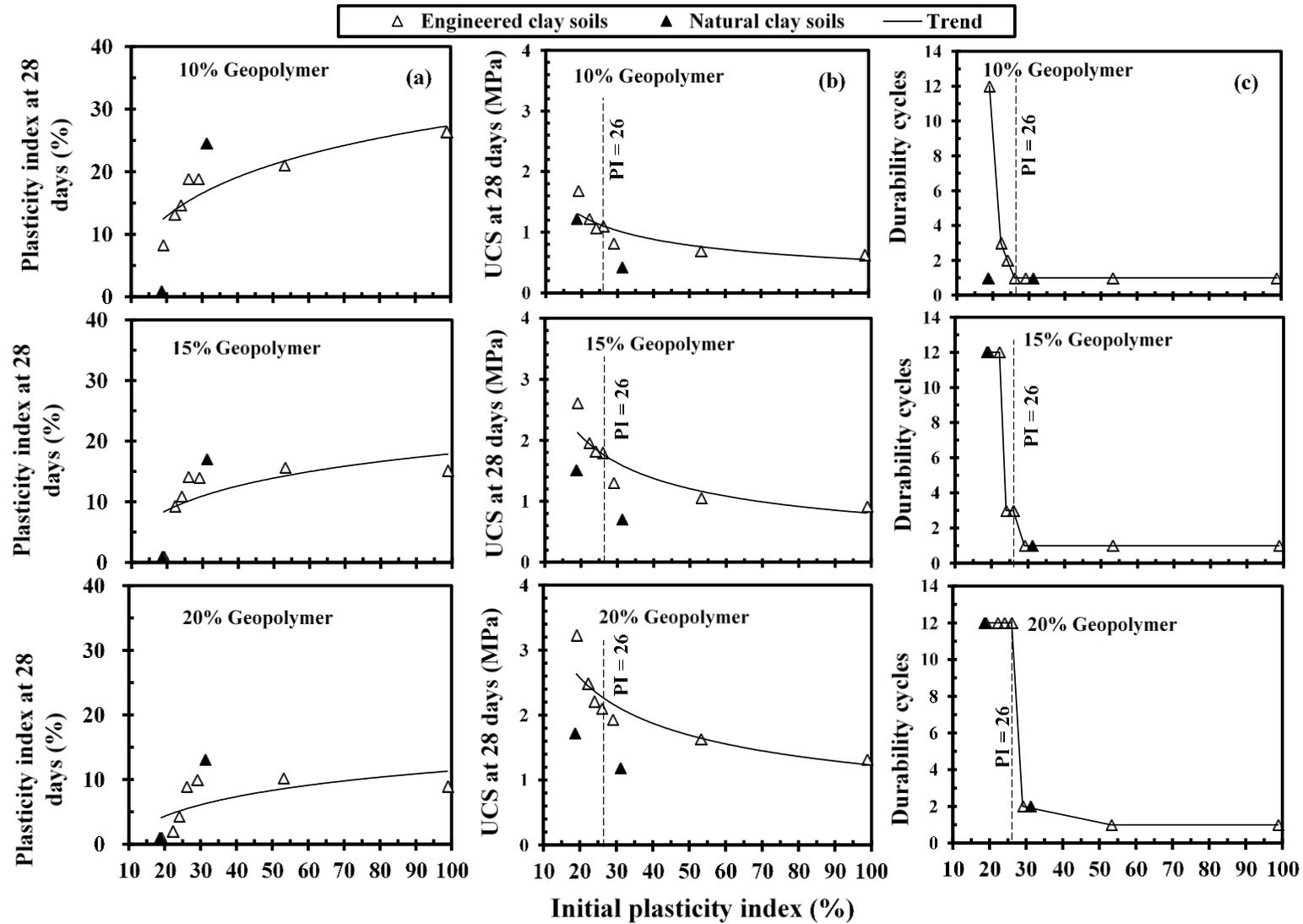


Figure 7.1: Relationship between the plasticity index of untreated clay against strength, durability and plasticity index of treated clay cured at 28 days.

Typically clay soils exhibit improved plasticity, strength and durability when mixed with geopolymer. Yet, as the plasticity of untreated soils increases, not all clays exhibit satisfactory improvement after treatment. As evident from Figure 7.1(a), most treated clays, especially those of high plasticity as tested in this study, produce a substantial reduction in PI at 28 days of curing when treated with geopolymer. However, the first increment of geopolymer addition (i.e. 10%) is generally most effective in reducing PI, with subsequent additions showing less benefit.

In terms of UCS (Figure 7.1b), kaolin and kaolin-sand tested clays with PI ranging from 18.8–26% and treated with 10% geopolymer, show adequate strength values of $UCS \geq 1$ MPa. It should be noted that 1 MPa strength threshold was suggested herein for treatment evaluation as it complies with the criterion mentioned in the literature for achieving a minimum strength increases of 0.35 MPa for treated soils over the strength of the corresponding untreated soils (Little et al. 2009). Nevertheless, the increase in UCS to greater than 1 MPa for kaolin-bentonite clays of very high plasticity range (PI = 29–98.8%) is still achievable but requires an increase in the geopolymer content to 15–20%.

Long-term stabilisation performance depends not only on strength improvement but also upon attaining adequate durability, as modelled by successive wetting-drying periods in this study (Chapter 5, Section 5.5). As can be seen in Figure 7.1(c), clays with $PI \leq 26\%$ were found to be adequately stabilised with 10-20% geopolymer as they survived the designated 12 wetting-drying cycles: the lower the PI, the lower the geopolymer content required for successful durability performance. In contrast, inadequate durability performance was detected for all tested clays with $PI > 26\%$. Soil mineralogy responsible for the diversity of plasticity index seems to significantly affect the ability to intimately mix geopolymer with the clay to produce a reasonably homogenous matrix of stabilised agglomerates. This, in turn, affects the strength and durability performance of the stabilised clays. Therefore, geopolymer treatment of very high plasticity clays for the purpose of long-term stabilisation (i.e. durable strength enhancement) should be considered sceptically, as clearly shown in Figure 7.1. Instead, the treatment might be considered for the purpose of short-term enhancement only (i.e. plasticity reduction and short term strength enhancement). Regarding favourable clay for stabilisation using geopolymer, the data in this study broadly supports a recommendation of $PI \leq 26\%$.

7.3 SELECTION OF GEOPOLYMER QUANTITY

While the plasticity index focuses on the soil type and its fundamental ability to be stabilised, the pH (as discussed previously in Section 5.8, Chapter 5) provides a guide to the geopolymer dosage required for effective treatment. This ensures that sufficient geopolymer is added to the clay to ensure adequately high pH to sustain the geopolymerisation reaction and the associated strength development throughout the curing period. Eades et al. (1966) proposed a similar criterion for lime treatment, also adopted for lime-fly ash treatment (Sharma et al. 2012), such that the minimum lime content required for effective treatment produced a minimum pH of 12.4 after 1 hour of curing. The results discussed previously (Chapter 5, Figure 5.32) suggest a similar pH level requirement for clay soils stabilised with fly-ash based geopolymer. To confirm this, two natural clays were examined after 1 hour of mixing at various geopolymer contents and the results are shown in Figure 7.2. It can be observed that the initial pH values of untreated NatSoil (1) and NatSoil (2) are 4.3 and 8.5, respectively, indicating acidic and alkaline clay soils. With the high alkalinity of the geopolymer used (i.e. $\text{pH} \approx 13$), a content of geopolymer of 12.5% is required to raise the initial pH of NatSoil (1) to the suggested pH threshold of 12.4. For NatSoil (2), a minimum geopolymer content of 22.5% was required to achieve a similar pH increase; this clearly indicates that different clays respond differently to geopolymer treatment.

It should be noted that the geopolymer contents derived from the pH test (Figure 7.2) satisfies only the minimum requirement of geopolymer content and does not necessarily produce adequate cured strength and durable performance. Therefore, a suitable geopolymer content must be determined through strength and durability testing together on trial mixtures utilising geopolymer content equal or higher than the minimum requirements (i.e. pH test).

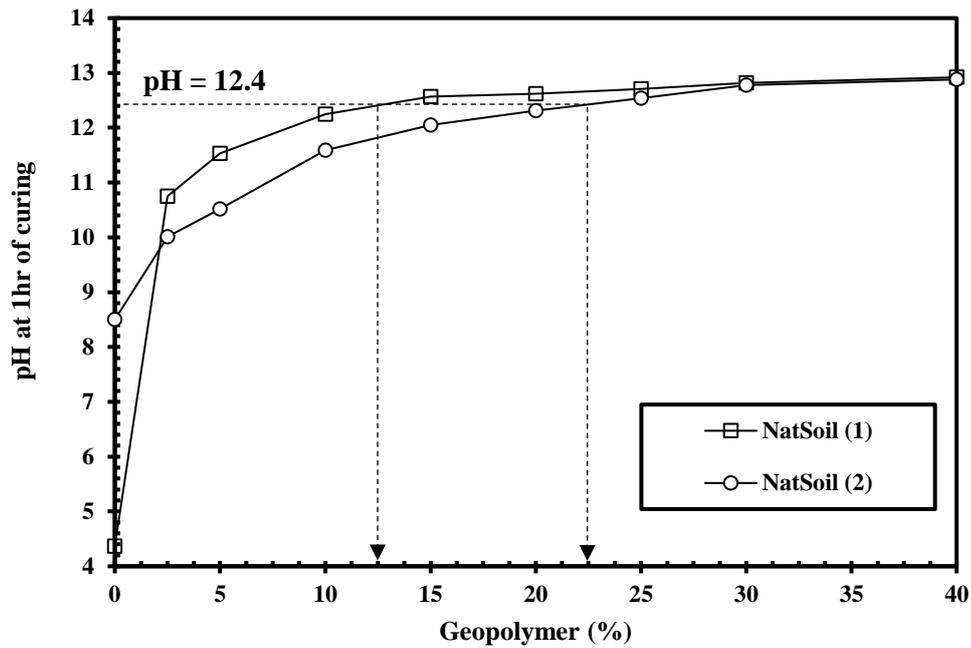


Figure 7.2: pH of natural clays mixed with different geopolymer contents and cured for 1 hr.

7.4 PREPARATION AND EVALUATION METHODS OF GEOPOLYMER-SOIL MIXTURE

To evaluate the potential for a soil to be treated, the geopolymer involves a process of multiple stages, including mixture preparation, specimen preparation, curing and testing. The geopolymer treatment in the thesis, as suggested throughout the methodology presented earlier in Chapter 3, was based on mixing and testing at the optimum compaction properties, as required for the optimum surface mixing. From this, the following can be suggested for field use by practitioners:

7.4.1 Mixing preparation

The fly-ash and granulated ground blast furnace slag, GGBFS can first be added as a partial replacement of pulverised dry clay soil to form the total dry material in the mixture, similarly to that suggested in Chapter 3. Based on a set of trial fly-ash and GGBFS ratios, premixed activator solution and free water can be determined then added before mixing. The volume of free water should be varied for each trial mixture to allow for an investigation of the optimum moisture content. For practical use, typical details of mixture proportions of fly-ash, GGBFS, activator and free added water to reach an optimum compaction of 1000 g treated clay corresponding to various geopolymer contents are presented in Tables 7.1 and 7.2.

Table 7.1: Details of geopolymer mixture proportions to stabilise 1000 g of clay.

Geopolymer (%)	Fly-ash+GGBFS (g)	Fly-ash (g)	GGBFS (g)	Sodium silicate^a (g)	Sodium hydroxide^{a,b} (g)
2	20.4	16.3	4.1	5.7	2.5
4	41.7	33.3	8.3	11.7	5.0
6	63.8	51.1	12.8	17.9	7.7
8	87.0	69.6	17.4	24.3	10.4
10	111.1	88.9	22.2	31.1	13.3
12	136.4	109.1	27.3	38.2	16.4
14	162.8	130.2	32.6	45.6	19.6
16	190.5	152.4	38.1	53.3	22.9
18	219.5	175.6	43.9	61.4	26.4
20	250.0	200.0	50.0	70.0	30.0
22	282.1	225.6	56.4	78.9	33.9
24	315.8	252.6	63.2	88.4	37.9
26	351.4	281.1	70.3	98.3	42.2
28	388.9	311.1	77.8	108.8	46.7
30	428.6	342.9	85.7	119.9	51.5

^a Solution form; ^b 14 Molar concentration

Table 7.2: Free water in (g) required to reach a designated moisture content in 1000 g of treated clay.

Moisture content (%)	Geopolymer (%)														
	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30
8	76.8	73.4	69.9	66.2	62.3	58.3	54.1	49.7	45.1	40.3	35.2	29.8	24.2	18.2	11.9
9	87.0	83.8	80.5	77.1	73.5	69.7	65.8	61.6	57.3	52.8	48.0	43.0	37.7	32.1	26.2
10	97.2	94.2	91.1	87.9	84.6	81.1	77.4	73.5	69.5	65.3	60.8	56.1	51.2	46.0	40.5
11	107.4	104.6	101.8	98.8	95.7	92.4	89.0	85.4	81.7	77.8	73.6	69.3	64.7	59.9	54.7
12	117.6	115.0	112.4	109.7	106.8	103.8	100.6	97.3	93.9	90.3	86.5	82.4	78.2	73.8	69.0
13	127.8	125.5	123.0	120.5	117.9	115.1	112.3	109.3	106.1	102.8	99.3	95.6	91.7	87.6	83.3
14	138.0	135.9	133.7	131.4	129.0	126.5	123.9	121.2	118.3	115.3	112.1	108.8	105.2	101.5	97.6
15	148.2	146.3	144.3	142.3	140.1	137.9	135.5	133.1	130.5	127.8	124.9	121.9	118.8	115.4	111.9
16	158.4	156.7	155.0	153.1	151.2	149.2	147.2	145.0	142.7	140.3	137.7	135.1	132.3	129.3	126.2
17	168.6	167.1	165.6	164.0	162.3	160.6	158.8	156.9	154.9	152.8	150.6	148.2	145.8	143.2	140.5
18	178.8	177.5	176.2	174.9	173.5	172.0	170.4	168.8	167.1	165.3	163.4	161.4	159.3	157.1	154.7
19	189.0	188.0	186.9	185.7	184.6	183.3	182.0	180.7	179.3	177.8	176.2	174.6	172.8	171.0	169.0
20	199.2	198.4	197.5	196.6	195.7	194.7	193.7	192.6	191.5	190.3	189.0	187.7	186.3	184.9	183.3
21	209.4	208.8	208.2	207.5	206.8	206.1	205.3	204.5	203.7	202.8	201.8	200.9	199.8	198.8	197.6
22	219.6	219.2	218.8	218.4	217.9	217.4	216.9	216.4	215.8	215.3	214.7	214.0	213.4	212.6	211.9
23	229.8	229.6	229.4	229.2	229.0	228.8	228.5	228.3	228.0	227.8	227.5	227.2	226.9	226.5	226.2
24	240.0	240.0	240.1	240.1	240.1	240.1	240.2	240.2	240.2	240.3	240.3	240.3	240.4	240.4	240.5
25	250.2	250.5	250.7	251.0	251.2	251.5	251.8	252.1	252.4	252.8	253.1	253.5	253.9	254.3	254.7
26	260.4	260.9	261.3	261.8	262.3	262.9	263.4	264.0	264.6	265.3	265.9	266.7	267.4	268.2	269.0
27	270.6	271.3	272.0	272.7	273.5	274.2	275.1	275.9	276.8	277.8	278.8	279.8	280.9	282.1	283.3
28	280.8	281.7	282.6	283.6	284.6	285.6	286.7	287.8	289.0	290.3	291.6	293.0	294.4	296.0	297.6
29	291.0	292.1	293.3	294.4	295.7	297.0	298.3	299.7	301.2	302.8	304.4	306.1	307.9	309.9	311.9
30	301.2	302.5	303.9	305.3	306.8	308.3	309.9	311.6	313.4	315.3	317.2	319.3	321.5	323.8	326.2
31	311.5	313.0	314.5	316.2	317.9	319.7	321.6	323.5	325.6	327.8	330.0	332.4	335.0	337.6	340.5
32	321.7	323.4	325.2	327.1	329.0	331.1	333.2	335.4	337.8	340.3	342.9	345.6	348.5	351.5	354.7
33	331.9	333.8	335.8	337.9	340.1	342.4	344.8	347.3	350.0	352.8	355.7	358.8	362.0	365.4	369.0
34	342.1	344.2	346.5	348.8	351.2	353.8	356.5	359.3	362.2	365.3	368.5	371.9	375.5	379.3	383.3
35	352.3	354.6	357.1	359.7	362.3	365.1	368.1	371.2	374.4	377.8	381.3	385.1	389.0	393.2	397.6
36	362.5	365.0	367.7	370.5	373.5	376.5	379.7	383.1	386.6	390.3	394.2	398.2	402.5	407.1	411.9
37	372.7	375.5	378.4	381.4	384.6	387.9	391.3	395.0	398.8	402.8	407.0	411.4	416.1	421.0	426.2
38	382.9	385.9	389.0	392.3	395.7	399.2	403.0	406.9	411.0	415.3	419.8	424.6	429.6	434.9	440.5
39	393.1	396.3	399.6	403.1	406.8	410.6	414.6	418.8	423.2	427.8	432.6	437.7	443.1	448.8	454.7
40	403.3	406.7	410.3	414.0	417.9	422.0	426.2	430.7	435.4	440.3	445.4	450.9	456.6	462.6	469.0

7.4.2 Specimen preparation

The specimen preparation methods used to evaluate geopolymer-soil mixtures are generally similar to those usually used for OPC and lime-soil mixtures. For the surface mixing application, all test specimens (i.e. strength and durability) shall be compacted considering the maximum dry unit weight (MDU). It should be noted that a small unit weight variation may cause some difficulties to accurately evaluate the effect of other variables such as geopolymer content and curing time. Attention should be taken to compact any treated soil within 30 min of mixing to avoid any possible effects of the initial binder setting.

7.4.3 Curing conditions

As for soil stabilisation using traditional binders, all test specimens of geopolymer treated clays should be cured in a sealed container at humid environment and room temperature; current study adopted curing conditions of 60% humidity and 20-25 °C temperature. For wetting-drying durability tests, curing period of 7 days is required, whereas for unconfined and triaxial tests curing should be extended to 28 days for effective evaluation.

7.4.4 Testing

The test methods used to evaluate geopolymer-soil mixtures to satisfy specific design requirements are generally identical to those usually used for OPC and lime-soil mixtures. In this research, these tests were investigated to evaluate the properties of the geopolymer-soil mixture as described in Chapter 3. These include:

1. Atterberg limits;
2. Wetting-drying durability tests;
3. Unconfined Compressive Strength; and
4. Compressive static triaxial strength – undrained/drained conditions.

Based on the treatment objective, specific testing, e.g. undrained triaxial tests subjected to cyclic loading for road and railway structures (Hoyos et al. 2004), are recommended for the evaluation of the stabilised soil. However, since such advanced testing methods have not been extensively investigated in geopolymer-clay literature, satisfactory performance has not been considered here as it is outside the scope of this thesis.

7.5 SYSTEMATIC APPROACH FOR SOIL TREATMENT USING GEOPOLYMER

The PI and pH criteria discussed previously in this chapter are used to develop a systematic approach for the effective treatment of clay soils with fly-ash based geopolymer, as shown in Figure 7.3. This approach divides the clay soil stabilisation performance into two categories (i.e. durable and non-durable) and the clay to be stabilised into two plasticity groups (i.e. high and low). The first phase of the proposed systematic approach considers the suitability of the clay for geopolymer treatment; plasticity index (PI) was employed as a suitability index for treatment. In the systematic approach, clay soils with $PI \leq 26\%$ can achieve durable stabilisation with geopolymer, whereas the use of geopolymer for higher plasticity clays is only recommended for PI and strength enhancements, not for durability. The second phase of the systematic approach determines the minimum geopolymer content required for treatment. The pH test presented previously in Figure 7.2 serves to ensure that a suitable percentage of geopolymer is added to the clay to satisfy all of the immediate occurring reactions and to provide adequate stabiliser to sustain geopolymerisation throughout curing. The suggested pH method briefly devises a series of pH tests to be performed on the clay after 1 hour of mixing with different geopolymer contents. A pH value of 12.4 is then suggested as a threshold to determine the minimum geopolymer content. It should be noted that this pH-method is restricted for the determination of the pH requirement for geopolymerisation. Strength and durability enhancements are related to the formation of the geopolymer cementitious product, which is shown here to be affected by the clay mineralogy, pulverisation level and curing time. Thus, different tests are recommended in the proposed procedures as a third phase to evaluate the percentage of improvement and to comply with any specific design requirements. It should be noted that the proposed systematic approach is specific for certain clays treated with a specific mixture of fly-ash based geopolymer and further future investigation is thus necessary to expand the present findings by including other types of clay. The proposed approach is not necessarily applicable for all geopolymer types or mixtures since the rate of formation and amount of cementitious products of treated clay might be different. However, the rigorous and validated approach presented in this thesis will promote the use of geopolymer as a more sustainable chemical stabiliser for geotechnical applications.

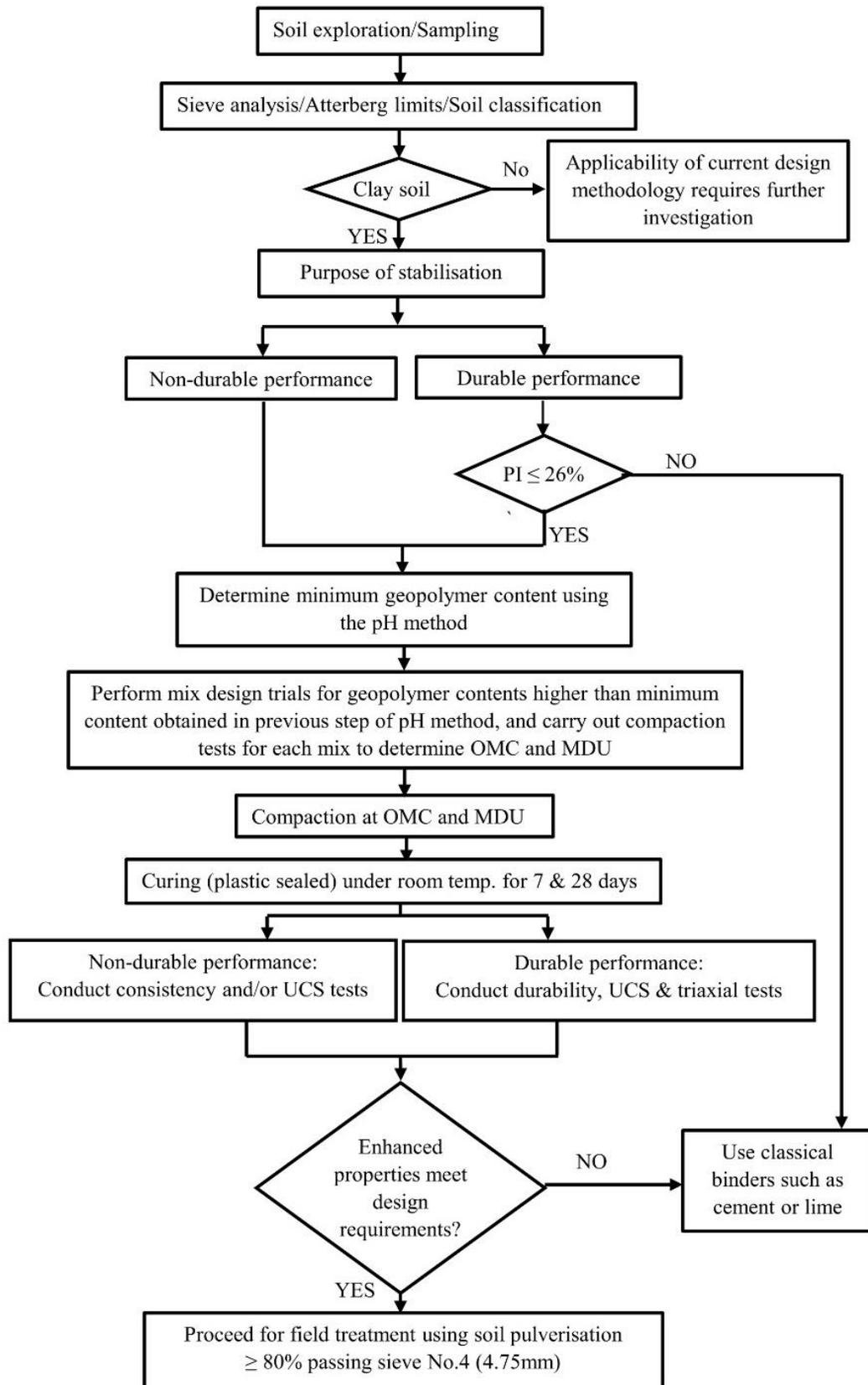


Figure 7.3: Proposed design practical procedures for geopolymer-treated clay soil.

7.6 GENERAL PRACTICAL CONSTRUCTION CONSIDERATIONS

For geotechnical applications where surface mixing is employed, geopolymer may be applied to the soil (after considering of the flow chart shown in figure 7.3) following some basic steps, including soil preparation (i.e. pulverisation), fly-ash and GGBFS spreading, mixing and activating, compacting and curing. The following section outline pertinent aspects of these processes. It is important to highlight that such general aspects are a replicate to those adopted in the laboratory control conditions of this investigation; thus they required future research.

7.6.1 Pulverisation

As previously discussed in Chapter 5, one of the most important aspects to control during surface mixing of clay soils using geopolymer is soil pulverisation. The test results of the current investigation (Chapter 5, Section 5.7) confirmed the degree of pulverisation as an influential factor in the successful geopolymer treatment of soil. The test results broadly support the use of OPC-soil related Portland Cement Association (2001) specification for geopolymer treatment. The specification states that the soil must be pulverised to ensure 80% passes through sieve No. 4 (4.75 mm) and 100% through 2.5mm. For soils of heavy clay content, adequate pulverisation can best be achieved through multi-stages.

7.6.2 Geopolymer content

When geopolymer is applied to the pulverised soil, the content can be easily determined as a weight percentage of oven-dry soil material. Written instructions can be made available for field engineers for the correct fly-ash, GGBFS, activator and water contents. Tables 7.1 and 7.2 can be used for this purpose, considering the total weight of the treated soil layer.

7.6.3 In-situ mixing

The basic objective of surface mixing is to provide thorough mixing of geopolymer ingredients and soil. Mixing of the geopolymer-soil system is generally similar in concept to that of other binders. This usually involves pre-wetting the pulverised soil below optimum moisture content, spreading the binder and mixing with water addition using special mixers (Portland Cement Association 1995). However, a slight modification to the mixing method should be considered due to the presence of a liquid

activator. A detailed study on in-situ mixing methods is beyond the scope of this thesis and should be considered in any future research.

7.6.4 In-situ compaction

It is well documented in the literature of traditional binder that strength of treated soils is correlated to the unit weight of treated mixture. To maximise the strength for surface mixing, treated soils should be compacted to the maximum unit weight using appropriate machines (Portland Cement Association 1995). Since the mechanism of soil stabilisation using geopolymer is based on the formation and hardening of cementitious products, it is essential to start compaction as soon as possible after final mixing has been completed. No compaction for layers exceeds the depth that can be compacted, usually 250 mm (Portland Cement Association 1995); otherwise, the mixing and the compaction should be conducted in multiple layers. Optimum compaction parameters can be checked after compaction of each layer using related field specifications.

7.6.5 In-situ curing

Following compaction, a moisture-retaining cover should be used immediately to cover the soil surface to retain the moisture necessary for adequate geopolymerisation and achieving targeted enhancement. Bitumen materials, waterproof paper or plastic sheets can be used to achieve satisfactory curing (Portland Cement Association 1995).

7.7 SUMMARY

This chapter presents a systematic practical procedure to check the suitability and to quantify the use of geopolymer for an effective clay treatment. Plasticity index was suggested to check the suitability of clay for the geopolymer treatment. The pH test for the geopolymer-soil slurry was introduced to determine the minimum content of geopolymer required for an effective treatment. Other practical construction considerations were also presented and discussed. The proposed practical procedures are expected to provide a significant contribution to the current soil stabilisation practice of clay soils.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

8.1 CONCLUSIONS

Geopolymer is a cementitious material that can replace ordinary Portland cement in several geotechnical engineering applications such as soil stabilisation, with the advantage of much lower harmful emissions and energy consumption. Despite these advantages, the use of fly-ash based geopolymer for soil stabilisation as a stabiliser is still not widely recognised within the geotechnical industry. This underutilisation is generally attributed to the lack of practical geopolymer mixtures suitable for ambient curing. Also, the literature still lacks systemic information in relation to the suitability of this binder for stabilisation of different soil types and the determination of dosages required for effective treatment. In response, this study has developed and tested a novel geopolymer mixture for stabilisation of clay soils at ambient temperature. To accomplish this goal, a series of experimental tests were conducted on engineered and natural clay types. The clays used were examined and evaluated for compaction, plasticity and unconfined compressive strength. Further evaluation included the wetting-drying, freezing-thawing and leaching durability performance, permeability, pH level and effect of pulverisation on treatment. Moreover, the static shear strength behaviour was investigated by conducting triaxial tests. This investigation allowed developing the current state of knowledge by identifying some influential factors and reliable practical procedures for effective stabilisation. Several important conclusions have been drawn based on the results of this research, as presented in the following sections.

8.1.1 Enhanced geopolymer mixture for soil stabilisation at ambient temperature

Progressive strength enhancement of kaolin clay treated with different combinations of fly-ash, granulated ground blast furnace slag, GGBFS and activator was evaluated in Chapter 4 of this thesis by conducting UCS tests and SEM analysis on treated specimens at different curing periods. It was found that strength and stiffness characteristics of kaolin clay treated with fly-ash based geopolymer could be enhanced significantly with the addition of GGBFS, on both the short- and long-term base. The short-term (i.e. 7 days) strength enhancement of geopolymer-treated specimens incorporating GGBFS was found to be equivalent to that obtained from soil stabilised

with OPC, whereas the enhancement was more prominent for the long-term basis (i.e. 90 days). However, this experimental study found no optimum GGBFS content, and over the range of the GGBFS content used, it was found that the strength increases with the increase of the GGBFS content in the mixture. This is also true for the activator content, where its increase resulted in persistent strength gain in the treated specimens. Based on the tests results, GGBFS/ash-GGBFS percentage of 20% and activator/ash-GGBFS percentage of 40% were found to promote strength level in the geopolymer treatment that fulfils the requirements of most geotechnical engineering applications, thus were selected for future investigation.

8.1.2 Engineering properties of geopolymer-treated clay soils

A comprehensive series of experimental tests were undertaken in Chapter 5 to characterise the engineering properties of treated clay soils using geopolymer mixture synthesised at GGBFS/ash-GGBFS of 20% and activator/ash-GGBFS of 40%, as per Section 8.1.1. The main variables investigated herein was the effect of geopolymer content, curing period and clay type. The parametric study yielded the following observations:

- The addition of geopolymer showed great potential for upgrading clay soils with a broad range of mineralogy/plasticity. Geopolymer addition was found to reduce plasticity, increase the strength and durability of treated clays.
- The geopolymer content and curing time are the main determinant of plasticity reduction and strength enhancement. Increasing the geopolymer content generally decreased plasticity and increased strength; this impact became more prominent with the curing time. The effect of curing time on strength development was seen to be more prominent from 7 days to 28 days than observed from 28 days to 90 days.
- Strength development with the increase of geopolymer content and curing time is unique for each clay type as it seems to be highly driven by the plasticity/activity and mineralogy of treated clays. For specific geopolymer content and curing time, low plasticity/activity clays showed increased strength gain compared to high plasticity/activity clays.
- Insufficient geopolymer content within a treated clay soil may result in problems related to the strength and durability performance under wetting-drying conditions. However, despite the utilisation of high geopolymer

content, unsatisfactory durability performance was detected in some treated soils. For specific geopolymer content, low plasticity/activity clays showed durable performance compared to high plasticity/activity clays.

- The durability of geopolymer-treated clays seems to exhibit less stable performance (i.e. high volumetric changes and low strength) in the freezing-thawing than wetting-drying tests, confirming the retardation of the geopolymerisation reaction at very low temperature. During curing and under an extreme field wetting condition, leaching of binder was found to be a minor concern for geopolymer clay stabilisation in terms of the residual strength.
- Increasing the geopolymer content slightly increased the permeability of the soil with heavy clay content which contradicts the effect of cement and lime in treated soils. It was postulated that the role of non-plastic silty fly-ash and GGBFS particles in decreasing the overall surface area of the particles within the mixture has dominated the effect of cementation in blocking voids.
- The level of enhancement of treated clays was found to be significantly impacted by the variation in the pulverisation level of clay. High pulverisation level (i.e. 100% < 4.74 mm sieve) of clay before treatment was found to achieve high strength gain compared to lower levels. The effect of pulverisation level on the strength enhancement was also found to be more pronounced at long-term curing periods. In terms of durability performance, the pulverisation level showed no pronounced influence on the ability of treated clays to withstand wetting-drying cycles. However, a slight loss of material was observed throughout the successive wetting-drying durability cycles for mixtures at medium and low pulverisation. Interestingly, the tested mixtures at high pulverised level showed a significant increase in UCS values within the first six cycles before stabilising for the remaining six cycles due to the heat effect induced by the drying phased of the durability test.
- The pH performance of geopolymer-treated clays has a significant impact on the treatment. After 1 hour of curing, it was found that the addition of geopolymer tends to increase the initial pH of all treated clays considerably. As curing continues, the pH level decreased slightly. The reduction in pH was found to be soil dependent. However, the degradation in the pH of treated clays coincided with an increase in strength performance. Considering the pH

degradation of geopolymer-treated soils with curing time, it is was found necessary to increase the geopolymer content to maintain higher pH throughout curing, to ensure the continuity of geopolymer reaction and the corresponding formation of cementitious products.

8.1.3 Undrained Shear strength of geopolymer-treated clays

The shear behaviour of geopolymer-treated clays was investigated in Chapter 6 with the help of the triaxial compression tests under static loading. The main variables investigated were the effect of geopolymer content, confining pressure, clay type and loading type on the shearing response. The findings may be summarised as follows:

- It was found that the geopolymer content has an essential role of influence on the undrained shearing behaviour of geopolymer-treated clays. With the addition of geopolymer, the undrained stress-strain behaviour of geopolymer treated clays was found to be progressively strain-softening in nature.
- It was found that the level of cementation induced by geopolymer content increases both the peak strength and initial stiffness of treated clays. Increasing the level of cementation resulting from increasing geopolymer content likely resulted in an increased amount of cementitious bonds which can be held responsible for the high undrained shear performance at the initial stages of loading (i.e. before yield).
- The effect of pre-shear consolidation pressure was also found to have a significant effect on the stress-strain behaviour of treated clays. Increasing the confining pressure generally increased the peak strength and initial stiffness of treated clay. It was suggested that an increase in the confining pressure (at the consolidation phase) might causes a slight de-structure in the cemented soil, resulting in a behaviour controlled by friction as well as cementitious bonds. With the increase of confining pressure, the potential damage of the cementitious structure of the specimen from the consolidation phase was suggested to be insignificant. This implied that the strength was still governed by the cementitious bonds with very little contribution from the mobilised friction associated with the change in the specimen volume due to consolidation. The residual post-peak strength was attributed to the frictional response.

- With the addition of geopolymers, the peak positive excess pore water pressure was found to be developed at low shear strain near the peak stress value, and thereafter the excess pore water pressure decreased to negative values at the higher level of shear strain. This indicated an initial specimen contraction at low strain followed by slight dilation at higher strain up until failure.
- The results revealed that the addition of 10% geopolymers by weight could change the initial quasi-over-consolidated characteristics of the remoulded clays to that of heavily over-consolidated. Clay with very high plasticity/activity indices was an exception, as the performance after treatment was less prone to dilation upon shearing than other treated clays. However, at high geopolymer content, all treated clays gave a typical heavily over-consolidated response.
- Linear failure envelopes could be used to approximate the undrained peak shear strength of treated clays in the $q-p'$ space. For all clay types, the ability of geopolymer treatment to move the stress states beyond the untreated clay failure envelope was also evident, consistently generating a geopolymer treatment failure envelope greater than that of the untreated clay.
- For each treated clay, a unique set of shear strength parameters can be derived corresponding to the peak stress condition. Generally, the cohesion intercept and the friction angle increased noticeably with the increase in geopolymer content.
- The level of enhancement in the strength, initial stiffness and shear strength parameters, as well as the change in response of pore water pressure of treated clays, were found to be significantly impacted by the variation in clay mineralogy and plasticity/activity indices. It was suggested that the difference in the shear performance between different geopolymer treated specimens might be related to the interactions between the geopolymer and individual clay mineralogy (type and associated volume present). These factors seemed to influence the geopolymerisation process and the particular characteristics of bonded clusters that formed along the shear plane. This suggested a need to treat high problematic clay (i.e. high plasticity/activity) with a higher amount of geopolymer to reach similar post-yield behaviour to that of the low problematic clay.

- A comparison of the peak failure envelopes of specimens subjected to drained/undrained conditions confirmed the suitability of undrained conditions for the short-term stability as the most critical case for geopolymer-treated clays.
- The overall shear performance of geopolymer-treated clays was found to be dependent on the loading stress path as a possible control factor.

8.1.4 Preliminary practical procedures for treatment of geopolymer- stabilised clay soils

Based on the outcomes of this thesis, a systematic approach in the form of an easy-to-use flowchart and procedures was presented and recommended for the use by practitioners.

8.2 RECOMMENDATIONS FOR FUTURE RESEARCH

Although this thesis has provided a useful contribution in regards to the use of geopolymer for stabilisation of clay soils, further experimental studies on the following aspects are recommended for future work.

- An experimental study for the effect of cyclic loading on the undrained shear performance of geopolymer treated clays need to be conducted to address the broader mechanical properties of treated soils under the full range of advanced loading conditions.
- Investigation into the undrained shear performance of geopolymer-treated clay in anisotropic consolidation.
- Further interpretations of the results of the undrained triaxial testing within the critical state framework are highly recommended.
- The effects of sulfate and organic content on the mechanical performance of geopolymer-treated clays, as this factor is known to have a significant influence on the behaviour of soil treated with traditional cement.
- With the view that the samples in this study were prepared at the optimum moisture content (for surface mixing), an additional study can be conducted at moisture contents higher than the optimum value (for deep mixing).
- Consolidation tests need to be conducted to capture the consolidation performance of geopolymer-treated clays and the impact of treatment on related test parameters. Further permeability testing is also recommended on

different treated clay types as it may provide future insights into the impact of porosity on the consolidation of geopolymer-treated clays.

- Chemical tests need to be conducted during wetting-drying durability tests to check the impact of binder loss during this process.
- It should be noted that the proposed systematic approach in this thesis is specific for certain clays treated with a specific mixture of fly-ash based geopolymer and further investigation is thus necessary to expand the present findings by including other clay types.

8.3 SUMMARY

In general, the current research suggests that fly-ash-based geopolymer can be used as an effective stabiliser of natural clay soils. The results of this study highlighted the impact of soil mineralogy and plasticity on geopolymer treatment and dosage required for adequate strength and durability performance, implying that fly-ash based geopolymer is not a universal binder for stabilisation of all types of clay. However, the results presented in the current study focussed on limited types of clays and further studies are needed for other soils.

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