

A thesis submitted in fulfillment of the requirement for the degree of Master of Science in Materials Engineering

School of Materials Engineering UNIVERSITI MALAYSIA PERLIS

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LIST OF ABBREVIATIONS

AAFA	Alkali Activated Fly Ash
Al	Alumina
Al ₂ O ₃	Aluminum Oxide
ASTM	American Society for Testing and Materials
Ca	Calcium
CaCO ₃	Calcium Carbonate
CaO	Calcium Oxide
CO_2	Carbon Oxide
CS	Calcium Silicate
CSH	Calcium Silicate Hydrate
H ₂ O	Calcium Carbonate Calcium Oxide Carbon Oxide Calcium Silicate Calcium Silicate Hydrate
ICDD	International Centre for Diffraction Data
JKR	Jabatan Kerja Raya
kPa	kilopascal
М	Molar Ratio
mm	Milimeter
MOH	Metal Hydroxide
MPa 🕜	Megapascal
Na ₂ SiO ₃	Sodium Silicate
NaOH	Sodium Hydroxide
ОН	Hydroxide
S/L	Solid/Liquid
SEM	Scanning Electron Microscopy
Si	Silica

SiO₂ Silica Oxide

UCS Unconfined Compressive Strength

- XRF X-Ray Fluorescence
- XRD X-Ray Diffraction

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Aplikasi Penstabilan Tanah Menggunakan Kaedah Pengeopolimeran

ABSTRAK

Tanah lembut seperti tanah liat dan kelodak telah dikaitkan dengan pelbagai masalah terutamanya dalam bidang kejuruteraan. Kebimbingan utama adalah untuk mencari penstabil tanah yang terbaik untuk mengatasi masalah ini. Tujuan penstabilan tanah bukan sahaja untuk memperolehi ciri-ciri kejuruteraan tanah yang dikehendaki, malah kos dan kesan kepada persekitaran juga perlu dipertimbangkan. Kajian yang berterusan dijalankan oleh ramai penyelidik untuk mencari kaedah-kaedah alternatif untuk menstabilkan tanah dan geopolimeran merupakan salah satu daripada kaedah yang mampu untuk memenuhi keperluan tersebut. Kajian ini dilaksanakan untuk menyelidik kaedah pengeopolimeran untuk aplikasi penstabilan tanah, dengan mencampurkan tanah secara terus dengan pelarut pengaktif alkali, menghasilkan geopolimer berasaskan tanah. Kaedah ini dijalankan ke atas tiga jenis tanah; kaolin, Tanah 1 dan Tanah 2. Tanah-tanah ini dianalisa daripada segi klasifikasi tanah, Atterberg Limits, komposisi kimia, fasa dan morfologi untuk penghasilan geopolimer. Manakala, untuk rekabentuk geopolimer berasaskan tanah, parameter-parameter yang terlibat adalah kemolaran NaOH, nisbah tanah/larutan pengaktif alkal dan nisbah Na₂SiO₃/NaOH. Nisbah optimum campuran (nisbah tanah/larutan pengaktif alkali) geopolimer berasaskan tanah diperolehi berdasarkan nilai kekuatan tertinggi dalam ujian Unconfined Compressive Strength (UCS) dan ujian California Bearing Ratio (CBR). Nilai kekuatan tertinggi bagi geopolimer berasaskan kaolin ialah 436 kPa untuk ujian UCS dan 46% untuk ujian CBR pada nisbah optimum campuran 1.5. Nisbah optimum campuran bagi geopolimer berasaskan Tanah 1 adalah 2.0 dengan nilai kekuatan tertinggi 500 kPa untuk ujian UCS test dan 55% untuk ujian CBR. Geopolimer berasaskan Tanah 2 juga menunjukkan nisbah optimum campuran yang sama, 2.0, dengan nilai kekuatan tertinggi 620 kPa dan 62% untuk ujian UCS dan ujian CBR. Geopolimer berasaskan tanah-tanah ini tidak efektif untuk aplikasi penstabilan tanah mengikut spesifikasi ASTM D4609 dan tidak memenuhi nilai minimum yang ditetapkan dalam Garis Panduan untuk Struktur Turapan Alternatif (Isipadu Trafik Rendah) Jabatan Kerja Rava (JKR) Malaysia. Geopolimer berasaskan kaolin menunjukkan penurunan indeks keplastikan sehingga 11.24%, manakala geopolimer berasaskan Tanah 1 dan Tanah 2 masing-masing menunjukkan penurunan sehingga 3.08% dan 4.31%. Pencirian geopolimer berasaskan tanah; analisa fasa dan analisa morfologi dilaksanakan pada nisbah optimum campuran. Peningkatan nilai kekuatan dan perubahan dalam pencirian geopolimer berasaskan tanah-tanah ini membuktikan bahawa kaedah pengeopolimeran boleh digunakan untuk aplikasi penstabilan tanah.

Soil Stabilization Application Using Geopolymerization Method

ABSTRACT

Soft soils such as clay and silt have been associated to countless problems especially in engineering field. The main concern is to search for the best soil stabilizers to overcome the aroused problems. The purpose of soil stabilization is not only to achieve the required soil engineering properties, in fact the cost and the effect towards the environment also should be considered. Continues studies are done by numerous researchers in order to find alternative methods for soil stabilization and geopolymerization is one of the method that can fulfill those requirements. This study has been conducted to investigate the geopolymerization method for soil stabilization application, by mixing the soils directly with alkaline solutions, producing soil based geopolymer. This method was conducted towards three types of soil; kaolin, Soil 1 and Soil 2. The soils were analyzed in terms of soil classification, Atterberg Limits, chemical composition, phase and morphology for geopolymer fabrication. Meanwhile, for the design of soil based geopolymer, the parameters involved were NaOH concentration, solid/liquid ratio and Na₂SiO₃/NaOH ratio. The optimum mixing ratio (solid/liquid ratio) of soil based geopolymers were obtained based on the highest strength values in Unconfined Compressive Strength (UCS) test and California Bearing Ratio (CBR) test. The highest strength value for kaolin based geopolymer was 436 kPa for UCS test and 46% for CBR test. Both values were at the same optimum solid/liquid ratio of 1.5. The optimum mixing ratio for Soil 1 based geopolymer was 2.0, with highest strength value of 500 kPa for UCS test and 55% for CBR test. Soil 2 based geopolymer also indicated the same optimum mixing ratio, 2.0, with highest strength value of 620 kPa and 62% for UCS test and CBR test, respectively. The soil based geopolymers were not effective for soil stabilization application according to ASTM D4609 specification and did not comply the minimum value specified in the Design Guideline for Alternative Pavement Structures (Low Volume Roads) of Malaysia Public Work Department (PWD). Kaolin based geopolymer indicated reduction of plasticity index up to 1.24%, meanwhile Soil 1 and Soil 2 based geopolymers indicated reduction up to 3.08% and 4.31%, respectively. The characterization of soil based geopolymers; phase analysis and morphology analysis were conducted at optimum mixing ratio. The increment of strength values and changes in the characterization of soil based geopolymers proved that geopolymerization method can be used for soil stabilization application.

CHAPTER 1

INTRODUCTION

1.1 Research Background

In most geotechnical projects, obtaining a construction site that met with the required design without any ground modification was impossible. The recent practice is modifying the soil engineering properties, so the design specification could be met. The aimed for soil stabilization was to improve the strength of soil and to increase the resistance to softening by water. These could be achieved by bonding the soil particles together, water proofing the particles or combination of both (Makusa, 2012).

The purpose of soil stabilization was not only limited to enhance the loadbearing of the soil capacity but also to improve the shear strength, filter, drainage system (Prabakar et al., 2004) and to meet specific engineering projects requirement (Kolias et al., 2005). In order to successfully withstand the load of the superstructure, soil stabilization technique was essential to certain the good stability of soil. At the same time a lot of time and money could be saved compared to the cutting out method and replacement of the unstable soils (Negi et al., 2013).

To highly improve the mechanical properties, weak soils can be chemically stabilized by using chemical agent such as lime or cement which able to create bonds between soil particles, remove excess moisture and fill the empty voids in soil skeleton (Sas and Gluchowski, 2013). For the purpose of increasing the soil strength parameters, increasing the loading capacity as well as decreasing the settlement, utilization of chemical stabilization seemed to be a more popular method compared to other method of soil stabilization. This was due to its low cost and convenience for a high volume of soil improvement that involved in geotechnical projects (Marto et al., 2013).

A new, practical and sustainable alternative is always been searched in civil engineering industry. Since geopolymers offered small shrinkage potential and also outstanding adhesion to aggregates, they could be an effective soil stabilizer. There were various low cost aluminosilicates or industrial wastes such as metakaolin, fly ash, furnace slag, red mud, and rice husk ash that could be used to synthesize geopolymer (Zhang, 2013).

Geopolymer has been broadly called 'inorganic polymer' and it has gained significant attention due to its numerous advantages such as low cost, outstanding properties of mechanical and physical, low energy consumption and greenhouse gas reduction (Muniz-Villareal et al., 2011). Apart from that, geopolymer also offered high compressive strength, low shrinkage, either fast or slow setting, acid resistance, fire resistance as well as low thermal conductivity (Duxson et al., 2007). Extensive researches have been done towards the development of geopolymer during the last decade and this was due to the various potential applications by using this material (Garcia et al., 2009; Bondar et al., 2010; Mohsen & Mostafa, 2010; Cristelo et al., 2013; Zhang et al., 2013).

Low manufacturing energy consumption as well as low carbon dioxide (CO₂) emission were highlighted as the main advantages which marked geopolymer binders as a kind of 'green material' (Mostafa et al., 2014). The global warming was resulted from the depletion of ozone layer that caused by greenhouse gases emissions during cement production which used high temperature techniques. However, the CO₂ emission could be reduced by 80% using geopolymer technology as the application of high temperature calcining was not needed in the production of cement (Milyaso et al., 2015). Geopolymer most likely has the potential since it offered smaller ecological footprint compare to cement (Habert et al., 2011).

The applications and procedures in engineering area could be explored with materials that have been designed with the help of geopolymerization reactions (Milyaso et al., 2015). The production of geopolymers was depending on the selection of the raw material as well as the processing conditions (Duxson et al., 2007).

Waste products such as fly ash, blast furnace slag and etc. can be converted into beneficial products especially in manufacturing of geopolymers thus, could be the solution to waste disposal problems (Damilola, 2013). These materials usually contained an aluminosilicate precursor which then will be activated in a concentrated alkali hydroxide solution and the final chemical composition usually will be controlled by adding alkali silicate (Li et al., 2013). The geopolymer could be formed by activating the aluminosilicate with alkaline or alkaline silicate solution either at ambient or higher temperature (Zuhua et al., 2009). Countless studies have shown that good mechanical properties also can be achieved by geopolymer materials synthesized at ambient temperature (Somna et al., 2011; Tashima et al., 2013, Kramar & Ducman, 2015). The existence of natural clay minerals in soil could be a crucial part of stabilizing mechanism since they inherited a source of aluminosilicates and most probably will be appropriated for geopolymerization (Maskell et al., 2014).

1.2 Problem Statement

Expansive soils contained clay minerals and they created problems especially to civil and geotechnical engineers since they could cause destruction to structures due to the serious volume changes that consistent with the changes in moisture content. When they absorbed water their volume will be increased or swell and they will become shrink or having reduction in their volume on water evaporation (Phanikumar, 2009).

Meanwhile, cohesive soils were referred to fine-grained soils; clays and silts (Briaud et al., 2004). They have particles that tend to stick together resulted from the interaction of water-particle and the existence of attractive forces between particles, so they were sticky, plastic and have intolerable engineering properties (Coduto, 1999).

In road construction, soil condition was one of the major factors that will be considered especially in pavement design and selection of the pavement material (Teh et al., 2015). This was due to the fact that the pavement components will be placed on the top of the soil (Geremew et al., 2016). Poor soil condition will lead to severe damages at the initial stage of road construction and the process of reconstruction will be very costly (Teh et al., 2005).

Another engineering problem related to soil was landslide and the risk of this problem to be happened in Malaysia was due to the type of the soil itself. It was also caused by heavy rain; either a single heavy thunderstorm or continuous days of moderate rain especially during the rainy season (Low, 2006). Other factors that attributed to this problem were subsurface investigation and laboratory tests that were not conducted to obtain the soil parameters, subsoil and groundwater profiles (Sew & Chin, 2006). The number of landslides failure and fatalies in Malaysia from 1973 to 2007 were described in Figure 1.1 (Jabatan Kerja Raya, 2009).

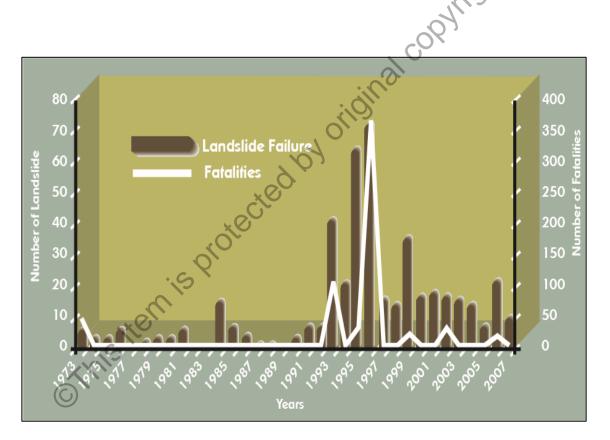


Figure 1.1: Landslide and fatalities from 1973 to 2007 (Jabatan Kerja Raya, 2009)

High cost and complex soil improvement usually involved in enhancing stability and reducing differential settlement of soft soil. The overall project cost also might be increased due to imported backfill materials that were required in most cases (Loke, 2000). The replacement of soft soils with suitable imported fill materials was a conventional method of soil improvement and due to the cost of excavation, dumping and filling materials, it became an expensive practice (Kazemian & Huat, 2010). Better techniques that are economical, less time and labour consuming and more effective treatment become the major concerns of the geotechnical engineers due to the challenges in improving the soil condition (Napiah et al., 2008).

1.3 Research Objectives

The objective of this research can be summarized as follows

- 1. To study the soils as raw materials for geopolymer fabrication
- 2. To investigate the optimum mixing ratio based on the soil based geopolymers strength
- 3. To characterize the soil based geopolymers at the optimum mixing ratio

1.4 Scope of Research

This research focused on using the geopolymerization method for soil stabilization application. The raw soils were analyzed in terms of soil classification, Atterberg Limits, chemical composition, phase and morphology.

The parameters involved in the design of soil based geopolymers were solid/liquid (soil/alkaline activator) ratio, sodium hydroxide (NaOH) concentration and sodium silicate (Na₂SiO₃)/sodium hydroxide (NaOH). Various mixing ratio (solid/liquid ratio) were carried out and the optimum mixing ratio was obtained based on the highest strength values in Unconfined Compressive Strength (UCS) test and California Bearing

Ratio (CBR) test. The soil based geopolymers soils were characterized by conducting Atterberg Limit test. The phase analysis and morphology analysis were conducted at the optimum mixing ratio.

There were some limitations in this research. First, only three types of soil were studied for soil stabilization application using geopolymerization method due to the time constraint. Second, due to the constraint on lab facilities, it was impossible to conduct all strength tests to the samples. Due to the reason, only two strength tests were original copy able to be conducted.

1.5 **Thesis Outline**

This thesis comprises a total of five chapters. Chapter 1 is an introduction of the research which discussed the research background, problem statement, research objectives, scope of research and outline of this thesis.

Chapter 2 highlighted the literature review, a brief summary of soil stabilization material and method, geopolymer, geopolymerization mechanism, geopolymer constituent, characterization and strength of soil stabilization material studied by previous researchers.

Chapter 3 focused on the methodology adopted in achieving the objectives of this research which included the preparation of raw materials, design of soil based geopolymers, mixing procedure and laboratory tests conducted.

Chapter 4 explained the results obtained from the tests that been carried out. Discussion regarding the characterization and strength of raw soils and soil based geopolymers were also provided in detail.

Chapter 5 presented the conclusion based on the results obtained throughout the research works as well as the recommendation for improvement and future study.

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CHAPTER 2

LITERATURE REVIEW

2.1 Problematic Soil

Soil materials such as clays and silts had destructive capabilities that might lead to life loss, property destruction and environmental damage (Torrenti & La Torre, 2016). Mostly, clays were flake-shaped microscopic and submicroscopic particles of mica, clay minerals and other minerals (Das, 2005) or they also could be described as plate-like (Wieffering and Fourie, 2009).

The attractions of water to clay were due to the (1) negative charge on the surface of clay and cations floating around the clay particles, (2) a water molecule acted like a small rod with a negative and a positive charges at each end and the dipolar water attracted by the negatively charged surface and the cations and (3) hydrogen bonding, the hydrogen atoms in the water molecules were shared with oxygen atoms on the surface of the clay. The attraction force of water was very strong near the clay surface and decreased with occurance of distance from the surface (Das, 2005).

Silts were microscopic soil fractions that comprised of very fine quartz grains and some fragments of flake-shaped micaceous minerals (Das, 2005). They were subjected to considerable shrinkage and expansion due to moisture change (Nikolaides, 2015). Other than that, silts also exhibited some plasticity, cohesion, adhesion and absorption due to the adhered film of clay and held much amount of water compared to sand but less than clay (Osman, 2013). In terms of particles, silt was smaller than sand and their shape was mainly spherical (Nikolaides, 2015).

However, fine grained soils were considered to be the good applicants for stabilization (Negi et al., 2013). They were also the easiest to be stabilized due to the large surface area in relation to the particle diameter (Sherwood, 1993). The application of soil stabilization products to stabilize them was quite extensive (Onyelowe and idinal copyright Okafor, 2012).

2.2 **Soil Stabilization Materials**

The invention of soil stabilization technology was started in 1960's by using cement, the oldest binding agent to stabilize soil. Since the used of cement alone able to generate the required stabilization action, it could be considered as the primary stabilizing agent or hydraulic binder (Sherwood, 1993).

Cement application in soil however, aroused some issues related to environment and durability (Cristelo et al., 2013) since it releases high levels of CO_2 during its production (Garcia et al., 2009) and chemical vulnerability due to the sulphates attack in ground or chemical wastes when it been used for soil improvement or structural foundations (Tomlinson, 2001).

Besides that, unacceptable effects on the properties of the stabilized soil were obtained when there was delayed in compaction after the soil was mixed with cement and water. Another problem was the undesirable consequences after the implementation of soil stabilization due to the incidence of shrinkage cracks on the compacted layers that mostly happened to fine-grained soils (Gharib et al., 2012). Due to the disadvantages, other alternatives or replacements of cement for soil stabilization were looked for through numerous research and investigations (Zhang et al., 2013).

The engineering properties of fine grained soils also could be changed by using lime stabilization, one of the oldest processes for soil stabilization which has been widely used and most effective especially for plastic clays treatment (Amu et al., 2011).

But the used of lime or any calcium-based materials comprising soluble sulfate salt in soil treatment will cause the soil to be distressed and heaving and also occurrence of disintegration which resulted to the loss of strength. Soil minerals, water used for mixing or ground water could be the source of sulfate. Other than that, the calcination of calcium carbonate was involved in the production of lime or any calcium-based material. Since the process of calcination occurred at very high temperature, it gave a negative impact to the environment due to the emission of carbon dioxide and consumption of high energy (Jawad et al., 2014).

Compared to lime and cement, fly ash had small amount of cementitious properties and it was produced as a byproduct in coal fired electric power generation facilities (Makusa, 2012). Since fly ash was an industrial byproduct, it had lower cost compared to cement and lime and it was attractive to be used (Ahmed, 2014).

The utilization of fly ash to stabilize soil also had some disadvantages; dewatering may be needed due to the less moisture content of the stabilized soil and