UNIVERSITI PUTRA MALAYSIA

STRENGTH AND COMPRESSIBILITY BEHAVIOR OF SOIL TREATED WITH PALM OIL FUEL ASH-BASED GEOPOLYMER

KHASIB ISAM ADNAN SOLIMAN

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By

KHASIB ISAM ADNAN SOLIMAN

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STRENGTH AND COMPRESSIBILITY BEHAVIOR OF SOIL TREATED WITH PALM OIL FUEL ASH-BASED GEOPOLYMER

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Chairman : Associate Professor Nik Norsyahariati binti Nik Daud, PhD
Faculty : Engineering

The use of geopolymer in soil stabilization has gained much attention recently due to its efficiency in improving the engineering properties of soils and being environmentally friendly at the same time. Common binders such as cement and lime yield in high stabilization potential but they were found to be uneconomical and contributed to some environmental issues. Hence, this research aimed to investigate the effect of palm oil fuel ash (POFA) based geopolymer on soft soil stabilization. The geopolymer was synthesized from POFA and an alkaline activator solution which was made from a mixture of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) at ambient temperature. The mechanical and microstructural behavior of two types of clayey soils stabilized with POFA-based geopolymer at four dosages (G10PA, G20PA, G30PA and G40PA) were investigated in this study. In this respect, a series of unconfined compression (UCS), one-dimensional consolidation, direct shear and California Bearing Ratio (CBR) tests was conducted to investigate the mechanical properties of soils treated with POFA-based geopolymer. The microstructural changes and mineralogy of the treated samples were analyzed by Field Emission Scanning Electron Microscopy along with Energy Dispersive X-Ray (FESEM-EDX) and X-Ray Diffraction (XRD) analyses. Furthermore, the physical properties of soils (Atterberg Limits, Plasticity Index, and Linear Shrinkage Limit), pH and compaction assessment were investigated before and after treatment with POFA-based geopolymer. The results indicated that shear strength and compressibility of both soils increased and decreased respectively by increasing the dosage of POFA-based geopolymer. Geopolymer with 40% POFA of the dry weight of soils (G40PA) yielded the highest UCS values and; least permeability and compressibility. Compressive strength of untreated soil 1(S1) and soil 2 (S2) increased from 0.26 MPa and 0.13 MPa reaching 4.18 MPa and 2.86 MPa for S1-G40PA and S2-G40PA, respectively. The permeability coefficient of S1 and S2 stabilized with G40PA is almost 300% and 200% smaller than that of the untreated samples, respectively. The potentiality of compressibility of natural S1 is considerably reduced from medium to a low level after geopolymer stabilization whereas S2 reduced from high to a medium level. In addition, it was observed that the longer the curing period (28 days) of the stabilized soils, the higher the compressive strength of the soil developed. However, the microstructural analysis (FESEM-EDX) revealed the material modifications can be related to the strength behavior in which geopolymer gel filled the interparticle voids in which a stiff soil structure was noticed. FESEM-EDX analysis has proved that geopolymer gel binding effect contributed to the improvement in the mechanical properties of stabilized soils. New minerals were found in the treated samples such as mullite and augite which are associated with geopolymerization reaction as indicated by XRD results. These results suggest the potentiality of using POFA-based geopolymer binder to stabilize soft soil.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KEKUATAN DAN KELAKUAN KEBOLEHMAMPATAN TANAH YANG DIRAWAT DENGAN ABU BAHAN BAKAR SAWIT-BERASASKAN GEOPOLIMER

Oleh

KHASIB ISAM ADNAN SOLIMAN

Jun 2021

Pengerusi : Profesor Madya Nik Norsyahariati binti Nik Daud, PhD
Fakulti : Kejuruteraan

Penggunaan geopolimer dalam penstabilan tanah telah mendapat perhatian baru-baru ini kerana kecekapannya dalam menambahbaikkan sifat kejuruteraan tanah dan mesra alam pada masa yang sama. Pengikat biasa seperti simen dan kapur menghasilkan potensi penstabilan yang tinggi tetapi didapati tidak ekonomi dan menyumbang kepada beberapa masalah persekitaran. Oleh itu, penyelidikan ini bertujuan untuk mengkaji kesan geopolimer berasaskan abu bahan bakar sawit (POFA) terhadap penstabilan tanah lembut. Geopolimer disintesis dari POFA dan larutan pengaktif alkali yang dibuat dari campuran sodium hidroksida (NaOH) dan sodium silikat (Na_2SiO_3) pada suhu bilik. Kelakuan mekanikal dan mikrostruktur bagi dua jenis tanah liat yang distabilkan dengan geopolimer berasaskan POFA pada empat dos (G10PA, G20PA, G30PA dan G40PA) telah disiasat dalam kajian ini. Sehubungan dengan itu, satu siri ujian kekuatan mampatan tak terkurung (UCS), pengukuhan satu dimensi, ricih langsung dan nisbah galas *California* (CBR) telah dijalankan untuk menyiasat sifat-sifat mekanikal tanah yang dirawat dengan geopolimer berasaskan POFA. Perubahan mikrostruktur dan hasil mineralogi dalam sampel yang dirawat telah dianalisis dengan ujian *Field Emission Scanning Electron Microscopy* bersama dengan *Energy Dispersive X-Ray* (FESEM-EDX) dan *X-Ray Diffraction* (XRD). Selanjutnya, sifat-sifat fizikal tanah (*Atterberg Limits, Plasticity Index, and Linear Shrinkage Limit*), pH dan penilaian pemadatan disiasat sebelum dan selepas rawatan dengan geopolimer berasaskan POFA. Hasil kajian kedua-dua tanah menunjukkan peningkatan dalam kekuatan ricih dan penurunan dalam kebolehmampatan dengan penambahan dos geopolimer berasaskan POFA. Geopolimer dengan 40% POFA dari berat kering tanah (G40PA) menghasilkan nilai UCS tertinggi dan kebolehtelapan dan kebolehmampatan yang paling rendah. Kekuatan mampatan S1 dan S2 yang tidak dirawat meningkat dari 0.26 MPa dan 0.13 MPa dan mencapai 4.18 MPa dan 2.86 MPa untuk S1-G40PA dan S2-G40PA, masing-masing. Pekali kebolehtelapan bagi S1 yang distabilkan dengan G40PA didapati meningkat 300% dan S2 menurun sebanyak 200% dibandingkan dengan sampel tidak dirawat, masing-masing. Potensi kebolehmampatan S1 yang asal menurun dari tahap sederhana ke tahap rendah setelah penstabilan dengan geopolimer, manakala S2 menurun dari tahap tinggi ke tahap

sederhana. Di samping itu, kajian juga didapati bahawa semakin lama tempoh pengawetan (28 hari) tanah yang distabilkan, semakin tinggi kekuatan mampatan tanah. Walau bagaimanapun, analisis mikrostruktural (*FESEM-EDX*) mendedahkan pengubahan bahan boleh dikaitkan dengan ciri-ciri kekuatan di mana gel geopolimer telah mengisi ruang antara partikel dan menghasilkan struktur tanah yang lebih kukuh. Analisis FESEM-EDX telah membuktikan bahawa kesan pengikatan gel geopolimer menyumbang kepada peningkatan sifat-sifat mekanikal tanah yang telah distabilkan. Mineral baru ditemui dalam sampel yang dirawat seperti *mullite* dan *augite* yang dikaitkan dengan tindakbalas geopempolimeran seperti yang ditunjukkan oleh keputusan XRD. Keputusan ini mencadangkan potensi penggunaan pengikat geopolimer berasaskan POFA untuk menstabilkan tanah lembut.

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Nik Norsyahariati binti Nik Daud, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Noor Azline binti Mohd. Nasir, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

____

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BS	British Standard
c	Cohesion
C-A-H	Calcium Aluminate Hydrate
C-A-S-H	Calcium Silicate Aluminate Hydrate
CBR	California Bearing Ratio
Cc	Compression Index
CH	High Plasticity Clay
CL	Low Plasticity Clay
Cs	Swelling Index
C-S-H	Calcium Silicate Hydrate
c_v	Coefficient of Consolidation
DST	Direct Shear Test
e	Void Ratio
EDX	Energy Dispersive X-ray
FESEM	Field Emission Scanning Electron Microscope
LL	Liquid Limit
LOI	Loss of Ignition
MDD	Maximum Dry Density
m_v	Compressibility Coefficient
Na_2SiO_3	Sodium Silicate
NaOH	Sodium Hydroxide
N-A-S-H	Sodium Silicate Aluminate Hydrate
OMC	Optimum Moisture Content

Øº	Angle of Internal Friction
PI	Plasticity Index
PL	Plastic Limit
POFA	Palm Oil Fuel Ash
S1-G10PA	Soil 1- Geopolymer Made from 10% POFA of Dry Soil Weight
S1-G20PA	Soil 1- Geopolymer Made from 20% POFA of Dry Soil Weight
S1-G30PA	Soil 1- Geopolymer Made from 30% POFA of Dry Soil Weight
S1-G40PA	Soil 1- Geopolymer Made from 40% POFA of Dry Soil Weight
S2-G10PA	Soil 2- Geopolymer Made from 10% POFA of Dry Soil Weight
S2-G20PA	Soil 2- Geopolymer Made from 20% POFA of Dry Soil Weight
S2-G30PA	Soil 2- Geopolymer Made from 30% POFA of Dry Soil Weight
S2-G40PA	Soil 2- Geopolymer Made from 40% POFA of Dry Soil Weight
SL	Shrinkage Limit
UCS	Unconfined Compressive Strength
XRD	X-ray Diffraction test
XRF	X-ray Fluorescence

CHAPTER 1

INTRODUCTION

1.1 Background

Population growth and space limitations all over the world, make soil improvement necessary to provide a strong underground layer to assist the infrastructure facilities such as road construction. Based on the origin, soils can be divided into residual and transported soils. Residual soils are the end product of the in-situ mechanical or chemical weathering of rock layers that have lost or deteriorated their natural fabric. The most important aspect of residual soils is their poor strength, which is caused by the weathering processes destroying the bonds and cementation of the original material (Faisal, 2000). Transported soils refer to soils that have been moved from their original location by gravity, wind, water, glaciers, or human action, individually or in combination. The characteristics of the resultant soil mass are greatly influenced by the manner of transportation and deposition. A very well-known example of transported soil is marine clay which is spread across the world in coastal areas. This soil is typically compatible with recognizable settlement and instability, as well as low soil properties which make it unsuitable for engineering purposes. Massive damage in the constructions on this soil is possible even with light loads and heavy cracks can be shown as a result of unequal moist distribution (Al-bared & Marto, 2017).

Soil stabilization has been known since many decades. It is an efficient and trustworthy technique to strengthen the soil, increase the bearing capacity of subgrade soil, slope stability and solving problems of foundations, which makes it very essential in the geotechnical engineering practice. The need of having a stable and strong underground to carry the applied loads is a challenging task that brought the idea of soil stabilization. Many researchers studied the treatment and stabilization of soil (Chen & Lin, 2009; Basha et al., 2005). Generally, soil stabilization is considered a great economical solution especially wherever weak soil is found; there is no need to landfill the weak and unsuitable soil from the site as well as no-cost of transferring soil from or to the site. The chemical stabilization method of soil was proved to be able to enhance the quality of soil throughout providing high strength and low permeability (Maaitah, 2012; Cabezas & Cataldo, 2019). It depends primarily on the chemical reaction between the additives and particles of soil, which develop a strong network that binds the soil grains to achieve the desired effect and improve geotechnical properties.

Chemical stabilization of soil has been widely investigated by many studies using traditional binders such as calcium-based binders (lime, cement or a combination of both) (Chinkulkijniwat & Horpibulsuk, 2012; Asgari et al., 2015; Wang et al., 2018). Although these binders have shown their ability in improving the soft soil performance for civil engineering applications, they are associated with several defects since they affect the environment and consume too much of the limited (finite) natural resources. Some other binders that have shown their effectiveness in soil improvement are alumina-silicate materials (pozzolanic materials). One of the most important pozzolanic materials used in soil stabilization is fly ash. The intent of using fly ash in soil stabilization is for industrial wastes disposal and the good strength associated when applied to the soil. However, due to the development and the necessity of higher strength binders and to overcome the shortages of calcium-based binders, a new idea was brought in what is called alkali-activated materials (geopolymers). These new binders can provide soil performances either comparable or even better than the other traditional stabilizers under similar conditions such as curing time and temperature. Under this category, the geopolymer technique is newly introduced in the geotechnical engineering field as a good replacement for the other binders. It utilizes solid waste or byproducts as a precursor in the alkaline activation process since they contain a substantial quantity of silica (SiO_2) and alumina (Al_2O_3) in an amorphous state to produce the geopolymer and gets synthesized. This technique has some advantages that the polymers are relatively easy to obtain, cheaper and bring less impact on the environment. In recent years, geopolymer has attracted considerable attention among all binders because of its high compressive strength, low permeability, good chemical resistance and excellent fire resistance behavior (Ji & Pei, 2019). Because of these advantageous properties, geopolymer is a promising alternative to cementitious materials in addressing various geotechnical problems and waste immobilization solutions for the industries. The geopolymerization process can be summarized as follows (Fernández-Jiménez et al., 2006; Van Riessen et al., 2013): dissolution of the reactive aluminosilicate chains in the precursor (aluminosilicate material) due to the increase in pH produced by the activator solution, and formation of monomers. Subsequently, polycondensation reactions take place to accumulate and precipitate the resulting products to form a three-dimensional amorphous structure which will tend to crystalize.

An extensive number of geotechnical studies have indicated the effectiveness of geopolymers synthesized from different aluminosilicate materials in soil stabilization (Sperberga et al., 2015; Hanegbi & Katra, 2020; Wang et al., 2020; Zhu et al., 2020; Khadka et al., 2020). Between the different aluminosilicate precursors, the use of palm oil fuel ash (POFA) merits more recognition due to its high potentiality to produce geopolymer and its abundance as a waste, especially in Southeast Asian countries such as Malaysia (Liu et al., 2014).

However, the utilization and application of geopolymers in soil stabilization are still in their early stages and require more demonstrations. Moreover, comprehensive studies about soft soil stabilization using geopolymers produced from POFA as a precursor with sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) as activators are still not well illustrated. In this study, the alkali-activated POFA (POFA-based geopolymer) is used to stabilize soft soils to lead to a positive effect on the engineering properties and performance of subgrade soils for pavement applications.

1.2 Problem Statements

Residual soils and marine clays are found in many places in this world, including Malaysia. Marine clay soils are famed for their poor geotechnical properties represented by high compressibility and low strength wherever they exist. Residual soils which are known as products of rock weathering sometimes need enhancements to fulfill the design standards. For example, in the case of road construction, the soil strength should be sufficient to carry the loads and avoid excessive volume change.

Although traditional binders (cement and lime) have shown their effectiveness in soil stabilization, they have some deficiencies. Too much consumption of energy and natural resources for the production process, make these binders unsuitable for stabilization as well as their financial and environmental issues. Cement manufacture produces a lot of CO_2 emissions, and CO_2 has been proved to be one of the main causes of global warming and high temperature associated (Gartner, 2004; Matthews et al., 2009). It was indicated that for producing 1 ton of cement, 1.5 tons of raw materials are consumed with 5.6 GJ/ton energy consumption and nearly 1 ton of CO_2 is emitted (Jafer et al., 2018; Du et al., 2016). Also, the production of 1 ton of lime releases approximately 0.86-ton CO_2 (Chang et al., 2015). Moreover, CO_2 emitted due to cement production forms around 7% of the total greenhouse gases in the atmosphere (Criado et al., 2007a; Aziz et al., 2015). In 2016, the approximated value was 1.45 ± 0.20 Gt CO_2 produced and it forms nearly 8% of the total global CO_2 release (Andrew, 2018).

In addition, agricultural waste such as palm oil fuel ash (POFA) is being produced in huge quantities in the world, especially in Malaysia. Planting palm oil trees has increased rapidly over the past decades in Malaysia and now it is considered one of the main exporters of palm oil products around the world (Valipour, 2015). Among the countries that plant palm oil trees, Malaysia is known to be the second-largest producing country of palm oil products in the world (Gan & Li, 2014). According to the Malaysian Palm Oil Board (MPOB), palm oil plantations occupied around 5 million hectares in Malaysia in 2011 (Malaysian Palm Oil Board, 2011). POFA is an agricultural waste resulting after burning palm oil residues such as palm fibers and shells at temperatures of about 800°C-1000°C to produce steam for electricity generation in power plants (Alengaram et al., 2013). In Malaysia, it was estimated that the produced amount of POFA to be approximately 3 million tons in 2007 (Johari et al., 2012). In Thailand, the estimated amount is more than 100,000 tons of POFA every year and it is increasing annually (Chindaprasirt et al., 2007). Moreover, it should be mentioned that the production of POFA is estimated to be 5% of the weight of solids waste (Aprianti et al., 2015; Sata et al., 2004). The need of using POFA in many applications is increasing in order to reduce the amount that needs to be dumped. Practically, this solid waste ash is dumped just near the palm oil mill inside the area of the factory. This light ash is easy to be transferred by the wind causing huge environmental pollution in the surrounding area. Also, the leaching of disposed ash can contaminate groundwater, natural supplies, and cultivation fields (Yilmaz et al., 2019; Mahvash et al., 2017). In closed disposal systems, fly ash stiffens in the presence of moisture root damage (Yilmaz et al., 2019).

1.3 Research Objectives and Research Questions

Aim – To investigate the effectiveness of using POFA-based geopolymer binder in improving the engineering properties of two types of soft soil to solve the issues and problems corresponding to weak soil. In order to achieve the aim, the specific objectives of this study are outlined below:

1. To investigate the physical, chemical and compaction properties of soft soil treated with POFA-based geopolymer.

2. To determine the effect of POFA-based geopolymer on shear strength and compressibility behavior of soil stabilized experimentally.

3. To evaluate the microstructural changes of soil after treatment with POFA-based geopolymer.

Research questions of this study are:

1. What is the effect of POFA-based geopolymer binder on the physical, chemical and compaction properties of the soil?

2. How does POFA-based geopolymer affect the compressibility and strength properties of the soft soil?

3. What are the microstructural changes that happen within the soil stabilized with POFA-based geopolymer?

1.4 Scope of Work and Limitations

The scope of the work in this study can be broken down into three phases, which are as follows:

i. Phase one, to improve the basic geotechnical characteristics and optimum conditions of residual and marine clay soils by using POFA-based geopolymer. Atterberg limits, shrinkage limit, plasticity index, optimum moisture content, maximum dry density and pH were tested on the original soils and POFA-based geopolymer treated soils.

ii. Phase two, in order to enhance the shear strength and compressibility behavior of soils stabilized by POFA-based geopolymer, mechanical testing was applied. In this regard, unconfined compression (UCS), direct shear, California bearing ratio (CBR) and one dimensional (1-D) consolidation tests have been performed.

iii. Phase three, to identify and understand the interaction between soil and POFA based geopolymer and its effect on the mechanical properties, microstructural analysis that employs Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X-ray Spectroscopy (EDX) and X-Ray Diffraction tests (XRD) was carried out.

The limitations of this study are as follows: source binder used in producing the geopolymer is limited to palm oil fuel ash (POFA) in this study. Alkaline activators used are sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) only. Regarding activation parameters, POFA to liquid alkali activator ratio and sodium silicate (Na_2SiO_3) to sodium hydroxide (NaOH) ratio were used with values 1.32 and 2.5 respectively. In addition, in this study, just four mixtures of POFA-based geopolymer were used using POFA to be 10%, 20%, 30% and 40% of the dry weight of soil. The curing period for the samples prepared was 7 and 28 days at ambient temperature. Two types of soft soil were used in this study.

1.5 Significance of Study

This study will illustrate and identify the efficiency of using POFA-based geopolymer in soft soil stabilization. Generally, using geopolymer in soil stabilization is relatively a new topic in geotechnical engineering and the majority of the researches done on POFA-based geopolymer stabilized soil focused on mechanical properties such as unconfined compressive strength; and microstructural properties of soil stabilized such as scanning electron microscope (SEM), fourier transform infrared (FTIR) and X-ray diffraction tests. Therefore, the identification of the physical properties (liquid limit, plastic limit and shrinkage limit) and chemical properties (pH) of soil stabilized with POFA-based geopolymer binder is required, which is done by this study. In addition to shear strength investigation, this study will provide a better understanding on other mechanical characteristics represented by compressibility behavior (1-D consolidation test) of soil stabilized with POFA-based geopolymer. These are considered the most important geotechnical properties that need to be studied and identified in soil stabilization. Finally, the efficiency of POFA-based geopolymer application on two types of clayey soils will be also compared.

Since POFA is being produced in huge quantities especially in Malaysia, the use of this binder will protect the environment from any possible pollution as well as low CO_2 emissions. Also, this research is going to open the doors to future research topics –new knowledge- since the illustration of compressibility behavior and permeability in POFA-based geopolymer stabilized soil is considered relatively new ideas. Comparing the results, types of soil and dosages of geopolymer will assist in finding the optimum dosage of POFA-based geopolymer regrading both compression and strength behavior. In conclusion, this agricultural waste geopolymer binder will be clearly studied to understand its impact on the properties of soft soil.

1.6 Organization of this Thesis

Chapter 1 illustrates a general background to this research work, problem statement, research objectives, research questions, the scope of work and limitations, significance of the study and the outline of this thesis.

Chapter 2 summarizes some literature review on soil stabilization techniques. A revision on previous researches conducted on binders used in soil treatment such as calcium-based binders (cement and lime), pozzolanic materials (alumina-silicate binders) and geopolymers (alkali-activated materials) are conducted. The gap is clearly identified to ensure to importance and necessity of this study.

Chapter 3 includes the detailed steps used to achieve the objectives set for this study. It starts with a flow chart showing the overall plan starting from soil sampling, geotechnical laboratory tests on the untreated soils, preparation of POFA-based geopolymer and some physical, chemical, mechanical and microstructural tests on the treated samples to investigate the effect of the binder on soils.

Chapter 4 displays the results obtained from all tests conducted in this study as well as the discussion of the findings. It shows the geotechnical characteristics of the untreated soils, properties and preparation of POFA and alkaline activators; and the performance of samples treated with POFA-based geopolymer undergoing physical, chemical, mechanical and microstructural analysis.

Chapter 5 is the summary and conclusions derived from the research work done. Also, it recommends some of the future work to continue the research started by this study.

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BIODATA OF STUDENT

Isam Adnan Khasib was born on 21st of May 1995 in Palestine. He has demonstrated superb work habits and high intelligence. He is a highly active student, always on time, open minded, friendly and enjoying teamwork. He has shown a high degree of intellectual effectiveness through absorbing and applying new information.

Mr. Isam has finished his bachelor degree in civil engineering at An-Najah National University in Palestine in 2017. He worked as a teaching and research assistant in the civil engineering department at An-Najah National University for almost 2 years. He enrolled geotechnical and geological engineering master's program at Universiti Putra Malaysia in September 2019

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