PHYSICO-CHEMICAL AND MICROSTRUCTURE OF ARTIFICIAL SOILS
STABILISED WITH LIME-ZEOLITE

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To my beloved father, mother, wife, daughters and siblings

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ABSTRACT

The chemical stabilisation by admixing soil with lime is widely used in soft soil stabilization but its effectiveness in organic soil is low. Studies have shown that besides the organic content, the type of organic matter also influences the effectiveness of lime stabilisation. Artificial soils were utilized in this study to minimize potential influential factors that may be affected by the geochemistry variability of natural organic soils. Two types of organic matter, namely organic acid (contains humic acid and fulvic acid) and coco peat were utilized to simulate humified and non-humified organic matters that are normally found in natural organic soils. Commercially processed kaolin with different fineness and nSi/nAl were chosen as the base soil. The base soil and organic matter were manually mixed in different ratios based on their dry unit weight. Lime was partially replaced with zeolite with the aim to produce additive with self cementing matrix. The effectiveness of blended lime zeolite was investigated in this study. The total amounts of additives ranged from 7.5% to 15% were utilized based on the results of the Initial Consumption of Lime (ICL). Half of the remoulded test specimens were cured with a constant temperature of 50°C in an oven while the other part of the samples was cured at room temperature. The curing periods were set as 7, 28 and 56 days before being tested for unconfined compressive strength. Strength is utilized to determine the effectiveness of additives in stabilising artificial soils. The morphology of the stabilised and unstabilised materials was examined using a Field Emission Scanning Electron Microscope (FESEM), while the mineralogy of the materials was determined using X-Ray Diffraction (XRD) in order to unveil the hardening mechanisms of stabilised soils. General Linear Model (GLM) was utilized to determine the significant main factors, and interactions factors on the strength of artificial soil. The significant factors were used as the input parameters for multiple regression analysis to develop the strength prediction model. The models were utilised to predict the strength of stabilised materials within the inference space defined by the experiment. Overall, the mixture of coco peat and organic acid showed a deleterious effect on the strength of lime-zeolite stabilised artificial soils by lowering the soil's pH and increasing its porosity. However, the results of the organic acid samples with different pH and cured in different curing temperature were inconsistent. The availability of needle-like structures or Calcium Silicate Hydrate (C-S-H) phase was used as an indicator of successful in the cementation process without being inhibited by organic matter. The 20% replacement of lime with zeolite was found to achieve the highest strength when used to stabilise artificial soils with 30% of organic acid under a laboratory environment. A 20% replacement of lime with zeolite was found to experience only a slight decrease in pH and was able to enrich the elemental properties of lime with silica and alumina, which are important for producing a cementing matrix in an alkaline environment.

ABSTRAK

Penstabilan kimia dengan kapur telah digunakan secara meluas dalam penstabilan tanah lembut tetapi keberkesanannya adalah rendah untuk tanah organik. Keberkesanan penstabilan kapur dipengaruhi oleh kandungan organik dan jenis bahan organik. Tanah tiruan yang digunakan dalam kajian ini bertujuan mengurangkan faktor pengaruh yang berpunca daripada sifat kepelbagaian geokimia tanah organik semulajadi. Dua jenis bahan organik, iaitu asid organik (asid humic dan asid fulvic) dan gambut kelapa digunakan sebagai pengganti bahan organik terurai dan tidak terurai yang lazim ditemui dalam tanah organik semulajadi. Kaolin komersial yang mempunyai kehalusan dan nilai nSi/nAl yang berbeza telah dipilih sebagai tanah asas. Tanah asas dan bahan organik telah dicampur secara manual pada nisbah yang berlainan berdasarkan kepada unit berat kering. Sebahagian kapur adalah digantikan dengan zeolit untuk menghasilkan bahan campuran yang mempunyai matriks pensimenan sendiri. Kebolehkesanan campuran kapur zeolit telah dikaji dalam kajian ini. Jumlah bahan campuran yang digunakan adalah dalam lingkungan 7.5% kepada 15% bergantung kepada Kadar Penggunaan Kapur (ICL). Separuh daripada spesimen yang dibentuk semula akan dirawat dalam ketuhar pada suhu 50°C. Manakala sebahagian lagi dirawatkan pada suhu bilik. Tempoh rawatan sampel adalah ditetapkan pada 7, 28 dan 56 hari sebelum diuji untuk menentukan kekuatan tak terkurung. Kekuatan digunakan sebagai penentu keberkesanan bahan pencampur dalam menstabilkan tanah tiruan. Morfologi bahan distabil atau tidak distabil telah diuji menggunakan alat Pelepasan Imbasan Mikroskop Elektron (FESEM), manakala mineralogi bahan telah dikaji menggunakan kaedah Pembelauan Sinar-X (XRD) untuk mengenali mekanisma pengerasan tanah. Model Linear Umum (GLM) telah digunakan bagi menentukan kesesuaian faktor-faktor utama, dan interaksinya sebagai parameter masukan untuk analisis regresi berganda bagi membangunkan model ramalan kekuatan yang boleh digunakan untuk meramal kekuatan bahan stabil dalam ruang inferens yang ditakrifkan oleh ujikaji. Secara amnya, campuran gambut kelapa dan asid organik telah menunjukkan kesan negatif ke atas kekuatan tanah tiruan dengan menurunkan pH dan meningkatkan keliangan. Namun demikian, kesan asid organik ke atas kekuatan tanah tiruan adalah tidak konsisten untuk nilai pH campuran dan suhu rawat yang berbeza. Penemuan struktur berbentuk jarum atau fasa Kalsium Silika Hidrat (C-S-H) digunakan sebagai penanda kejayaan proses pensimenan tanpa diberhentikan oleh bahan organik. Penukaran 20% kapur dengan zeolit didapati mencapai kekuatan paling tinggi apabila digunakan untuk merawat tanah tiruan yang mengandungi 30% asid organik dalam suasana makmal. Sebanyak 20% penukaran kapur dengan zeolit hanya mengalami penurunan nilai pH yang rendah dan dapat memperkayakan ciri-ciri asas kapur dengan silika dan alumina. Elemen silika dan alumina adalah penting dalam menghasilkan matriks simen dalam suasana alkali.

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LIST OF ABBREVIATIONS AND SYMBOLS

Al	-	Aluminum
Al_2O_3	-	Aliminium Oxide
ATR	-	Attenuated Total Reflectance
BS	-	British Standard
Ca^{2+}	-	Calcium Cation
CAH	-	Calcium Aluminum Hydrate
CaO	-	Calcium oxide
$CaCO_3$	-	Calcium Carbonate
$Ca(OH)_2$	-	Calcium hydroxide
$CaSO_4$	-	Calcium Sulphate
CSH	-	Calcium Silicate Hydrate
EDX	-	Energy Dispersive X-ray Analyzer
Fe_2O_3	-	Ferric Oxide
FESEM	-	Field Emission Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared
GPS	-	Global Position System
H_2O	-	Water
HCI	-	Hydrochloric Acid
IC	-	Inorganic Carbon
ICDD	-	International Center for Diffraction Data
ICL	-	Initial Consumption of Lime
JMG	-	Minerals and Geoscience Department Malaysia
K^+	-	Potassium Cation
LL	-	Liquid Limit
LOI	-	Loss of Ignition
MARDI	-	Malaysia Agricultural Research & Development Institute
MC	-	Moisture Content

Mg^{2+}	-	Ion Magnesium
MgO	-	Magnesium oxide
MP	-	Modified Peat
Na^{+}	-	Sodium Cation
NaOH	-	Sodium Hydroxide
$(OH)^{-}$	-	Hydroxide ion
OPC	-	Ordinary Portland Cement
PI	-	Plasticity Index
PL	-	Plasticity Limit
ppm	-	Parts per million
RECESS	-	Research Centre for Soft Soil
RHA	-	Rice Hush Ash
SiO_2	-	Silica
SG	-	Specific Gravity
SOM	-	Soil Organic Matter
TC	-	Total Carbon
TEM	-	Transient Electromagnetic
TG	-	Thermal Gravimetric
TGA	-	Thermal Gravimetric Analyzer
TOC	-	Total Organic Carbon
UCT	-	Unconfined Compression Test
UTHM	-	Universiti Tun Hussein Onn Malaysia
UTM	-	Universiti Teknologi Malaysia
VP	-	Virgin Peat
WDXRF	-	Wavelength Dispersive X-ray Fluorescence
XRD	-	X-ray Diffraction
XRF	-	X-ray Fluorescence
ZrO_2	-	Zirconium Oxide
θ	-	Failure angle

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Malaysia, as a tropical country is reported to experience humid and wet climates throughout the year. The organic materials in this region are found to be well preserved over a long period of time because of their physico-chemical and bio-chemical processes (Bujang and Faisal, 2007). Plants that were preserved and disintegrated under anaerobic and high water content conditions have resulted in an accumulation of organic soils in this region. Organic soil that is found to be low in strength and high in compressibility is categorized as soft soils.

Soft soil stabilisation by admixing it with chemical stabilisers namely lime and cement are able to improve its engineering properties. The improved engineering properties of stabilised soil are found to be better in strength and variability control, lower in compressibility and deformability, and increase in volume stability and durability (Hausmann, 1990). However, Koslanant *et al.* (2006) found that lime or cement stabilisation of soil are less effective when dealing with high organic content. Soils with high organic content normally experience lower strength increase after stabilisation, if compared with inorganic soils stabilised with the same types and amounts of additive (Ruis and Hansson, 2001).

Organic soils which are formed by disintegration of organic matter exhibit various engineering characteristics with the amount of organic contents and stages of decomposition. Hence, the organic soil is categorized based on its contents (British

Standards Institution, 2015; Department of Standards Malaysia, 2005; IKRAM, 1995)and stages of decomposition (Von post classification system) in order to better describe the soil behaviour. However, the engineering properties of organic soil with organic matter higher than 20% are not suitable to be estimated using the mechanical criteria of conventional mineral soil. (Bujang and Faisal, 2007). Indeed, the properties of organic soil are not only governed by the organic contents but also vary with types of organic matter (Huttunen and Kujala, 1996; Koslanant *et al.*, 2006; Kujala, *et al.*, 1996; Kuno, *et al.*, 1989).

1.2 Problem Statement

Lime has been found to be an effective stabiliser for soil as it is able to increase its strength and stability under the action of water (National Lime Association, 2004). However, lime is not very effective in stabilising organic soils (Ruis and Hasson, 2001) even though the successfulness of organic soil stabilisation is sometimes deduced from empirical test data. The major problem in the use of calcium based stabiliser (including lime and cement) in the stabilisation of organic soils is the effectiveness of stabiliser over time (Bujang, 2006; Chen *et al*., 2009; Koslanant *et al*., 2006). Thompson (1966) explained that the pozzolanic reactions were obstructed in organic soil because the soil particles (as the primary source of silica and/or alumina) used in the stabilisation process, were "coated" by organic cations, inhibiting the silica and/or alumina from dissolution, which results in an absence of silica and/or alumina in pozzolanic reaction. As a result, the desired improvement is hard to gain when dealing with high organic content. Sometimes, the improvement might also disappear over time.

Chen *et al.* (2009) found that calcium-based stabilisers like lime and cement are not suitable for soils with high organic content as the detailed mechanism involved in organic soil stabilisation is still not fully understood. It was explained that the organic molecules absorb calcium ions that are necessary for chemical reactions between stabilisers and soils thus inhibiting the growth of reaction products, such as Calcium Silicate Hydrate (C-S-H) and Calcium Aluminates

Hydrate (C-A-H). The reaction products of the chemical reaction (pozzolanic reaction) are the main contributor for strength enhancement. Thus, a detail study of its physico-chemical behavior is needed to understand the mechanisms involved in stabilisation.

In addition, Chen *et al.* (2009) also urged that alternative methods other than adding extra stabiliser must be used even with organic content as low as 1%. The effectiveness of lime or cement stabilisation can be also affected by the types of organic matter. Hence, it is practically difficult to determine the threshold of organic content that will retard the lime or cement stabilisation.

1.3 Objectives of Study

This research study aims to investigate and evaluate the use of Zeolite as pozzolan for lime stabilisation of artificial soils. Hence, the research study was undertaken with the following objectives:-

(i) To analyse the molecular structure in terms of functional group and its x-ray diffraction pattern in an attempt to identify the composition of natural organic soil as reference guide to design artificial soil;

(ii) To establish the use of artificial soils with organic acid and coco peat, and lime-zeolite as additives by determining its physico-chemical and microstructural properties;

(iii) To assess the changes induced on the mineralogy and microstructure of stabilised artificial soils with its unconfined compressive strength;

(iv) To evaluate the effect of various contributing factors, namely curing periods, curing temperature, types and contents of inorganic soil, types and contents of organic matter and percentage of additives (lime-zeolite) on strength enhancement of artificial soils;

(v) To establish strength prediction models for stabilising artificial soils with Lime- Zeolite

1.4 Significance of Study

The deleterious effect of organic matter on the strength of soil stabilised with lime and cement is not yet fully understood yet even though many studies have been completed. One of the main challenges is identifying the types and quantity of organic matter of the soil because the types and content of organic matter are found to change with the humification process and vary with time. Hence, there is a need for a comprehensive study of using artificial soil with specific organic matter and organic contents, especially the soil organic matter widely available in the peat/ organic soil of Malaysia.

In addition, even though natural zeolite (a kind of natural pozzolan that is widely available in our neighboring region) has a successful application in the cement industry as a pozzolan, there are no known studies on the use of it in soil stabilisation with lime, or in particular with organic soil. In fact, the concept of partially replacing lime with zeolite (rich in alumino-silicate) in order to produce a self-cementing matrix in aqueous environment with organic matter is also novel. Hence, there is a need to identify the possibilities of using zeolite to stabilise soil with organic matter.

Therefore, there is a need to identify the composition of local natural organic soils in order to design artificial soils that are able to address local problems. By identifying the physico-chemical and microstructure of artificial soils stabilised with lime-zeolite, this study will able to address the above mentioned gaps in knowledge and may be useful for geotechnical engineers and/ or those who wish to develop projects on local organic soil deposits.

1.5 Scope of Study

This study was carried out in four stages with the aim to investigate and evaluate the use of zeolite as pozzolan for lime stabilising soil with organic matters. The first stage focused on the composition properties of the natural organic soil in Pontian, and Batu Pahat, Johor which served as an input for designing artificial soil. Field exploration studies for peat and organic soils were carried out in Pontian, and Batu Pahat, Johor. The tropical peat and organic soil samples were collected using a peat auger or a tube sampler and tested for its mineralogy and organic composition. All the specimens were dried at the low temperature of 50°C in order to avoid the potential losses of volatile organic components in higher temperatures. The mineral composition of natural organic soils was determined using X-Ray Diffraction (XRD) and the elemental chemical composition was determined using X-Ray Fluorescence (XRF). The functional group of soils was identified by using Fourier Transform Infrared Spectroscopy (FTIR) which helped determine the organic composition of the soils.

The second stage was focused on examining the material properties and behaviour of artificial soils, natural zeolites and hydrated lime. The composition of artificial soils was chosen by the composition details of natural organic soils. However, the ratio of each component (organic matter vs. inorganic matter) was purposely changed with the aim to study the effect of its content. Two different kinds of processed soil (Kaolin S300 and Kaolin FM-C) with different ratios of silicate over aluminates and size distributions were chosen as the base soil for this study. The kaolin S300 and Kaolin FM-C are commercially processed inorganic soils from Kaolin (M) Sdn. Bhd. Two different types of organic matter: organic acid and coco peat were utilized in this study as substitute material of humified matter and non-humified matter in the natural organic soils. The organic acid, which consists of humic acid and fulvic acid, was originally imported by a local fertilizer shop from China as soil conditioner for agriculture, while the coco peat (also known as coir dust) was obtained by pre-drying and sieving the coir fiber/ coconut fiber through a sieve aperture of 2 mm. The use of organic acid and coco peat as substitute material of organic matter was also reported by other researches in horticulture studies (Abad, *et al.*, 2002; De Silva, and van Gestel, 2009; Sharma, *et al.*, 2014) and chemical stabilisation studies (Kujala, *et al.*, 1996; Tremblay, *et al.*, 2002; Modmoltin, *et al.*, 2004; Shao, *et al.*, 2007; Xu, *et al.*, 2007; Zhu, *et al.*, 2009; M. Yunos, *et al.*, 2013). In addition, the hydrated lime was also directly obtained from Lime Treat (M) Sdn. Bhd. in Pasir Gudang in order to insure freshness. The natural zeolite utilized in this study was imported from Indonesia and marketed as MECHASTONE by Anugerah Alam Sdn. Bhd.

The third stage included the parametric study which focused on the influence of organic matter (organic acid and coco peat), the percentage of additive (lime-zeolite), curing periods, and curing temperature on the mechanical properties of stabilised and unstabilised soil. A variety of artificial soils with known types and percentage of organic matter and inorganic matter was prepared and mixed with various percentages of lime and zeolite as additive for stabilisation. A series of parametric studies were planned and carried out in order to determine the effect of inorganic material (two types of kaolin soil), and organic matter (humified matter-organic acid; non-humified matter- coco peat) on the properties of soils. Besides that, various ratios of lime zeolite were utilized to stabilise the artificial soils with the aim to determine the suitability of zeolite as pozzolan in the lime stabilisation of soils. The amount of additives utilized in this study was in the range of 7.5% to 15% by referring to its initial consumption of lime (ICL). The remoulded specimens were subjected to an unconfined compressive test after curing for 7, 28 or 56 days. The specimens were either cured in a humid box (room temperature curing) or in an oven with a constant temperature of 50°c. It was found that the elevated temperature of 50°c is ideal for enhancing the strength of specimen stabilised with lime (George, et al., 1992). A total of 636 specimens were tested for its unconfined compressive strength by counting in 3 replicates for each mixture. A selective small portion of specimen obtained from tested specimen after a UCT test was further oven dried and grinded into small particles for microstructure tests. The changes in mineralogy and morphology were determined by using X-Ray Diffraction (XRD), and a Field Emission Scanning Electron Microscope (FESEM), respectively with the aim to identify its hardening mechanism. The findings of this study are served as a reference on the use of zeolite as pozzolan for lime stabilisation of organic matter that artificially added in.

Lastly, a group of strength prediction models were established by using a statistical approach in which the correlation between the various contribution factors namely organic content, curing periods, curing temperature and additive content were taken into consideration. The other factors namely, moulding water content, types of compaction and compactive effort are keep constant through controlled experiments. The General Linear Model (GLM) was utilized to determine the significance of the main factors, two-factor interactions, and three factor interactions. The significance factors and interactions were utilized in multiple regression analyses to develop a strength prediction model which can be utilized to predict the strength of stabilised materials within the inference space defined by the experiment.

1.6 Organisation of Thesis

This thesis aims to investigate the physico-chemical and microstructure of artificial soils stabilised with lime and zeolite. Overall, this thesis consists of six chapters with each chapter focusing on a different subject matter as follows.

Chapter 1 is the introduction for the whole thesis which gives an overview of the background of study, the problem statement, objectives of study, significance of study and scope of study. It covers a brief overview on the current problems and issues when stabilising soil with organic matter. In addition, this chapter also explains the approach taken in order to understand the mechanisms of organic matter stabilised with blended lime zeolite or in particular the role of zeolite as pozzolan.

Chapter 2 presents the review of literature related to this study, which includes the materials chosen in this study namely organic soils, lime and zeolite. This chapter also encompasses topic likes soil-lime reactions, the influence of organic matter on pozzolanic reactions, the use of humic acid and coco peat as a substitute in producing artificial soil, and the use of zeolite as pozzolan.

Chapter 3 focuses on the material characterisation and selection which includes the exploration study of organic soil in Pontian, and Batu Pahat, Johor as well as the methods and analysis of the physico-chemical, microstructure and mineralogy of artificial soils. Furthermore, this chapter also includes details of the characterization and selection of additive (hydrated lime and natural zeolite) that was used in this study.

Chapter 4 presents the compositional properties of natural organic soils in Pontian and Batu Pahat, Johor. The findings served as an input for designing artificial soils. In addition, the physico-chemical and mineralogical properties of a variety of artificial soils, and additives are also presented and discussed in this topic. The material properties reported in this topic unveiled the characteristics and behaviour of artificial soils and additives.

Chapter 5 presents the unconfined compressive strength of artificial soils with and without stabilisation by additives. The effect of various contributing factors on the strength of artificial soils such as the curing periods, curing temperatures, organic contents, organic types and percentage of additives (hydrated lime and zeolite) are analyzed using a statistical approach to determine the correlation. The findings from the study are also compared with other researchers' work. Besides that, the analysis and discussion on the morphology of the materials under the Scanning Electron Microscope, the bulk chemical composition and its phases determined via X-ray techniques, the functional group of materials via Infrared Spectroscopy are also presented in this chapter.

The last chapter in this thesis- Chapter 6 gives a summary of the whole result as an integral part of the study. The recommendations for future works or further works are also provided in this topic.

5. The nonlinear effects of various contributing factors on the unconfined compressive strength of stabilised artificial soil should also be focused with the aim to produce curvature of the response surface of unconfined compressive strength. This would further increase the accuracy of strength prediction models.

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