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CARBONATED ALKALI-ACTIVATED OLIVINE WITH GLASS FIBER FOR SOIL STABILIZATION

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By

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Chairman : Professor Bujang Kim Huat, PhD
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Soil stabilization is a universal approach commonly used in counter-balancing of soil ground under structures. This method of soil improvement utilizes binders such as cement and/or lime to enhance the mechanical properties of soil for construction purposes. However, the production of these binders has been known to increase the levels of carbon dioxide (CO_2) in the environment. Therefore, in an attempt to stabilize soil conditions, the search for sustainable materials which are essentially harmless to surrounding soils when treated and at the same time are cost-efficient, is justified. Olivine, with a chemical composition of [$(Mg,Fe)_2SiO_4$] can be considered as a sustainable material which has the natural capability of capturing CO_2 in the environment and creating carbonated minerals. The high amount of magnesium oxide (MgO) and aluminum oxide (Al_2O_3), as well as an adequate amount of silicon dioxide (SiO_2) form the chemical composition of olivine, making olivine a good choice for use in soil improvement activities in terms of its pozzolanic reaction and hydration. The present study was undertaken to emphasize some problems on the utilization of olivine as a newly proposed sustainable material for soil improvement programs. The study highlights the applicability of glass fiber with an alkali activated soil-olivine mixture, with and without carbonation, which helps in determining the Unconfined Compressive Strength (UCS), Indirect Tensile Strength (ITS), and the Flexural Strength (FS). In the study, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray spectroscopy (EDX), and X-ray Diffraction (XRD) analyses were also executed on pure soil and alkali activated soil-olivine mixtures with and without carbonation.
The first stage was to analyze the performance of the preliminary investigation in order to evaluate the effectiveness of olivine on some basic geotechnical characteristics of silty clay soil. The compaction test and the Unconfined Compression Strength (UCS) were used as a practical indicator to investigate the strength development. According to the test findings, utilizing 30% olivine resulted

in a sharp increase in the compaction and the UCS of the samples, in the same curing time.

In the second stage of this study, carbonated alkaline activation of soil+30% olivine, was adopted as a viable technique to evaluate binder formation due to CO_2 pressure change. In simpler terms, the binder formation is generally a synthetic alkali aluminosilicate which is produced from the reaction of a solid aluminosilicate with pre-designed concentrated aqueous alkaline solutes. After this, pressurized CO_2 is injected into it form the new binder ($MgCO_3$/$CaCO_3$). Based on the obtained UCS values at exposure pressure of up to 300 kPa, for a 7 day exposure period, using alkali-activated olivine, it was found that the peak strength of soil+30%olivine was increased by up to 55 times compared to that of host soil. Regarding exposure period, it was found that based on UCS results at an exposure period of up to 7 days, using alkali activated olivine, the peak strength of soil+30%olivine was increased by up to 55 times compared to that of host soil.

The third stage was to identify the effect of the alkali agent molarity on the strength development. The rules of alkali agent (NaOH) molarity in binder formation were examined (with and without carbonation). In accordance to UCS values, 10 M of NaOH after 7 days of exposure and 300 kPa CO_2 pressure, increased peak strengths by up to 55 times compared to that of host soil and 5 times to that of alkali activated samples (without carbonation).

In the fourth and last stage, besides the shear strength development, in order to increase the tensile strength and ductility of soil+30%olivine, the combined effect of fibre inclusion and alkaline activation (with and without carbonation) was described and reported. In this stage, along with the 30% olivine in presence of high alkali solutes, mineral glass fibers were used as a strong reinforcement inclusion. Besides the UCS test, indirect tensile strength and flexural strength tests were carried out at pre-designed curing regems. The test results indicated that the inclusion of glass fibers within alkali-activated soil+30%olivine caused a further increase in the peak stress and tensile strength, and a decrease in the loss of post-peak strength. The results show that the incorporation of carbonation in alkali activated soil+30%olivine+3%glass fiber, increased the peak strength by up to 1.2 times to that of a mixture without glass fiber.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KAJIAN OLIVINE TERAKTIF-ALKALI BERKABONAT DENGAN GENTIAN KACA UNTUK PENSTABILAN TANAH

Oleh

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Penstabilan tanah adalah satu pendekatan sejagat yang digunakan secara meluas bagi penyeimbangan tanah dibawah struktur. Kaedah penambahbaikan struktur tanah ini menggunakan pengikat seperti simen dan/atau kapur bagi meningkatkan ciri mekanikal tanah untuk tujuan pembinaan. Walaubagaimana pun, pengeluaran bahan pengikat tersebut diketahui meningkatkan paras karbon dioksida (CO_2) di persekitaran. Dengan itu, dalam usaha menstabilkan keadaan tanah, pencarian bahan mampan yang tidak berbahaya kepada tanah sekitar, dan pada masa yang sama, mempunyai kos yang cekap, adalah wajar. Olivine, dengan komposisi kimia [$(Mg,Fe)_2SiO_4$] dianggap sebagai bahan mampan yang mempunyai keupayaan semulajadi dalam memerangkap CO_2 dari persekitaran dan menjana mineral berkarbonat. Kandungan magnesium oksida (MgO) dan silikon oxsida yang tinggi dan jumlah silikon dioksida (SiO_2) yang mencukupi dalam komposisi kimia olivine, menjadikan olivine sebagai satu pilihan untuk digunakan dalam penambahbaikan tanah dari segi reaksi pozzolanik dan penghidratan. Kajian ini dilaksanakan untuk memberi penekanan keatas beberapa masalah terhadap penggunaan olivine sebagai satu bahan yang mampan untuk program penambahbaikan tanah. Kajian ini juga menonjolkan kebolehgunaan gentian kaca dengan tanah yang dirawat dengan olivine, menentukan kekuatan mampatan yang tiada had (UCS), kekuatan tegangan tidak langsung (ITS) dan kekuatan lenturan (FS). Dalam kajian ini, analisis Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Energy-dispersive X-ray Spectroscopy (EDX), dan X-ray Diffraction (XRD) analisis juga dilaksanakan keatas tanah tulen dan campuran tanah-olivine yang diktifkan alkali dengan dan tanpa pengkarbonan. Objektif pertama daripada empat objektif dalam kajian ini adalah untuk meneliti kesan olivine keatas kekuatan mekanikal tanah asal, mengenalpasti ciri tanah dan olivine dan menentukan peratusan tanah/olivine yang berkesan melalui kekuatan mekanikal. Objektif kedua kajian adalah untuk mengkaji mekanisme asas bagi matriks tanah-olivine dalam kewujudan

NaOH di bawah tekanan gas CO_2 dan dalam jangkamasa yang berbeza, menentukan kesan karbonasi olivine di bawah tekanan gas CO_2 yang berbeza dankesan karbonasi olivine didalam jangkamasa pemulihan CO_2 yang berbeza ke atas ciri mekanikal tanah. Objektif ketiga kajian ialah untuk mengenalpasti mekanisma asas matriks tanah-olivine dalam kewujudan NaOH yang berbeza molariti dan gas CO_2. Fokus kajian adalah untuk menentukan kesan pengkarbonan olivine dalam molariti NaOH yang rendah ke atas ciri mekanikal tanah. Objektif keempat dan terakhir adalah untuk menganggarkan nilai peratusan gentian yang ditambah ke dalam tanah, olivine dan NaOH dan kesan pengkarbonan ke atas interaksi antara gel geopolimerik dan gentian kaca. Dalam fasa pertama kajian, 30% olivine meningkatkan UCS sebanyak 12 kali ganda berbanding tanah tanpa rawatan. Keputusan struktur mikro menunjukkan bahawa olivine meningkatkan ciri kejuruteraan tanah, oleh itu, kestabilan tanah. Dalam fasa kedua, kesan pelbagai tekanan CO_2 dan jangkamasa pemulihan telah dikaji untuk memahami kesan-kesan tersebut ke atas campuran tanah-olivine yang diaktifan alkali. Keputusan UCS menunjukkan peningkatan ketara dalam kekuatan mampatan dengan peningkatan tekanan daripada 200 ke 300 kPa. Kadar peningkatan adalah masing-masing 1.9% dan 1.04% antara 330 dan 400 kPa. Keputusan UCS bagi pelbagai jangkamasa pemulihan menunjukkan peningkatan dalam kekuatan mampatan selepas pemulihan selama 7 hari. Analisis SEM pada 300 kPa CO_2 menunjukkan pengurangan tekanan menyebabkan pemberhentian tanah akibat pengkarbonan dan penghidratan MgO untuk mengeluarkan $MgCO_3$ dan $Mg(OH)_2$. Peningkatan yang sama dan ketara dalam ketumpatan direkodkan pada SEM selepas 7 hari pemulihan. Analisis XRD mengesahkan kiasan ini. Di fasa 3, keputusan UCS menunjukkan 10M NaOH merekodkan nilai tertinggi 5035 kPa bagi sampel berkarbonat,iaitu 4.7 kali ganda lebih tinggi daripada sampel tanpa pengkarbonan. Dalam analisis SEM, kajian menunjukkan perpecahan olivine daripada NaOH bagi mendapatkan struktur tanah yang sama. Analisis EDX menunjukkan bahawa sementara nisbah Si/Al and Na/Al meningkat, kekuatan mampatan tanah turut meningkat. Analisis FTIR menunjukkan puncak Si-O, Si-O-Al, Al-O, H-O-H, -OH, dan C-O masing-masing membuktikan fungsi olivine dan NaOH sebagai pengikat pengaktif.

Di fasa yang keempat, keputusan ujian UCS, ITS dan FS menunjukkan pengkarbonan tanah-olivine yang diaktifkan alkali, sebagai pengaktif, merekodkan kekuatan kemampatan yang lebih tinggi berbanding tanpa pengkarbonan. Apabila dibandingkan dengan kerja sebelum ini, kajian ini mendapati bahawa lebih banyak kandungan tanah liat di dalam tanah, maka lebih banyak olivine diperlukan. Seterusnya, lebih banyak olivine diperlukan, lebih tinggi tekanan CO_2 diperlukan untuk pengkarbonan sepenuhnya. Lapan dan sepuluh molar NaOH memberi UCS yang ketara bagi campuran tanah-olivine dalam kewujudan CO_2, sementara 10 and 12 M NaOH adalah lebih ketara dalam sampel yang diaktifkan alkali. Perlakuan CSOF 3% adalah ketara yang menunjukkan keberkesanan peningkatan pengkarbonan dalam meningkatkan interaksi antara gel yang terbentuk dan yang diperkuatkan.

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LIST OF ABBREVIATIONS

ASTM	American Socity for Testing and Materials
GHG	Greenhouse gases
CSH	Calicum Silicate Hydrates
CAH	Calicum Aluminate Hydrates
XRD	X-ray diffractograms
SEM	Scanning Electron Microscopy
EDX	Energy-Dispersive X-ray spectroscopy
GGBS	Ground Granulated Blast furnace Slag
CKD	Cement Kiln Dust
POFA	Palm Oil Full Ash
CASH	Calicum Aluminate Silicate Hydrates
OWC	Optimum Water Content
MDD	Maximum Dry Density
UCS	Unconfined Compression Strength
LL	Liquid Limit
PL	Plastic Limit
PI	Plasticity Index
S	Soil
SO_{10}	Soil mixed with 10%Olivine
SO_{20}	Soil mixed with 20%Olivine
SO_{30}	Soil mixed with 30%Olivine
SO_{40}	Soil mixed with 40%Olivine
ASO_4	Alkali activated Soil Olivine mixture with 4M of NaOH
ASO_6	Alkali activated Soil Olivine mixture with 6M of NaOH
ASO_8	Alkali activated Soil Olivine mixture with 8M of NaOH
ASO_{10}	Alkali activated Soil Olivine mixture with 10M of NaOH
ASO_{12}	Alkali activated Soil Olivine mixture with 12M of NaOH
CSO_4	Carbonated alkali activated Soil Olivine mixture with 4M of NaOH

CSO_6	Carbonated alkali activated Soil Olivine mixture with 6M of NaOH
CSO_8	Carbonated alkali activated Soil Olivine mixture with 8M of NaOH
CSO_{10}	Carbonated alkali activated Soil Olivine mixture with 10M of NaOH
CSO_{12}	Carbonated alkali activated Soil Olivine mixture with 12M of NaOH
MSH	Magnesium Silicate Hydrate

CHAPTER 1

INTRODUCTION

1.1 Introduction

The stabilization process of soil always plays a significant role in the civil engineering field, in that it designates reliability of each consequent which can be established. By its own nature, soil is a completely adjustable material, yet also complex in its own way. Soil materials could offer great prospects that in civil engineering, and hence, they are being widly employed for the dexterous usage, easy worldwide availability and low costs of implementation. However, the most commonly occuring soil materials are not exactly suitable for construction or engineering purposes. Several materials are available for application to different kinds of soils that depending on their characteristics, modify its composition and properties. The general rules indicate that several soils do not possess sufficient strength. Therefore, utilizing certain materials by adding them to soils, may result in adjustment and fortification of the soils themselves. Concrete is a broadly utilized material, used for the purpose of soil stabilization.

The annual growing average in the utilization for coal, oil, and natural gas, was assessed to be about 1.4% also 1.6% and 2.4%, respectively. The greenhouse gasses (GHG) emissions which are predicted to intensify even further as anthropogenic activities are on the rise. Estimations indicate that by 2030, the emissions would be beyond 70%. It is predicted that the energy associated with CO_2 expanse could be more than 38 Billion at a worldwide circle in 2030, which could lead to universal climate modifications (Ke Mcneil Price and Khanna, 2013).

It was predicted that the needs and usage of numerous fuels would increase by 1.7% each year, until 2030 (Birol, 2002). An early sample of study in climate change estimated concentration levels of CO_2 to be between 430 ppm and 530 ppm by the year 2100. To keep up with those predictions, it would require both, long term and medium term cuts of CO_2 emissions cumulatively, and other GHG restrictions. To bring the concentration levels of CO_2 from 430 ppm to 480 ppm instead of 530 ppm, would need a reduction in the discharge of GHG from 70% to 40% from 2010 through 2050 (Edenhifer et al, 2014).

Figure 1.1 presents an overview of global CO_2 emission in 2015, which highlights that two sectors, namely Transport and Electricity & Heat, produced nearly two-thirds of global CO_2 emissions. . Other sectors include energy industries other than electricity, residential industry and others such as commerical fishing, forestry and agriculture.

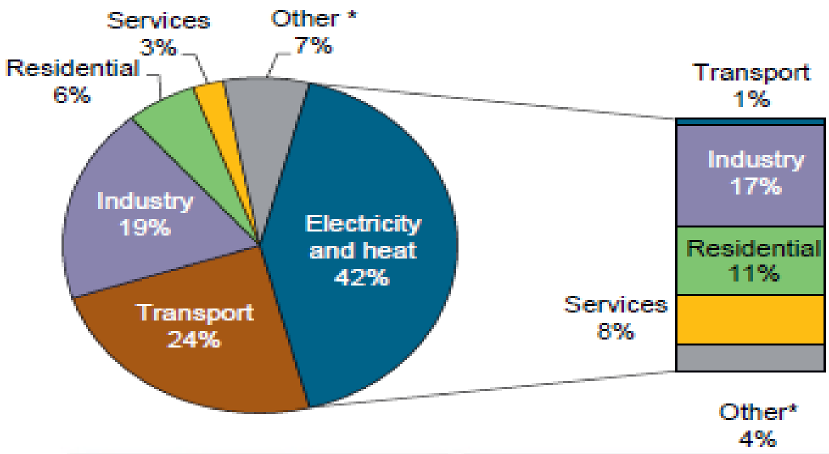

Figure 1.1: World CO2 Emissions from Fuel Combustion By Sectors.
(Source: IEA, 2015)

Recent studies have demonstrated the usage of some substances, such as MgO, as better substitutions of cement for soil stabilization (Ahnbreg and Johanssin, 2003; Jegandan, Al-Tabbaa, Lisk and Osman, 2010). The optimum rate of MgO, as the examination showed, could produce properties similar to that of pure cement. Furthermore, mixing soil with some waste substances in such combinations has proven to yield excellent results.

The American Society for Testing and Materials (ASTM) states that certain waste materials which are also pozzolanic in nature, could help in the process of soil stabilization. The utilization of MgO as an additive in the soil stabilization process has many advantages including, increasing the soil strength, decreasing the amount of cement used in soil, and capturing CO_2 in the soil, atmosphere, and rain. The utilization of CO_2 sequestration techniques offer a promising technology in the field of CO_2 fixation, by helping in the, fixing of CO_2 as a mineral (mineralization). This can be attributed to the minerals which can capture CO_2 and produce stable carbonates, resulting in permanent CO_2 sequestration. The process of chemical weathering by alkaline earth minerals include a process called mineral carbonation, which helps in capturing CO_2 (Kwon, 2011).

In the potential CO_2 capturing methods the usage of olivine $(Mg\text{-}Fe)_2$ SiO_4 is seen as a copious mineral technique. It was the employment of Mg and iron which led to the production of the Olivine matter. The outcome was mainly discovered in the mafic alongside the ultramafic igneous on rocks from the olivine production process, where other metamorphic rocks where typically founded (Jeas, 2011).

Earth olivine products were very rare to find, as they occur only in mantle xenoliths, which are the confident Basalt type in Dunite, and Peridotite massifs and phenocrysts, which are formed as a result of volcanic procedures (Schuiling, 2001).

In view of the environmental points, these byproducts and natural resources ought to be considered for the soil stabilization process. For a complete control of climate change olivine was considered to be the main contender for its superstation of the CO_2, the burning of 1L to relief CO_2 would require less than 1L of olivine typically, 1M of olivine burns 2M of CO_2 (Schuiling, 2001).

The above described CO_2 outcome from olivine happens as a result of mineral stabilization of the produced carbonates. In accordance with ASTMD5370 olivine is considered equal to a pozzolanic material, due to the enormous quantity of Al_2O_3, Fe_2O_3 also SiO_2, which could hypothetically be used to could advance soil products. Additionally, using MgO in a similar ratio as olivine creates a high possibility of carbonation of soil through the hydration process, giving it extra strength.

The worldwide distribution of olivine production as indicated in the Malaysian Geological Survey, is mostly concentrated along specific volcanic rocks, a great sum of which are present on the Andesite Dacite Basalt, which is located in the Mountains of Tawau in Sabah which is deliberated to be a key olivine foundation site (Tahir Musta and Rahim, 2010).

The first part of this research presents study of the olivine on the stabilization of soil and its benefits on engineering characteristics. A study is also done on key properties of olivine by analyzing some structures which have been treated by olivine using micro techniques. The second part of this research studies the carbonation effects of olivine on the alkaline stability of clayey soil which has been treated with olivine at different CO_2 pressures and at different curing times. The third stage of this research shows the function of the alkaline activator, sodium hydroxide (NaOH), at range of molarities on clayey soil which has been treated with olivine. The last part of this study explores the effect of carbonation in increasing the interaction between glass fibers (as the reinforcement member) and alkaline activated soil which has been treated by olivine.

In this research, the investigation used the CO_2 sequestration technique to stabilize the olivine soil products. Although in Malaysia, these soil products can still be detracted as unstable soils. Additionally, the research which added the alkaline activation by incorporating sodium hydroxide (NaOH) alongside olivine in order to increase the sequestration of CO_2, also ended up breaking and destroying the chemical bonding of SiO_2, for the stabilization process of soil. Similarly MgO, through a carbonation and hydration method of the olivine and MgO mixture, released SiO_2 pozzolanic materials.

The impact of this research drives from:

1. Decreasing the environment cost of constructing buildings.
2. Decreasing the time required to complete the construction activities.
3. Storage of CO_2 from atmosphere into a stable chemical form ($MgCO_3$/$CaCO_3$).
4. Using sustainable materials (olivine/glass fibers) for soil stabilization.
5. Strengthening silty clay soil.
6. Increasing the ductility of alkali activated material (with and without carbonation).

1.2 Problem Statement

Unstable soils are a geotechnical dilemma in Malaysia. Lime and/or cement were considered the most largely used stabilizers. However, these materials pose a few environmental problems resulting from CO_2 emission in the outside atmosphere; hence, there is a need to use aterials which contain fewer damaging side effects on the natural environmental stabilization of soil, and furthermore decrease the content of waste materials on the surface of the earth. Moreover, some of the lime/cement-based products are also considered to be dangerous and harmful for soil surroundings.

Furthermore, the use of cementitious binders in soil stabilization results in low flexural strength and poor tensile as well as a brittle behaviour (Sukontasukkul and Jamsawang 2012; Correia, Oliveira, and Custódio 2015). Moreover, the stabilized soil tends to fail under tension, due to its brittleness (Sukontasukkul and Jamsawang 2012; Correia et al. 2015).

Therefore, this study introduces olivine and carbonated glass fibers as a contemporary sustainable materials, according to the range of the molarities of "NaOH", for soil stabilization through CO_2 sequestration.

1.3 Objectives

Finding out the efficiency of olivine as a maintainable material is the main objective of this study, by examining and investigating the complete and distinct alkaline instigation procedure designed for the "soil stabilization". Accordingly, the subsequent objectives which will be specifically addressed in the course of this study are:

1. To evaluate the effect of olivine on the mechanical strength of silty clay soil.

2. To assess the underlying mechanisms of soil-olivine matrix in the presence of NaOH with different CO_2 gas pressures in different exposure times.
3. To assess the underlying mechanisms of soil-olivine matrix in the presence of NaOH with a range of molarities with and without carbonation.
4. To evaluate the effect of carbonation on the strength and underlying mechanism of soil-olivine-glass fiber mixture in the presence of an alkaline activator.

1.4 Organization of this Thesis

This thesis will be organized by chapters; Chapter two will present a literature review on soil stabilization with the usage MgO and its by-products alongside the customary binders.

Furthermore, this chapter will also cover the key principles in the olivine mineral carbonation processes. The examination of alkaline activation on the maintenance of soils will be also mentioned in this chapter.

The third chapter will address in further detail the different effects of the olivine percentages on few engineering properties, and will also present a microstructure analysis of soil treated by olivine before carbonation in addition to alkaline activation process.

The third chapter also describes the techniques used in the research for soil stabilization. This chapter chapter further describes the materials used, the classification tests as well as the chemical and physical, unconfined compressive strength, indirect tensile strength, flexural strength and micro-structural tests.

The fourth chapter gives an overview of the results of the testing programme, along with the analysis and discussion of these test results, aided with drown curves.

The fifth chapter presents a brief summary of the research methods and the results obtained, followed by a description on the full conclusion of this study and additional recommendations purposed for future studies.

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LIST OF PUBLICATIONS

Wisam Dheyab, Afshin Asadi, Bujang B.K. Huat, Mohd Saleh Jaafar, and Lokmane Abdeldjouad (2018) “Soil Stabilized with Geopolymers for Low Cost and Environmentally Friendly Construction” GEOMATE conf. (accepted).

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Wisam Dheyab, Afshin Asadi, Bujang Kim Huat, Haslinda N., Lokmane Abdeldjouad, and Ahmed Giuma Elkhebu (2018) “Application of Alkali-Activated Olivine Reinforced with Glass Fibers in Soil Stabilization” (in process).

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