KAOLIN STABILISATION BY UTILISING GROUND GRANULATED BLAST SLAG FOR CARBON DIOXIDE REDUCTION

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DEDICATION

To my late Mother Somaya

Who taught me to believe in myself, who had been the kindest soul and the gentle voice calling me everyday to infuse me with patience, courage and determination to complete this research. You are not here with me today to witness the results of my work but I know you will be proud.

To my Father Mohammed

Who taught me hard work always pays off and to strive for the best. Who has been the source of strength and resilience for me and my siblings in the most difficult of times. Your love and faithful prayers carried me to complete this thesis to the end.

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ABSTRACT

The aim of this study is to identify the effect of ground granulated blast furnaces slag (GGBS) in stabilising kaolin using the accelerated carbonation process while sequestering CO_2 to reduce its level in the atmosphere. The effects of incorporating GGBS and carbonated GGBS on mechanical properties of soil and the underlying mechanisms of stabilisation are investigated by carrying out physical characterisations, strength test and microstructure analysis (i.e., Field Emission Scanning Electron Microscope, X-Ray Diffraction). In addition, thermogravimetric analysis (TGA) and Acid Digestion method were used to quantify the CO_2 that has been sequester in the soil. The results show that GGBS was found to be a good candidate for kaolin stabilisation (under ambient condition) in which the strength keeps increasing with time, and as the content of GGBS increases. 20% and 25% GGBS-kaolin mixtures strength have exceeded the value requires for the subgrade layer at 7 curing days. The highest strength recorded was for 25% GGBS-kaolin mixture at 28 days curing as the strength reached 8.4 MPa. For the mineral carbonation process, the outcome indicates that the strength rises as the carbonation period rises, and a substantial strength gain was noted during the first hour of the carbonation phase. Likewise, unconfined compressive strength (UCS) rises as the CO_2 pressure rises from 100 kPa to 200 kPa. Acid digestion method and TGA shows that the GGBS-kaolin mixtures sequester between 4.5 to 5% CO_2 from its original dry weight. XRD analysis shows no peaks were observed for the magnesium-carbonated products. On the other hand, a single peak of calcium-carbonated products was seen in the diffractogram of the carbonated GGBS-kaolin mixture. Field emission scanning electron microscopy (FESEM) analysis for carbonated samples shows a needle-like structures seen on the surface of the kaolin particles which is Aragonite ($CaCO_3$). It could be concluded that augmentation of the strength is due to carbonated calcium and magnesium products which stuff the soil voids.

ABSTRAK

Tujuan kajian ini adalah untuk mengenal pasti kesan terak tungku letupan berbutir tanah (GGBS) dalam proses menstabilkan kaolin dengan menggunakan kaedah proses karbonasi dipercepatkan disamping mengasingkan CO_2 supaya dapat mengurangkan tahap intensitinya di atmosfera. Kesan kombinasi GGBS tidak berkarbonat dan GGBS berkarbonat pada sifat mekanikal tanah dan mekanisme penstabilannya telah dikenalpasti dengan menjalankan ujian ciri fizikal tanah, ujian kekuatan dan analisis struktur (mikro Imbasan mikroskop Electron (FESEM), Serakan Tenaga X-Ray). Sebagai tambahan, analisis Gravimetrik Terma (TGA) dan kaedah Pencernaan Asid digunakan untuk mendapatkan kuantiti CO_2 yang telah diasingkan dalam pengstabilan tanah. Hasil kajian menunjukkan bahawa GGBS terbukti menjadi bahan yang baik dalam proses pengstabilan kaolin (dalam keadaan tidak berkarbonat) dimana kekuatannya terus meningkat seiring tempoh masa yang semakin lama, dan pada kandungan GGBS yang kian meningkat. Kekuatan campuran 20% dan 25% GGBS-kaolin telah melebihi nilai yang diperlukan untuk lapisan paling bawah permukaan tanah pada 7 hari tempoh pengawetan. Kekuatan GGBS-Kaolin tertinggi yang dicatatkan adalah pada campuran 25% GGBS-kaolin dalam 28 hari pengawetan iaitu mencapai kekuatan sehingga 8.4 MPa. Manakala bagi proses karbonasi mineral, keputusan kajian menunjukkan bahawa kekuatan meningkat sepanjang tempoh karbonasi meningkat dan peningkatan kekuatan yang banyak dicatat pada jam pertama fasa karbonasi (3jam). Begitu juga keputusan kekuatan mampatan tidak terkurung kekuatan mampatan yang tidak terkawal (UCS) semakin meningkat ketika tekanan CO_2 meningkat daripada 100 kPa hingga 200 kPa. Kaedah pencernaan asid dan TGA pula menunjukkan bahawa GGBS-kaolin mengasingkan antara 4.5% hingga 5% CO_2 daripada berat asal kering. Analisis XRD menunjukkan tidak ada puncak yang diperhatikan untuk produk berkarbonat magnesium. Sebaliknya, satu puncak produk kalsium berkarbonat dilihat dalam diffractogram campuran GGBS-kaolin berkarbonat. Analisis mikro Imbasan mikroskop electron (FESEM) untuk sampel berkarbonat menunjukkan struktur seperti jarum yang dilihat pada permukaan zarah kaolin yang merupakan Aragonite ($CaCO_3$). Kesimpulannya, peningkatan kekuatan disebabkan oleh kandungan kalsium dan magnesium berkarbonat yang mengisi kekosongan partikel dalam tanah

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Al	-	Aluminum
ASTM	-	American society of testing material
BS	-	British standard
CaO	-	Calcium oxide
$CaCO_3$	-	Calcite
CSH	-	Calcium silicate hydrate
CO_2	-	Carbon dioxide
E_{50}	-	Secant Modulus
G_s	-	Specific Gravity
MgO	-	Magnesium Oxide
$MgCO_3$	-	Magnesite
PI	-	Plasticity Index
PL	-	Plastic limit
SEM	-	Scanning electron microscopy
SiO_2	-	Silica
TGA	-	Thermogravimetric analysis
UCS	-	Unconfined compressive strength test
XRD	-	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background

Malaysia is known as a developing country that will develop more in the next few years. This development is due to population growth, which required more building, roads, and other facilities. Tropical countries such as Malaysia experience humid and wet weather. Ground tropical soils are generally affected by moisture content fluctuation, which causes a volume change. Hence, it leads to a low strength capacity that causes structural failure.

Chemical stabilisation using pozzolanic material that has involves a significant amount of calcium, silica, and aluminum in the presence of water was proven to be very useful in improving soil strength (Walker and Pavia, 2011). The most common pozzolanic materials used for this purpose are cement and lime. Cement is preferable as it is cheaper and more effective than lime (Raftari et al., 2014). Nevertheless, in Malaysia, lime in the form of hydrated lime is widely used; it can be found easily and abundantly, making it an economical choice for stabilising tropical soil. However, cement and lime production comes with disadvantages, such as the high amount of carbon dioxide (CO_2) emits during the process and the fact that they are not sustainable materials. (Phummiphan et al., 2018). In the late 1940s, the need to find alternative sustainable materials becomes essential.

The necessity to find alternative materials to replace ordinary Portland cement (OPC) partially or fully is essential, especially in Malaysia, where green and recycled material has been suggested. Among them, fly ash and bottom ash from the power plant (Abubakar A.U. & Baharudin K.S., 2012), ground granulated blast furnace slag (GGBS), which is a by-product from iron production, and other by-product materials (Pathak et al., 2014). Fly ash and ground granulated blast furnaces dust are pozzolanic materials according to ASTM: C-618 and ASTM: C-989, respectively (Hashim et al., 2015). Bagasse ash, a by-product of natural agricultural waste generated during bagasse combustion in the sugar industry (Jamsawang et al., 2017), and waste marble powder is a by-product of the marble industry (Choksi, Mishra and Patel 2018).

Malaysia generates about 22,000 tons of waste material per day, as reported in 2012; this quantity will rise annually to reach more than 30,000 tons per day by 2020. (Zainuddin et al., 2016) The essential sources for those waste materials are construction sector solid waste, representing about 41% of Malaysia's total waste, followed by industrial, household, commercial, and institutions, representing about 25% and 33%. It has been recorded that 76% of solid waste is successfully collected in Malaysia. 5% of the collected waste has been recycled while the remaining have been disposed to 112 landfills (Papargyropoulou et al., 2011).

It is well known that waste or by-product materials are considered to be most likely a toxic, ignitable, corrosive, or reactive pose that can cause severe damage to human health and the environment. Alternatively, the utilisation of these materials is an option in soil stabilisation instead of been disposed of (Rasool and Kapoor, 2016).

Currently, amidst the world climate change consideration, mineral carbonation (MC) is the most promising methodology for permanently storing CO_2. It encompasses minerals comprising calcium (Ca) and magnesium (Mg) with CO_2 to produce Ca/Mg carbonated products. It has been proven to enhance soil strength (He et al., 2013; Mun and Cho, 2013; Hu et al., 2017; Jimoh et al., 2018).

GGBS is considered as a waste material generates from cooling molten slag in water or steam it followed by drying it (Mujtaba et al., 2018). It is obtainable in Malaysia, and it is mainly composed of calcium, magnesium, silicates, and aluminum (Islam et al., 2014). GGBS is the primary stabiliser in this study.

1.2 Problem Statement

Kaolin clay is a tropical clay associated with volume change due to the changes in moisture content. It is also known for its low shear strength, high plasticity, and poor workability; such a soil cannot afford the load from structures and vehicles to a certain extent. For this matter, the need for soil improvement/stabilisation is essential to overcome the problems.

OPC is used globally to treat different soil types. However, OPC's global production is responsible for 5% to 8% of the total CO_2 emission worldwide. Producing 1 ton from OPC leads to 0.85 to 0.95 t CO_2 emission. CO_2 is considered the major greenhouse gas, including methane (CH_4) and nitrous oxide (N_2O). The CO_2 causes 50% of the global warming impacts on the climate change temperature and sea level; those impacts is considered one of the main causes of extinction. The other concern about OPC is the energy consumed, about 12 to 15% of the industrial area's energy. Compared to OPC, 1 ton of GGBS leads to 0.05 to 0.07 t CO_2 emission, much lower than OPC.

Globally annual emission of CO_2 increased from 17.78 billion tons in 1980 to reach 32.1 billion tons. This increment is about 100% in 35 years and is expected to reach double this value in 2035. In Malaysia, CO_2 emission increased by 69% from 2000 to 2020 to reach about 285.7 million tons (Jorat et al., 2018).

In the recent years, a new chemical stabilization technique that is more environmentally friendly has been introduced. The technique involves the use of calcium (Ca^+) and magnesium (Mg^+) rich material (cement, serpentine, olivine, wollastonite, steel slag, and reactive magnesia cement). These materials have the potential to absorb and sequester CO_2 permanently. Thus, cause the reduction of CO_2 in the atmosphere while beneficial for increasing the soil strength quickly. The process also is favorable as it does not rely much on pozzolanic reaction as conventional soil stabilisers.

1.3 Aim and Objectives of Study

This study aims to identify the effect of ground granulated blast furnaces slag in stabilising kaolin using the accelerated carbonation process while sequestering CO_2 to reduce its level in the atmosphere. In order to achieve this aim, the following objectives are conducted:

1. To evaluate the effect of GGBS contents on the strength of the kaolin clay under different curing period.

2. To evaluate the effect of carbon dioxide pressures on the treated kaolin clay with the effective content of GGBS under different carbonation period.

3. To quantify the content of carbon dioxide that has been sequestered by the stabilise soil.

4. To identify the changes occurs in the microstructure and mineralogical characteristics for the treated soil under ambient and carbonation condition.

1.4 Scope of Study

The scope of this study can be divided into the material to be used in this research and the laboratory tests:

1.4.1 Material

The soil used in this study is kaolin clay. It is considered as an unsuitable material for construction purposes as it is considered a weak soil due to its low strength. It is available in Malaysia and can be obtained in two colors; white without a sufficient amount of iron and changes to brown when iron is acceptable. GGBS is used as a stabiliser to improve the engineering characteristics of kaolin. GGBS has both calcium (Ca^+) and magnesium (Mg^+) in its chemical composition, known for their ability to react and sequester CO_2 as carbonated products.

1.4.2 Testing

Physical tests were carried out on the untreated kaolin clay to determine its properties and classification. A compaction test will be carried out on the treated kaolin clay with different content (5%, 10%, 15%, 20%, and 25%) of GGBS to determine the optimum moisture content and the maximum dry density, which were used later to prepare the samples for unconfined compressive strength (UCS). No further addition of GGBS was conducted (beyond 25%) as the strength already achieved the requirement limit at earlier curing. Also, the fact that the load cell capacity reaches its highest limit but the sample did not fail at 25% GGBS cured kaolin for 14 days. After preparing the samples, those samples were cured for 0, 7, 14, and 28 days under ambient conditions before the testing. The effective content of GGBS stabilised kaolin was then identified.

Chemical compositions of lime and cement from various studies show that lime has calcium oxide (CaO) between 80% to 90% from its total chemical composition, while cement with its different types has a CaO content between 60% to 70%. The chemical composition of GGBS from previous literature shows that the content of CaO in this waste material is between 40% to 45%, which half of the amount of CaO in lime or cement, respectively. Thus, it can be concluded that the initial content of GGBS for soil stabilisation to occur was 5%, which is twice the one used in lime and 1% greater than cement.

Next, different samples were prepared with the effective content of GGBS (20% and 25%) and cured for 0 and 7 days under ambient conditions. Those samples were then placed in a carbonation cell; the carbonation was performed by applying different CO_2 pressure (100 and 200 kPa) for other carbonation period (1, 3, 6, and 24 hours).

The quantification of CO_2 sequestered within GGBS-kaolin mixtures was determined using two methods: The acid digestion method using 10% nitrite acid solution (chemical method) and thermogravimetric analysis (TGA) (thermal method). Microstructure analyses such as X-ray Diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM), and X-ray fluorescence (XRF) are conducted on selected samples to determine the changes in soil structure and its chemical composition.

1.5 Significance of Study

This study is contributed to the chemical stabilisation of kaolin clay with the non-traditional stabiliser represent in this study by GGBS. GGBS has a significant amount of Calcium (Ca^+), so it can be used alone as a stabiliser due to its cementitious and pozzolanic properties. However, since GGBS has a low hydration rate, it leads to a low strength gained at the early stages. GGBS is considered as waste material with low CO_2 emission and low energy during manufacturing. Thus, it leads to a dual advantage; it will be cost-effective and environmentally friendly.

Previous studies have shown that magnesium and calcium can react with CO_2 and sequester it in the form of carbonates (He et al., 2013; Hu et al., 2017; Jimoh et al., 2018). This process is known as carbonation or mineral carbonation. It has been used effectively for the permanent storage of CO_2. Calcium carbonation that involves the reaction between calcium hydroxide ($Ca(OH)_2$) and carbon dioxide (CO_2) to produce calcium carbonate known as calcite with formula $CaCO_3$ have shown a more significant effect in enhancing soil strength at early stages. However, the reaction between calcium silicate hydrate C-S-H with CO_2 reduces soil strength. Materials rich with calcium can be used to carbonate and improve soil strength, such as cement (Nakarai and Yoshida, 2015; Cao et al., 2017; Ho et al., 2017). Magnesium carbonation using magnesium-rich material (reactive magnesia and natural olivine) have been proven to be very useful in soil stabilisation (Yi et al., 2013a; Yi et al., 2013b; Y.L et al., 2013; Cai et al., 2015; Guang-hua et al., 2015; Yi et al., 2016; Cai and Liu, 2017; Liu et al., 2017; Song-yu et al., 2017).

GGBS, with its high content of CaO about 47.5% and reasonable MgO of about 4.9%, can be used as an alternative for cement, reactive magnesia, and olivine as it has another advantage (by-product material) over them. Calcium (Ca) or magnesium (Mg) rich materials as cement, reactive magnesia cement or olivine have the ability to sequester the CO_2 by the reaction between CO_2 and calcium or magnesium. This reaction is a chemical expansion reaction that fills the soil's voids, which increases soil strength and durability. The ability of such an effect is being tested for ground granulated furnaces slag by conducting different laboratory tests.

Nonetheless, limited studies was conducted on soil stabilisation using carbonation where only cement (industrial material) (Nakarai and Yoshida, 2015; Ho et al., 2017) reactive magnesia (industrial material) (Yi et al., 2013a; Yi et al., 2013b; Y.L et al., 2013; Cai et al., 2015; Guang-hua et al., 2015; Yi et al., 2016; Cai and Liu, 2017; Liu et al., 2017; Song-yu et al., 2017) and olivine (natural material) (Fasihnikoutalab et al., 2016a; Fasihnikoutalab et al., 2016b; Fasihnikoutalab et al., 2017) have been used previously. . Up to date, there are no studies emphasized on the utilisation of GGBS as stabiliser material with consideration of its effect on carbonation. This study contributes to calcium and magnesium carbonation to enhance the soil strength while reducing CO_2 levels in the atmosphere by using sustainable material (GGBS). GGBS has been used widely in soil stabilisation due to its significant amount of calcium alone and other materials. GGBS can be used as an alternative for cement, reactive magnesia, and olivine as it has another advantage (by-product material) over them. Therefore, in this study, the abovementioned is covered.

1.6 Thesis Outline

This thesis consists of five chapters. Chapter one consists of an introduction on waste generation, the probability of using pozzolanic waste material as a stabiliser in soil stabilisation, and the new method for soil stabilisation using minerals carbonation, enhancing soil strength and sequestering CO_2. This chapter also includes the problem statement, objectives of the study, scope of the study, and significant of the study.

Chapter two provides an overview of kaolin formation, GGBS, waste management in Malaysia, and ground improvement role in CO_2 reduction. The role of calcium and magnesium carbonation in enhancing soil strength and the factors affecting it from previous findings is discussed in detail. Moreover, the methods which are used in the quantification of sequester CO_2 are introduced.

Chapter three includes the laboratory tests used in this study to achieve its objectives and the standards used for each test. The methodology used to set up the carbonation cell used to carbonate different mixtures of GGBS-kaolin. The criteria used to accelerate the carbonation process is also discussed.

Chapter four analyses and discuss the result obtained from the laboratory testing designed in chapter three. Macrostructure and microstructure behaviour, together with an integration of the basic engineering properties of treated kaolin and the strength behaviour under ambient conditions and carbonation process, are also discussed and compared to previous findings are included in this chapter.

The last chapter summarizes the whole result as an integral part of the study. It also provides recommendations for future works related to this topic.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Ahmed Mohammed Awad Mohammed**, Nor Zurairahetty Mohd Yunus, Muhammad Azril, Ahmad Safuan A Hezmi (2021). Sequestration of Carbon Dioxide Using Ground Granulated Blast Furnaces Slag and kaolin mixtures. "*Global NEST Journal*". **DOI:** https://doi.org/10.30955/gnj.003487. **(Q4, IF: 0.983).**

2. **Ahmed Mohammed Awad Mohammed**, Nor Zurairahetty Mohd Yunus, Muhammad Azril, Dayang Zulaika Abang Hasbollah, Ahmad Safuan A Hezmi (2021). Ground improvement and its role in carbon dioxide reduction: a review. "*Environmental Science and Pollution Research*". **DOI:**10.1007/s11356-021-12768-2 **(Q2, IF: 3.056).**

3. **Ahmed Mohammed Awad Mohammed**, Nor Zurairahetty Mohd Yunus, Muhammad Azril Hezmi, Ahmad Safuan A. Rashid & Suksun Horpibulsuk (2021). Carbonated ground granulated blast furnace slag stabilising brown kaolin. "Environmental Science and Pollution Research". **DOI:** https://doi.org/10.1007/s11356-021-14718-4 **(Q2, IF: 3.056).**