POZZOLANA CHARACTERISATION AND PROPERTIES OF MORTAR CONTAINING CALCINED MARINE CLAY

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DEDICATION

This thesis is dedicated to Dr. Salihu Jamari.

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ABSTRACT

The production of cement is highly associated with the emission of carbon dioxide into the atmosphere which has generally lead to the use of pozzolanic material to partially replace cement in construction industries with the motive of reducing the emission. The use of high-grade kaolinite as pozzolanic materials in construction industries has long been established. However, the high cost and depletion of high-grade material has led to the use of low-grade kaolinite material in cement production. This research aims to explore the use of a low-grade kaoline – marine clay, as a potential pozzolanic material through a series of experimental studies. These studies are done to assess the characteristics of marine clay; examine its pozzolanic reactivity and the effect of calcination: this study also investigates the properties of mortar containing the calcined marine clay. The characterisation process was done based on its index properties, which included its natural moisture content, liquid limit, plastic limit, and plasticity index. The microstructure was determined via the particle size analysis (PSA), the x-ray fluorescence analysis (XRF), the thermogravimetric analysis (TGA), the differential thermal analysis (DTA), and the x-ray diffraction (XRD) test. The reactivity of marine clay was studied through its conductivity. The effect of calcination ranging from 600 - 1000°C on marine clay was done to examine the response of its embedded chloride and sulphate content and loss on ignition. Finally, the properties of mortar containing calcined marine clay were investigated via the compressive strength, strength activity index, and microstructure. The characterization result indicated that the marine clay had fulfilled the basic properties of a pozzolanic material - where it belongs to the kaoline group with over 40% kaoline content. The conductivity test has indicated that marine clay that was calcined at 700 °C for 1-hour yielded the highest pozzolanic reactivity, measured based on the loss in conductivity due to the formation of C-S-H. A decrease in compressive strength was recorded with the replacement level increased from 5% to 30%; the control specimen consistently exhibited the highest compressive strength. This is attributed to the dilution effect and the ease at which marine clay absorbs water from the mix to yield a more porous blend. Nevertheless, marine clay has achieved the strength activity index requirement for pozzolanic materials. Lastly, the scanning electron microscopy, XRD, TGA, and DTA results had conclusively justified the use of marine clay as a potential pozzolanic material, which was proven through the consumption of portlandite for C-S-H formation. As such, even though marine clay has a bad reputation due to its instability and presence of undesirable organic material, it was still thermally activated to produce a good pozzolana. The results of this study are expected to provide valuable insights into the existing literature on the application of low-grade kaoline as a potential pozzolanic material.

ABSTRAK

Pengeluaran simen yang sering dikaitkan dengan pelepasan karbon dioksida ke persekitaran menyebabkan penggunaan bahan pozzolanik digunakan untuk menggantikan simen dalam industri pembinaan. Penggunaan kaolinit bermutu tinggi sebagai bahan pozzolanik telah lama wujud .Walau bagaimanapun, kos yang semakin tinggi dan perosotan bahan gred tinggi yang semakin serius kini mewajibkan penggunaan bahan kaolinit kelas rendah. Penyelidikan ini bertujuan untuk meneroka penggunaan kaolin kelas rendah, iaitu tanah liat laut sebagai bahan pozzolanik melalui kajian-kajian eksperimen untuk menilai ciri-cirinya, mengkaji kereaktifan pozzolanik dan kesan kalsinasi serta menyiasat sifat mortar yang mengandungi tanah liat laut yang terkalsinasi. Kajian ke atas ciri-ciri bahan ini telah dilakukan untuk mengenalpasti sifat indeksnya seperti kandungan kelembapan semula jadi, had cecair, had plastik, dan indeks keplastikan. Struktur mikro telah ditentukan melalui analisis ukuran zarah (PSA), analisis pendarfluor sinar-x (XRF), analisis termogravimetrik (TGA), analisis termal pembezaan (DTA), dan ujian difraksi sinar-x (XRD). Kesan kalsium antara 600 – 1000 °C di tanah liat marin telah dilakukan untuk memeriksa tindak balas klorida terbenam dan kandungan sulfat dan kehilangan pencucuhan. Akhirnya, sifat-sifat mortar yang mengandungi tanah liat marin terkalsinasi yang telah disiasat meliputi kekuatan mampatan, indeks aktiviti kekuatan, dan struktur mikro. Hasil ujian-ujian pencirian menunjukkan bahawa tanah liat laut telah menepati sifat-sifat asas bahan pozzolanik; ia mengandungi kandungan kaolin lebih daripada 40%. Ujian kekonduksian menunjukkan bahawa tanah liat laut yang telah dikalsinasi pada suhu 700 ^{o}C selama sejam menghasilkan kereaktifan pozzolanik tertinggi berdasarkan kehilangan kekonduksiannya akibat pembentukan C-S-H. Malangnya, kekuatan mampatan telah menurun apabila tahap penggantian meningkat daripada 5% sehingga 30%. Spesimen kawalan tetap menunjukkan kekuatan mampatan tertinggi. Fenomena ini telah dikaitkan dengan kesan pencairan dan kesenangan tanah liat laut menyerap air dari campuran untuk menjadi lebih berpori. Walaupun demikian, tanah liat laut telah mencapai indeks aktiviti ujian-ujian mikroskopi elektron imbasan, XRD, TGA, dan hasil DTA secara konklusifnya menunjukkan potensi tanah liat laut sebagai bahan pozzolanik, terbukti melalui penggunaan portlandit untuk pembentukan C-S-H. Oleh itu, walaupun tanah liat laut mempunyai reputasi buruk kerana ketidakstabilan dan kehadiran bahan organik yang tidak diingini, ia masih dapat diaktifkan secara termal untuk menghasilkan pozzolana yang baik. Hasil kajian ini diharapkan dapat menyumbang kepada literatur yang sedia ada mengenai penerapan kaolin kelas rendah sebagai bahan pozzolanik yang berpotensi

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LIST OF ABBREVIATIONS

AASHTO - American Association of State Highway and Transportation Officials
ASTM - American society for testing and materials
CASH - Calcium Alumina Silicate Hydrate
CSH - Calcium Silicate Hydrate
DMS - Dredge Marine Soil
DTA - Differential Thermal Analysis
E - Ettringite
K - kaolinite
LL - Liquid Limit
LOI - Loss On Ignition
MC - Marine Clay
OHP - Other Hydration Products
OPC - Ordinary Portland Cement
PI - Plasticity Index
PL - Plastic Limit
PSA - Particle Size Analysis
Q - Quartz
SEM - Scanning Electron Microscopy
TGA - Thermogravimetric Analysis
UTM - Universiti Teknologi Malaysia
XRD - X-Ray Diffraction
XRF - X-Ray Fluorescence

LIST OF SYMBOLS

Δw - Change in weight

A - Cross-sectional area

D - density

F - Compressive strength

p - Maximum load

CHAPTER 1

INTRODUCTION

1.1 Background of Study

After water, cement is said to be the most widely used construction material in the world. The superiority of cement in the construction industry is its exceptional ability to bind other construction materials like sand, water, and aggregate to form a composite material called concrete which undoubtedly has higher durability and strength as compared to other construction materials like timber and steel. However, the production of cement notably Portland cement is a major contributor to carbon dioxide (CO_2) emission in the construction industry.

Carbon dioxide emitted into the atmosphere during the production of cement leads to global warming. The CO_2 is released mainly from the combustion of raw materials ($CaCO_3 \rightarrow CaO+CO_2$) and from electric and fuel consumption in generating high temperatures for the calcination in the kiln. For every one ton of cement produced, an estimated 0.8 to 1 ton of CO_2 is emitted into the atmosphere which is an estimated 5 to 8% of the total anthropogenic CO_2 and it is coming from the production of cement alone (Pina, Ferrão, Fournier, et al., 2017). In fact, Armstrong (2013) estimated an increase in CO_2 emission from 3737 Metric tons (Mt) in 2012 to a projected 4368 Mt emission for 2016 due to the high demand for cement. These estimates are in line with the predictions by Gómez-Pozuelo, Sanz-Pérez, Arencibia, et al. (2019) which suggest an estimated 1.5 ˚C increase in global temperature within the 21st century. However, the partial replacement of cement with pozzolanic materials in concrete has been recognized to curb the excessive production of cement thereby reducing the emission of CO_2 into the atmosphere.

Volcano ash found near the modern town of Pozzuoli in Italy was originally refer to as pozzolana. Pozzolans have been used since the reign of the Roman Empire to produce cementitious products. Due to the fineness and high constituent of amorphous silica, pozzolana would often react with lime in the presence of water to form a relatively soluble compound. This compound gives concrete high mechanical strength because of its exceptional ability to bind lime and aggregate allowing concrete to set hydraulically (harden underwater). Generally, pozzolana became an ideal name for any material exhibiting the aforementioned characteristics, that is to say, that any siliceous and aluminous material that reacts with calcium hydroxide (lime) to produce a cementitious compound that enhances strength and durability is referred to as pozzolana (Gillot, 2014; Grist, Paine, Heath, et al., 2013).

There are two types of pozzolanic materials, the naturally occurring pozzolans e.g volcanic ashes, pumices, and Diatomaceous earth, we also have industrially made pozzolans such as metakaolin (MK), palm oil fuel ash (POFA), slag, fly ash, silica fumes, etc. In addition to the reduction of CO_2 emission and increasing the strength of concrete, the use of pozzolans also reduces the cost of construction since they are mostly industrial wastes. Furthermore, it serves as a solution to environmental pollution coming from the dumping of those industrial waste materials.

In places like Malaysia, Singapore, Indonesia, China, and many other countries around the world, there has been a growing need for infrastructural development in coastal areas due to insufficient land spaces, among others. However, structures built in coastal areas were observed to have issues due to the presence of a certain poor clay material underneath the ground, later termed “marine clay”.

Marine clay largely found in coastal areas around the world in countries like Singapore, Malaysia, China, Indonesia, etc. is often excavated before the commencement of construction due to the poor properties it exhibits (Du and Pang, 2018). These poor properties of marine clay are functions of the presence of the organic materials in marine clay which includes the likes of chloride and sulphate. Hence it is often regarded as a profitless material (Thomas, 1996). In fact, the dredging of marine clay has become prohibited in some countries like Singapore due to dumping issues and poor characteristics of marine clay (Du and Pang, 2018). However, as highlighted by Shahri and Chan (2015), a large volume of marine clay is dredged every year in Malaysian water bodies. These dredged marine deposits also referred to as dredged marine soil (DMS) or soft clay are not significantly dissimilar with ideal soil but the presence of biological and chemical contaminants gives marine clay poor characteristics (Emmanuel, Lau, Anggraini, et al., 2019; Hosein, Shekarchi, and Tadayon, 2016; Song, Zeng, and Hong, 2017). As reported by Pakir, Marto, Yunus, et al. (2015), one of these poor properties is the swelling and shrinking of marine clay as a function of the moisture content which makes the use of marine clay challenging, this report is in line with Lee, Tan, Lim, et al. (2016) investigation of marine clay found at a construction site in Kedah (Malaysia) where the authors also concluded that marine clay has an undrained shear strength of less than 25 KPa.

Yunus, Marto, Pakir, et al. (2015) reported that marine clay when saturated exhibits significantly different engineering properties when compared to other moist soil. The exceptional ability of marine clay to change in property with varying moisture content makes it unpredictable and dangerous to be used in foundations and subgrade for the pavement of roads. Marine clay was further described by Rao (2011) as a type of soil with a liquid limit below its natural moisture content and so even though marine clay is very strong when dry, it cracks easily when it comes in contact with moisture.

Although there have been numerous reports about the poor characteristics of marine clay because it is considered too soft to be used even as backfill materials (Bergado, Youwai, Teerawattanasuk, et al., 2003; Bo, Arulrajah, Sukmak, et al., 2015), Some researchers have however taken bold steps to re-energize and transform these marine clay into a more suitable material since soil can generally be stabilized either chemically using chemical additives (Pakir et al., 2015), mechanically by compaction (Burroughs, 2007; Ekinci, 2019; Kaproth, Kacewicz, Muhuri, et al., 2016; Mahvash, López-querol, and Bahadori-jahromi, 2017), biologically by bacteria (Attramadal, Tøndel, Salvesen, et al., 2012; Burroughs, 2007; Kheirfam, and Asadzadeh, 2020), hydrologically by controlling the temperature (Khalil, Charef, Khiari, et al., 2018; Lim, and Cachier, 1996; Wu, Deng, Zheng, et al., 2019; Yanguatin, Ramírez, Tironi, et al., 2019) or cement treated (Kang, Tsuchida, and Kim, 2017; Kang, Tsuchida, and

Athapaththu, 2016). The much research interest on the stabilization of marine clay is because of the increasing demand in infrastructural development in coastal areas leading to many encounters with marine clay deposits during dredging or excavation works around the world like in Asia, Australia, Europe, and North America (Gingele, Deckker and Hillenbrand, 2001; Kalscheuer, Bastani, Donohue, et al., 2013; Ohtsubo, Egashira, and Kashima, 1995; Poulos, 1996)

Pakir et al. (2015) looked at the effect of sodium silicate as a liquid based stabilizer on the shear strength of marine clay. This was an attempt to improve the strength and reliability of marine clay using sodium silicate acting as a liquid stabilizing agent. It worked out well since the result showed a reduction in plasticity and an increase in the unconfined compressive strength of the soil. furthermore, Rahman, Yaacob, Rahim, et al. (2013) looked into the geotechnical characteristics of marine clay to be used as a filler material. The permeability test on several samples revealed that the hydraulic conductivity ranges between 1.10×10^{-9} and 2.44×10^{-9} m/s. The low permeability is evidence of the dominance of finer fractions of silt and clay (78 - 88%) and just (12 - 22%) sand constituent of marine clay. A study has been proposed by Khalid, Ye, Kumar, et al. (2019) on the easy way to reconstruct structured clay from a troubled natural state to aid fast and easy quantitative analysis on marine clay. This is because the quantitative laboratory studies on the structural behaviour of natural unharmed marine clay require many similar natural samples due to the difference in mechanical behaviour with the difference in the location which makes it a tedious and expensive task. The study proposed mixing marine clay with a low cement content of about 1- 6% to help generate inter-particle bonds and hence modify clay structure to be similar to natural intact clay for easy quantitative analysis

Although there are recent reports on the possible reutilization of marine clay, there are, however, limited studies on the use of marine clay as a potential pozzolanic material. A study by Du and Pang (2018) has shown that marine clay belongs to the kaolinite group and hence can be used as supplementary cementitious material after undergoing treatment to eliminate the aforementioned contaminant. Another very recent study by Dang, Du, and Pang (2020) also agreed with the possible re-utilization of marine clay in mortar but failed to properly assess its pozzolanic behaviour as it dwells on just the thermal activation. It is very prudent to note that a deep understanding of the nature of marine clay, its properties, and the transformation mechanism to a pure pozzolana is imminent since a poor treatment would not yield a suitable material and the bid to avoid the aforementioned problems associated with marine clay would not be achieved.

It is in that regard that this research seeks to explore the best possible conditions of re-energizing naturally super flow marine clay deposits to a potential pozzolanic material as a function of its reactivity through not just thermal activation but its physical and mechanical properties, conductivity measures, chloride, and sulphite content, and its consequent influence in blended mortar.

1.2 Problem Statement

There has been numerous research on the use of kaolinite clay as a pozzolana (Kanimozhi, Rajkumar, Kumar, et al., 2021; Mgbemene, Akinlabi, and Ikumapayi, 2019; Sternik, Galaburda, Bogatyrov, et al., 2020). Kaolin clay exhibits pozzolanic behaviour after being transformed into metakaolin via the calcination process. Metakaolin is considered unique because it is neither entirely artificial nor it is entirely natural since it is formed as a result of proper thermal treatment of natural kaolin clay (Ilić, Mitrović, and Miličić, 2010). The thermal treatment dissipates water from mineral kaolinite ($Al_2O_3.2SiO_2.2H_2O$), which is the major constituent of kaolin clay. This water dissipation leads to the destruction of the material structure thereby forming an amorphous aluminosilicate ($Al_2O_3.2SiO_2$), otherwise known as metakaolin. The water dissipation process is referred to as dehydroxylation represented by the equation $Al_2O_3.2SiO_2.2H_2O \rightarrow Al_2O_3.2SiO_2 + 2H_2O$.

In the early days, high-grade kaolinite clay was used as supplementary cementitious material but in recent years, the high cost and limited availability of high-grade kaolinite clay deposits have necessitated the use of low-grade kaolinite clay to partially replace Portland cement. Among the various low kaolinite clay, a very limited study has shown that marine clay also has pozzolanic properties (Du and Pang, 2018).

However, the unstable nature of marine clay and the presence of impurities like sulphate and chloride has rendered the transformation of marine clay rather tricky compared to high-grade kaolinite clay because improper calcination treatment will not lead to the ideal transformation of marine clay to good pozzolanic material. While a recent study by Dang, Du, and Pang (2020) had just agreed with the potential value-added utilization of marine clay as a cement replacement through thermal activation, the study has, however, failed to produce a proper insight on the reactivity of marine clay at different temperature ranges through conductivity which is quite critical when dealing with an unstable material like marine clay. The previous study has also failed to determine how the thermal treatment affects the embedded contaminants like chloride and sulphate. It is in light of all these lapses that this research seeks to look into the reactivity behaviour of thermally treated marine clay at different temperature ranges through very vital experimental procedures like conductivity. Since marine clay from different location have different mechanical properties and may hence require different thermal treatment, it has, therefore, became very eminent to provide more insight on the proper treatment measures and wider understanding of the reactive nature of marine clay considering that improper calcination process of marine clay would not lead to its best possible transformation to a highly pozzolanic material.

On the other hand, urban development and or lack of land spaces have also necessitated mega construction projects in coastal areas around the world. However, marine clay found largely in those areas would have to be dredged out by contractors before the commencement of construction because it is reported that marine clay has poor properties like poor bearing capacity which affects the foundations of structures. After dredging, the marine clay being invaluable material is often disposed of in an open field which has become a great concern in many countries like Singapore where dumping of marine clay has now become prohibited. Prohibiting the dumping of marine clay is an indirect way of restricting construction in coastal areas since construction cannot be done without dredging and replacing the marine clay with a more suitable material. The dumping of marine clay has become a major issue that requires determining the re-utilization of this profitless marine clay for construction to go on smoothly in coastal areas of countries around the world. Hence the quest to determine various ways of value-added utilization of marine clay.

1.3 Research Aim and Objectives

This research aim is to assess the use of marine clay as a potential pozzolana material through the following objectives.

(i) To characterize marine clay through index properties and microstructure examination.

(ii) To evaluate the pozzolanic reactivity of calcined marine clay.

(iii) To investigate the effect of calcination on the impurities in marine clay.

(iv) To investigate the properties of mortar containing calcined marine clay.

1.4 Scope of Study

This study involves numerous experimental tests in other to answer the aforementioned objectives i.e. to characterize the marine clay, to evaluate its pozzolanic reactivity after calcination, to investigate the effect of calcination on the impurities, and lastly to investigate the properties of mortar containing calcined marine clay.

The characterization was done in two stages, the first stage was through the soil properties (Atterberg limit examination) where the natural water content, liquid limit, plastic limit, and plasticity index was examined to have an idea on the engineering property of marine clay which is a function of its critical water content. The second stage looked at the characterization of marine clay through microstructure examination which was limited to the particle size analysis (PSA), x-ray fluorescence (XRF), thermogravimetric and differential thermal analysis TGA/DTA, and x-ray diffraction (XRD) to study the microstructural features of the marine clay under magnification in an attempt to examine its potential use as a pozzolanic material.

The pozzolanic reactivity of marine clay previously calcined at 450 °C for 1 hour, 700 °C for 1 hour, 700 °C for 2 hours, and 1000 °C for 1 hour were examined through conductivity to study C-A-S-H formation which would consequently result in loss of conductivity. Calcination at 450 °C was chosen for the examination because the consumption of portlandite is said to occur at the temperature range of 400 – 500 °C, 700 °C was chosen because calcite is formed at the range of 600 -800 °C however more emphasis was laid on 700 °C because it appeared promising which lead to its examination at 1 and 2 hours of calcination, finally, 1000 °C was also chosen to be examined because mullite is formed with calcination beyond 950 °C.

The effect of calcination on marine clay was examined after the calcination process at different temperatures. This is in line with the temperature ranges associated with dehydroxylation which would eventually indicate the characteristics of kaolinite and its transformation mechanism. What ensued was a conductivity test on the different calcined clay in a bid to determine the calcium hydroxide consumption, loss in conductivity which is ultimately a measure of its reactivity.

The strength and reactivity of mortar containing calcined marine clay were examined by first looking into its workability at 0.5, 0.55, and 0.6 w/c ratios. Next, the compressive strength at a replacement level of 5%, 10%, 15%, 20%, 25%, and 30% at 3,7,14, and 28 days of curing age was examined followed by water absorption and strength activity index was determined. Finally, the mortar reactivity was checked using microstructure examination through scanning electron microscopic (SEM), x-ray diffraction (XRD), and thermogravimetric (TGA/DTA).

1.5 Significance of the Study

As this research was aimed at assessing the engineering properties of marine clay as a potential pozzolanic material through systematic investigation, it can then provide a platform for the development of a standard specification for a blended system involving marine clay which would be essential for practical applications. This research would also provide more insight on the use of low-grade kaolinite material as an alternative to the high-priced, unavailable high-grade kaolinite material which would be highly beneficial to construction industries. This will also contribute to the development of eco-friendly material that has a wide range of applications while also solving the problem of dumping issues associated with the excavation of marine clay during construction. Finally, it is envisioned that this study would add more substance to the pool of knowledge as regards the use of low-grade kaolinite material as potential pozzolanic materials.

1.6 Thesis Organisation

Chapter 1 provides the general appraisal for conducting the research, backed by the problem background, problem statement, research aim, research objectives, scope, and significance of the study.

Chapter 2 provides the review of literature on binders like Portland cement, pozzolana, and metakaolin, it also gives insight on literature regarding the effect of calcination, index properties, and pozzolanic reactivity of kaolinite clay through microstructure examination.

Chapter 3 describes in detail the methods of the experiments carried out during the research including some pictorial representations of the steps to provide more insight. It covers the methodology for raw material preparation, experiments on the characterization of marine clay, pozzolanic reactivity, the effect of calcination on the impurities embedded in marine clay, alongside the strength and reactivity of calcined marine clay in mortar.

Chapter 4 describes the results obtained from the experimental procedure experiments on the characterization of marine clay, pozzolanic reactivity, the effect of calcination of marine clay, alongside the properties of mortar containing calcined marine cay.

Chapter 5 gives an overall conclusion and overview on the result obtained in line with the objectives which include the characterization of marine clay, the pozzolanic reactivity of marine clay, the effect of calcination on the impurities, and finally the strength of marine clay mortar alongside its pozzolanic reactivity.

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