ASSESSMENT OF POZZOLANIC REACTIVITY FOR CALCINED SPENT BLEACHING EARTH ASH (SBEA) USING STRENGTH ACTIVITY INDEX (SAI), FRATTINI TEST AND X-RAY DIFFRACTION (XRD)

SYAZA SYAZVINA BINTI ANWAR

FACULTY OF ENGINEERING
UNIVERSITI MALAYSIA SABAH
2022

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SYAZA SYAZVINA BINTI ANWAR

THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE DEGREE OF BACHELOR OF CIVIL ENGINEERING

FACULTY OF ENGINEERING
UNIVERSITI MALAYSIA SABAH
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Mr. Lim Chung Han
SUPERVISOR

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NAME	:	**SYAZA SYAZVINA BINTI ANWAR**
MATRIC NO.	:	**BK18110240**
TITLE	:	**ASSESSMENT OF POZZOLANIC REACTIVITY FOR CALCINED SPENT BLEACHING EARTH ASH (SBEA) USING STRENGTH ACTIVITY INDEX (SAI), FRATTINI TEST AND X-RAY DIFFRACTION (XRD)**
DEGREE	:	**BACHELOR OF ENGINEERING**
FIELD	:	**CIVIL ENGINEERING**
VIVA DATE	:	**21 JULY 2022**

CERTIFIED BY:

<u>SINGLE SUPERVISION</u> Signature

SUPERVISOR

Mr. Lim Chung Han ANDREW

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Syaza Syazvina binti Anwar
21 July 2022

ABSTRACT

This research briefly determines and compares the test methods on assessing the pozzolanic reactivity. As the application of concrete in this industry had been widely known and applied, the replacement of cement content is needed as cement had been contributed to the higher amount of releasing gas carbon dioxide (CO_2) to the atmosphere. As Spent Bleaching Earth is one of the solid wastes that need to be reduced, the recycle product of SBE which is Spent Bleaching Earth Ash (SBEA) can be the solution. The utilization of SBEA in construction industry is widely justified, however, the calcination process is adding to the SBEA and questioned the capability of the calcined SBEA to partially substitute the cement content. Therefore, there are two (2) main objectives of this study which are to determine the pozzolanic reactivity of calcined SBEA using the Strength Activity Index, Frattini Test and X-Ray Diffraction (XRD) methods and to investigate the pozzolanic reactivity of uncalcined and calcined SBEA. This study is focus on SBEA where the samples used are divided into control mortar, uncalcined SBEA, and calcined SBEA. The calcined SBEA were obtained from the heating process in the furnace at the laboratory for 700°C in 4 hours. The research study had been found that the calcined SBEA shows less improvement of pozzolanic reactivity compared to uncalcined SBEA. The reduction of pozzolanic reactivity is due to two (2) main factors which are the percentage of water absorption and the intensity of the silica in the samples. Therefore, the calcination process of SBEA is not needed to improve the pozzolanic reactivity of SBEA.

ABSTRAK

Penyelidikan ini secara ringkas menentukan dan membandingkan kaedah ujian untuk menilai kereaktifan pozzolanic. Memandangkan penggunaan konkrit dalam industri ini telah diketahui dan digunakan secara meluas, penggantian kandungan simen diperlukan kerana simen telah menyumbang kepada jumlah pelepasan gas karbon dioksida (CO2) yang lebih tinggi ke atmosfera. Memandangkan Spent Bleaching Earth merupakan salah satu sisa pepejal yang perlu dikurangkan, produk kitar semula SBE iaitu Spent Bleaching Earth Ash (SBEA) boleh menjadi penyelesaiannya. Penggunaan SBEA dalam industri pembinaan adalah wajar secara meluas, namun, proses pengkalsinan menambah kepada SBEA dan mempersoalkan keupayaan SBEA yang dikalsinkan untuk menggantikan sebahagian kandungan simen. Oleh itu, terdapat dua (2) objektif utama kajian ini iaitu untuk menentukan kereaktifan pozzolanic SBEA terkalsin menggunakan kaedah Indeks Aktiviti Kekuatan, Ujian Frattini dan X-Ray Difraction (XRD) dan untuk menyiasat kereaktifan pozzolanic bagi uncalcined dan calcined. SBEA. Kajian ini tertumpu kepada SBEA di mana sampel yang digunakan terbahagi kepada mortar kawalan, SBEA tidak berkalsin, dan SBEA berkalsin. SBEA terkalsin diperolehi daripada proses pemanasan di dalam relau di makmal selama 700°C dalam masa 4 jam. Kajian penyelidikan telah mendapati bahawa SBEA yang dikalsinkan menunjukkan kurang peningkatan kereaktifan pozzolanic berbanding dengan SBEA yang tidak dikalsinkan. Pengurangan kereaktifan pozzolanic adalah disebabkan oleh dua (2) faktor utama iaitu peratusan penyerapan air dan keamatan silika dalam sampel. Oleh itu, proses pengkalsinan SBEA tidak diperlukan untuk meningkatkan kereaktifan pozzolanic SBEA.

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LIST OF ABBREVIATION

PC	-	Portland Cement
POFA	-	Palm Oil Fuel Ash
GGBS	-	Ground Granulated Blast Furnace Slag
MK	-	Metakaolin
SBE	-	Spent Bleaching Earth
SBEA	-	Spent Bleaching Earth Ash
SAI	-	Strength Activity Index
XRD	-	X-Ray Diffraction
ASTM	-	American Society for Testing and Materials
CH	-	Calcium Hydroxide
CSH	-	Calcium Silicate Hydrate
CS	-	Compressive Strength
Q	-	Quartz
SF	-	Silica Fume
ISSA	-	Incinerated Sewage Sludge
FA	-	Fly Ash

CHAPTER 1

INTRODUCTION

1.1 Overview

In the construction industry, the utilization of concrete is significant to construct various of structures as the characteristics of concrete which are high durability, strong, versatile and low cost. The concrete is obtained from the mixture of Portland Cement (PC), aggregates and water (Hasan, 2015). Based on Equation 1.1, the hydration process of cement is a chemical reaction whereas occurred when PC react with water to develop the strength. This reaction will mainly produce the calcium silicate hydrate and calcium hydroxide. The production of calcium silicate hydrate will contribute to the strength of concrete while the calcium hydroxide will not be affecting the strength. Calcium hydroxide is not a desired product in concrete since it is soluble in water and can be filtered away, making the concrete porous and reducing the durability of concrete. The strength in concrete is essential to sustain the load to construct various facilities for the public needs. The higher the strength in concrete, the better the performance of it. However, the application of PC contributed to the higher amount of carbon dioxide (CO_2) in the atmosphere. The environmental effect into the ecosystem should be reduced to ensure the habitat is well-lived.

Equation 1.1: Cement Hydration Process

Cement Hydration Process

$$OPC + H_2O \text{ -------------------------------------> } CSH + CH$$

According to the research study done by Sohrab in 2016, partially substitution of pozzolan into the cement will reduce the CO_2 emission into the atmosphere. This process will generate more sustainable outcomes as it can reduce the environmental issues with the similar objective. In addition, most of pozzolan can be obtained from a recycled materials such as eggshell, recycled glass and so on. The usage of pozzolan will reduce the cost of production of concrete as the cement usage will be reduced as well. Generally, pozzolan can be defined as a siliceous substance that can be utilised in mortar blends as a low-cost replacement for cement (Sohrab, 2016). When pozzolan was added into the concrete mixture and partially replaced the cement, pozzolanic reaction will be occurred as shown in Equation 1.2.

Equation 1.2: Pozzolanic Reaction Process

Pozzolanic Reaction Process

CH + S --------------------> CSH

In the pozzolanic reaction process, the silicious substance which is pozzolan will react with the calcium hydroxide to produce the calcium silicate hydrate. The entire amount of calcium hydroxide that pozzolanic material may mix, according to Massazza (1998), is determined by the nature and composition of the reactive phase in the pozzolan, the SiO_2 concentration of reactive phases, the proportion of calcium hydroxide to pozzolan, and the curing time. According to the research study by Altwair, the hydration process will produce 70% of calcium silicate hydrate, 20% of calcium hydroxide and 10% of other substances. The addition of pozzolan in concrete mixture which act as the replacement of cement will react with the 20% of calcium hydroxide and increase the percentage of calcium silicate hydrate to 90%. Thus, the rising amount of calcium silicate hydrate will improve the strength of the concrete. Therefore, the substitution of pozzolan into the cement is not only reducing the CO_2 emission but also reduce the cost of producing and enhance the quality of concrete.

1.2 Background of Study

Spent bleaching earth (SBE) is a solid waste product of the palm oil industry's bleaching process. This solid waste is currently disposed of without treatment in landfills, resulting in significant water and air pollution. Most of the countries around the world had forbidden the direct disposal of SBE at the landfills due to the worst pollution that might be occurred. A crude palm oil refinery plant produces roughly 2 million tonnes of SBE each year across the world (Farahiyah, 2020). Statistically, after Indonesia, Malaysia is the world's second-largest producer of palm oil. These two (2) countries had produced more than 80% of the supplies for palm oil to the whole world (Hirschmann, 2021). The direct disposal of SBE might be impossible as the production of SBE is huge. Therefore, the acceptable method for SBE is the application of recycle.

The recycled product of SBE is called as Spent Bleaching Earth Ash (SBEA). SBEA is the product which is extracted from the SBE as shown in Figure 1.1. The product of SBEA is recycled by a company at Lahad Datu, Sabah called as EcoOils Sdn Bhd. The utilization of calcined SBEA in the construction industry as pozzolan to partially substitute the cement content have not been widely explored as the research study for calcined SBEA is limited. Therefore, the observation of calcined SBEA is significant to ensure the capability of calcined SBEA to be pozzolan to partially replaced the cement content.

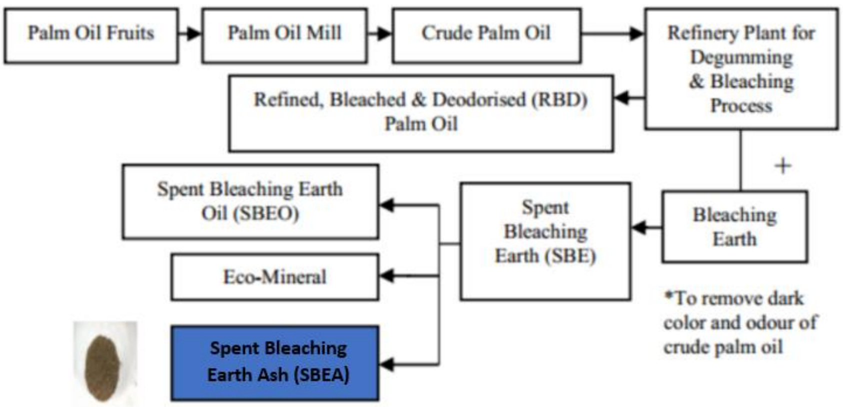

Figure 1.1: Spent Bleaching Earth Ash (SBEA) Process

Source: Farahiyah *et al.* (2020)

1.3 Problem Statement

In the construction industry, there are numerous types of pozzolans including the natural and artificial pozzolans. The ability of pozzolans to partially substitute the cement content is widely proven by several site-testing and their usage in this industry. The previous study proved that the calcined pozzolan had improved the pozzolanic reactivity of the pozzolan such as Palm Oil Fuel Ash (POFA), Metakaolin, and Clays. The improvement of pozzolanic reactivity is a benefit to the construction industry as it will enhance the characteristics of the concrete.

Generally, as the disposal of Spent Bleaching Earth (SBE) increased in Malaysia, the production of Spent Bleaching Earth Ash (SBEA) also increased. The pozzolanic reactivity of uncalcined SBEA is proven by numerous researchers and no longer questioned. However, the improvement is needed to fully utilize the usage of SBEA. The calcined SBEA is not explored yet when compared to other pozzolans. Therefore, the observation of calcined SBEA is significant to ensure the capability of calcined SBEA to partially replace the cement content.

Figure 1.2: Spent Bleaching Earth Ash (SBEA)

1.4 Objectives

The aim of this research is to assess the pozzolanic reactivity of Spent Bleaching Earth Ash (SBEA). Below are the objectives that need to be fulfilled to ensure the completion of this research.

1) To determine the pozzolanic reactivity of calcined SBEA using the Strength Activity Index, Frattini Test and X-Ray Diffraction (XRD) methods.
2) To investigate the pozzolanic reactivity on uncalcined and calcined SBEA.

1.5 Scope of Study

The scope of this study focused on achieving the two (2) objectives stated on this research which are to determine the pozzolanic reactivity of calcined SBEA using the Strength Activity Index, Frattini Test and X-Ray Diffraction (XRD) methods and to investigate the pozzolanic reactivity on uncalcined and calcined SBEA. Basically, the results are obtained from the laboratory testing of Compressive Strength Test, Frattini Test and X-Ray Diffraction Test. The comparison of three (3) different methods which are SAI, Frattini and XRD on the uncalcined and calcined SBEA is tabulated and analysed based on the results obtained. Therefore, the objectives will be achieved.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter present the reviews of the previous research study related to pozzolanic reactivity. Pozzolanic reactivity can be determined by using various of test methods. When the pozzolanic reactivity increase, the characteristics of concrete also will be improved.

2.2 Pozzolanic Reactivity on Calcined Pozzolan

Pozzolan is a silica mineral which do not possess the cement properties. However, the silica in the pozzolan will react with the CH which is the product of cement hydration process and produce the C-S-H. The C-S-H produced will contributes to the strength of the concrete and improve the characteristics of the concrete. The calcined pozzolan which is produced by the calcination process in the furnace with the very high temperature will change the properties of the pozzolan and affect the pozzolanic reactivity as well.

2.2.1 Metakaolin and Calcined Clays as Pozzolans for Concrete

As in the research study by Sabir in 2001, the metakaolin and calcined clays had been used as the pozzolan to partially substitute the cement content. The replacement had executed to minimize the usage of Portland cement (PC) as the production of PC had been damaging the environment by releasing the numerous amounts of carbon dioxide (CO_2). This study had been discussing several topics including the calcining temperature. The right temperature is significant to ensure the pozzolanic reactivity in its maximum level. As for the clay, the most reactive state is when the temperature of calcination in the range of 600°C to 800°C. It causes hydroxyl loss, which results in a crumpled and disorganised clay structure. According to this study, the optimum temperature for the calcination clay is 700°C. The temperature below 700°C will resulting less pozzolanic reactivity while above 850°C will declines the reactivity. The study was focusing on the clays and other pozzolans were not tested yet. Therefore, the tested on SBEA is significant to ensure the best replacement for cement content is served.

2.3 The Comparison of SAI and Frattini Test in Pozzolanic Reactivity

The Strength Activity Index (SAI) and Frattini Test are two (2) test methods which retain the high accuracy of testing in pozzolanic reactivity. Both methods are frequently used to determine the pozzolanic reactivity of the pozzolan and will conclude the ability of pozzolans to partially substitute the cement content.

SAI is obtained by the compressive strength test. The results from the test will be calculated based on Equation 2.1 where A is the compressive strength test of samples and B is the compressive strength test of control mortar.

Equation 2.1: Strength Activity Index (SAI) Formula

$$\boxed{SAI = A/B \times 100}$$

Frattini Test is a chemical testing where it observes the amount of CH in the samples by calculating the [OH^-] and [CaO] concentration by using the titration method.

2.3.1 Assessment of Pozzolanic Reactivity of Different Calcined Clays

In the research study by Tironi in 2013, there were seven calcined clays used which consist of five (5) kaolinites and two (2) bentonites. The calcined clays were obtained from the calcination of natural clays and tested for the pozzolanic reactivity by four (4) different tests and Strength Activity Index (SAI) is one of them. The calcination process of clays was done in the laboratory programmable furnace by using a bed technique. The samples were heated at temperature up to 700˚C in remained for 5 minutes. As for the test methods, the SAI and Frattini Test were used due to the higher accuracy to assess pozzolanic reactivity. The accuracy of the methods was already validated by a lot of research studies. This study was executed to examine the test methods in assessing pozzolanic reactivity of the calcined clays.

As the objective of this study is to analyse the test methods on assessing pozzolanic reactivity, the SAI and Frattini Test were having the most accurate result showing the pozzolanic reactivity. According to the ASTM C618, the SAI values of minimum 75% are considered active in pozzolanic reaction. The compressive strength (CS) value will not determine the pozzolanic reactivity due to dilution effect. The increase of the water to cement ratio will reduce the compressive strength but not the SAI values. Therefore, the SAI will define the pozzolanic reactivity of the calcined clays. As referred in Table 2.1, the most active sample is MK2 where it shows from the SAI value on 7, 28 and 90 days. The MK2 is having the highest SAI value compared to other samples. Therefore, it is concluded that the MK2 is active as in the pozzolanic reactivity.

Table 2.1: Compressive Strength and SAI

Compressive strength of mortars (CS in MPa) and strength activity index (SAI) at 7, 28 and 90 days.

Age (days)	PC	Q	MK1	MK2	MK3	MK4	MK5	BLPc	BNc
7									
CS	30.6	21.0	24.7	32.2	27.1	20.5	20.6	22.2	22.6
SAI		*0.69*	*0.81*	*1.05*	*0.89*	*0.67*	*0.67*	*0.73*	*0.74*
28									
CS	38.4	25.2	37.8	49.0	41.2	36.8	30.9	29.6	33.7
SAI		*0.65*	*0.98*	*1.28*	*1.07*	*0.96*	*0.80*	*0.77*	*0.88*
90									
CS	40.8	26.7	45.1	57.4	48.0	41.5	34.2	39.6	40.3
SAI		*0.65*	*1.11*	*1.41*	*1.18*	*1.02*	*0.84*	*0.97*	*0.99*

Source: Tironi *et al.* (2013)

The result of Frattini Test will be presented in a graph of [CaO] versus [OH^-]. The calcium isothermal curve will be defined the pozzolanic reactivity. The plotted calcined clay below the curve will shows the pozzolanic reactivity while the plotted calcined clays on and above the curve will shows no pozzolanic reactivity. As shown in Figure 2.1, the plotted points on and above the line which are Portland Cement (PC), Quartz (Q) and MK5 are not active as in their pozzolanic reactivity. Meanwhile, the farthest plotted point below the line which is MK2 is the most active as in pozzolanic reactivity.

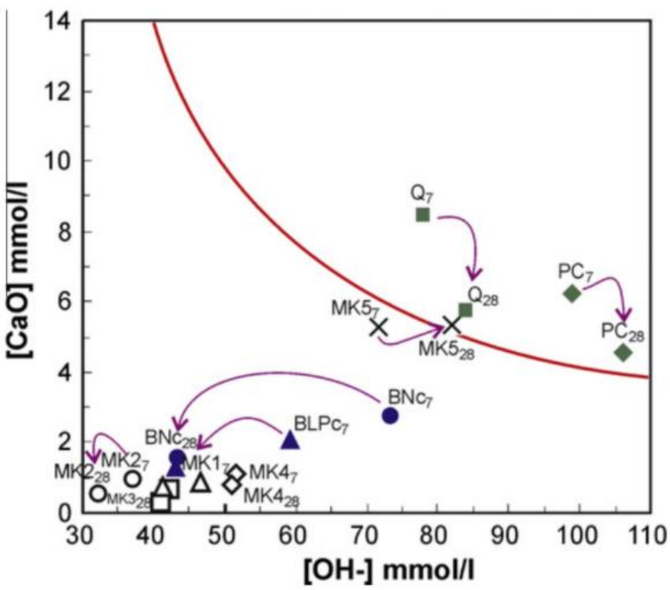

Figure 2.1: Frattini Test Graph

Source: Tironi *et al.* (2013)

The result of Frattini Test was complied with the SAI whereas MK2 was the most active in the pozzolanic reactivity. In addition, the reactivity sequence of calcined clays was the same in SAI and Frattini tests: MK2, MK3, MK1, and MK4. The accuracy of both methods was confirmed in several research study that have been done.

2.3.2 Comparison of Test Methods to Assess Pozzolanic Activity

The research study of comparing the test methods to assess pozzolanic reactivity by Donatello in 2010 had been referred. In this study, there were five (5) pozzolans which are Metakaolin (MK), Silica Fume (SF), Coal Fly Ash (FA), Incinerated Sewage Sludge Ash (ISSA) and Sand (S) used to assess the pozzolanic reactivity. The methods used in this study were consists of the Frattini Test and Strength Activity Index (SAI). The Frattini Test and SAI were used due to the common usage in previous study and known as the suitable methods that have the standard procedures to assess the pozzolanic reactivity. The different pozzolans were having their own reactivity. Therefore, the assessment of each pozzolan is significant to ensure the best quality of pozzolan used to partially substitute the cement mixture. The objective of this research study is to test whether the methods correlate with each other.

According to the research study, the SAI required to have a value greater than 0.75 after 7 and 28 days with the 20% replacement of cement with fly ash and natural pozzolans referring to ASTM C618. The results were reporting a decrease on strength on the 7 days of curing period comparing to the control cement mortar. However, all pozzolans excluding the Sand were achieving the minimum requirement of SAI in 28 days of curing period as shown in Figure 2.2. Then again, the ISSA considered for not showing the pozzolanic reactivity as the result was similar to the control cement mortar. The other pozzolans were showing their pozzolanic reactivity especially MK as it was resulting a considerable value of strength activity indices.