COMPARING COMMON METHODS OF ASSESSING POZZOLANIC REACTIVITY ASSESSMENT OF POZZOLANIC REACTIVITY FOR GRINDING SPENT BLEACHING EARTH ASH (SBEA)

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

FACULTY OF ENGINEERING
UNIVERSITI MALAYSIA SABAH
2022

DECLARATION

I hereby declare that this thesis submitted to University Malaysia Sabah is a partial fulfillment of the requirements for the degree of Bachelor of Civil Engineering. Hence, it has not been submitted to any other university for any degree. I also certify that the work described herein is entirely my own, except for the equations, quotations, and references of which have been duly acknowledged.

28 January 2022

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CERTIFICATION

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DEGREE : **BACHELOR DEGREE OF CIVIL ENGINEERING**

FIELD : **CIVIL ENGINEERING**

VIVA DATE : **21 JULY 2022**

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ACKNOWLEDMENT

First and foremost, I'd want to thank the Almighty God for his blessings and for granting me good health during the thesis writing process. This thesis would not have been possible without the tremendous aid and collaboration of everyone who assisted and supported me during the research. I'd like to use this occasion to convey my heartfelt gratitude and special thanks to Mr. Lim Chung Han, my Final Year Project Supervisor, for his advice and support. He generously offered his thoughts, ideas, and recommendations to help me enhance my thesis writing, and he provided me with helpful advice.

Bearing in mind what has come before, I would want to express my heartfelt gratitude to my beloved family and friends for their unwavering external support and encouragement during the thesis process. Without them, the thesis for the final year project would not have been completed.

Finally, I'd want to express my heartfelt appreciation to everyone who helped me, both indirectly and directly, from the beginning to the completion of my thesis writing process, allowing it to run smoothly and on schedule.

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30 JUNE 2022

ABSTRACT

In order to reduce greenhouse gas emissions due to natural anaerobic degradation, SBE was used as a cement substituent on concrete, in addition, helping in improving the quality of the concrete formed. The significance of this study is to learn about the performance of SBEA in compressive strength, chemical, and physical properties. Besides that, this study was using SBEA as the waste material is becoming useful and makes benefits to the construction industry for a better and greener environment. To determine the potential of pozzolanic reactivity of SBEA in concrete, strength activity index (SAI), Chapelle test, and X-ray diffraction, (XRD) was carried out. In this project, the main objective is to determine the reactivity of untreated and ground SBEA using SAI, XRD, and Chapelle tests then compare the overall result of the tests. From the XRD test, Quartz is found as the main component in SBEA sample. Furthermore, the SAI value for untreated SBEA mortar, 120 and 240 minutes of ground SBEA mortar at later age is higher than the early age. Futhermore, in Chapelle test, the 240 minutes ground SBEA exhibits highest pozzolanic activity which is 911 mgCa(OH)2/g while the untreated SBEA exhibits lowest pozzolanic activity which is 146 mgCa(OH)2/g. This is due to the difference grinding time affect the particles size of pozzolan. The higher the total surface area of the pozzolan, the higher the pozzolanic reactivity. Therefore, SBEA is proved to exhibit high pozzolanic reactivity.

ABSTRAK

Bagi mengurangkan pelepasan gas rumah hijau akibat kemerosotan anaerobik semulajadi, SBE digunakan sebagai pengganti simen pada konkrit, di samping itu, membantu dalam meningkatkan kualiti konkrit yang terbentuk. Untuk menentukan potensi kereaktifan pozzolanic SBEA dalam konkrit, indeks aktiviti kekuatan (SAI), ujian Chapelle, dan pembelauan sinar-X, (XRD) telah dijalankan. Dalam projek ini, objektif utama adalah untuk menentukan kereaktifan SBEA yang tidak dirawat dan dikisar menggunakan ujian SAI, XRD, dan Chapelle kemudian membandingkan keputusan keseluruhan ujian. Daripada ujian XRD, Kuarza didapati sebagai komponen utama dalam sampel SBEA. Tambahan pula, nilai SAI untuk mortar SBEA yang tidak dirawat, 120 dan 240 minit mortar SBEA yang dikisar pada usia lanjut adalah lebih tinggi daripada usia awal. Tambahan pula, dalam ujian Chapelle, SBEA yang digiling selama 240 minit menunjukkan aktiviti pozzolanik tertinggi iaitu 911 mgCa(OH)2/g manakala SBEA yang tidak dirawat mempamerkan aktiviti pozzolanic terendah iaitu 146 mgCa(OH)2/g. Ini disebabkan oleh perbezaan masa pengisaran mempengaruhi saiz zarah pozzolan. Semakin tinggi jumlah luas permukaan pozzolan, semakin tinggi kereaktifan pozzolan. Oleh itu, SBEA terbukti mempamerkan kereaktifan pozzolanic yang tinggi.

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LIST OF SYMBOLS

SBE	-	Spent bleaching earth
SBEA	-	Spent bleaching earth ash
CO_2	-	Carbon dioxide
SAI	-	Strength activity index
XRD	-	X-Ray diffraction
SBO	-	Oil industry solid residue
POFA	-	Palm oil fuel oil
CS	-	Corn starch
PAI	-	Pozzolanic activity index
G	-	Ground-glass pozzolan
UHPC	-	Ultra-high-performance concrete
HPC	-	High-performance concrete
CC	-	Conventional concrete

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Usually, bricks are comprised of sand, aggregates, and cement. Cement is the major constituent, and a minimum of 210 kg/m^3 is utilized to make pavements bricks (Ganjian *et al.* 2015). Cement is made from a variety of basic ingredients, including sand, clay, and limestone. The procedure necessitates a significant quantity of heat, which is typically supplied by fuel sources like tires, natural gas, coal, and toxic materials. Cement is readily manufactured, and due to the massive quantity of carbon dioxide (CO_2) generated during the process, it is acknowledged to be one of the biggest contributors to negative environmental consequences (Hamada *et al.* 2018).

ASTM C 125-13 defines a pozzolan material as a product with a chemical structure based on siliceous or siliceous and aluminous material, with little or no cement, that reacts chemically with calcium hydroxide at normal temperatures in finely divided form and with water to form compounds with cementitious properties. According to Sutan, N.M. *et al* (2018), the pozzolanic reaction occurs when the reactive aluminous and siliceous phases combine with calcium hydroxide (CH) at ambient temperature to create a new calcium silicate hydrate (C-S-H), which possesses cementitious properties. Pore filling effect is defined as the capacity of materials to fill voids, resulting in a compact, dense microstructure and increased strength of concrete structures. The cement hydration process hypothesis, which is affected by partial substitution of pozzolans.

Currently, pozzolanic materials made from by-products such as fly ash, silica fume, rice husk ash, and others are commonly employed in concrete. Its use not only enhances the qualities of concrete, but it also helps to conserve the environment. Silica fume is composed of very tiny solid spherical particles and contains a high concentration of the glassy phase of silicon dioxide, SiO_2. It has long been used as a mineral addition in high-strength concrete. Fly ash, another common pozzolan, can increase hardened concrete qualities such as ultimate strength, durability and workability. Significant fineness fly ash has high pozzolanic activity and may be utilized to make high strength concrete (Haque MN and Kayali O., 1998).

The production of palm oil refinery plants generates spent bleaching earth (dubbed as SBE) which is the by-product in the bleaching process of refined palm oil. It has been known that SBE contains a lot of phosphorus and potassium and is also available for plants to absorb (A. Beshara and C. R. Cheeseman, 2014). In the pretreatment process which is in the degumming and bleaching of crude palm oil stage, bleaching earth is added to produce refined palm oil. SBE is one of the products of the crude palm oil refining process. SBE is a fine yellowish-white powder that has the function of improving the yield strength of polymers, controlling viscosity, and functioning as a filler in plastic and rubber industries. In Malaysia, the disposal of refinery solid waste and drainage effluents (SBE) at landfills can affect greenhouse gas emissions due to natural anaerobic degradation. Leaching of soluble alkalinity, SA and carbonaceous material released into the landfill soil could result in the formation of greenhouse gas emissions which could then increase the concentration of atmospheric CO_2. This can have an adverse impact on global climate change (S. K. Loh *et al.* 2013). In order to mitigate this, a study was conducted at a refinery plant in Peninsular Malaysia to produce a pozzolanic reaction product from SBEs that was used as cementing material for stabilizing landfill leachate. To give an overview on the use of pozzolanic reaction products for the stabilization of landfill leachate, results obtained from laboratory investigations are presented.

SBEA is the main product of the EcoOils' company and it is produced by high-temperature calcination of slag that contains 0.4~0.8% CaO + 0.1~0.3% MgO + >30% SiO_2 + <3% Al_2O_3 based on the total weight of SBE. The major products in SBEA are Si, Ca, and Mg minerals which help to improve durability and counteract chemical attacks.

Additionally, SBEA also has a high reactive silica content (65 wt%) which enables SBEA to be used as a reactive pozzolanic building material. The dramatic drop in oil prices recently was one of the catastrophic events for oil companies, but it could be one more opportunity for new industries to develop. For example, this price drop would cause traditional hydraulic fracturing will not to have economic feasibility to build new wells; however, other techniques may be more economical like CO2 addition stimulation in the presence of natural pozzolan binder derived from oil industry solid residue, SBO. The figure 1.0 showed the sustainable products extracted from SBE.

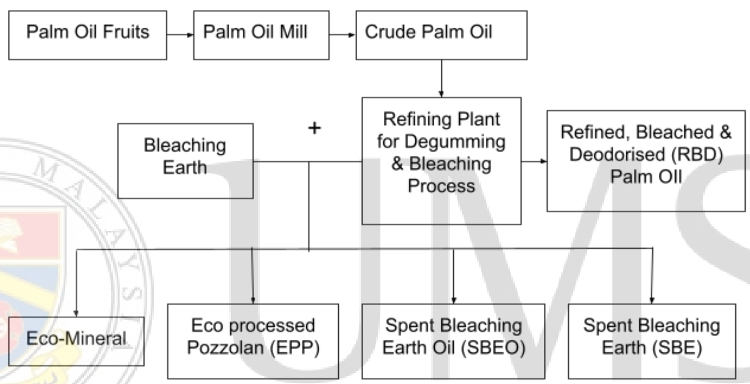

Figure 1.0 : SBEA Production Process

Few studies have been performed on the SBE samples despite the variety of materials produced, which demonstrate great potential for sustainability applications. The high-calcium, low-sulfur compound with pozzolanic reaction can be used as an efficient substitute for Portland cement in concrete. By replacing the conventional fine sand with 5–10% SBE material, artificial island construction becomes more economical and eco-friendly.

A pozzolanic reaction can be exhibited by various substances and materials. However, in this research, a pozzolanic reaction was examined by SBEA. The sand was prepared according to the ASTM C778 standard. The mixture with a water-to-binder (W/B) ratio of 0.48 was prepared. The mortar specimens with the dimension of 50 mm × 50 mm × 50 mm in size were cast. The pozzolanic activity was investigated by determining its strength activity index, SAI value based on the ASTM C311 standard.

1.2 Background of study

For years, cementing materials are commonly employed in the construction of diverse constructions. Concrete is extremely important in the building sector, particularly in the early nineteenth century, when it was used at a rate of up to 10,000 million tonnes a year. Cement is man-made, long-lasting, resistant to most exposure, requires little care, and hence is cost-effective. Furthermore, the manufacture of one tonne of Portland cement produces about one tonne of CO2 (Pacheco-Torgal, 2011). In Type 1 cement, 1 kilogram produces approximately 800g of CO2 and emits SO2 and NOx ranging from 1.1 to 3.4g (Pacheco-Torgal, 2011). As a result, it has an effect on the environment. Spent Bleaching Earth (SBE) is an industrial by-product of the bleaching earth refining process used in the production of edible oils. It is estimated that roughly 600,000 million tonnes of bleaching earth are used globally, producing approximately 60 million tonnes of oil (Loh S K *et al.* 2006), and it was stated in 2014 that 1 to 2 million tonnes of SBE were generated globally, including 25-40% oil and other pollutants. It frequently ends up in landfills, where it has a detrimental influence on the environment and can be hazardous, such as being very combustible. As a result, a sustainable solution was developed, which is to process and calcine SBE at high temperatures to produce SBE ash and SBE oil.

SBE is mostly made up of bleaching clay and residual oil. An SBE sample was analyzed and found to have a calorific value of 15.8J/g and an ash concentration of roughly 55%. The resultant ash met the Kenya Standard chemical composition standards for pozzolans after 4 hours of calcination at 550° (Muthengia J *et al.*, 2005). According to another study, the complete oil content from 10g of SBE was extracted through heating at high temperatures of 1000°C until no more sample weight loss occurred, and the average oil content was later estimated to be 24 percent (Al-Zahrani A A and Daous M A, 2000). SBEA is a pozzolanic substance with comparable cementitious qualities that may be used as a partial substitute for cement in the production of concrete, therefore contributing to a better environment while also saving money. SBEA is a pozzolanic substance with comparable cementitious qualities that may be used as a partial substitute for cement in the production of concrete, thus leading to a healthier environment while simultaneously saving material costs. Its sole constraint is its workability/consistency, which is critical for hot-climate areas like Brunei. One of the key motivations for this study is to learn more about the capabilities of SBEA, as there has been little research on this pozzolan. SBEA is a pozzolanic substance with comparable cementitious qualities that may be used as a partial substitute for cement in the production of concrete, therefore contributing to a healthier environment while simultaneously saving expenses. Its main restriction is its workability/consistency, which is critical for hot-climate areas like Brunei. One of the key motivations for this study is to learn more about the capabilities of SBEA, as there has been little research on this pozzolan.

Therefore, this study is to determine the pozzolanic characteristics of SBEA as cement replacement through a series of experiments such as the SAI, XRD, and Chapelle test. The sample preparation is SBEA with different fineness.

1.3 Problem Statement

Industry activities generate a lots of waste products. Concerns over the disposal of created garbage have been greatly exacerbated by people's growing environmental awareness. One of the world's top environmental challenges is the handling of solid waste. Waste utilization has emerged as a desirable substitute for disposal due to the lack of landfill space and the rising expense of disposal. On the use of waste materials in mortar research is being done. Rice husk, spent bleaching and glass are a few examples of such waste materials. Each of these waste materials had a unique impact on the characteristics of mortar. Mortar is made more affordable by using waste materials, and it also helps with disposal issues. Bulky garbage reuse is thought to be the most environmentally friendly solution to the disposal issue. Through this study, the issue of glass trash produced by construction and demolition activity is solved. Reusing glass waste materials to replace some of the cement used in regular portland cement has been suggested as a way to reduce the amount of spent bleaching produced. The goal of this study is to examine the viability of employing recycled earth spent bleaching in mortar manufacturing.

1.4 Significance of study

The significance of this study is to learn about the performance of SBEA in compressive strength, chemical, and physical properties. Besides that, this study was using SBEA as the waste material is becoming useful and makes benefits to the construction industry for a better and greener environment.

1.5 Objective of Research

The objective of this study is to:

1. To determine the pozzolanic reactivity of untreated and ground SBEA using SAI, XRD and Chapelle test.
2. To investigate the effect of difference grinding period of SBEA against pozzolanic activity

1.6 Scope of Study

The most popular waste-based supplementary cementitious materials, SCMs include fly ash, ground granulated blast-furnace slag, spent bleaching earth, and silica fume. The purpose of this research is to investigate how the difference grinding time of SBEA affect the pozzolanic reactivity. The SBEA used is ground and unground which 120 minutes and 240 minutes ground pozzolan is considered as ground pozzolan. In this study, 20% of SBEA is used as a cement replacement. It focuses on the effect of SBEA curing on compressive strength at 24 hours. The research is organized into four major phases, which are as follows:

1. Phase 1 is to prepare Portland cement, sand, water and ground SBEA for cement replacement in the mortar mixture.
2. Phase 2 is to determine the elemental composition of SBEA by XRD test.
3. Phase 3 is to prepare and analyze the compressive strength of SBEA by using SAI test.
4. Phase 4 is to test the performance of EPP using the Chapelle test.

All the materials and specimens preparation is conducted based on the standard code of practice design requirement of ASTM at Faculty of Civil Engineering, University Malaysia Sabah, Malaysia.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter discussed the background of the study and the literature review of the project. A literature study is essential for achieving an effective strategy since it aids in the identification of problems that happened in the present study. Aside from that, it aids in determining the optimal way to achieving the project's goal based on the research. This chapter concentrates on the application of ground SBEA with different periods and its comparison between ground SBEA added in the concrete and the control concrete in order to improve its strength by strength activity index, X-ray diffraction, and Chapelle test.

Concrete technology has evolved significantly over time. Several very notable developments include the invention of novel chemical pozzolanic materials to affect the characteristics of hardened and fresh concrete, as well as mineral additives or supplementary cementitious materials (SCMs) to enhance and assist Portland cement (Perraki, Kontori, Tsivilis, & Kakali, 2010; Al-Amoudi, Maslehuddin, Shameem, & Ibrahim, 2007). Variety forms of SCMs have various impacts on the characteristics of fresh and cured concrete. Currently, fly ash, granulated blast furnace slag, and silica fume, are the most often utilized SCMs. The ferrous-silica alloy production generates silica fume, whereas steel manufacturing generates granulated blast furnace slag. There are several raw materials of SCMs, like natural volcanic pozzolans and clays (metakaolin), that seems to be easily accessible throughout some areas such as Europe and the Mediterranean. According to Sabir, Wild and Bai, 2001, it is frequently used in certain of these nations (Sabir, Wild, & Bai, 2001).

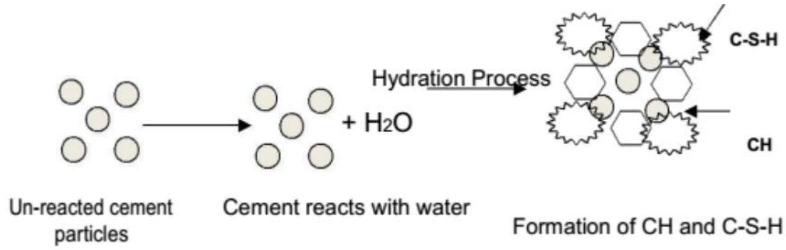

Figure 2.0 : Hydration Process of Ordinary Portland Cement

Source : Sutan, N.M. *et al*, 2018

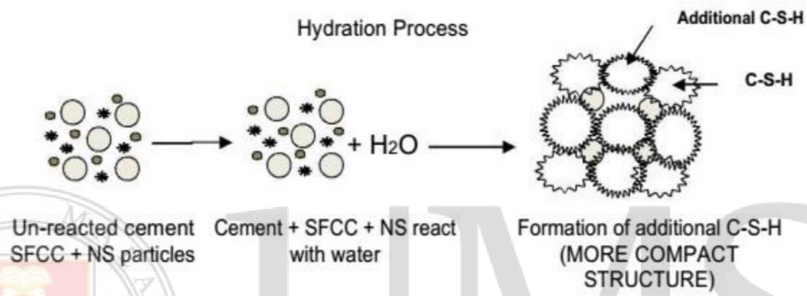

Figure 2.1 : Hydration Process of Partial Replacement of Cement by SFCC and NS

(Source : Sutan, N.M. *et al*, 2018)

There are few quantities of calcium hydroxide (CH) that stay in the cement structure after the hydration process. That residual CH is then substituted by reacting them with silica oxide to generate extra C-S-H, which contributes significantly to cement strength. This silica oxide may be found in the pozzolanic substance known as pozzolan. The chemical reaction that happens during the cement hydration process is characterised as follows:

$Chemcal\ equation\ Normal\ Cement\ Hydration\ Process$:

$$Cement\ (C_3S) + H_2O \rightarrow Ca^{2+} + H_2SiO_4{}^{2-} + OH^-$$

$$Ca^{2+} + H_2SiO_4{}^{2-} \rightarrow C - S - H$$

$$H_2SiO_4{}^{2-} + OH^- \rightarrow Ca(OH)_2\ (remaining\ CH)$$

Chemical Reaction in Pozzolan Replacement Cement Hydration Process:

$$SiO_2 + H_2O \rightarrow H_2SiO_4{}^{2-}$$

$$Ca(OH)_2\ (remaining\ CH) + H_2O \rightarrow Ca^{2+} + OH^-$$

$$H_2SiO_4{}^{2-} + Ca^{2+} \rightarrow C - S - H\ (additional)$$

2.2 Pozzolan

According to ACI Concrete Terminology, Pozzolan is a siliceous or silico-aluminous substance which reacts chemically with calcium hydroxide at normal temperatures to produce compounds with cementitious characteristics when it is finely split and present with moisture (there are both natural and artificial pozzolans).

2.2.1 Spent Bleaching Earth Ash, SBEA

SBEA has recently been utilized as a partial replacement of cement. In fact, previous research on SBEA as a pozzolanic material was limited. The pozzolan especially SBEA is used as a substituent for cement to help in reducing carbon dioxide, CO_2 emissions from the cement sector, as cement manufacturing accounts for 5% to 7% of worldwide CO_2 emissions (E. Benhelal *et al*., 2013). Raihana *et al*., (2020) strongly support the above statements after testing the mineralogical, chemical, physical, and microstructural characteristics of SBEA. In Set A, SBEA was introduced as a cement substitution in pavement formulation at 20 percent to 90 percent, whereas in Set B, the addition of Fly Ash (FA) and SBEA was employed as a part of the substituent of the mortars. From the findings, it showed that the highest amount of SBEA that could be added to the pavement mix was 20% with high compressive strength as shown in Figure 2.2. The researchers wrap up the article by concluding that SBEA from EcoOils, Lahad Datu exhibits pozzolanic capabilities due to the high content of SiO_2 and has the ability to replace cement in the concrete and mortar.

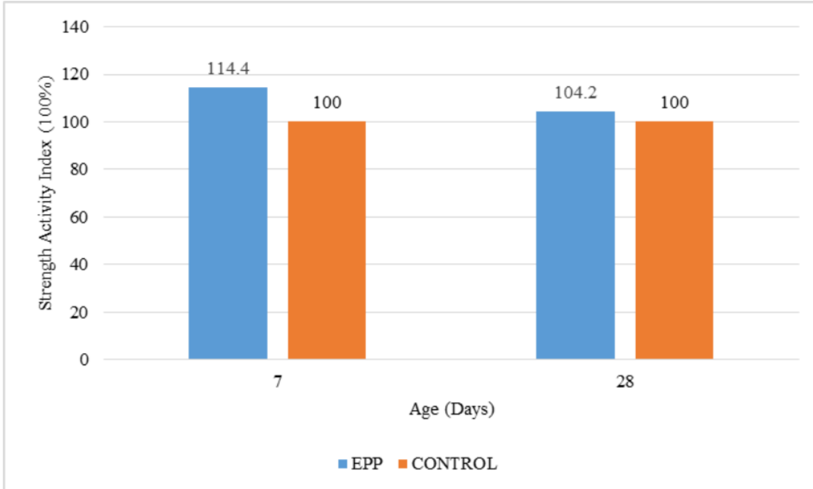

Figure 2.2 : Strength activity index, SAI at 7 and 28 days

Source : Raihana *et al.*, (2020)

The most significant challenges in the topic of long-lasting concrete constructions are sulfate and chloride resistance on cement. Therefore, Yunus *et al.* (2019) did research on the influence of SBEA on the strength characteristics of concrete subjected to chloride and sulfate surroundings. 10%, 20%, and 30% SBEA ash by concrete replaced the Ordinary Portland cement in this investigation, and a water-to-binder ratio of 0.45 was used in all concrete compositions. After demoulding, sample specimens were immersed in water for 28 days to cure. Next, the samples were immersed in 3.0% Sodium sulfate (Na2So4) solutions and 3.5% Sodium chloride (NaCl) for one, two, and four weeks. The quick impacts of chloride and sulfate on the hardened concrete were investigated in terms of compressive strength variation and weight change. When immersed in sulfate chloride and sodium chloride for 28 days, it was discovered that the replacement of cement by SBEA results in a stronger strength compared to the reference mix. The loss in weight of the SBEA is lower means that the SBEA has stronger resistance to chloride and sulfate. Figures 2.3 and 2.4 showed the result of the SBEA weight change against time. As a result, 10% SBEA has the greatest strength when compared to the other SBEA mixing ratio. According to this study, using 10% SBEA as SCM in concrete can minimize the harmful impacts of chloride and sulfate ions.