MODELLING SULPHATE RESISTANCE OF MORTAR CONTAINING ECO-PROCESSED POZZOLAN

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FACULTY OF ENGINEERING UNIVERSITI MALAYSIA SABAH
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ABSTRACT

Sulphate attack on concrete is the most common issue occur in the construction industry. The effect of the sulphate on the concrete for a short term was evaluated in terms of the expansion of mortar. However, the laboratory test of mortar expansion owing to sulphate is taking longer duration to complete. Therefore, the purpose of this research is to identify the optimum percentage of Eco-Processed Pozzolan (EPP) in mitigating the sulphate attack in concrete and to determine the expansion of EPP mortar when exposed to long-term sulphate exposure using Response Surface Methodology (RSM) from Design Expert 13 software. The models in RSM are developed according to the experimental design, where the RSM describe the relationship between the parameters which are testing duration, size of EPP, percentage of EPP and expansion of mortar. From the research, it can be identified that the addition of EPP as a partial replacement in concrete reduce the expansion of mortar compared to the control mix when exposed to sulphate environment for 56 days. In addition, the fineness of EPP influence the expansion mortar as partial replacement of ground EPP in mortar lower the expansion compare to unground EPP. Based on the optimization using RSM, the optimum percentage of EPP obtained is 40%. In addition, a cubic equation is developed from the Analysis of variance (ANOVA) where this equation is used to predict a long-term sulphate exposure in term of expansion of mortar for 91 days, 105 days, 120 days and 180 days. The expansion-predicted data obtained increased until 56 days and shrinkage of mortar occur up until 180 days except for 40% of ground EPP where the mortar expand on 105 days of testing and shrink after reach 120 days of testing. This might due to the error occur during the simulation process. This is due to the equations generate from the RSM was limit for only 56 days. Therefore, the prediction for a long-term expansion after 56 days was no relevant in this study. However, this research shows that using EPP as supplementary replacement in cement can give positive impact to the durability and strength of mortar. It also improves the sulphate resistance of mortar when exposed to sulphate environment. Hence, the concern on sulphate attack that commonly occur in construction industry can be reduce.

ABSTRAK

PEMODELAN MORTAR MENGANDUNGI EKO POZZOLAN YANG DIPROSES TERHADAP RINTANGAN SULFAT

Serangan sulfat pada konkrit adalah isu yang paling biasa berlaku dalam industri pembinaan. Kesan sulfat pada konkrit untuk jangka pendek dinilai dari segi pengembangan mortar. Walau bagaimanapun, ujian makmal sulfat mengambil tempoh yang lebih lama untuk disiapkan bagi mengkaji pengembangan mortar akibat daripada sulfat. Oleh itu, tujuan penyelidikan ini dilakukan untuk mengenal pasti peratusan optimum Proses Eko Pozzolan (EPP) dalam mengurangkan serangan sulfat dalam konkrit dan untuk meramal pengembangan mortar mengandungi EPP apabila terdedah kepada pendedahan sulfat bagi jangka masa yang panjang dengan menggunakan Metodologi Permukaan Tindak Balas (RSM), daripada perisian Design Expert 13. Model-model dalam RSM digunakan mengikut reka bentuk eksperimen, di mana RSM menerangkan hubungan antara parameter iaitu tempoh ujian, saiz EPP, peratusan EPP dan pengembangan mortar. Daripada penyelidikan, dapat dikenalpasti bahawa penambahan EPP sebagai penggantian separa dalam konkrit mengurangkan pengembangan mortar berbanding konkrit tanpa EPP apabila terdedah kepada persekitaran sulfat selama 56 hari. Di samping itu, kehalusan EPP mempengaruhi mortar pengembangan kerana penggantian separa menggunakan EPP yang telah dikisar dalam mortar mengurangka pengembangan mortar berbanding dengan EPP yang tidak dikisar. Berdasarkan pengoptimuman menggunakan RSM, peratusan optimum EPP yang diperolehi ialah 40%. Di samping itu, persamaan padu dihasilkan daripada Analisis varians (ANOVA) di mana persamaan ini digunakan untuk meramalkan pendedahan sulfat jangka panjang dari segi pengembangan mortar selama 91 hari, 105 hari, 120 hari dan 180 hari. Berdasarkan data yang diperoleh, data ramalan pengembangan yang diperoleh meningkat sehingga 56 hari dan pengecutan mortar berlaku sehingga 180 hari kecuali 40% EPP yang dikisar. 40% EPP yang dikisar ini mengembang pada 105 hari ujian dan mengecut selepas mencapai 120 hari ujian. Ini mungkin disebabkan oleh kesilapan yang berlaku semasa proses simulasi. Penyelidikan ini menunujukkan bahawa formula yang dihasilkan daripada RSM dihadkan untuk hanya 56 hari. Oleh itu, ramalan untuk jangka panjang selepas 56 hari tidak relevan dalam kajian ini. Walau bagaimanapun, kajian ini menunjukkan bahawa penggunaan EPP sebagai penggantian tambahan dalam simen boleh memberi impak positif kepada ketahanan dan kekuatan mortar. Perkara ini juga meningkatkan rintangan sulfat mortar apabila terdedah kepada persekitaran yang mengandungi sulfat. Oleh itu, kebimbangan terhadap serangan sulfat yang biasa berlaku dalam industri pembinaan dapat dikurangkan.

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LIST OF ABBREVIATIONS

EPP	-	Eco – Processed Pozzolan
RSM	-	Response Surface Methodology
ANOVA	-	Analysis of Variance
OPC	-	Ordinary Portland Cement
SCM	-	Supplementary Cementitious Materials
SBE	-	Spent Bleaching Earth
SBEA	-	Spent Bleaching Earth Ash
SBEO	-	spent Bleaching oil
GHG	-	Greenhouse Gas
SEM	-	Scanning Electron Microscope
SCC	-	Self-compacting concrete
DEF	-	Delayed Ettringite Formation
ESA	-	External sulphate attack
ISA	-	Internal sulphate attack
CSH	-	Calcium silicate hydrate
GBA	-	Coal bottom ash
RHA	-	Rice Husk Ash
DOE	-	Design of experiments
BBD	-	Box-Behnken Design
CCD	-	Central composite design
R2	-	Coefficient of determination
PRESS	-	Prediction error sum of squares
FFD	-	Full factorial 3-level design

CHAPTER 1

INTRODUCTION

1.1 Background of Research

The construction industry is one in every of the industries that contributes considerably to the growth and development of economy as well as the development of the country. Concrete is widely used in construction industry due to its property that can give strength and durability during the construction. Cement, water, sand, fine and coarse aggregates are the materials used to produce cement where all these materials are mix together meanwhile cement is made from a raw material, including limestone, clay, and sand. The process to produce cement requires a significant amount of heat, which it is usually generated from coal, natural gas, and hazardous waste. Cement is widely produced, and it released the emission of carbon dioxide during the process into the environment which known to be the biggest contributor to environmental degradation (Hamada *et al.*, 2018). According to Moetaz E. *et al.*, (2016), the annual production of Portland cement in the worldwide is estimated to be around 3 billion tonnes. When 1 tonne of cement is produced, around 0.9 tonne of carbon dioxide is released into the atmosphere.

Concrete is widely used nowadays to strengthen building structures and increase its durability. However, the deterioration of Ordinary Portland Cement (OPC) can occur rapidly when the concrete exposed to aggressive environment condition such as underground structures, marine structures, and wastewater treatment plant structures (Mwiti and Marangu, 2013). As a result, the concrete performance of the concrete structure may be influenced by its surroundings. In that circumstance, sulphate and chloride attacks are the most pressing concerns as it can affect concrete strength and durability (Golizadeh *et al.,* 2015).

Hence, to reduce environmental impact due to the large amount of production of cement which can affect the environment, pozzolan which is a siliceous and aluminous material has been proposed as a partial replacement in cement. Pozzolan define as a finely powdered pumice that reacts chemically with calcium hydroxide in the presence of moisture to generate cementitious silicate or aluminate compounds (Rathi and Modera, 2007). Natural pozzolan and human made pozzolan are two (2) different types of pozzolans. Silica fume, blast furnace slag, and fly ash are by-product of human-made pozzolans whereas natural pozzolans are based on natural minerals and volcanic deposits. The waste products contain pozzolan properties are used as a partial replacement in cement to reduce the usage of cement in industry. The emission of carbon dioxide in the atmosphere could be minimise when using the pozzolan materials as the production of carbon dioxide in cement contribute to 5%-7% (Benhelal E. *et al.*, 2013).

These pozzolan also known as Supplementary Cementitious Materials (SCM). SCM were incorporated into concrete construction to improve its strength and endurance for long-term concrete building (Osei and Jackson, 2012). SCM such as Fly Ash, Ground granulated blast furnace, and Palm oil fuel ash are the waste materials that usually used as a partial replacement in cement. These SCM have pozzolanic properties, mostly because they contain a large amount of silica, which plays an important function when incorporated into the OPC at a specific proportion. The usage of SCM in concrete improves the concrete's compressive strength and durability. The SCMs have the ability to reduce the permeability of concrete. As a result, there is a significant resistance to reinforcement corrosion, sulphate attack, and acid attack (Yunus, E. *et al.*, 2019).

According to Malaysian Palm Oil Council (2021), the world’s largest palm oil exporters is the industry of palm oil in Malaysia where Malaysia produced 25.8% of global palm oil production and 34.3% of global palm oil exports in 2020. It is reported that the Malaysia accounted for 9.1% and 19.7% of the world's total production and exports of oils and fats respectively in the same year. A solid waste known as Spent Bleaching Earth (SBE) in the palm oil industry usually disposed to landfills (Wangrakdiskul *et al.*, 2015). Approximately 2 million tonnes of spent bleaching earth (SBE) is produced annually by crude palm oil refining plants around the world (Beshara A. *et al.*, 2014) and Malaysia known as a producer of a large number of palm oil fruits. To make refined palm oil, bleaching earth is added to the pre-treatment process, which is in the degumming and bleaching of crude palm oil stage. The major purpose of bleaching earth is to remove colourants, soap, gums, metals, and oxidising chemicals during the oil refining process. This waste usually just dumped in landfill without any treatment and as the waste contain 20% to 40% residual oil by weight, metallic contaminants and organic compounds, SBE can create a fire which led to risk in air pollution. SBE is a by-product of the refining of crude palm oil (Raihana F. *et al.,* 2020). The SBE that is disposed into landfills can affect the greenhouse gas emission due to the natural aerobic degradation. Hence it is important to not dumped the SBE in landfills or public disposal sites without treatment to protect the environment.

Currently, Spent Bleaching Earth (SBEA) or Eco process pozzolan (EPP) is used as a partial replacement of cement in the making of concrete due to its cementitious properties. EPP is a recycled product from SBE that produced by a refinery company in Malaysia and also contribute to a better environment and economical. It is important to use the pozzolanic material as a partial cement substitution as cement deterioration always occur when exposed in aggressive environment such as when attack by a sulphate. Pozzolanic material helps to improve the quality of cement in terms of compressive strength, durability and sorpotivity. A few previous researchers had done a research paper regarding this problem. In this research, the sulphate resistance of mortar containing EPP was determined using the Response Surface Method (RSM) to achieve the objectives of this research.

1.2 Problem Statement

Despite pozzolanic materials improve the properties of concrete, the study and data on the durability of the EPP concrete is still limited. Research on the effect of EPP as a pozzolanic material for cement replacement was limited on the sulphate resistance of mortar. In addition, the laboratory test to determine the expansion of mortar due to sulphate took a longer time to complete. Hence, this study is conducted to determine the expansion of mortar when attack by sulphate for a long-term as it was time consuming for a laboratory work. Although there were a few technologies that can help to investigate the long-term expansion of mortar when expose to sulphate with different percentage of EPP, it is not enough to detect the response. Therefore, a better improvement in computerised modelling via data collecting such as Response Surface Methodology (RSM) using Design Expert 13 software might make the study easier.

1.3 Objectives of Research

The aim of this research is to determine the impact of Eco-Processed Pozzolan on the sulphate resistance of mortar. The specific objectives to achieve this research are:

1. To determine the expansion of EPP mortar when exposed to long-term sulphate exposure using Response Surface Methodology.
2. To identify the optimum percentage of EPP in mitigating the sulphate attack in concrete.

1.4 Scope of Work and Limitation

The primary data were obtained from the previous lab work data which consist of six (6) experiments data on mortar expansion. The parameters used were percentage of EPP, size of EPP which were ground and unground, testing duration, and expansion of mortar. Using these parameters, the mix proportion of EPP would be obtained using the RSM. There were six (6) experimental data from the lab work. All the percentage of EPP used in the study and testing duration would be input into the Design Expert 13 software for RSM model analysis to determine the optimum percentage of EPP as well as the predicted data for mortar expansion when exposed to sulphate. The equation under formula was created to predict the long-term expansion of mortar which was 180 days from the RSM method. The data would be processed using the RSM and compared to the predicted data to determine the similarities and relevance of the data.

1.5 Significance of Research

This study identifying the prediction of expansion of mortar for a long-term expansion when expose to the sulphate with different percentage of EPP. Furthermore, there is lack of research regarding modelling in research paper and this study conducting a simulation using Response Surface Method (RSM) by Design Expert 13 software to determine sulphate resistance of mortar containing EPP. Equation is developed from this model would help in prediction of long-term expansion of mortar due to sulphate. This generated formula also help to ease researcher to obtain the response variable of this research and it saved more time and faster compare to conducting laboratory work to achieve the main objective of this study. The effectiveness of EPP in term of expansion is predicted and the data then compared for accuracy checking.

1.6 Summary

Chapter 1 mainly discussed the introduction of this study for more understanding and as a guide for the researcher. In this chapter, the research background, the problems of the research, objectives, scope of work and significant of study were explain in order to achieve an outstanding result and outcome without facing any obstacles. This chapter focussing on the introduction of the pozzolana material, which is EPP, its importance to the construction industry, the problems occur related to the large amount of cement production and more. All these info was explained on why and how this study is conducted. The software exposure of the Response Surface Methodology will be discussed more detail on the next chapter, literature review.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Concrete is the most common material used in the construction industry. It required a mixture of materials which are cement, sand, water, and aggregates to produce concrete. Concrete provides strength, better durability, and versatility in the concrete structure and due to these properties, it is very suitable as a construction material. Portland cement is the cement that is widely used in the construction industry to build a building, bridges, and dams for instance. It is widely used in the industry due to its availability and cost-efficiency.

However, a concrete structure built with Ordinary Portland Cement (OPC) tends to damage faster when exposed to aggressive environmental conditions such as marine structures and underground structures. This shows that the surrounding environment of the structure can affect the property of concrete in terms of strength and durability. Sulphate and chloride attacks are the most common issues that affect the properties of concrete which led to deterioration.

Recently, pozzolanic materials are used as a partial replacement in concrete as it helps to improve the performance of concrete for more sustainable concrete construction. Therefore, a few researchers had done research regarding the usage of pozzolana materials such as fly ash, silica fume, and palm oil fuel ash. This study focuses on the Spent Bleaching Earth Ash (EPP) as pozzolanic material in cement replacement. Hence, Chapter two (2) will discuss in more detail the literature review on the effect on the sulphate resistance of the cement using the RSM.

2.2 Overview of Eco-Processed Pozzolan

Spent Bleaching Earth (SBE) is a waste product from the Palm Oil Refinery (POR) that contains a high amount of residual oil, typically between 20% and 40% (Aziz *et al.*, 2001). SBE has been burned for cement production, but because of the high oil content in SBE, it is not easy to maintain good cement quality (Park *et al.*, 2004). To keep the durability and stability of cement, the mixing and curing water should be free of crude oil spills, as this will impair the compressive strength of the materials. However, a study from Ting Q. C. (2017) showed that 30% of processed SBE outperformed foamed concrete exposed to chloride in terms of strength and durability. Meanwhile, a study by Chia K. (2010) stated that 60% SBE concrete has a lower outcome than 50% SBE concrete, concluding that 60% SBE concrete has a lower result than 50% SBE concrete.

Spent bleaching earth ash (SBEA) which is also known as Eco-process pozzolan (EPP) is a solid waste material that extracts from the waste product of refinery plants' crude palm oil degumming and bleaching processes. Figure 2.1 presents the production of EPP from SBE. Based on the figure, the bleaching earth was used to eliminate impurities colour, and odour of crude palm oil as well as other pollutants that are hazardous to people's health, during the degumming and bleaching processes of crude palm oil. This process yields refined, bleached, and deodorized (RBD) palm oil as well as spent bleaching earth (SBE), a waste product that is typically dumped in landfills and is categorised as solid waste. SBE absorbs some oil throughout the extraction and refinement process, and when the oil is recovered, SBE produces additional products including spent bleaching oil (SBEO), eco-mineral, and a sustainable product called EPP. EPP is mostly utilised as a cement substitute in the construction industry as it contains a high amount of silica and has pozzolanic properties (Kho J. H., 2021). Waste material issues can be avoided and can be reduced by replacing the cement with EPP as can be made at a lower cost.

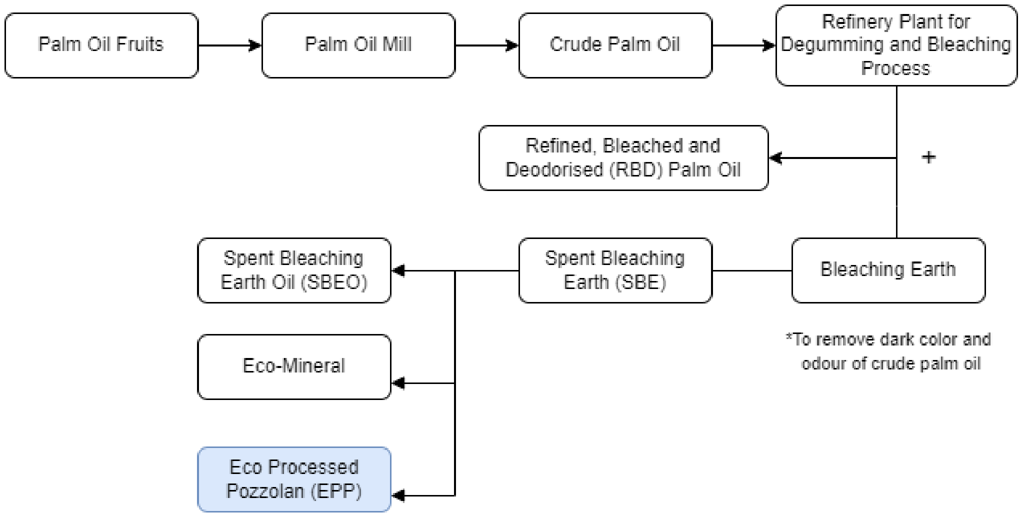

Figure 2.1 : Production of Eco-Processed Pozzolan
Source : Raihana F. *et al*., (2020)

2.2.1 Impact of The SBE Waste on The Environment

The disposal of SBE usually done by incineration, incorporation in animal feeds, land filling, or concrete manufacture. Currently, the most popular method of disposal in Malaysia is landfills, which creates a fire and pollution risk due to the degradation of residual oil and accompanying greenhouse gas (GHG) emissions during disposal. Meanwhile in Japan, SBE also disposed by incinerated for the manufacturer of cement but due to the high concentration of oil in SBE, it is hard to maintain the good quality of cement (Park *et al*., 2004). The increasing of the environmental regulatory restrictions causes the lack of new dump sites and the most significantly, the emission of GHG into the atmosphere which will affect the environment. Hence, incineration or landfill disposal is likely to become impractical in the future (Soh K. *et al*., 2013).

According to Loh S. K. *et al.,* (2006), bleaching earth is estimated being used globally 600,000 million tons and produced approximately 60 million tons of oil. 1 to 2 million tons of SBE that was produced globally contain 25-40% of oil and other contaminants was reported in 2014. This waste will be disposed in landfills most of the time and can give bad impact to the environment which can be hazardous, for instance the waste can be highly flammable (Kho J. H., 2021).

2.3 Chemical and Physical Properties of Eco-Processed Pozzolan

2.3.1 Physical Properties

Figure 2.2 and Figure 2.3 show the physical property of EPP and Scanning Electron Microscope (SEM) of EPP respectively. The original EPP was observed to have coarser and porous particles as indicated in Figure 2.1. The microstructure of EPP was further studied by utilizing SEM. The SEM image indicates that EPP has uneven, pointy, rough, and porous structure (Raihana F. *et al*., 2020).

Table 2.1 shows a mean particle size and specific gravity of EPP and OPC according to a study by (Raihana F. *et al.,* 2020). For particle size, d_{50} of EPP and OPC are 29.3 µm and 27.4 µm, respectively. Meanwhile for d_{90} of EPP and OPC are 1.93 µm and 3.27 µm, respectively. The specific gravity of EPP is lower compared to OPC based on the Table 2.1.

Figure 2.2 : Eco Process Pozzolan Ash
Source : Yunus, E. *et al*., (2020)