SULPHATE ATTACK RESISTANCE OF MORTAR CONTAINING SPENT BLEACHING EARTH ASH

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DECLARATION

I hereby declare that the material in this thesis is my own except for quotations, equations, summaries, and references, which have been duly acknowledged.

12 January 2023

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ABSTRACT

In this study, the spent bleaching earth ash (SBEA) was used as partial cement replacement to determine its effects on the sulphate resistance of mortar. Sulphate attack resistance of concrete containing SBEA has been studied previously and acknowledged to enhance the sulphate resistance of concrete. However, the knowledge on the effect of particle size and level of replacement of SBEA on sulphate attack resistance of mortar is still limited. The physical, chemical, mineralogical and microstructural properties of unground spent bleaching earth ash (USBEA) and ground spent bleaching earth ash (GSBEA) were characterized by using laser particle analysis, X-ray fluorescence (XRF), X-diffraction (XRD), and scanning electron microscope (SEM). The use of USBEA and GSBEA as cement replacement at 10, 20, 30, 40, and 50% were prepared for mortar mixture in this study. The pozzolanic activity of SBEA was evaluated through strength activity index (SAI) test at 7, 28, and 90 days. The effect of USBEA and GSBEA on the expansion of mortar bar after being immersed in 5% sodium sulphate solution was evaluated according to ASTM C1012 by measuring the length change of mortar bars at 1, 2, 3, 4, 8, 13, 15 weeks and subsequent length change was measured at 4 and 6 months. The grinding process of GSBEA resulted in smaller particle size (704 m^2/kg) and higher specific surface area than USBEA (390 m^2/kg). It was found that 40% of GSBEA showed the highest SAI with 135.16% and 136.17% at 28 and 90 days compared to other specimens. The expansion of mortar bar containing 40% GSBEA also showed the lowest expansion value (0.01%) at 6 months. Lower amount of calcium hydroxide (CH) was determined on mortar containing 40% GSBEA showing that it was consumed through pozzolanic reaction to form additional calcium silicate hydrate (CSH). Based on the SEM analysis, CSH was found in mortar containing 40% of GSBEA. This study also discovered that replacing cement with 40% of GSBEA reduced the expansion of mortar due to sulphate attack. Therefore, 40% of GSBEA as partial cement replacement has the potential to improve the sulphate resistance of mortar.

ABSTRAK

KETAHANAN SERANGAN SULFAT MORTAR YANG MENGANDUNGI SPENT BLEACHING EARTH ASH

Dalam kajian ini, spent bleaching earth ash (SBEA) digunakan sebagai penggantian separa simen untuk menentukan kesannya terhadap rintangan sulfat mortar. Rintangan serangan sulfat konkrit yang mengandungi SBEA telah dikaji sebelum ini dan diakui boleh meningkatkan rintangan sulfat konkrit. Walau bagaimanapun, pengetahuan tentang kesan saiz zarah dan tahap penggantian SBEA terhadap rintangan serangan sulfat mortar masih terhad. Sifat fizikal, kimia, mineralogi dan mikrostruktur unground spent bleaching earth ash (USBEA) dan ground spent bleaching earth ash (GSBEA) telah dicirikan dengan menggunakan analisis zarah laser, pendarfluor sinar-X (XRF), pembelauan-X (XRD), dan pengimbasan mikroskop elektron (SEM). Penggunaan USBEA dan GSBEA sebagai pengganti simen pada 10, 20, 30, 40, dan 50% telah disediakan untuk campuran mortar dalam kajian ini. Aktiviti pozzolanik SBEA dinilai melalui ujian indeks aktiviti kekuatan (IAK) pada 7, 28, dan 90 hari. Kesan USBEA dan GSBEA pada pengembangan bar mortar selepas direndam dalam larutan natrium sulfat 5% dinilai mengikut ASTM C1012 dengan mengukur perubahan panjang bar mortar pada 1, 2, 3, 4, 8, 13, 15 minggu dan perubahan panjang seterusnya diukur pada 4 dan 6 bulan. Proses pengisaran GSBEA menghasilkan saiz zarah yang lebih kecil (704 m^2/kg) dan luas permukaan spesifik yang lebih tinggi daripada USBEA (390 m^2/kg). Didapati 40% GSBEA menunjukkan IAK tertinggi iaitu 135.16% dan 136.17% pada 28 dan 90 hari berbanding spesimen lain. Pengembangan bar mortar yang mengandungi 40% GSBEA juga menunjukkan nilai pengembangan yang paling rendah (0.01%) pada 6 bulan. Jumlah kalsium hidroksida (CH) yang lebih rendah ditentukan pada mortar yang mengandungi 40% GSBEA menunjukkan bahawa ia telah digunakan melalui tindak balas pozzolanik untuk membentuk kalsium silikat hidrat (CSH) tambahan. Berdasarkan analisis SEM, CSH didapati dalam mortar yang mengandungi 40% GSBEA. Kajian ini juga mendapati bahawa menggantikan simen dengan 40% GSBEA mengurangkan pengembangan mortar akibat serangan sulfat. Oleh itu, 40% GSBEA sebagai penggantian simen separa berpotensi untuk meningkatkan rintangan sulfat mortar.

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LIST OF SYMBOLS

%	-	Percentage
°C	-	Degree Celcius
θ	-	Theta
g	-	Gram
L	-	Liter
ml	-	Mililiter
m/kg^2	-	Meter per square kilogram
mm	-	Milimeter
min	-	Minute
μm	-	Micrometer
MPa	-	Megapascal

LIST OF ABBREVIATIONS

Al_2O_3	-	Aluminium oxide
ASTM	-	American Society for Testing and Materials
BS	-	British Standard
C	-	Calcite
Ca	-	Calcium
CaO	-	Calcium oxide
CH	-	Calcium hydroxide
C_2S	-	Dicalcium silicate
C_3A	-	Tricalcium aluminate
C_4AF	-	Tetracalcium aluminoferrite
CSH	-	Calcium silicate hydrate
Et. al	-	And others
Fe_2O_3	-	Iron oxide
LOI	-	Loss on ignition
OPC	-	Ordinary Portland Cement
Q	-	Quartz
SAI	-	Strength activity index
SBEA	-	Spent bleaching earth ash
SDF	-	Strength deterioration factor
SEM	-	Scanning electron microscope
SiO_2	-	Silicon oxide
SO_3	-	Sulfur trioxide
TGA	-	Thermogravimetric analyzer

UMS	-	Universiti Malaysia Sabah
UTM	-	Universiti Teknologi Malaysia
XRD	-	X-ray diffraction
XRF	-	X-ray Fluorescence

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CHAPTER 1

INTRODUCTION

1.1 Overview

Over the past decades, the reuse of waste products from the palm oil industry has been applied to achieve a more sustainable environment. These waste products are usually sent to landfills and cause environmental problems due to the degradation of substances contained in the wastes. The palm oil industry generates wastes that cause major disposal problems. Malaysia is known as one of the largest producers of palm oil among several countries, such as Indonesia and Thailand, throughout the world (Kamil & Omar, 2016).

In a palm oil refinery, bleaching earth is used to remove the dark colour and other impurities of crude palm oil. Bleaching earth is a fine clay powder that mainly consists of silicon oxide (Loh et al., 2013; E. Y. Park et al., 2004). The estimated amount of bleaching earth used in refinery plants is around 600,000 metric tonnes or more based on the production of more than 60 million tonnes of palm oil worldwide (Kaimal et al., 2002; Loh et al., 2013). After the phase of bleaching and degumming of crude palm oil, the residual waste, which is also called spent bleaching earth (SBE), is generated (Loh et al., 2013). In that process, bleaching earth absorbs just about 0.5% by weight of oil (Kheang et al., 2007). Dumping of SBE waste at landfills could lead to fire and pollution hazards (Loh et al., 2013). It might be due to the degradation of the residual oil as well as metallic contaminants and organic compounds in the SBE waste (O. Rokiah et al., 2019).

Over 2 million tonnes per year of SBE is generated by the palm oil industry globally (Beshara & Cheeseman, 2014). In Malaysia, approximately 240 000 tonnes of SBE have been generated annually from the palm oil refinery plant (Loh et al., 2017; O. Rokiah et al., 2019). It is challenging to manage SBE waste due to its large volume generated from the refinery plant. Due to the insufficient solutions to reuse or recycle for beneficial use, SBE is usually disposed of at landfills improperly and without treatment. Disposing of by-products from the industry at landfills may also cause environmental problems such as greenhouse effects (Loh et al., 2013). Besides that, it requires a high cost to dump SBE into landfills (Kheang et al., 2007). Therefore, SBE should be reused and recycled in the industry for better waste management.

Despite being discarded in landfills, SBE is extracted to produce residual oil and de-oiled SBE. Several researchers have used the residual oil to develop solutions for its disposal problem by recycling SBE into useful material, such as the application of SBE to produce biodiesel (Kheang, Loh Soh et al., 2010; Kheang et al., 2007) and non-food application such as soap (Daous & Al-Zahrani, 2000; Kheang, Loh Soh et al., 2010). Apart from the recovery of residual oil, de-oiled SBE is also the product of the extraction process of SBE. De-oiled SBE is obtained after the oil from the SBE wastes is recovered. It has been used in clay blocks (Beshara & Cheeseman, 2014; Eliche-Quesada & Corpas-Iglesias, 2014). Furthermore, de-oiled spent bleaching earth (SBE) is calcined at high temperatures to produce a spent bleaching earth ash (SBEA), which is also referred to as eco-processed pozzolan in various studies (Kho, 2021; Kusaimi et al., 2020; Yunus et al., 2019). It has also been mentioned as a processed SBE by Othman et al. (2019b) and Wei Chong et al. (2021). SBEA has been utilized as a partial replacement for cement in a mortar (Wei Chong et al., 2021) and concrete in studies conducted by Kho (2021), Othman et al. (2020), and Yunus et al. (2020).

Based on the previous studies, it appears that using SBEA as a partial cement replacement in concrete can effectively improve its resistance against sulphate attack (Othman et al., 2020; Yunus et al., 2020), chloride attack (Yunus et al., 2020), and acid attack (Othman et al., 2019a). Sulphate attack is a common cause of concrete