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PERFORMANCE OF MORTAR CONTAINING GROUND SPENT BLEACHING EARTH ASH AGAINST SULFATE ATTACK

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ABSTRACT: Recently, spent bleaching earth ash (SBEA) which is extracted and calcined from spent bleaching earth waste from a palm oil refinery plant, has been used as partial cement replacement. In this study, the SBEA was ground into smaller particle sizes through a grinding process. The effect of unground spent bleaching earth ash (USBEA) and ground-spent bleaching earth ash (GSBEA) on the sulfate attack resistance of mortar was investigated in this study. The characterization of USBEA and GSBEA was determined in terms of particle size, specific surface area, chemical compositions, and microstructural properties. Their effects on pozzolanic activity were determined in terms of the strength activity index. Then, the expansion of the mortar bar containing 40% of USBEA and GSBEA due to sulfate attack was determined to investigate the sulfate attack resistance of mortar containing USBEA and GSBEA. The microstructural changes of mortar against sulfate attack were determined by using x-ray diffraction, scanning electron microscopy and thermogravimetric analysis. Results indicated that the strength activity index (SAI) value of mortar containing 40% of GSBEA was the highest among the specimens. Furthermore, the expansion of mortar bars containing 40% of GSBEA was the lowest, with 0.01% at 6 months. The microstructural results of mortar also confirmed that the incorporation of 40% of GSBEA with the compact microstructure with calcium silicate hydrate (CSH) and less ettringite was found compared to other specimens. This shows that the inclusion of GSBEA enhances the sulfate attack resistance of mortar.

Keywords: Spent bleaching earth ash, Sulfate attack resistance, Pozzolanic activity, Cement replacement

1. INTRODUCTION

A sulfate attack can cause the deterioration of mortar and concrete. Sulfate ions are often found in soil, groundwater, seawater and industrial mining plants. The occurrence of sulfate attacks can be divided into internal and external sulfate attacks [1]. Internal sulfate attack can occur due to the existence of sulfate ions in the materials during mixing. On the other hand, in an external sulfate attack, sulfate ions penetrate into the cement paste to react with the hydration products when the concrete is exposed to a sulfate environment. A sulfate attack can occur when sulfate ions react with the components of hydration of cement, such as calcium aluminate hydrate (C_3A) and calcium hydroxide (CH), to form ettringite and gypsum [2], which lead to expansion, cracking and spalling of concrete. It is known that the sulfate attack reaction could be mitigated when the main hydration product of cement calcium hydroxide and calcium aluminate hydrate could be reduced [2].

Recently, many studies have been done on the effect of using pozzolans in sulfate attacks of mortar and concrete. Past studies reported that the use of pozzolans as cement replacement enhances the performance of mortar and concrete against sulfate attack by reducing the C_3A content in cement and the CH content through pozzolanic reaction [2–4].

The pozzolanic reaction of pozzolans can be attributed to the impermeability of concrete due to pore refinement [3, 5]. This would prevent the ingress of sulfate solution into mortar and concrete. The resistance of mortar and concrete against sulfate attack can be enhanced by a high cement replacement level of pozzolan [5] and finer particle size of pozzolan [6]. The finer particle of pozzolan with a high specific surface area provides a larger surface for the reaction of silica with CH to produce additional calcium silicate hydrate [7] and can act as a filler to enhance the pore refinement by filling the voids [8]. Therefore, finer pozzolan is recommended to be used as a partial cement replacement to improve the sulfate resistance of mortar and concrete.

Spent bleaching earth ash (SBEA) has been used as partial cement replacement in mortar and concrete [9-13]. The effect of SBEA on the sulfate resistance of mortar and concrete has been evaluated in terms of expansion [14], loss in compressive strength [14, 15], and microstructural changes in deteriorated mortar and concrete [14]. The inclusion of SBEA enhances the sulfate resistance of concrete [15]. However, the effect of the particle size of SBEA on sulfate attack resistance has not been explored yet. The aim of this paper is to investigate the effect of unground spent bleaching earth ash (USBEA) and ground spent bleaching earth ash (GSBEA) on the sulfate attack resistance of mortar in terms of expansion of mortar bar and the microstructural changes after being immersed in 5% sodium sulfate solution for 6 months.

2. RESEARCH SIGNIFICANCE

The use of SBEA as cement replacement on sulfate resistance of concrete has been investigated in terms of loss in compressive strength. However, the effect of SBEA, when ground into finer particle size and in high replacement level, on the sulfate resistance of mortar has not been explored yet. Therefore, this study focuses on the usage of unground and ground SBEA as partial cement replacement at higher replacement levels in order to provide a new understanding regarding the role of particle size and level of SBEA replacement in improving the sulfate attack resistance of mortar.

3. MATERIALS AND METHOD

3.1 Preparation of Materials and Samples

Ordinary Portland cement (OPC) was used in this study. The unground spent bleaching earth (USBEA) was supplied from Lahad Datu, Sabah. The ground spent bleaching earth ash (GSBEA) was prepared by grinding the USBEA in the planetary grinding ball mill for 30 minutes. The physical properties of materials are shown in Table 1. The particle size of GSBEA (18.6μm) was smaller than OPC (27.4μm) and USBEA (29.3μm) while the specific surface area of GSBEA (704 m^2/kg) was larger than USBEA (390 m^2/kg) and OPC (635 m^2/kg).

Table 1 Physical properties of materials

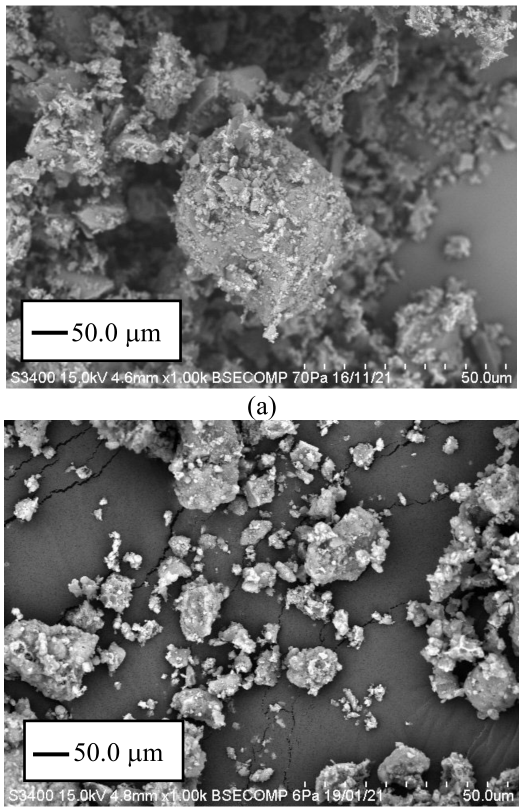
Physical Properties	Median particle size, d_{50} (μm)	Specific surface area (m^2/kg)	Specific gravity
OPC	27.4	635	3.27
USBEA	29.3	390	1.93
GSBEA	18.6	704	1.9

Table 2 shows the chemical properties of materials. The major component of USBEA and GSBEA was silica. The total sum of SiO_2, Al_2O_3, and Fe2O3 of USBEA and GSBEA was more than 50%. Thus, USBEA and GSBEA can be considered as Class C pozzolan as specified in ASTM C618. The value was different from SBEA in previous studies, in which the total sum of SiO_2, Al_2O_3, and Fe2O3 was similar to [11] but lower than [10], [13]. It differs depending on SBEA production.

The micrograph images of USBEA and GSBEA are shown in Fig.1. The microstructure of particles before and after grinding was obviously different. It shows that USBEA contains irregularly shaped particles and large spherical-shaped particles and consists of pores and agglomerates. Meanwhile, GSBEA contained mostly finer, irregularly shaped, spherical shaped particles in crushed form with fewer pores compared to particles in USBEA.

Table 2 Chemical properties of materials

Chemical Properties (%)	OPC	USBEA	GSBEA
SiO_2	14.4	47.57	48.43
Al_2O_3	3.6	11.64	11.01
Fe_2O_3	3.2	9.77	10.03
CaO	72.3	12.46	12.68
SO_3	3.66	2.13	1.76
$SiO_2 + Al_2O_3 + Fe_2O_3$	-	68.98	69.47
Loss on ignition (LOI)	5.78	3.3	3.2



(a)

(b)

Fig.1 Micrograph images of: (a) USBEA and (b) GSBEA

The mix proportion for the strength activity index test is shown in Table 3. The binder-to-sand ratio for all mixtures was 1:2.75. The water-to-cement ratio for the mortar was 0.485 for the control mortar, while for mortar containing USBEA and GSBEA, the water was adjusted to obtain ± 5 control flow. The flow and water requirement of each sample is shown in Table 4. The water requirement increased with the inclusion of USBEA. However, the water requirement of mortar with GSBEA was lower than USBEA. This might be due to the lower pores in GSBEA than in USBEA, as shown in Fig. 1, due to the grinding process. Pozzolan, with fewer pores, will have less water demand. This finding is in good agreement with previous studies [16], [17].

The sulfate attack resistance of mortar containing USBEA and GSBEA was investigated by using an expansion test as specified in ASTM C1012 [18]. For each mixture, six mortar bars with dimensions 25×25×285 mm and nine mortar cube specimens with dimensions 50×50×50 mm were prepared. The mix proportion for the expansion test was as stated in Table 3. For 1L of distilled water, 50 g of sodium sulfate technical grade was used to make a 5% sodium sulfate solution. The solution was made 24 hours before the mortar bars were immersed in a sodium solution, and the pH was kept relatively constant at 6 to 8.

Table 3 Mix the proportion of mortar specimen for strength activity index and expansion test

Sample	Binder (%)		
	OPC	USBEA	GSBEA
Control	100	0	0
USBEA20	80	20	0
USBEA40	60	40	0
GSBEA20	80	0	20
GSBEA40	60	0	40

Table 4 Flow and water requirement

Sample	Flow	Water requirement (%)
Control	97	100
USBEA20	101	117
USBEA40	99	127
GSBEA20	98	104
GSBEA40	98	104

3.2 Testing Procedures

3.2.1 Strength activity index

The strength activity index (SAI) of control mortar (reference mortar), and mortar containing 20% and 40% USBEA and GSBEA were determined according to ASTM C311 [19]. The SAI was calculated by using Eq. (1).

$$\mathbf{SAI}\ (\%) = \left(\frac{\boldsymbol{A}}{\boldsymbol{B}}\right) \times \mathbf{100\%} \tag{1}$$

Eq. (1) is the formula to determine the SAI, where A is the average compressive strength of the test mortar and B is the average compressive strength of the control mortar.

3.2.2 expansion of mortar bar

Mortar bars containing 20% and 40% of USBEA and GSBEA as partial cement replacement were tested for expansion test due to external sulfate attack by measuring the length change of mortar bar according to ASTM C1012. The mortar bar was placed in saturated lime water until the compressive strength of the cube mortar sample reached 20 MPa. Then, the mortar bar was placed in a 5% sodium sulfate solution for 6 months. The length change of the mortar bar was measured at 1, 2, 3, 4, 8, 13, and 15 weeks and subsequent length change was measured at 4 and 6 months by using a length comparator.

3.2.3 Microstructural changes

The microstructural changes of selected samples with high level of replacement (40%) of USBEA and GSBEA were determined by using x-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA) after the sample was immersed in 5% sodium sulfate solution for 6 months. The testing was done to determine the hydrated products formed due to sulfate attack. For XRD and TGA, the mortar sample was immersed in isopropyl alcohol and dried in a vacuum desiccator until it reached the constant weight to stop the hydration. Then, it was crushed, ground and sieved until the powdered mortar achieved a 45μm particle size. For SEM, the mortar sample was cut into smaller pieces with a diameter of approximately 1 cm. This step was followed by immersing them in isopropyl alcohol prior to testing.

4. RESULTS AND DISCUSSIONS

4.1 Pozzolanic Activity

The strength activity indices (SAI) of mortar specimens at 7, 28, and 90 days are shown in Fig.2. The SAI values of mortar containing USBEA and GSBEA increased as the curing age increased. The SAI values of mortar containing GSBEA were higher than mortar containing USBEA. At 28 days, the SAI values of control, USBEA20, USBEA40, GSBEA20, and GSBEA40 were 100%, 88.87%, 101.2%, 109.77%, and 135.16%, respectively. Meanwhile, the SAI values of control, USBEA20, USBEA40, GSBEA20, and GSBEA40 at 90 days were 100%, 96.98%, 107.96%, 127.48%, and 136.17%, respectively. All specimens obtained SAI values more than 75%, which was the minimum limit of SAI as specified in ASTM C618. This shows that GSBEA40 obtained the highest pozzolanic reaction. It might be due to the finer particle size of GSBEA. It is obvious that the finer particle size of GSBEA enhanced the pozzolanic reaction rate. It might be because finer-sized particles have higher specific surface areas, which allow the silica to react with calcium hydroxide (CH) to produce calcium silicate hydrate (CSH), which makes the mortar denser, thus increasing its pozzolanic reaction. Jaturapitakkul et al. [20] also agreed that grinding enhanced the reactivity of pozzolans.

4.2 Expansion of Mortar Bar

Fig. 3 shows the expansion of the control mortar and mortar bar containing 20% and 40% of USBEA and GSBEA as partial cement replacement after being immersed in 5% sodium sulfate solution for 6 months, as specified in ASTM C1012. According to the ASTM C618, the maximum expansion value of mortar bars exposed to a moderate sulfate environment and high sulfate environment after 6 months were 0.05% and 0.1%, respectively. Based on the result, for the control specimen, an obvious increment of expansion value was observed up to 6 months of immersion. For USBEA20 and GSBEA20, the expansion of the mortar bar gradually increased up until 6 months of immersion. Meanwhile, the expansion patterns of USBEA40 and GSBEA40 increased linearly up until 4 months and then increased gradually until 6 months. At the end of immersion (6 months), the expansion values of control, USBEA20, USBEA40, GSBEA20, and GSBEA40 were 0.140%, 0.027%, 0.014%, 0.018% and 0.010%, respectively. The expansion of the control specimen was more than 0.1% at 6 months. While for USBEA20, USBEA40, GSBEA20 and GSBEA40, the expansion values were less than 0.05% at 6 months. Thus, all of the specimens can be considered as high sulfate resistant as specified in ASTM C618 [21]. The expansion of GSBEA40 (0.01%) was the lowest among the specimens. This might be due to the smaller particle size of GSBEA with a high specific area, which accelerated the pozzolanic reaction rate to produce CSH. Furthermore, the smaller-sized particles can act as fillers to fill the pores, which prevents the ingression of sulfate solution. These findings are in good agreement with previous studies, which found that ground pozzolans reduced the expansion of mortar bars [8].

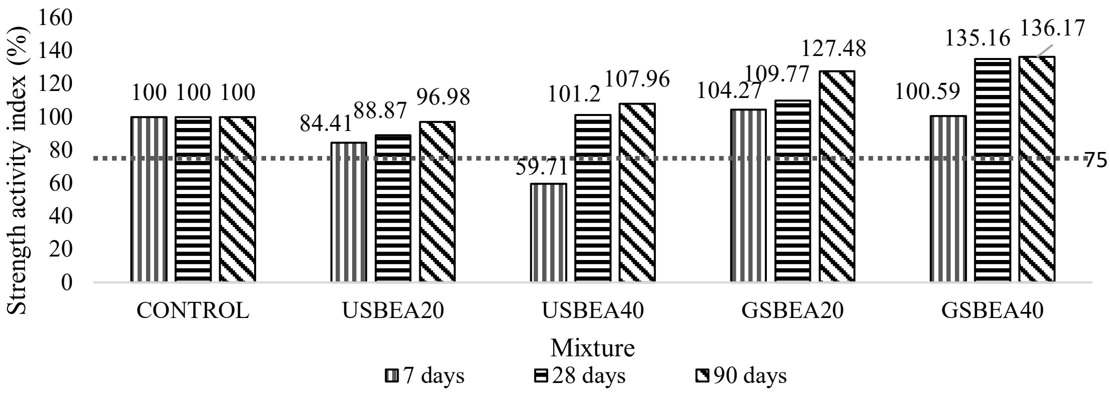


Fig.2 Strength activity index

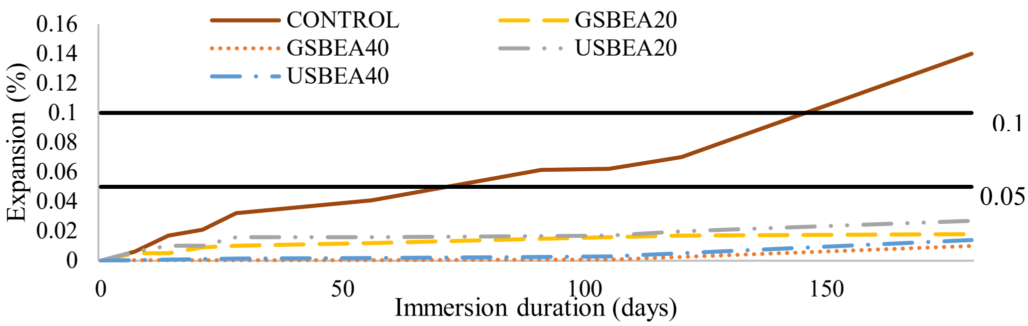


Fig. 3 Expansion of mortar bar

4.3 Microstructural Changes

The micrograph images of control, USBEA40 and GSBEA40 specimens after being immersed in 5% sodium sulfate solution for 6 months are shown in Fig. 4. The formation of needle-like forms of ettringite and elongated rode shapes of gypsum were observed on micrograph image of control and USBEA40 specimens. Meanwhile, crowded tiny cotton shapes of calcium silicate hydrate (CSH) and only a few amounts of gypsum were found on the GSBEA40 specimen. The microstructure of GSBEA40 was more compact than the control and USBEA specimen.

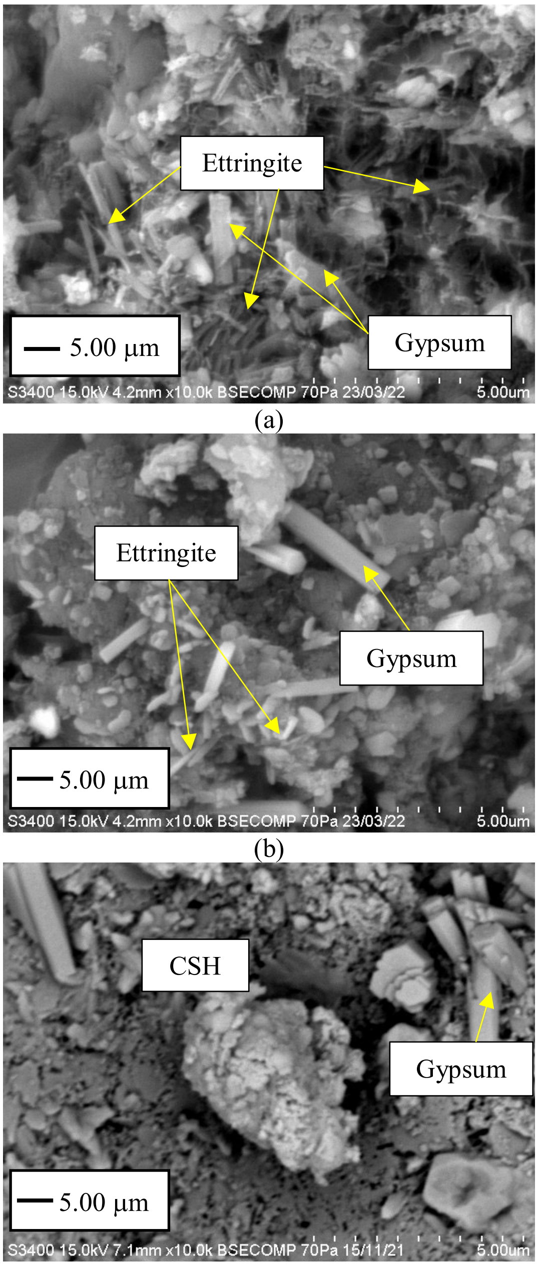


Fig. 4 Micrograph images of: (a) Control, (b) USBEA40 and (c) GSBEA40

From the XRD results in Fig. 5, the ettringite, gypsum, quartz, and calcite intensities were labeled as E, G, Q, and C. The minerals' peaks based on 2-theta values for the control, USBEA40 and GSBEA40 are shown in Table 5. For control specimen, gypsum (20.83°) and ettringite (9.1°, 15.85°, 18.1°, 18.87°, 22.97°, 24.26°, 25.6°, 26.62°, 29.34°, 31.12°, 33.27°, 33.39°, 34.39°, and 36°) were detected at 6 months of immersion in 5% sodium sulfate solution. This finding supported the microstructural observation via SEM, where the formation of gypsum and ettringite might be the reason for the highest expansion value (0.14%) of the control specimen. The ettringite was found in USBEA40 at 9.07°, 15.77°, 17.78°, 18.84°, 22.05°, 22.89°, 25.59°, 26.59°, 29.44°, 32.26°, 34.91°, 35.86°, and 39.44°. The presence of ettringite and gypsum might be attributed to the reaction between sulfate ions with C_3A and calcium hydroxide (CH). Usually, the presence of quartz was detected due to the presence of fine aggregate, which was not removed completely by the sieving process. For specimens containing USBEA and GSBEA, the quartz detected might be due to the presence of a high amount of silica in USBEA and GSBEA. Calcite (C) was present due to a little carbonation reaction on the surface of the specimens. Carbonation must have happened during the preparation for testing due to the small portions of samples. The peak of ettringite and gypsum were not detected in GSBEA40. This might be due to the pozzolanic reaction of finer-sized particles of GSBEA, which led to the reduction of CH. This reduced the reaction of sulfate ions with CH to form ettringite.

Fig. 6 shows the TGA curves of all specimens after 6 months of immersion in 5% sodium sulfate solution. The mass loss at 450°C to 550°C can be attributed to the dehydroxylation of calcium hydroxide (CH). It is well known that the presence of CH negatively affects the expansion caused by the sulfate attack. The mass loss of CH is shown in Fig. 7. The mass of CH of GSBEA40 was the lowest at 0.95%. The consumption of CH is usually due to the pozzolanic reaction and sulfate attack. This statement is generally in agreement with the previous study of Elahi et al. [22]. However, since there was no ettringite and less gypsum found on GSBEA40 based on XRD and SEM results, therefore, the CH consumption might be due to the pozzolanic reaction to produce CSH. The lower amount of CH and increased production of CSH, along with the filler effect of finer particle size of GSBEA, might increase the resistance of mortar against sulfate attack. This finding was supported by the lowest expansion value obtained by GSBEA40 in Section 4.2.

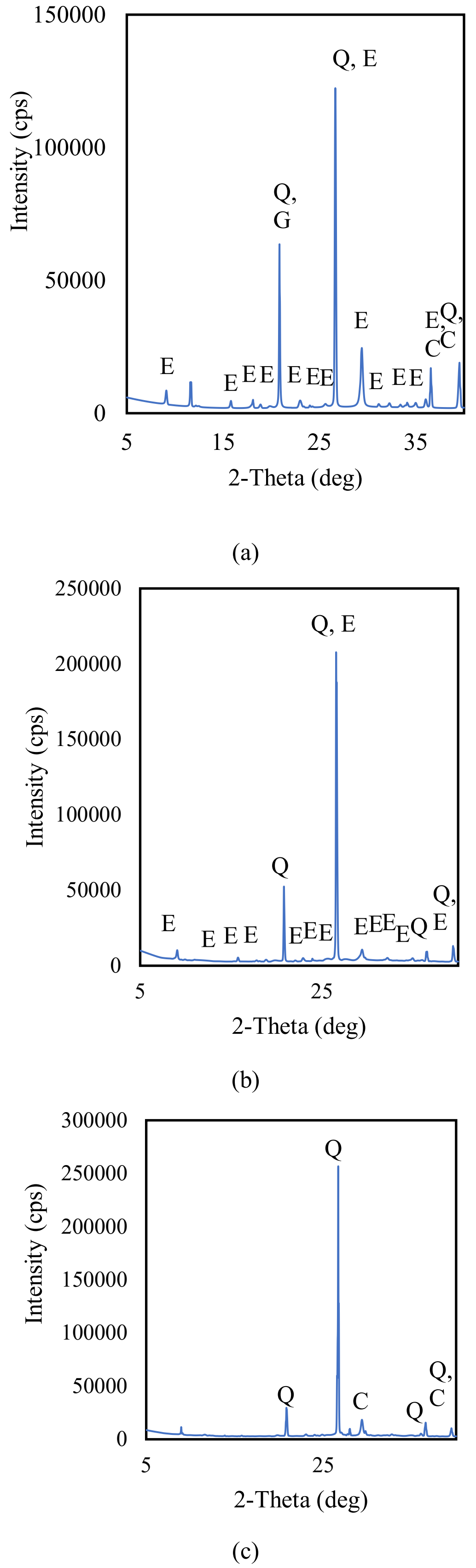


Fig. 5 XRD patterns of: (a) Control, (b) USBEA40 and (c) GSBEA40

Table 5 Mineral peaks based on 2-theta values

Sample	Minerals	2-Theta
Control	Ettringite	9.1°, 15.85°, 18.1°, 18.87°, 22.97°, 24.26°, 25.6°, 26.62°, 29.34°, 31.12°, 33.27°, 33.39°, 34.39°, 36°
	Gypsum	20.83°
USBEA40	Ettringite	9.07°, 15.77°, 17.78°, 18.84°, 22.05°, 22.89°, 25.59°, 26.59°, 29.44°, 32.26°, 34.91°, 35.86°, 39.44°
GSBEA40	No ettringite and gypsum were detected.	-

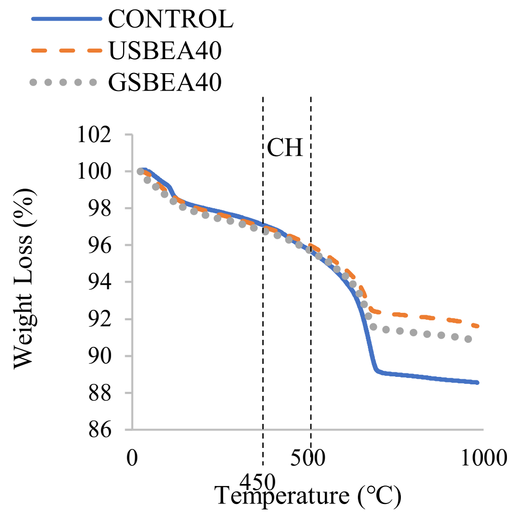


Fig. 6 TGA result

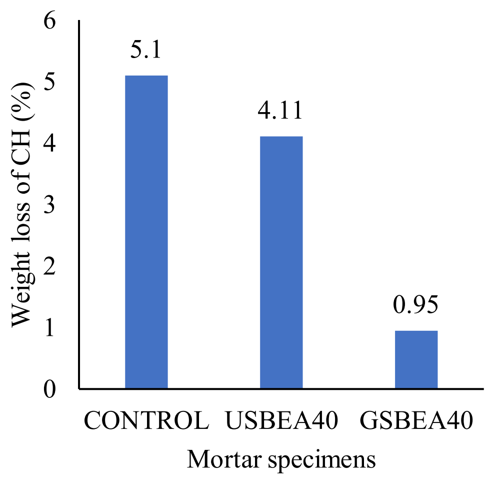


Fig. 7 Weight loss of CH

5. CONCLUSIONS

Based on the results, the following conclusions can be drawn:

1. Both USBEA and GSBEA can be used as partial cement replacements due to their pozzolanic properties. The pozzolanic reactivity of mortar containing GSBEA was better than mortar containing USBEA. Mortar containing 40% GSBEA achieved the highest value of strength activity index with 135.16% and 136.17% at 28 and 90 days, respectively.
2. The expansion of mortar bars containing 40% of GSBEA was the lowest, with 0.01%. The finer particle size of SBEA enhanced the resistance of mortar against sulfate attack.
3. The substitution of GSBEA as partial cement replacement at 40% improved the resistance to sulfate attack by reducing calcium hydroxide content through a pozzolanic reaction to produce more additional calcium silicate hydrate. Thus, little or no formation of gypsum and ettringite was found in the mortar containing 40% of GSBEA against sulfate attack. It is concluded that GSBEA has the potential to be used as a partial cement replacement due to its ability to resist sulfate attack.

6. ACKNOWLEDGMENTS

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