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STRENGTH ACTIVITY INDEX AND PROPERTIES OF SPENT BLEACHING EARTH ASH

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ABSTRACT: Spent bleaching earth ash (SBEA) is a recycled product from spent bleaching earth (SBE) waste. Despite the disposal of SBE waste at landfills which can cause pollution, the waste can be processed to produce SBEA which is currently used in the construction industry. The strength activity index of ground spent bleaching earth ash (GSBEA) has been investigated in this study. The chemical, mineralogical, physical, and microstructural properties of GSBEA have been determined. The GSBEA was used at 10, 20, 30, 40, and 50% as partial cement replacement. The strength activity index (SAI) of GSBEA was determined according to ASTM C311 at 7, 28, and 90 days. SA scanning electron microscope equipped with energy dispersive x-ray spectroscopy (SEM/EDX) was used to determine the effect of GSBEA on the microstructural properties of mortar paste at 90 days. The GSBEA can be used up to 40% due to its better performance in SAI values compared to other mortars which were 100.58%, 135.17%, and 136.17% at 7, 2,8 and 90 days respectively. This finding is supported ban a SEM/EDX micrograph image of a mortar containing 40% of GSBEA, which revealed the calcium silicate hydrate formation after 90 days of curing.

Keywords: Spent bleaching earth ash, Pozzolan, Strength activity index, Cement replacement

1. INTRODUCTION

Spent bleaching earth ash (SBEA) is a by-product of the calcination process of the de-oiled spent bleaching earth that is derived from the degumming and bleaching process of palm oil at the refinery plant. The de-oiled spent bleaching earth is the product from the extraction process after the oil is recovered. More than 2 million tonnes of spent bleaching earth is generated per year globally due to huge palm oil production [1]. The spent bleaching earth waste is usually dumped at landfills which can cause environmental pollution. Hence, the waste is extracted to increase its beneficial use. Recently, SBEA was used as partial cement replacement in mortar [2], [3], concrete [4], [5], foamed concrete [6], and pavement [7]. Cement replacement material should consist of pozzolanic properties as specified in ASTM C618 [8]. Pozzolans are amorphous siliceous or siliceous and aluminous minerals that react with calcium hydroxide to generate cementitious hydration products in the presence of water. The pozzolanic activity of pozzolanic material could be determined through the strength activity index (SAI) test according to ASTM C311[9]. Pozzolan with high silica content would increase the SAI by the pozzolanic reaction of pozzolan with the hydration products of cement which results in increased strength of mortar and concrete. The SAI of pozzolans also increases as the curing age increases [10]. Many pozzolans have been used as partial cement replacements due to their high silica and alumina content such as fly ash [11], [12], palm oil fuel ash [13], [14], waste glass powder [15], and corncob ash [16]. The SBEA also known as eco-processed pozzolan [5] and processed spent bleaching eacontains contain a high percentage of silica and alumina which satisfies the pozzolanic properties as specified in ASTM C618 [4].

Besides the amorphous silica content in pozzolans, the specific surface area and particle size of a pozzolan also play an important in the pozzolanic activity. In previous studies, ground and treated pozzolans have been used as partial cement replacements in mortar and concrete. The inclusion of finer particle size of ground pozzolan in mortar improved its pozzolanic reaction over the curing age [17]. A previous study reported that mortar containing fine particle size of pozzolan ha as higher SAI value than the larger particle size [18]. High specific surface area can lead to high rate of pozzolanic reaction. The pozzolan with smaller particle size usually has larger specific surface area which could allow the silica to react with the calcium hydroxide ($Ca(OH)_2$) at high rate of reaction to produce more calcium silicate hydrate (CSH). Furthermore, the inclusion of treated pozzolan enhanced the formation of CSH gel shown on the SEM image and depletion of calcium hydroxide ($Ca(OH)_2$) was analysed from thermogravimetric analysis result [10]. Based on the microstructural analysis of the mortar, the CSH was produced from the consumption of silica oxide and $Ca(OH)_2$ [14]. Incorporation of smaller particle size in the cement paste would fill the pores of the microstructure which makes it denser and enhances the strength [19].

The increase in replacement level of pozzolan with larger specific surface area could reduce the content of $Ca(OH)_2$ in paste [20]. This might be due to the consumption of $Ca(OH)_2$ through the pozzolanic reaction to produce secondary CSH. Up to 40% replacement with finer pozzolan enhanced the strength of concrete at 28 days compared to 7 days of curing age [21]. Furthermore, cement paste containing large specific surface area has lower porosity than specimen with low specific surface area. Past study reported that the paste which has lower porosity with high specific surface area particles improved the pozzolanic reaction rate and refined the pore network of the paste [20]. Zeyad et al. [22], stated that high specific surface area, smaller particle size, low carbon content, low loss on ignition and high amorphous content are the factors to improve the properties of pozzolan.

2. RESEARCH SIGNIFICANCE

The benefit of incorporating SBEA as partial replacement of cement should be acknowledged to increase its utilization in cement and concrete industry. As a result, utilization of SBEA could also reduce its waste disposal at landfill. To date, few studies have been done on the SBEA to be used in cement and concrete production. There is also limited study investigating the pozzolanic activity of SBEA presently. Therefore, the effect of incorporating 10%-50% of ground spent bleaching earth ash (GSBEA) on the SAI of mortar was determined in this study. The enhancement of the properties of SBEA by grinding has also been provided in this study.

3. MATERIALS AND METHOD

3.1 Preparation of Materials

The ordinary Portland cement, river sand, and ground spent bleaching earth ash (GSBEA) were used in this study. The river sand was prepared according to ASTM C778 [23]. Fig. 1 shows the ground spent bleaching earth ash (SBEA). GSBEA was supplied from Lahad Datu, Sabah. The SBEA was oven-dried in the oven for 24 hours. Then, it was ground at the planetary grinding ball mill for 30 minutes to obtain smaller particle size of SBEA, as also known as GSBEA.

Fig.1 Ground spent bleaching earth ash

3.2 Mix Proportion of Mortar Samples

Table 1 shows the mix proportion of mortar samples. The mix proportion of mortar samples were prepared according to ASTM C109 [24]. There were 6 mixtures including the control specimen and specimens containing GSBEA at 10%, 20%, 30%, 40%, and 50%. The specimens were prepared for conducting the strength activity index (SAI) of mortar. The water to cement ratio used in this study was 0.485 for the control sample. For mortar containing GSBEA, the water was adjusted to obtain flow ± 5 of the value of control mortar according to ASTM C311. The size of mould used for the mortar mixture was 50 × 50 × 50 mm. For each mixture, three (3) samples were prepared for each age of curing. The flow table test was conducted according to ASTM C230 [25]. The flow table was dropped 25 times after the mould was lifted. The average diameter of the mortar was obtained from measurements taken at four (4) different dimensions to calculate the flow of mortar.

Table 1 Mix proportion of mortar sample

Sample	Binder (%)		Binder to Sand Ratio
	OPC	GSBEA	
CONTROL	100	0	1:2.75
GSBEA10	90	10	1:2.75
GSBEA20	80	20	1:2.75
GSBEA30	70	30	1:2.75
GSBEA40	60	40	1:2.75
GSBEA50	50	50	1:2.75

3.3 Testing Procedures

The chemical, mineralogical, physical, and microstructural properties of materials were determined in this study. The particle size and specific surface area of the materials were determined by using laser diffraction particle size analyzer (MALVERN MASTERSIZER). Furthermore, the chemical, mineralogical, and microstructural properties were determined by X-Ray Fluorescence (XRF) (EPSILON 1), X-Ray Diffraction (XRD) (RIGAKU), and scanning electron microscope (SEM) (Hitachi S3400N) respectively.

The strength activity index of GSBEA were determined by following ASTM C311 [9]. The analysis for every mixture was tested at three (3) different curing ages which were 7, 28, and 90 days to obtain the accurate values for the pozzolanic activity of GSBEA. The specimens were de-moulded after 24 hours before cured in saturated lime water for 7, 28, and 90 days. Then, the compressive strength test was conducted on the specimens at 7, 28, and 90 days according to ASTM C109. The value of SAI was determined by using Eq. (1).

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

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

For scanning electron microscope equipped with energy dispersive x-ray spectroscopy (SEM/EDX) testing, the sample was cut into smaller pieces with diameter approximately 1 cm to observe the micrograph image of the paste. Then, it was subjected to isopropanol alcohol to stop the hydration before being dried in a vacuum desiccator. SEM/EDX testing was conducted to produce the micrograph image of the mortar paste at 90 days of curing age.

4. RESULTS AND DISCUSSIONS

4.1 Chemical Properties

The chemical properties of the ordinary Portland cement (OPC) and ground spent bleaching earth ash (GSBEA) are shown in Table 2. The percentages of SiO_2, Al_2O_3, and Fe_2O_3 of GSBEA were 47.6%, 11.6%, and 9.8% respectively. While for OPC, the percentages of SiO_2, Al_2O_3, and Fe_2O_3 were 14.4%, 3.6%, and 3.2% respectively. It shows that the percentages of SiO_2, Al_2O_3, and Fe_2O_3 of GSBEA were higher than OPC. The total sums of SiO_2, Al_2O_3 and Fe_2O_3 (S+A+F) of GSBEA was 69% which was more than 50%. Thus, it is classified as Class C Pozzolan according to ASTM C618. A similar finding was reported by Yunus *et al.* [4] which the percentage sum of SiO_2, Al_2O_3 and Fe_2O_3 of GSBEA was 69% which can be classified as Class C pozzolan. Meanwhile Rokiah *et al.* [6] reported that SBEA might be grouped as Class N pozzolan as specified in ASTM C618.

The percentages of silica and alumina content of GSBEA are 47.6% and 11.6% respectively, which are higher than the percentages of silica and alumina content of OPC, which are 14.4% and 3.6% respectively. These chemical properties generally comply with those reported in previous studies [4], [6]. Pozzolans with high silica content will react with $Ca(OH)_2$ to produce more calcium silicate hydrate (CSH). This finding is also supported by evidence from previous observations [10], [12], [14]. The alumina content in the pozzolan will react with $Ca(OH)_2$ to produce calcium aluminate silicate hydrate.

The CaO content in chemical composition of GSBEA (12.5%) was lower than OPC (72.3%). The result was similar as reported by Yunus *et al.* [4] which the percentage of CaO in SBEA was lower than OPC. Low CaO content may cause retardation and dilution effects especially at an early age where the hydration of cement occurs. This is because the CaO is required for early age strength while silica and alumina are required for later age strength. Using pozzolan at high replacement percentages might reduce the production of CSH due to lower CaO content, thus reducing the strength of mortar at early age. It is reported that CaO has a major role as binding part in OPC [26]. Therefore, the low CaO content might affect the strength development of mortar [27]. The loss on ignition (LOI) percentage of GSBEA was 3.3% which was lower compared to OPC. It was also in the range for a Class C pozzolan as specified in ASTM C618. The LOI value would affect the pozzolanic reactivity of pozzolan. Previous study on other pozzolans found that a high LOI value of a pozzolan would result in a lower SAI value of pozzolan [28].

Table 2 Chemical compositions of materials

Chemical Properties (%)	OPC	GSBEA	ASTM C618 (Class C Pozzolan)
SiO_2	14.4	47.6	
Al_2O_3	3.6	11.6	
Fe_2O_3	3.2	9.8	
CaO	72.3	12.5	
SO_3	3.66	2.13	5.0 (max.)
S + A + F	-	69.0	50.0 (min.)
LOI	5.78	3.3	6.0 (max.)

4.2 Mineralogical Analysis

Fig. 2 shows the mineralogical analysis of GSBEA and OPC. Minerals such as α-quartz was spotted at 2θ angles of 20.86°, 26.63°, and 50.20° as a major crystalline phase of GSBEA. The α-quartz as a major crystalline phase confirms the presence of silica as the main constituent which was in line with the chemical composition in Table 2. Furthermore, the other minerals such as cristobalite, hematite, calcite, and mullite were detected at 2θ of 28.03°, 33.21°, 29.44°, and 31.31° respectively as a minor crystalline phase of GSBEA. Minerals such as α-aluminium oxide (α-Al_2O_3) was also detected as minor crystalline at peak of 35.68° and 39.44°. From the XRD analysis, the broad peak can be seen at 20° to 30° which could indicate the amorphous phase in GSBEA. The result is in agreement with the chemical oxides of GSBEA as mentioned in Section 4.1 where GSBEA mainly consists of SiO_2.

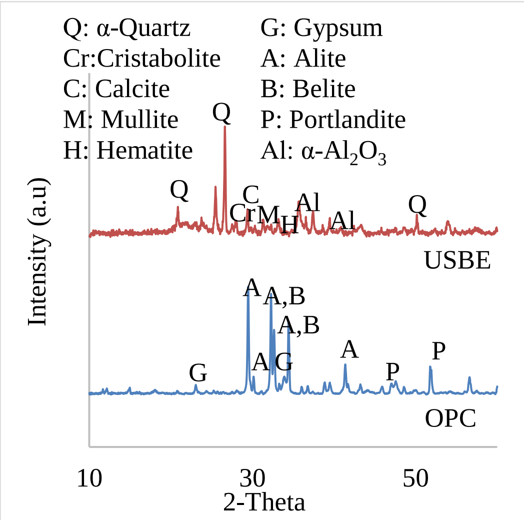

Fig.2 XRD patterns of OPC and GSBEA

4.3 Physical Properties

Table 3 shows the physical properties for the OPC and GSBEA. It shows that the particle size of GSBEA was 32.1% smaller than OPC. It was revealed that 50% of particle sizes of OPC and GSBEA were under 27.4 and 18.6 µm respectively. The specific surface area of OPC and GSBEA were 635 and 704 m^2/kg respectively. The specific surface area of GSBEA was higher than OPC. The grinding process of the pozzolan causes its porous cellular structure to collapse and break down into smaller fractures. This behavior generally agrees with previous findings where the pozzolans with smaller particle sizes usually have higher surface areas [21], [29]. The specific gravity values of OPC and GSBEA are 3.27 and 1.9 respectively. The specific gravity of OPC was higher than GSBEA. It shows that GSBEA was the lighter material compared to OPC.

Table 3 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
GSBEA	18.6	704	1.9

4.4 Microstructural Properties

Fig. 3 displays SEM images that were used to assess the microstructural properties of OPC and GSBEA. The particles of OPC are agglomerated, irregularly shaped, and in a rough texture. While for GSBEA, the particles appear in spherical shape, a crushed, smaller form, less rough and are less porous. This result is different with the previous studies on the unground SBEA which stated that it has a porous and rough texture [4]. This might be due to the grinding process effect on the GSBEA. According to Asrah et al. [30], the porosity of larger particle size of pozzolan decreased when the pozzolan was ground into a smaller particle size.

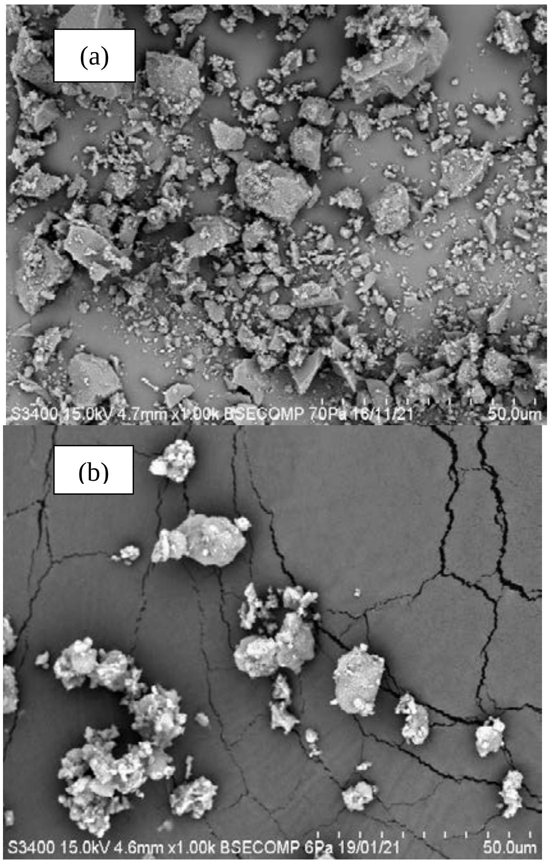

Fig. 3 SEM images of materials (a) OPC and (b) GSBEA

4.5 Flow and Water Requirement of Mixtures

Table 4 shows the flow and the water requirement of the mixture. The flow of control sample was recorded at water to cement ratio of 0.485 as specified in ASTM C109. While for samples with GSBEA as partial cement replacements, the amount of water was adjusted to obtain ±5 of control flow. Based on the result, as the level of replacement of GSBEA increased, the water requirement also increased.

Table 4 Flow and water requirement of mixtures

Mix	Flow	Water requirement (%)
CONTROL	97	100
GSEBA10	99	100
GSBEA20	98	104
GSBEA30	99	104
GSBEA40	98	104
GSBEA50	97	107

Fig. 4 shows the water requirement for every sample. The water requirement of GSBEA10, GSBEA20, GSBEA30, GSBEA40, and GSBEA50 were 100%, 104%, 104%, 104%, and 107% respectively. This shows that high content of GSBEA in the mixture would cause more water to be absorbed by the particles due to the porous texture as mentioned in Section 4.4. Thus, more water was provided to achieve the desired workability and fluidity of the mixture.

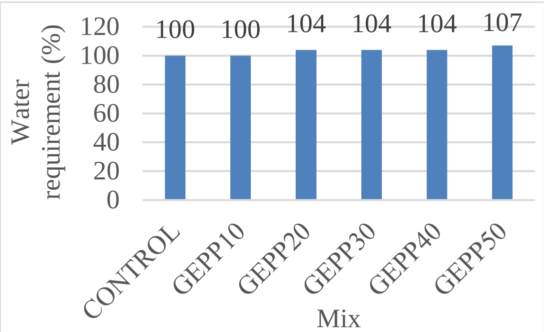

Fig. 4 Water requirements of mixture

4.6 Strength Activity Index

Fig. 5 displays the strength activity indices of all of the mixtures. The strength activity index (SAI) of mortar containing GSBEA were determined at 7, 28, and 90 days. The SAI of reference mortar (CONTROL) was expected to be 100% at 7, 28, and 90 days. At 7 days of curing, the SAI values of GSBEA10, GSBEA20, GSBEA30, GSBEA40, and GSBEA50 are 94.43%, 104.25%, 103.15%, 100.58%, and 99.74% respectively which were more than the minimum requirement of 75% as specified in ASTM C618. It shows that the grinding process of GSBEA influences the properties of GSBEA which has high specific surface area leads to positive impact on the pozzolanic reactivity of GSBEA.

At 28 days of curing, the SAI values of GSBEA10, GSBEA20, GSBEA30, GSBEA40, and GSBEA50 were 98.26%, 109.77%, 122.14%, 135.16%, and 124.69% respectively. The SAI values of all specimens achieved more than 75% as specified in ASTM C618. Furthermore, SAI values of GSBEA20, GSBEA30, GSBEA40, GSBEA50 were more than SAI value of the CONTROL specimen. It shows that GSBEA40 has the highest SAI value with 135.16% compared to other specimens at 28 days. All specimens at 28 days of curing achieved higher SAI values compared to SAI values at 7 days of curing.

At 90 days, the SAI values of GSBEA10, GSBEA20, GSBEA30, GSBEA40, and GSBEA50, were 107.91%, 127.48%, 135.08%, 136.17%, and 124.74% respectively. As can be seen from the figure, as curing age increased, the SAI values of GSBEA specimens increased. The SAI values of GSBEA10, GSBEA20, GSBEA30, GSBEA40, and GSBEA50 were more than the SAI value of the CONTROL specimen. The result shows that GSBEA40 achieved the highest SAI value at 90 days of curing which was 136.17%. This might be due to the high content of amorphous silica and alumina in GSBEA as mentioned in Section 4.2. The SiO_2 and Al_2O_3 in GSBEA react with calcium hydroxide to produce calcium silicate hydrate and calcium aluminate silicate hydrate respectively. This increased the pozzolanic reaction and improved the strength development hence increasing the SAI value. The inclusion of 40% of GSBEA consist of sufficient reactive silica to react with $Ca(OH)_2$ for pozzolanic reaction. Even though, the GSBEA50 has a higher amount of GSBEA, but it also has less CaO due to the lower amount of cement which contributes to the low formation of $Ca(OH)_2$. The amount of $Ca(OH)_2$ was not enough to react with the reactive silica in GSBEA50.

The grinding process increases the specific surface area of GSBEA which was also attributed to the dissolution of silica. High specific surface area of GSBEA leads to the high rate of pozzolanic reaction. As a result, the larger surface area of GSBEA might allow more silica to react with $Ca(OH)_2$ and produce more CSH which accelerate the pozzolanic activity of mortar. This finding is in agreement with findings by Altwair et al. [10]. It shows that ground particle material consisting of fine particle size, high specific surface area, and high amorphous content enhanced the properties of GSBEA, thus increasing its pozzolanic activity.

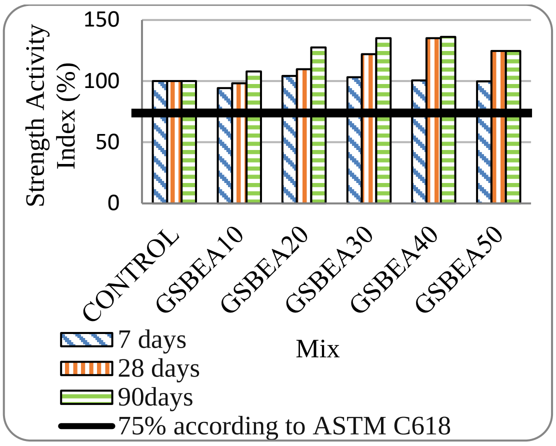

Fig.5 Strength activity index of mortars at different curing ages

SEM/EDX results obtained in this study confirmed the results of SAI shown in Figure 6. The mortar pastes with 0%, 40%, and 50% of GSBEA were chosen to compare the microstructure of CONTROL sample (without GSBEA) with GSBEA40 (sample with the highest SAI value) and GSBEA50 (sample with the highest replacement of GSBEA). Fig. 6 (a), (b), and (c) display the SEM images of CONTROL, GSBEA40, and GSBEA50 at 90 days of curing age respectively while Fig. 7 (a), (b), and (c) display the EDX images of CONTROL, GSBEA40, and GSBEA50 at 90 days of curing age respectively. Formation of $Ca(OH)_2$ was identified based on the hexagonal shape of $Ca(OH)_2$ shown in SEM image in the finding reported by Shao et al. [31]. The formation of CSH was detected based on the appearance of crowded tiny cotton shapes on the SEM image [6]. From the SEM image of the control sample, the hexagonal shape indicated that calcium hydroxide ($Ca(OH)_2$) was detected at 90 days of curing age. The existence of $Ca(OH)_2$ in the paste might be attributed to the high CaO content in cement in the CONTROL specimen. Therefore, there were more $Ca(OH)_2$ than CSH because pozzolanic reaction cannot occur without pozzolan so there was no further consumption of $Ca(OH)_2$ to produce additional CSH.

The formation of CSH was shown in the SEM images of GSBEA40 and GSBEA50, where the microstructure of the paste mostly appeared in tiny crowded cotton shapes and more compact than the CONTROL paste at 90 days of curing. The EDX result shows that GSBEA40 and GSBEA50 contained higher amount of CSH in the pastes due to the high peaks of calcium and silica. The smaller particle size of GSBEA may also contribute to the formation of CSH gel since it provided a larger surface to allow the silica to react with $Ca(OH)_2$. Ground material also acts as a filler where the smaller particles fill the void in the paste which make it denser. This shows that a pozzolanic reaction between silica in GSBEA with hydration product of cement increased the pozzolanic activity of GSBEA mortar. The SEM/EDX result shows that the highest SAI value (136.17%) of GSBEA40 was confirmed by the development of CSH in GSBEA40. The SAI value GSBEA50 was lower than GSBEA40. This might be due to the increased amount of GSBEA in the total mass of paste. Thus, it results in lower $Ca(OH)_2$ due to lower CaO content, hence less CSH was produced in the pozzolanic reaction. Unreacted particles of GSBEA were also shown in GSBEA50 paste. It can act as an inert filler to fill the void in the paste without contributing to the strength.

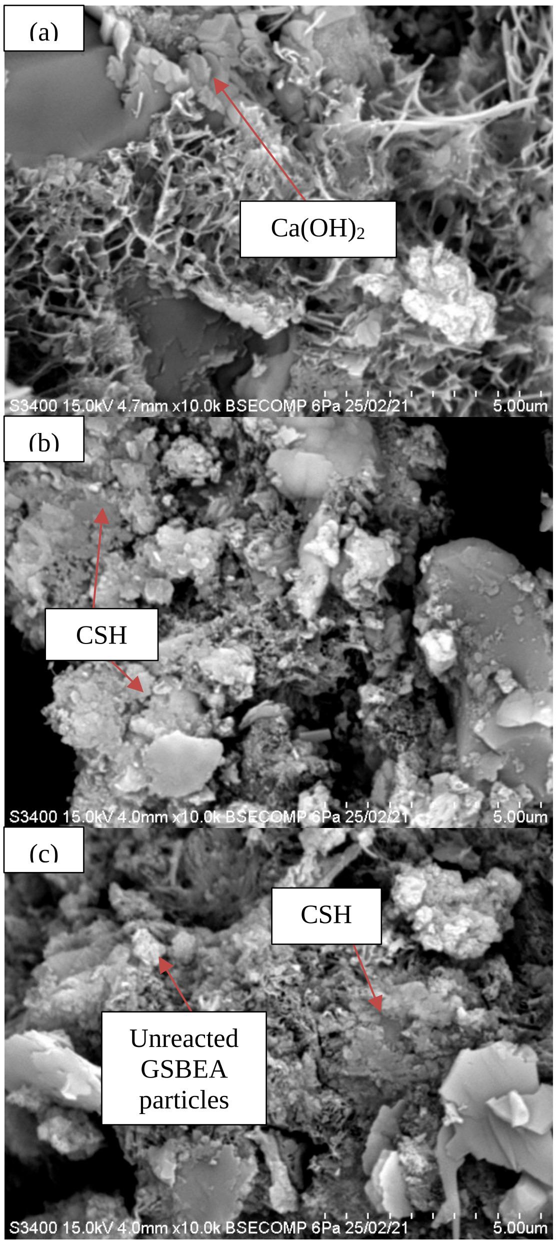

Fig.6 SEM images of mortar paste at 90 days (a) CONTROL, (b) GSBEA40, and (c) GSBEA50

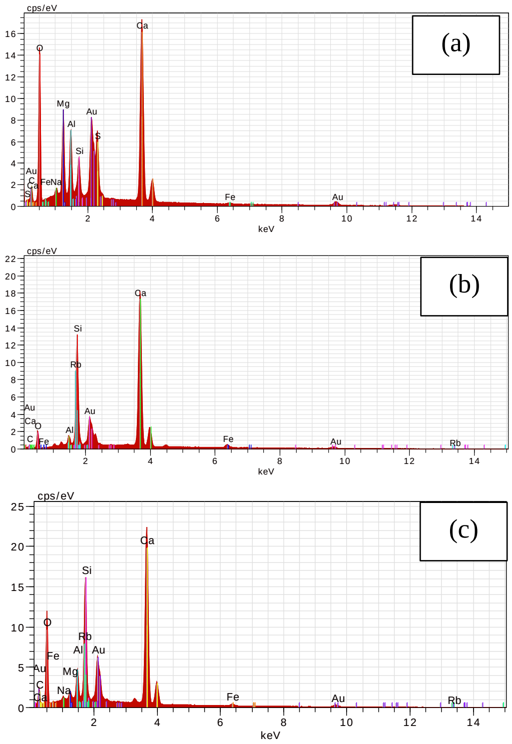

Fig.7 EDX images of mortar paste at 90 days (a) CONTROL, (b) GSBEA40, and (c) GSBEA50

5. CONCLUSION

Based on this research, the conclusions can be drawn as following:

The utilization of ground spent bleaching earth ash (GSBEA) as cement replacement could be beneficial for construction. The main components of GSBEA were silica oxide (47.6%), aluminium oxide (11.6%) and ferric oxide (9.8%). The sum of (SiO_2 + Al_2O_3 + Fe_2O_3) of GSBEA was 69% so it can be classified into Class C pozzolan according to ASTM C618. The The inclusion of GSBEA at high level of replacement, especially at 40%, showed positive results in pozzolanic activity which obtained the strength activity index (SAI) values of 100.58%, 135.17%, and 136.17% at 7, 28 and 90 days respectively. The SAI values were more than 75% of the minimum SAI value as specified in ASTM C311. The SAI value increased as the curing age increased.

This result was confirmed by the formation of calcium silicate hydrate (CSH) that was observed on the scanning electron microscope/energy dispersive x-ray spectroscopy (SEM/EDX) result of paste containing 40% of GSBEA. High peak of calcium and silica on the EDX result indicated the existence of CSH. The grinding process of spent bleaching earth ash for 30 minutes into smaller particle size (d_{50}: 18.6 μm) and higher specific surface area (704 m^2/kg) improved its pozzolanic activity in terms of SAI test. The high specific area of GSBEA allows the silica to react with calcium hydroxide to produce more calcium silicate hydrate, which accelerates the pozzolanic reaction.

6. ACKNOWLEDGMENTS

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