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Resistance of lignite bottom ash geopolymer mortar to sulfate and sulfuric acid attack

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ABSTRACT

This paper presents an investigation of the compressive strength and the durability of lignite bottom ash geopolymer mortars in 3% sulfuric acid and 5% sodium sulfate solutions. Three finenesses of ground bottom ash viz., fine, medium and coarse bottom ash were used to make geopolymer mortars. Sodium silicate, sodium hydroxide and curing temperature of 75 °C for 48 h were used to activate the geopolymerization. The results were compared to those of Portland cement and high volume fly ash mortars. It was found that the fine bottom ash was more reactive and gave geopolymer mortars with higher compressive strengths than those of the coarser fly ashes. All bottom ash geopolymer mortars were less susceptible to the attack by sodium sulfate and sulfuric acid solutions than the traditional Portland cement mortars.

1. Introduction

It is known that 0.7–1.1 ton of CO_2 is emitted for every ton of cement produced. Approximately 50% of carbon dioxide emission by cement industry is from the calcination of limestone and the other 40% from combustion of fuel in the kiln, and 10% from transportation and manufacturing operations [1]. In order to reduce the amount of CO_2 emission from the cement industry, the manufacturing process has been improved and the growth in the Portland cement usage has to be reduced. For cement consumption, there are several efforts to reduce the use of cement in concrete. These include the utilization of supplementary cementitious materials such as fly ash, blast-furnace slag, silica fume, metakaolin, natural pozzolans, and biomass ash [2–4]. In the last decade, geopolymer binders have emerged as one of the possible alternative to conventional cement binders for concrete industry [5,6]. Geopolymer is an inorganic binder material and can be produced by a geopolymeric reaction of alkali activating solution with silica and alumina rich source materials from geological origin or pozzolanic materials such as metakaolin, fly ash, and rice husk ash [7]. Geopolymers exhibit many excellent properties such as high early strength, low creep, low shrinkage, and good resistance against acid and sulfate attack in addition to its environment friendliness [8–12].

Bottom ash (BA) is a by-product of the combustion of pulverized coal in the power plants. It is generated from the coagulation of the melting fly ash particles. The BA particles are therefore relatively large. The world production of coal ash was approximately 459 million tons in 1992 [13]. The BA comprises approximately 20% of the coal ash and almost all of it is disposed of as landfill. The annual output of lignite BA at Mae Moh power plant in the north of Thailand is about 0.8 million tons and is disposed of in the landfill and ash ponds which causes an environmental problem. BA contains high silica and alumina contents similar to fly ash. Several researchers [14,15] found that grinding of BA increases its pozzolanic activity and makes it suitable for use as partial replacement in Portland cement. In addition, BA is a suitable source material for making geopolymer [16–18]. Previous researches [19] studied the effects of fineness of BA, sodium hydroxide, sodium silicate, water, and naphthalene-based superplasticizer on workability and strength of geopolymer mortars. The results indicated that the flow and strength of geopolymers were improved with an increase in the fineness of BA.

In this study, the effect of BA fineness on compressive strength and the durability of BA geopolymer mortars in 5% sodium sulfate and 3% sulfuric acid solutions were presented. The results were compared to those of Portland cement mortar, and mortar containing 40% fly ash and ground BA. The obtained data of this work would be beneficial for the utilization of BA in the geopolymer mortar and concrete. This should lay a good foundation for the increased use of BA and thus lead to the reduction in the cement consumption and environmental problems.

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2. Experimental details

2.1. Materials

Materials used in this study consisted of Portland cement Type I (OPC), fly ash (FA), and lignite bottom ash (BA) from Mae Moh power plant in northern Thailand. The chemical and mineral compositions are summarized in Table 1 and Fig. 1, respectively and the physical properties of these materials are shown in Table 2.

The specific gravity of cement was 3.18 with the median particle size (d_{50}) of 17.3 μm. The major oxide ($SiO_2 + Al_2O_3 + Fe_2O_3$) of the FA was 74.7% which is higher than the minimum requirement (70%) specified by ASTM C 618 [20] for Class F fly ash. The XRD pattern of FA shows substantial amorphous phase with some peaks of crystals of anhydrite, lime, magnesioferrite, quartz, and mullite. In order to study the effect of BA fineness, three finenesses of ground BA viz., fine BA (FBA), medium BA (MBA) and coarse BA (CBA) with corresponding median particle sizes of 15.7, 24.5 and 32.2 μm and Blaine finenesses of 5300, 3400 and 2100 cm^2/g, respectively were used. The mineral compositions of FBA, MBA, and CBA were similar suggesting that the grinding of BA had little effect on the crystallinity. Sodium silicate solution (NS) with 13.8% Na_2O, 32.2% SiO_2 and 54.0% H_2O, and 10 molar (M) sodium hydroxide (NH) solution were used as alkaline activators. A local river sand with specific gravity of 2.62 and fineness modulus of 2.85 was used for making the mortar.

2.2. Mix proportions and mixing

In order to study the resistance of lignite BA geopolymer mortars on sulfuric acid attack and to compare the results to cement mortar, three Portland cement mortars and three geopolymer mortars were used. The mix proportions are shown in Table 3.

For cement mortar (PC, PFA40, and PFBA40), the ratio of a binder material to fine aggregate was set at 1–2.75 by weight. The PC mortar consisted of only OPC as cementitious materials. The PFA40 and PFBA40 mixes were Portland cement mortars with OPC replaced by FA and FBA at the dosage of 40% by weight of binder. The mortars were mixed and the flows were determined in accordance with the procedure given in ASTM C 1437 [21].

The BA to sand ratio of 1–2.75, liquid alkaline/BA ratio of 0.597 and NS/NH ratio of 1.50 were the geopolymer mortar mix proportions used. GFBA, GMBA, and GCBA were geopolymer mortars made from FBA, MBA, and CBA, respectively. The mixing of geopolymer mortar was done in a controlled room at 25 °C. The BA was mixed with NH for 5 min to allow the leaching of silica and alumina ions [22]. Sand was then incorporated and mixing was done for 5 more minutes. This was followed by the addition of NS with a final mixing of 5 min. After mixing, the flows of BA geopolymer mortars were tested.

Table 1
Chemical composition of materials.

Chemical composition (%)	OPC	FA	FBA	MBA	CBA
SiO_2	21.5	38.7	39.3	40.3	35.5
Al_2O_3	4.5	20.7	21.3	17.4	16.8
Fe_2O_3	3.1	15.3	13.5	13.6	15.1
CaO	62.7	16.6	16.5	11.8	13.3
MgO	1.1	1.5	2.6	7.6	8.4
Na_2O	1.5	1.2	1.0	2.8	3.8
K_2O	0.7	2.7	2.1	1.5	1.8
TiO_2	0.1	0.4	0.4	0.3	0.3
P_2O_5	–	0.2	0.2	0.2	0.2
SO_3	3.5	2.6	1.5	–	–
LOI	1.3	0.1	1.4	4.4	4.7

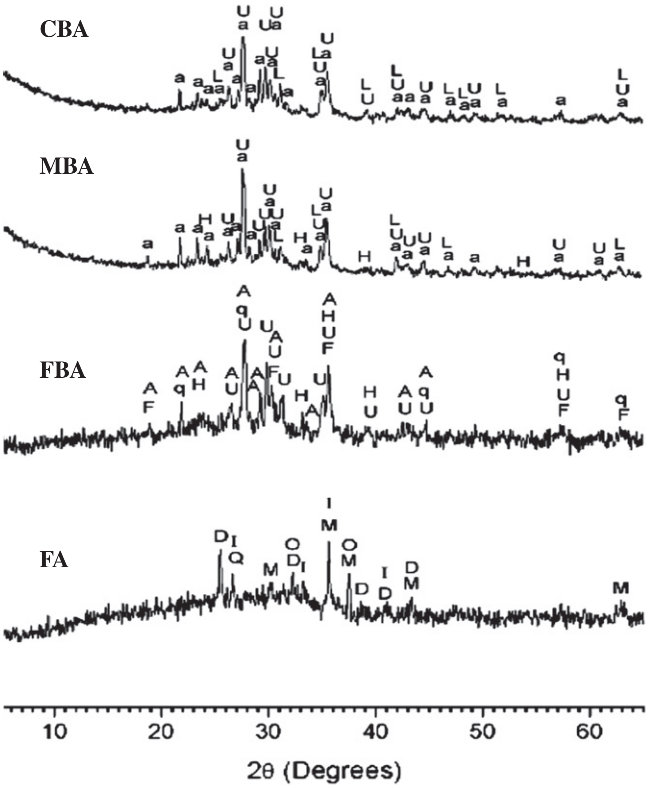


Fig. 1. The XRD pattern of materials; D-anhydrite; $CaSO_4$; O-lime: CaO; A-anorthite, Na-rich intermediate: $(Ca,Na)(Si,Al)_4O_8$; F-magnetite: Fe_3O_4; H-hematite: Fe_2O_3; Q-quartz: SiO_2; U-augite, Al-rich: $Ca(Mg,Fe,Al)(Si,Al)_2O_6$; I-Mullite: $3Al_2O_3.2SiO_2$.

Table 2
Physical properties of materials.

Sample	Specific gravity	Median particle size, d_{50} (μm)	Blaine fineness (cm^2/g)	Retained on a sieve No. 325 (%)
OPC	3.18	17.3	3400	9
FA	2.51	37.1	2100	33
FBA	2.89	15.7	5000	3
MBA	2.87	24.5	3400	18
CBA	2.86	32.2	2100	33

2.3. Testing detail

2.3.1. Compressive strength test

The cement mortars were cast in 50 × 50 × 50 mm cube molds. The specimens were cover with damped cloth and polyurethane sheet for moist curing. They were demolded at one day and stored in water at 23 ± 2 °C until the testing age. For geopolymer mortars, they were placed into the 50 × 50 × 50 mm cube molds and wrapped with cling film to avoid the moisture lost and left in the laboratory for 1 h and then temperature cured at 75 °C for 48 h [19]. The specimens were demolded and kept in the controlled room at 23 ± 2 °C and 50% RH until the testing age. All samples were tested for compressive strength at 7, 28, 90, 180 and 360 days in accordance with ASTM C 109 [23]. The reported compressive strengths were the average of three samples.

2.3.2. Sulfate resistance

In order to determine the durability of geopolymer mortar, the expansions of mortar bars immersed in sodium sulfate solution were tested. Mortar bars with 25 × 25 × 285 mm in dimension

Table 3
Mix proportions of mortars.

Mix	Type of fly ash	OPC (g)	FA (g)	BA (g)	NS (g)	NH (g)	Water (g)	Sand (g)	Flow (%)
PC	–	100	0	0	0	0	47	275	114
PFA40	FA	60	40	0	0	0	47	275	146
PFBA40	FBA	60	0	40	0	0	47	275	112
GFBA	FBA	0	0	100	35.8	23.9	0	275	62
GMBA	MBA	0	0	100	35.8	23.9	0	275	51
GCBA	CBA	0	0	100	35.8	23.9	0	275	35

Note: 10 M of NH, liquid alkaline/BA = 0.597, NS/NH = 1.50.

were prepared in accordance with the procedures given in Section 2.3.1 and ASTM C 1012 [24]. The mortar bars were immersed in 5% sodium sulfate solution and were tested for expansions. The sulfate solution was replaced with fresh solution after the immersion of 7, 14, 28, 56, 84, 120 and 240 days.

2.3.3. Sulfuric acid resistance

The sulfuric acid resistances of mortars were tested by modified test method B in accordance with ASTM C 267 [25]. The 50 × 50 × 50 mm cube mortar specimens were immersed in 3% sulfuric acid solution. After immersed time of 7, 14, 28, 56, 84, and 120 days in the sulfuric acid, the samples were brushed softly under water with a nylon brush to remove loose surface debris before weighting. The weight losses of specimens after immersion in the acid were evaluated. The sulfuric acid solution was replaced with fresh solution after the immersions of 7, 14, 28, 56, 84, and 120 days.

3. Results and discussion

3.1. Compressive strength

The results of the compressive strength tests of mortars are summarized in Table 4 and Fig. 2. The compressive strength of cement mortars (PC, PFA40, and PFBA40) increased with the curing age in accordance with the strength development of Portland cement. For example, the compressive strengths of PFBA40 mortar at 7, 28, 90, 180, and 360 days were 31.5, 43.5, 63.5, 68.5, and 71.5 MPa, respectively. The strength development of geopolymer mortars (GFBA, GMBA, and GCBA) increased at a lower rate compared to those of Portland cement mortars. This was due to the fact that the curing at temperature of 75 °C for 48 h increased the early age compressive strengths [26].

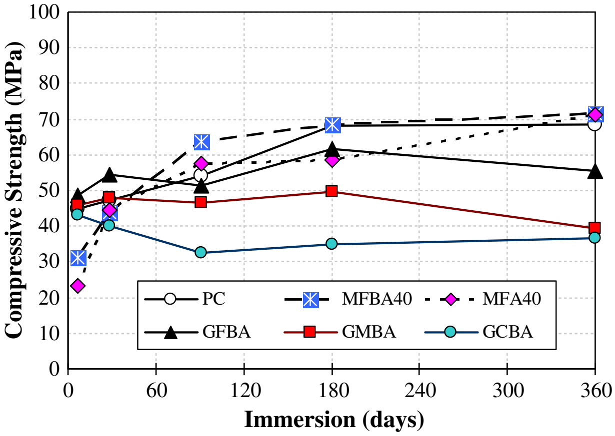


Fig. 2. Compressive strength of mortars and time of curing.

The development of compressive strength was also affected by the BA fineness. The use of FBA in geopolymer mortar seemed to give higher compressive strengths than those of the coarser ones. Since, the BA with high finenesses and high surface areas could dissolve more silica and alumina [19] and thus resulted in the increase in geopolymerization and compressive strength. In addition, a large portion of as-received BA particles were porous and contributed to the low strength of geopolymer mortars [12]. Grinding helped destroy the pores of BA particles and thus improved the strength of geopolymer mortars [19]. The compressive strengths of GFBA mortars at 7, 28, 90, 180 and 360 days were 48.5, 54.5, 51.5, 61.5 and 55.5 MPa, respectively. For the coarser BA, the compressive strengths of GCBA mortars at the same ages were 43.0, 40.0, 32.5, 35.0 and 36.5 MPa, respectively.

Table 4
Compressive strengths of mortars.

Mix	Compressive strength (MPa)				
	7 D	28 D	90 D	180 D	360 D
PC	44.7	47.4	54.0	68.0	68.5
PFA40	23.0	44.5	57.5	58.5	71.5
PFBA40	31.0	43.5	63.5	68.6	71.5
GFBA	48.5	54.5	51.5	61.5	55.5
GMBA	46.0	48.0	46.5	49.5	39.5
GCBA	43.0	40.0	32.5	35.0	36.5

3.2. Resistance to sodium sulfate attack

The results of the length change of mortars bars subjected to 5% Na_2SO_4 solution are shown in Fig. 3. The early-age expansions of PFA and PFBA mortars immersed in sodium sulfate solution were higher than that of OPC mortar due to the lower strengths of the PFA and PFBA mortars compared to that of OPC mortar. After 105 days of immersion, the expansions of PC mortar exceeded those of the PFA40 and PFBA40 mortars. This was due to the incorporation of pozzolanic materials in the mixture which helped

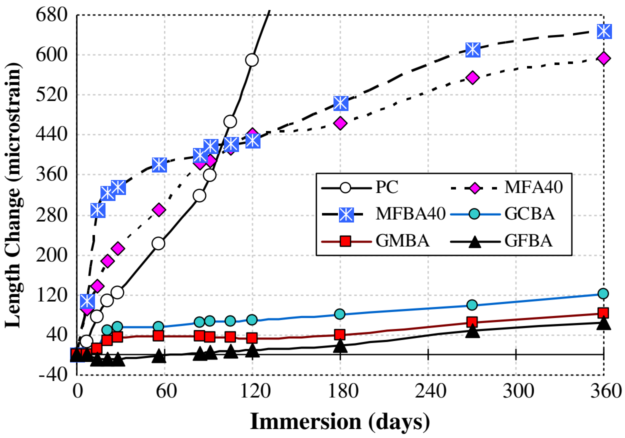
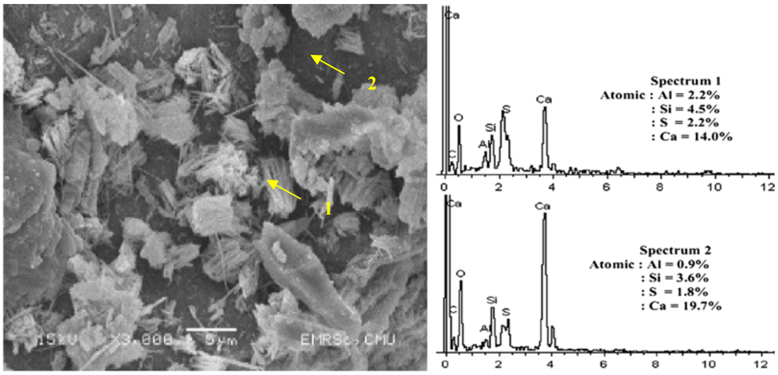
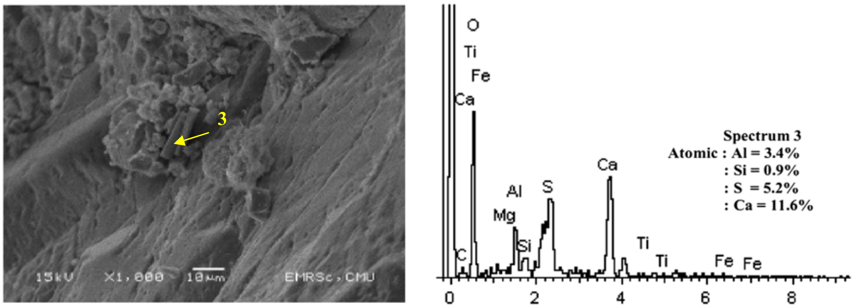


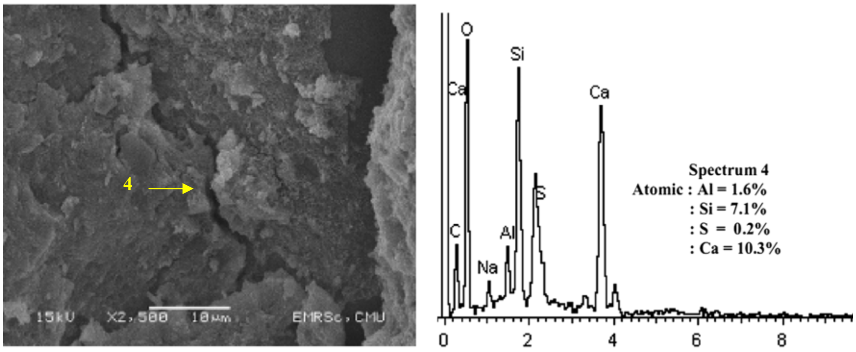
Fig. 3. Length change of mortars after immersion in 5% Na_2SO_4 solution.



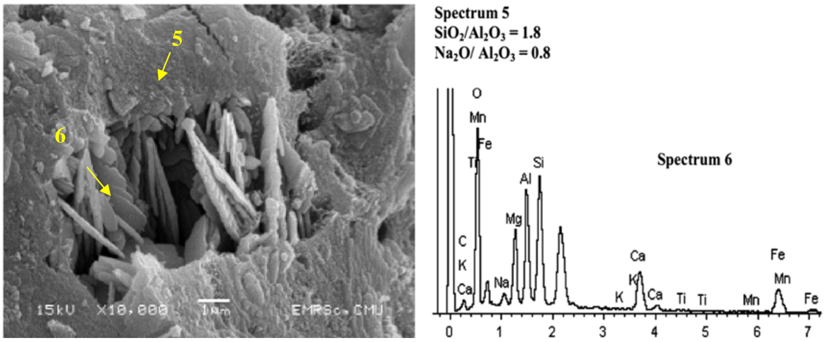
(a) PC mortar



(b) PFA40 mortar



(c) PFBA40 mortar

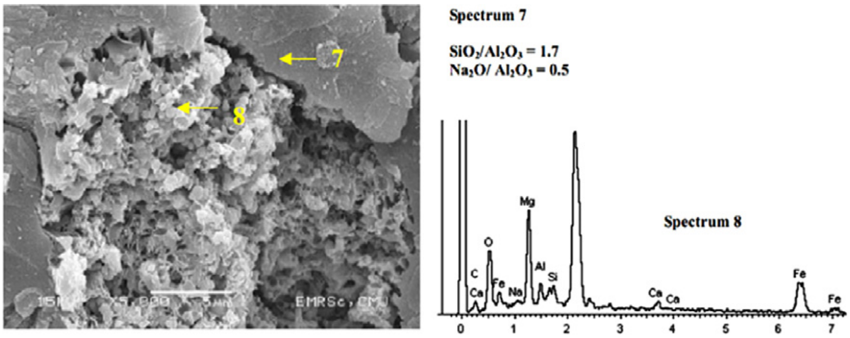


(d) GFBA mortar

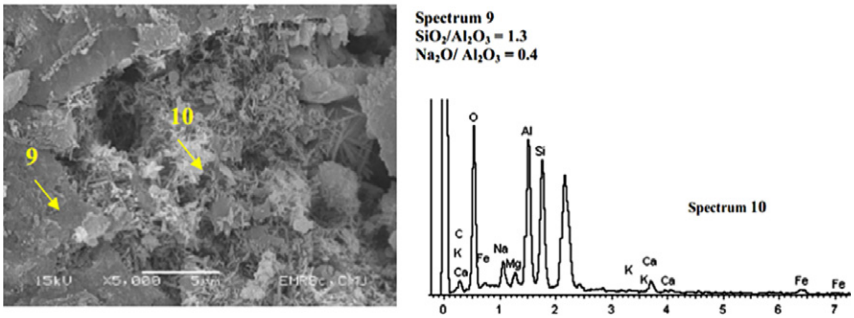
Fig. 4. SEM image and EDX spectrum of mortars after 270-day immersion in 5% Na_2SO_4 solution.

decrease the amount of $Ca(OH)_2$ and the total amount of calcium in the matrices of the PFA40 and PFBA40 mortars.

In case of the geopolymer mortars, the BA geopolymer mortars gave excellent resistance to sodium sulfate attack. After exposure to 5% Na_2SO_4 solution for 360 days, the length changes of geopolymer mortars were only 65–121 microstrain, while those of the PFA40 and PFBA40 mortars were 595 and 648 microstrain, respectively. The PC mortar exhibited the highest expansion of 7600 microstrain. The high deterioration of cement based mortars were due to the reaction of sulfate with calcium hydroxide and calcium monosulfoaluminate to form gypsum and ettringite which could lead to strength loss, expansion, cracking, and scaling of



(e) GMBA mortar



(f) GCBA mortar

Fig. 4 (*continued*)

the surface layers in mortar [27,28]. The geopolymer mortars, however, exhibited a different mechanism. The main geopolymerization products were different from Portland cement hydration products and were less susceptible to sulfate attack compared to the normal cement hydration product. Bakharev [10] found the good performance of fly ash based geopolymer in sodium and magnesium sulfate solutions and attributed this to a more stable cross-linked aluminosilicate polymer structure.

The microstructures of the matrices near the external surface of mortars after 270 days of immersion in 5% Na_2SO_4 solution were studied using Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Analysis (EDX) and the results are shown in Fig. 4. The SEM of cement based mortars (Fig. 4a) showed some cracks and the presence of ettringite in the samples. The EDX spectrum at area 1 showed the existence of the ettringite which could be observed in substantial quantity. The EDX spectrum at area 2 showed the background of the normal hydration products of calcium silicate and calcium aluminate hydrate. The sulfate attack on the cement matrix was associated with the ettringite and gypsum formation and the decalcification. At pH of 12–12.5 only ettringite formation took place and at pH of 8.0–11.5 gypsum formation and decalcification occurred [29]. For the PFA40 and PFBA40 mortars (Fig. 4b and c), the needle-like crystals of ettringite were not easily seen. The mass of hydration products of calcium silicate and aluminate hydrates remained intact. The contents of Ca were slightly lower at around 10.3–11.6% as compared to the values of 14–19.7% for the normal Portland cement hydration products. The attack on the $Ca(OH)_2$, calcium sulfoaluminates, and the CSH with a high C/S ratio in hardened PC paste resulted in a very porous layer whereas the attack on the low C/S CSH resulted in a protective layer of silica gel [30]. The Portland cement mortars containing pozzolans such as fly ash and rice husk ash with low calcium content, therefore, were more durable to the sulfate attack than the conventional Portland cement mortars [31].

The microstructure of BA geopolymer mortars are shown in Fig. 4d–f. The EDX spectrum showed the presence of unreacted BA particles (with the usually observed Al, Si, Fe, O, Ca, Mg, and

Table 5
Weight change of mortars after immersion in 3% of H_2SO_4.

Mix	Weight loss (%)						
	0 D	7 D	14 D	28 D	56 D	84 D	120 D
PC	0.0	−9.8	−23.1	−42.2	−66.5	−85.9	−95.7
PFA40	0.0	−1.7	−7.7	−26.1	−48.4	−71.7	−91.8
PFBA40	0.0	−0.1	−1.4	−7.3	−28.5	−52.5	−77.2
GFBA	0.0	−1.2	−2.0	−2.5	−2.8	−3.2	−3.6
GMBA	0.0	0.0	−0.4	−0.9	−1.0	−1.3	−1.7
GCBA	0.0	−0.1	−0.4	−0.8	−0.9	−1.1	−1.4

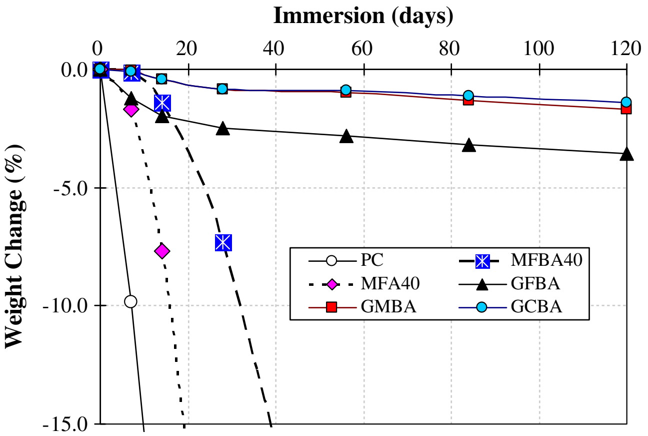
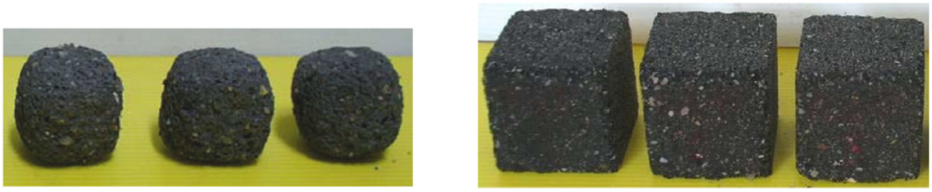
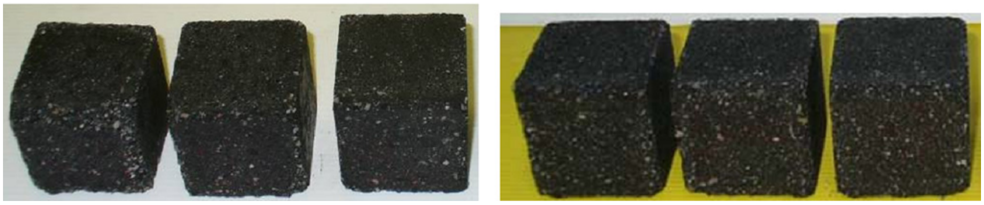


Fig. 5. Weight loss of mortar specimens after 120-day immersion in 3% H_2SO_4 solution.

(a) PC mortar (b) PFA40 mortar



(c) PFBA40 mortar (d) GFBA mortar



(e) GMBA mortar (f) GCBA mortar

Fig. 6. Physical appearance of mortar specimens after 120-day immersion in 3% H_2SO_4 solution.

Na elements) embedded in the geopolymer matrix. These elements were the basic elements of the source BA. As compare to Portland cement mortar, this system contained significantly lower Ca content. The needle-shape like particles were also presented but only in a limited amount and could be observed in the some of the available space as shown in Fig. 4f. These needle-shape like particles were from the calcium presented in the source material as the BA was a high calcium lignite coal ash. The results confirmed that the main geopolymerization products were less susceptible to sulfate attack compared to the normal Portland cement product.

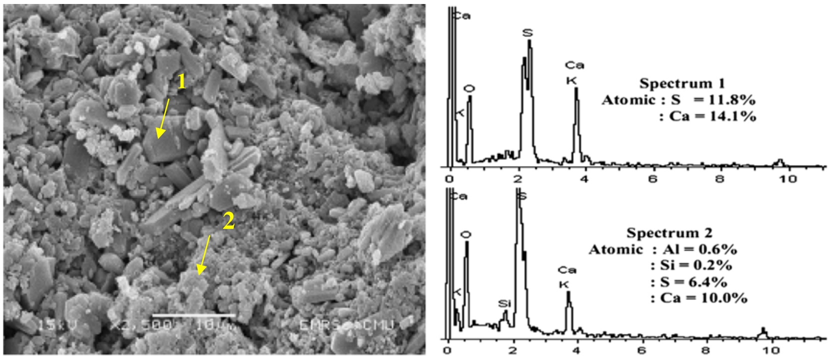
3.3. Resistance to sulfuric acid attack

Table 5 and Fig. 5 show the relationship between the weight change due to sulfuric acid attack on mortars and the immersed time. As for cement based mortars, their weight losses were due primarily on the reaction between calcium hydroxide ($Ca(OH)_2$) presented in the specimens and the acid, which can induce tensile stress, resulting in cracking and scaling of mortar. Among the cement based mortars, the PFA40 and PFBA40 mortars exhibited higher acid resistance than that of PC mortar. After 120 days of immersion in sulfuric acid solution, the weight loss of PFA40 and PFBA40 mortars were 91.8% and 77.2%, respectively, while that of PC mortar was 95.7%. The small particles of these ashes allowed a denser packing of the mixture. In addition, the siliceous compounds in FA and FBA reacted with free lime to form a more stable product of C–S–H that further filled the pores in mortars [32]. Therefore, mortars containing FA and FBA yielded lower weight loss than PC mortar.

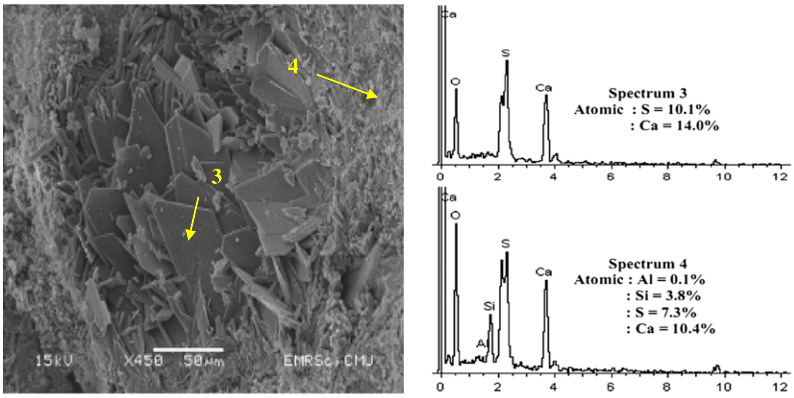
Compared to the cement based mortars, the weight losses of all of geopolymer mortars were much lower. After 120 days of immersion, GFBA, GMBA and GCBA mortars had lost only 3.6%, 1.7%, and 1.4% of their weights, respectively. The geopolymer mortars had low weight loss under acid attack due to the low water absorption of these binders and also to their low calcium (Ca) contents compared to those of cement based mortars [33]. This result conformed with that of Thokchom et al. [34] who reported that after exposure in sulfuric acid solution for 18 weeks, the fly ash based geopolymer mortars showed a very low weight loss in the range from 0.41% to 1.23% of the initial weight. In addition, Bakharev [9] studied the resistance of fly ash geopolymer materials against 5% acetic and sulfuric acids up to 5 months exposure and found that geopolymer materials had better resistance than cement based specimens. Fig. 6 shows the physical appearance of the mortar specimens after 120 days of immersion. The cement mortars were severely deteriorated, while the geopolymer mortars seemed to be slightly damaged at the surface and around the edges of specimens.

The SEM and EDX spectrum of mortars after 120 days of immersion in 3% sulfuric acid solution are shown in Fig. 7. The EDX spectrums at areas 1 and 2 of Fig. 7a–c revealed the presence of elongated crystalline structures of gypsum ($CaSO_4 \cdot 2H_2O$) as indicated by the large presences of Ca, O, and S. The PC mortar had soft white depositions on the surface and became very porous after exposure to sulfuric acid. In the PFA40 mortar after the acid attack (Fig. 7b), abundant gypsum crystals could be seen in the morphological features similar to PC mortar. For the PFBA40 mortar (Fig. 7c), the results confirmed that the low calcium hydration products were more durable in the acid environment compared to that of high calcium hydration products in PC mortar.

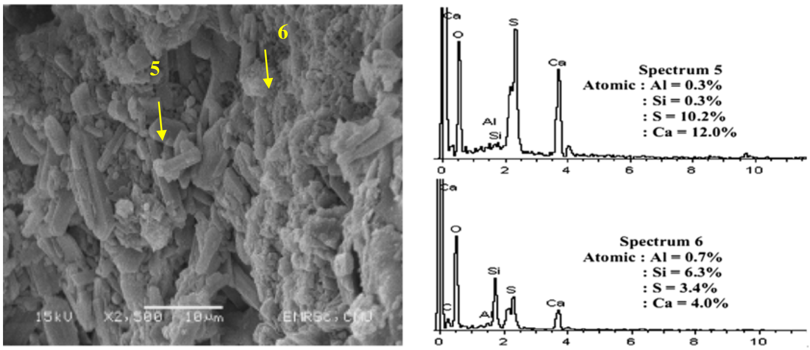
The geopolymer mortars showed some delaminations at the surface, but the damage was significantly less than the cement based mortars as shown in Fig. 7d–f. The presence of $CaSO_4$ was also observed at the surface. The geopolymer mortars showed better performance than the cement based mortars when exposed to sulfuric solution due to difference in chemical and phase compositions [35]. The major components from the EDX spectrum of geopolymer mortars in the deterioration area were Si, Ca, Al, Mg, and O. The ratios of SiO_2/Al_2O_3 and Na_2O/Al_2O_3 varied between 11.5–15.7 and 0.2–0.3, respectively. The high SiO_2/Al_2O_3 and low Na_2O/Al_2O_3 ratio after deterioration were due to the leaching of Al and Na from geopolymer structure in the presence of sulfuric acid solution. This result conformed to the work of Bakharev [9] which reported that after the exposure to sulfuric acid, there was an increase of the Si/Al ratio in the fly ash based geopolymer.



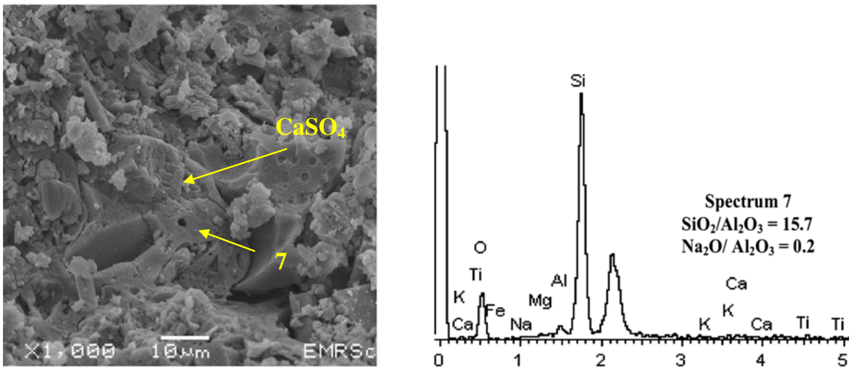
(a) PC mortar



(b) PFA40 mortar



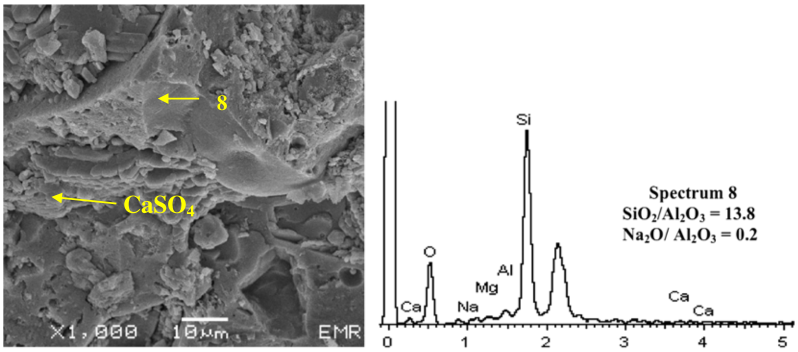
(c) PFBA40 mortar



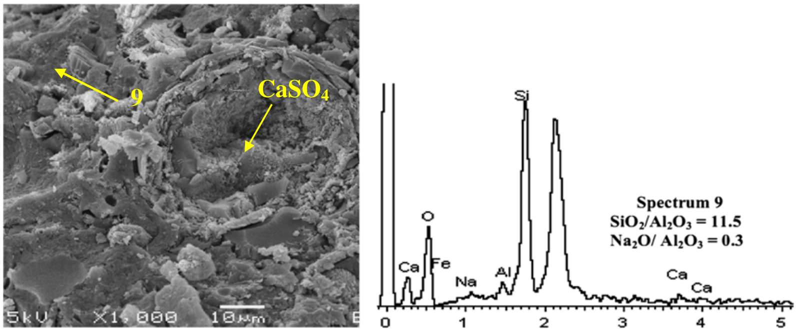
(d) GFBA mortar

Fig. 7. SEM images and EDX spectrum of mortars after 120-day immersion in 3% H_2SO_4 solution.

Among geopolymer mortars, GCBA mortar performed significantly better than the others and showed the lowest SiO_2/Al_2O_3 ratio of 11.5. The fine BA with high surface area was more susceptible to the attack in sulfuric acid solution and thus GFBA mortar seems to deteriorate more as compared with GMBA and GCBA mortars. It is likely that the coarse BA underwent less geopolymerization and less hydration associated with the calcium as compared to those of the fine BA as indicated by the strength results. The



(e) GMBA mortar



(f) GCBA mortar

Fig. 7 (*continued*)

calcium hydration product was susceptible to the attack of the acid solution and if present in large quantity should cause the deterioration of the mortar. Hence the coarse BA geopolymer mortars performed better in the acid solution as compared to the fine BA geopolymer mortars. The good performance of Portland cement mortars containing coarse fly ash immersed in sulfuric acid as compared to that containing fine fly ash was also reported [36].

4. Conclusions

This study presents the investigations of the resistance on sulfuric acid and sulfate solutions of OPC and FA mortars, and BA geopolymer mortars. Based on the results, the following conclusions can be drawn.

(1) The compressive strengths of BA geopolymer mortars were improved with increasing the fineness of BA.
(2) The BA geopolymer mortars were less susceptible to the attack by 5% sodium sulfate solutions compared to the Portland cement based system.
(3) The deterioration of BA geopolymer mortars immersed in 3% sulfuric acid solution also showed better performance than those of PC mortars and mortars containing 40% of FA and FBA. All BA geopolymer mortars showed weight loss less than 3.6% at 120 days.
(4) The better performance of the geopolymer mortars in the sulfate and sulfuric solutions were due to the more stable cross-linked aluminosilicate polymer structure as compared to the normal Portland cement hydration structure.

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