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Workability and strength of lignite bottom ash geopolymer mortar

Apha Sathonsaowaphak[a], Prinya Chindaprasirt[a,*], Kedsarin Pimraksa[b]

[a] Department of Civil Engineering, Faculty of Engineering, Khon Kaen University, Khon Kaen, 40002, Thailand
[b] Department of Industrial Chemistry, Faculty of Sciences, Chiang Mai University, Chiang Mai, 50200, Thailand

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ABSTRACT

In this paper, the waste lignite bottom ash from power station was used as a source material for making geopolymer. Sodium silicate and sodium hydroxide (NaOH) were used as liquid for the mixture and heat curing was used to activate the geopolymerization. The fineness of bottom ash, the liquid alkaline/ash ratio, the sodium silicate/NaOH ratio and the NaOH concentration were studied. The effects of the additions of water, NaOH and napthalene-based superplasticizer on the workability and strength of the geopolymer mortar were also studied. Relatively high strength geopolymer mortars of 24.0–58.0 MPa were obtained with the use of ground bottom ash with 3% retained on sieve no. 325 and mean particle size of 15.7 μm, using liquid alkaline/ash ratios of 0.429–0.709, the sodium silicate/NaOH ratios of 0.67–1.5 and 7.5–12.5 M NaOH. The incorporation of water improved the workability of geopolymer mortar more effectively than the use of napthalene-based superplasticizer with similar slight reduction in strengths. The addition of NaOH solution slightly improves the workability of the mix while maintaining the strength of the geopolymer mortars.

1. Introduction

Bottom ash (BA) is a solid waste from the combustion of coal. The annual output of lignite bottom ash at Mae Moh power plant in the north of Thailand is around 0.8 million tons and is disposed of as landfill near the power plant as shown in Fig. 1. The bottom ash contains several toxic elements, such as lead (Pb), zinc (Zn), cadmium (Cd) and copper (Cu) [1]. These toxic elements can leach out and contaminate soils as well as surface water and groundwater [2]. Several researches on the utilization of coal bottom ash for use as cementitious material have been conducted. The bottom ash has to be ground to increase the pozzolanic activity and used to partially replace Portland cement [3,4]. The utilization of bottom ash as a cementitious replacement material has not yet been well received as it needs grinding and only a partial replacement of cement is possible. Moreover, the bottom ash itself is porous and increases the water requirement of the mix [3]. On the other hand, the grinding of coal bottom ash results in a prolonged setting time and causes a reduction in the workability of the paste [5].

Geopolymer materials are alkali-activated aluminosilicates, with a much smaller CO_2 footprint than traditional Portland cements, and display very good strength and chemical resistance properties as well as a variety of other potentially valuable characteristics [6,7]. Geopolymerization processes can, therefore, be applied to utilize solid waste and by-products containing silica and/or alumina. It is environmentally friendly and need moderate energy to produce. It also provides a mature and cost-effective solution to many problems where hazardous residue has to be treated and stored under critical environmental conditions [8].

Fly ash is a good source material for making geopolymer owing to its high content of silica and alumina [9–11]. The fly ash geopolymer can totally substitute the use of normal Portland cement. Similar to fly ash, bottom ash contains high contents of silica and alumina and should be suitable for use as source materials in making geopolymer. Its use to totally replace the Portland cement system is therefore very attractive. The physical characteristics and phase properties of the bottom ash are, however, different to fly ash even from the same source. For example, bottom ash is composed of large angular fragments with only a small amount of semi-spherical particles [12] and less glassy constituent phase [13].

Workability and strength are two important properties of the mortar, concrete and geopolymer. Water can be used to effectively increase the workability of the geopolymer mortar with a slight reduction in strength [14]. It has also been shown that superplasticizer can be used to increase the workability of geopolymer concrete [10]. The limited study on the effects of superplasticizer on alkali-activated slag paste and mortars, however, shows that several superplasticizers except the naphthalene-based lose their fluidity properties in mortar activated with NaOH as a result of the change in their chemical structures in high alkaline media [15].

The objective of this research is, therefore, to study the effects of fineness of bottom ash, sodium hydroxide solution, sodium

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[*] Corresponding author. Tel.: +66 4320 2355; fax: +66 4320 2355x12.
E-mail address: prinya@kku.ac.th (P. Chindaprasirt).

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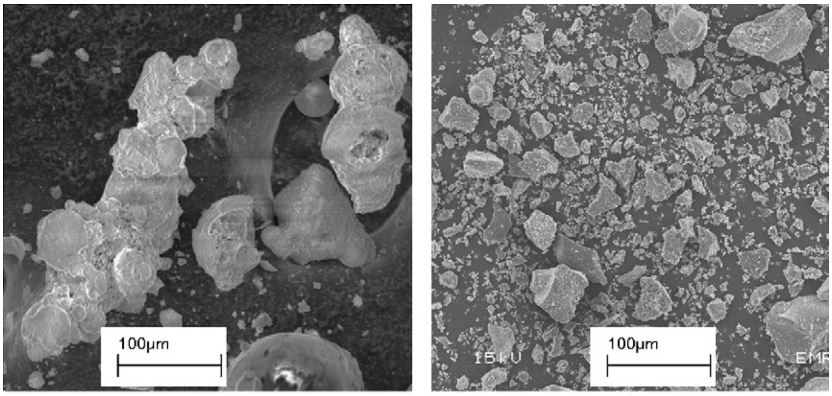
(a) Fresh bottom ash (b) Landfill of bottom ash

Fig. 1. Fresh bottom ash and landfill of bottom ash at Mae Moh power plant. (a) Fresh bottom ash and (b) landfill of bottom ash.



(a) As-received bottom ash (b) ground bottom ash

Fig. 2. SEM of as-received bottom ash and ground bottom ash. (a) As-received bottom ash and (b) ground bottom ash.

silicate solution, water and naphthalene-based superplasticizer (NSP) on workability and strength of the geopolymer mortar. The work would lay a foundation for the future utilization of bottom ash in the geopolymer mortar and concrete products instead of treating the bottom ash as waste material.

2. Experimental details

2.1. Materials

Lignite bottom ash came from the Mae Moh power station in the north of Thailand. The as-received BA particles were relatively large and very irregular showing agglomeration of some spherical particles and other fragments with observable pores as shown in Fig. 2(a). The ground BA particles were angular in shape with much less pores in comparison to those of the as-received BA as shown in Fig. 2(b). The chemical compositions of BA were 39.3% SiO_2, 21.3% Al_2O_3, 13.5% Fe_2O_3, 2.1% K_2O, 16.5% CaO, 1.0% Na_2O, and 1.4% loss on ignition. The XRD pattern of BA as shown in Fig. 3 shows substantial amorphous phase in its structure with the peaks of crystals of quartz, anorthite, augite, magnetite and hematite. The ash was ground until 3, 18 and 33% retained on sieve no. 325 (45 μm) were obtained with the corresponding mean particle sizes of 15.7, 24.5 and 32.2 μm and Blaine finenesses of 5000, 3400 and 2100 cm^2/g, respectively.

Sodium silicate solutions with 13.8% Na_2O, 32.2% SiO_2 and 54.0% H_2O, and 5, 7.5, 10, 12.5 and 15 M NaOH were used. Local river sand with specific gravity of 2.62 and fineness modulus of 2.85 in saturated surface dry condition was used for making geopolymer mortar. Distilled water, 10 M NaOH and NSP, solution were incorporated to the mixes to study their effects on the workability of the mix. The NSP was selected as other superplasticizers were reported to lose their fluidity properties in mortar activated with NaOH [15].

2.2. Mixes of bottom ash geopolymer mortar

2.2.1. Effects of BA fineness

This series was designed to test the effects of fineness of BA on the workability and strength of geopolymer mortars. Bottom ashes with 3, 18 and 33% retained on sieve no. 325, liquid alkaline/ash ratio of 0.597, 10 M NaOH, and sodium silicate/NaOH ratio of 1.5 were selected.

2.2.2. Effects of different compositions

Based on the result of Section 2.2.1, BA with 3% retained on sieve no. 325 was selected. Five series of mixtures were designed to test the effects of the liquid alkaline/ash ratio, the sodium silicate/NaOH ratio, the concentration of NaOH solution, the water/ash ratio, and the additions of 10 M NaOH solution and NSP on workability and strength of mortars. The details of the mixes are shown in Table 1 and the initial molar ratios of Al_2O_3, SiO_2, H_2O and Na_2O are shown in Table 2.

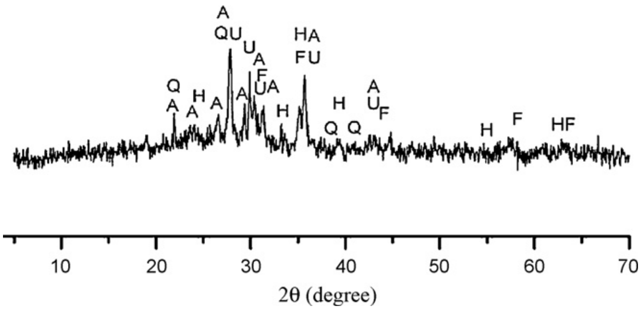


Fig. 3. The XRD pattern of ground bottom ash. A—anorthite,sodian,intermediate: $(Ca,Na)(Si,Al)_4O_8$; F—magenetite: Fe_3O_4; H—haematite: Fe_2O_3; Q—quartz: SiO_2; U—augite, aluminian $Ca(Mg,Fe,Al)(Si,Al)_2O_6$.

Table 1
Weight ratios of mixtures of bottom ash geopolymer mortars.

Series	NaOH concentration (M)	Liquid alkaline/ash ratios	Sodium silicate/NaOH ratios	Water/ash ratios	10 M NaOH or NSP/ash ratios
A	10	0.325, 0.429, 0.518, 0.597, 0.709	1.50	0	0
B	10	0.597	0.4, 0.67, 1.0, 1.5, 2.5	0	0
C1	5, 7.5, 10, 12.5, 15	0.597	0.67	0	0
C2	5, 7.5, 10, 12.5, 15	0.597	1.50	0	0
D	10	0.429	1.50	0, 0.03, 0.06, 0.09, 0.15	0
E	10	0.429	1.50	0.03	0, 0.01, 0.03, 0.06, 0.09

Table 2
Initial molar ratios of Al_2O_3, SiO_2, H_2O and Na_2O in mixtures.

Series	Variables	SiO_2/Al_2O_3	Na_2O/Al_2O_3	H_2O/Na_2O
A	Liquid alkaline/ash ratios 0.325, 0.429, 0.518, 0.597, 0.709	3.63, 3.79, 3.93, 4.05, 4.23	0.44, 0.59, 0.71, 0.82, 0.97	12.27
B	Sodium silicate/NaOH ratios 0.40, 0.67,1.00, 1.50, 2.50	3.57, 3.75, 3.90, 4.05, 4.23	0.96, 0.90, 0.86, 0.82, 0.76	11.65, 11.86, 12.05, 12.27, 12.56
C1	NaOH concentration (M) 5, 7.5, 10, 12.5, 15	3.75	0.62, 0.77, 0.90, 1.03, 1.17	18.75, 14.52, 11.86, 9.95, 8.42
C2	NaOH concentration (M) 5, 7.5, 10, 12.5, 15	4.05	0.63, 0.75, 0.82, 0.90, 0.99	16.96, 13.63, 12.27, 10.78, 9.50
D	Water/ash ratios 0, 0.03, 0.06, 0.09, 0.15	3.79	0.59	12.27, 13.64, 15.00, 16.36, 19.08
E	10 M NaOH/ash ratios 0, 0.01, 0.03, 0.06, 0.09	3.79	0.59, 0.60, 0.64, 0.69, 0.75	13.64, 13.57, 13.44, 13.27, 13.12
	NSP/ash ratios 0, 0.01, 0.03, 0.06, 0.09	3.79	0.59	13.64, 13.86, 14.32, 15.00, 15.68

1. *Series A*: To study the effects of the liquid alkaline/ash ratio, the liquid alkaline/ash ratios were varied between 0.325 and 0.709 with the constant sodium silicate/10 M NaOH ratio of 1.5.
2. *Series B*: To study the sodium silicate/NaOH ratio, the sodium silicate/10 M NaOH ratios of 0.4, 0.67, 1.0, 1.5 and 2.5 were used. The liquid alkaline/ash ratio was kept constant at 0.597 (based on the result of series A).
3. *Series C*: To study the concentration of the NaOH solution, 5, 7.5, 10, 12.5 and 15 M NaOH, and the sodium silicate/NaOH ratios of 0.67 (C1) and 1.5 (C2) were used (based on the result of series B). The liquid alkaline/ash ratio was kept constant at 0.597.
4. *Series D*: To study the water content, the water/ash ratios of 0, 0.03, 0.06, 0.09 and 0.15, the liquid alkaline/ash ratio of 0.429, and the sodium silicate/10 M NaOH ratio of 1.50 were used (based on the results of series A, B and C).
5. *Series E*: To study of the improvement on workability, the sodium silicate/10 M NaOH ratio of 1.50, the liquid alkaline/ash ratio of 0.429, and water/ash ratio of 0.03 were used as a control mix. The mixes with the 10 M NaOH or NSP/ash ratios of 0, 0.01, 0.03, 0.06 and 0.09 were used. The samples of geopolymer mortars were analyzed with energy-dispersive X-ray scanning electron microscope.

2.3. *Mixing, flow and strength tests*

All geopolymer mortars were made with sand to fly ash ratio of 2.75. The mixing was done in an air conditioned room at 25 °C to eliminate the possible effect of temperature variation. The mixing procedure involved mixing of NaOH solution and BA for 5 min in a pan mixer. Sand and other liquids (water, NaOH and NSP) were then added to the mixture and the mixing was done for another 5 min. This was followed by the addition of sodium silicate solution with a final mixing of 5 min. Right after the mixing, the flow value of fresh geopolymer mortar was determined in accordance with ASTM C1437 [16].

After the flow test, fresh mortar was placed in the 50 mm cube mould and compacted as described in the ASTM C109 [17]. This was followed by an additional vibration of 10 s using a vibrating table in the final step. The moulds with mortar specimens were wrapped with vinyl sheet to prevent moisture loss. The specimens were left standing for 1 h and then cured at 75 °C in the oven for 48 h. The specimens were then left in the laboratory to cool down. Demoulding was done the next day and then kept in the 23 ± 2 °C and 50% RH room. The compressive strengths of mortars were tested at the age of 7 days in accordance with ASTM C109. The reported strengths were the average of the three tests.

3. Results and discussions

3.1. *Fineness of bottom ash*

The effects of fineness of BA on the workability and strength of mortar as shown in Fig. 4 indicated that ground BA could be used as a source material for making geopolymer. The workability and strength of mortars were improved with the increase in the fineness of BA. The as-received BA particles were relatively large and very irregular with observable pores and needed to be ground to increase their surface area and the reactivity [18]. The workability improved with the increase in the grinding since the pores were destroyed by grinding. These pores could absorb the liquid of the

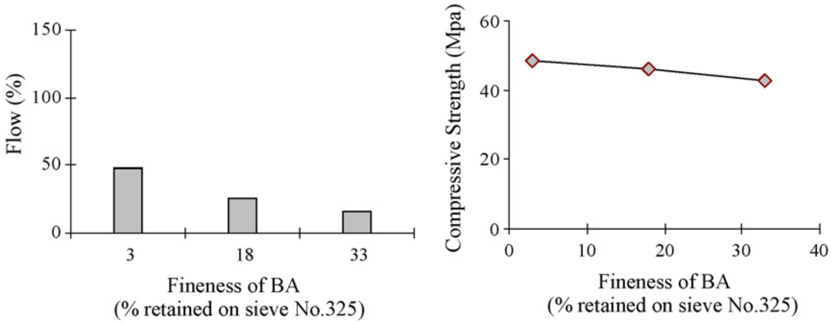


Fig. 4. Flow and strength of mortar with various BA finenesses.

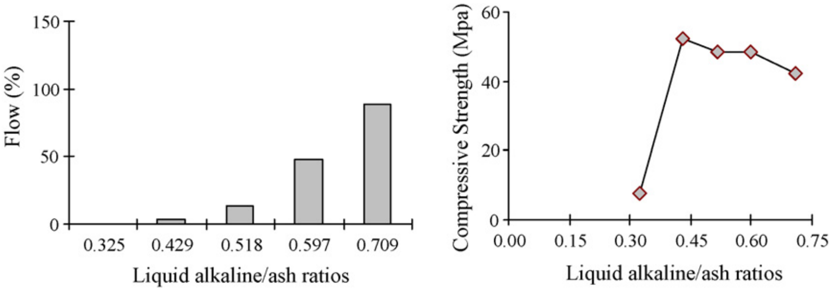


Fig. 5. Flow and strength of mortar with various liquid alkaline/ash ratios.

fresh mortar and lower the workability of the mixes. For the ground coarse BA with 33% retained on sieve no. 325, the strength of the mortar was reasonable at 43.0 MPa. However, the flow of the fresh mortar was low at 16%. With the increase in finenesses of BA to 18 and 3% retained on sieve no. 325, the flows improved to 25 and 49% and the strengths increased to 46.0 and 48.0 MPa, respectively. The mortar made of BA with 3% retained on sieve no. 325 showed the highest workability and compressive strength.

3.2. *Liquid alkaline/ash ratio*

The effects of different liquid alkaline/ash ratios are shown in Fig. 5. The workability of the mixes increased with the increase in the liquid alkaline/ash ratio. The mortar was very stiff with no flow value at the liquid alkaline/ash ratio of 0.325. The mortar was not homogenous and difficult to mix and compact. With the liquid alkaline/ash ratio of 0.429, the mortars became less stiff and a low flow value of 5% was obtained. The workable mortars with flow values of 49 and 89% were obtained with the liquid alkaline/ash ratios of 0.597 and 0.709, respectively. With an increase in fluid medium content, particle to particle interaction of BA was less as a result of a larger interparticle distance and lower particle interference.

With regard to compressive strength, the mixes with liquid alkaline/ash ratios of 0.429–0.709 showed high compressive strength of 42.0–52.0 MPa. The low compressive strength of 8.0 MPa was obtained with the very dry mix of liquid alkaline/ash ratio of 0.325 as a result of difficulties in compaction.

3.3. *Sodium silicate/NaOH ratio*

In this test, the liquid alkaline/ash ratio was kept constant at 0.597. The results as shown in Fig. 6 revealed that the workability of the mixes reduced with an increase in the sodium silicate content. This was expected since sodium silicate was more viscous than sodium hydroxide. The concentration of NaOH solution was constant at 10 M. The sodium silicate/NaOH ratios of 0.4–1.5 produced workable mortars with flow values of 49–97%. However, the flow of the mix with high sodium silicate/NaOH ratio of 2.5 was very low at 5%. As shown in Fig. 6, the strength was also affected by the increase in the sodium silicate/NaOH ratio. The optimum sodium silicate/NaOH ratio was 1.5 and the maximum strength of 48.0 MPa was obtained. An increase in the sodium silicate/NaOH ratio from 0.4 to 1.5 increased the strength. However, when the sodium silicate/NaOH ratio was 2.5, the strength started to drop due to the difficulty in compaction. As shown in Table 2, the mixes contained initial SiO_2/Al_2O_3 molar ratios of 3.57, 3.75, 3.90, 4.05, 4.23 and Na_2O/Al_2O_3 ratios of 0.96, 0.90, 0.86, 0.82, 0.76 were within the normal range of geopolymer [6,20,23]. It should be noted here that NaOH cost less than sodium silicate and the mix should therefore contain low sodium silicate/NaOH ratio while still giving the required strength and workability.

3.4. *Concentration of NaOH solution*

The results of the concentration of NaOH solution are shown in Fig. 7. The workability decreased while the strength increased with the increase in the NaOH concentration. The NaOH concentration of 5, 7.5, 10, 12.5 and 15 M corresponded to mixes with initial SiO_2/Al_2O_3 molar ratios of 3.75 and Na_2O/Al_2O_3 ratios of 0.62, 0.77, 0.90, 1.03 and 1.17, respectively. Mixes with 5 M NaOH had high flow values of 92–123%, whereas those with 15 M NaOH had low flow values of 34–56%. The increase in the concentration of NaOH increased the stiffness of the fresh mortars. The strength increased with the increase in NaOH concentration mainly through the leaching out of silica and alumina with the high concentration of NaOH [19] and high Na_2O/Al_2O_3 ratios. The increased NaOH concentration increased Na ions in the system which was important for the geopolymerization as Na ions were used to balance the charges and formed the alumino-silicate networks.

3.5. *Water/ash ratio*

The effects of various water/ash ratios are shown in Fig. 8. Incorporation of water, as expected, improved the workability of geopolymer mortars. Water/ash ratios of 0, 0.03, 0.06, 0.09 and 0.15 corresponded to mixes with initial SiO_2/Al_2O_3 molar ratios of 3.79, Na_2O/Al_2O_3 ratio of 0.59 and H_2O/Na_2O ratios of 12.27, 13.64, 15.00, 16.36 and 19.08, respectively as shown in Table 2. The water/ash

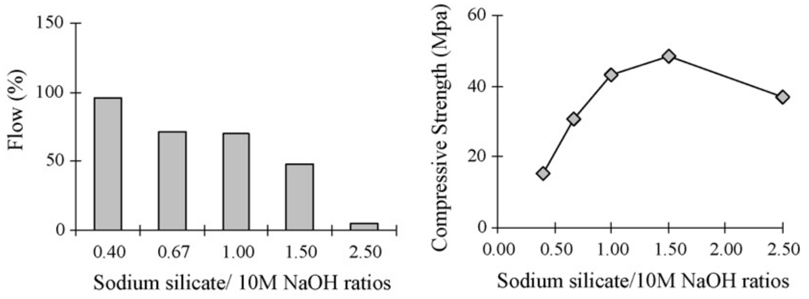


Fig. 6. Flow and strength with various sodium silicate/10 M NaOH ratios.

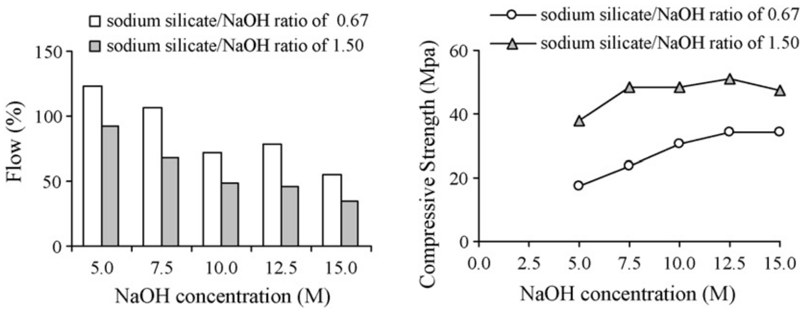


Fig. 7. Flow and strength with various NaOH concentrations.

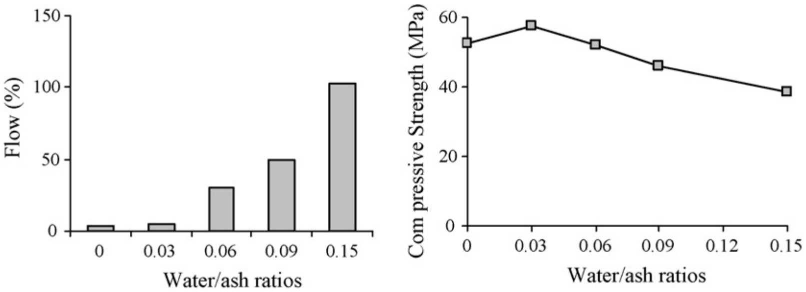


Fig. 8. Flow and strength of mortar with various water/ash ratios.

ratios of 0.09–0.15 produced mixes with flow of 50–103% which was highly desirable. The strengths of these mixes were also high at 38.5–46.0 MPa. The incorporation of water effectively improved the flow of fly ash geopolymer mortar with some reductions in strength [14]. The H_2O/Na_2O ratios of mixes with water/ash ratios of 0.09 and 0.15 were rather high at 16.36 and 19.08. Excess water also diluted the liquid and thus slowed down the dissolution and reaction of the geopolymer. Similar reduction in strength with the increase in H_2O/Na_2O ratio was reported [20]. The large improvement in workability with a small reduction in strength was very attractive.

3.6. Additions of 10 M NaOH and NSP solutions

The additions of 10 M NaOH and NSP were done to test the effect of other liquids other than water on the workability and strength of geopolymer mortars. The results are shown in Fig. 9. The incorporation of 10 M NaOH solution improved the flow while maintaining the strength of mortar. The 10 M NaOH addition reduced the sodium silicate/NaOH ratio and increased the liquid content of the mix and thus resulted in improved workability. In addition, the NaOH solution contains Na ion and water molecules which were the basic ingredients for the geopolymerization [21,22] and thus had no negative effect on the strength. The incorporation of NSP/ash ratios of 0.01–0.03 slightly improved the flow to 16–17% while maintaining the high strengths of 54.0–57.0 MPa. However, the addition of NSP/ash ratios of 0.06–0.09 resulted in slight decreased in the strengths of mortar. At NSP/ash ratio of 0.09, the strength of the mortar reduced to 40.0 MPa. The effect of incorporation of NSP was similar to the incorporation of water in term of strength, but the workability of the mixes with the addition of water was much better. The use of NSP in the geopolymer system activated with NaOH and sodium silicate solutions was not effective. The improvement in the workability was a result of the increase in the water content of the mix from the NSP solution.

Microstructure images of the control BA geopolymer mortar obtained with the water/ash ratio of 0.03 are shown in Fig. 10(a). The mortar appeared as a dense geopolymer matrix with a high strength of 58.0 MPa. The physical and mechanical properties of geopolymers were a function of the SiO_2/Al_2O_3 and Na_2O/Al_2O_3 ratios [23]. The samples of the matrix with SiO_2/Al_2O_3 ratios of 2.1 and 2.7, and Na_2O/Al_2O_3 ratios of 1.30 and 1.34 confirmed the presence of continuous mass of strong geopolymer. For the addition of 10 M NaOH/ash ratio of 0.09, the dense matrix was also obtained as shown in Fig. 10(b) with corresponding high strength geopolymer mortar of 53.0 MPa. The maintaining of high compressive strength

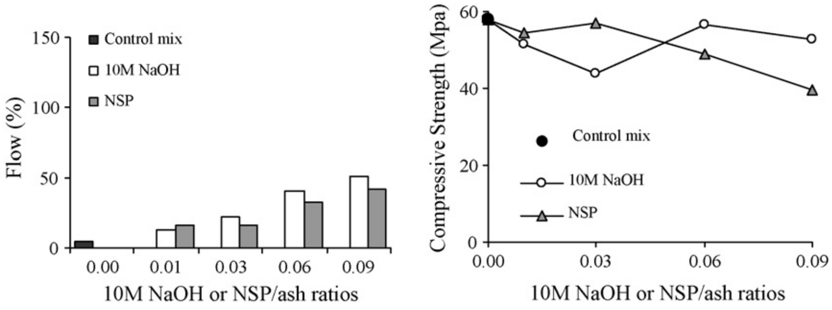
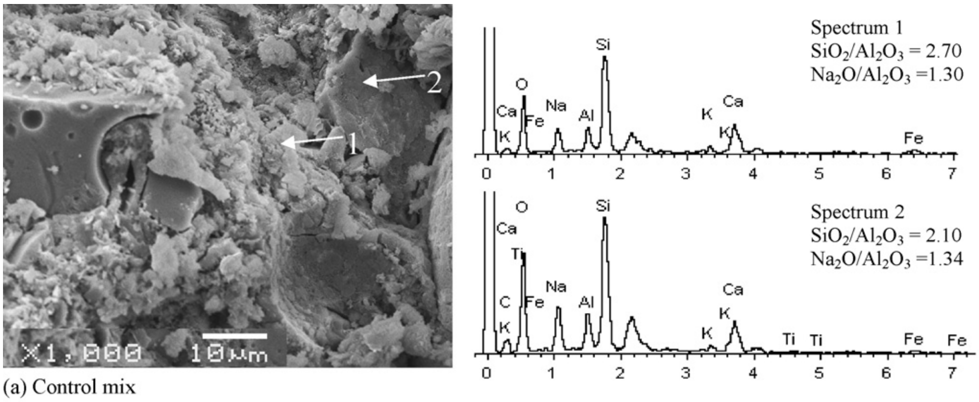
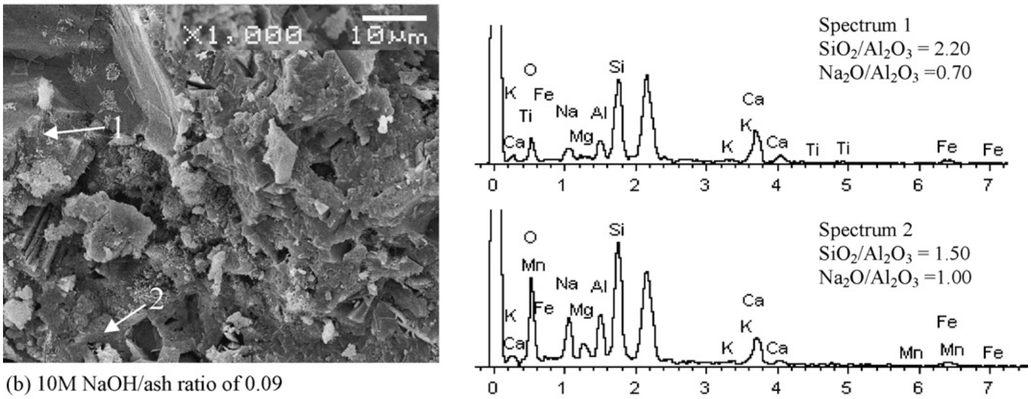


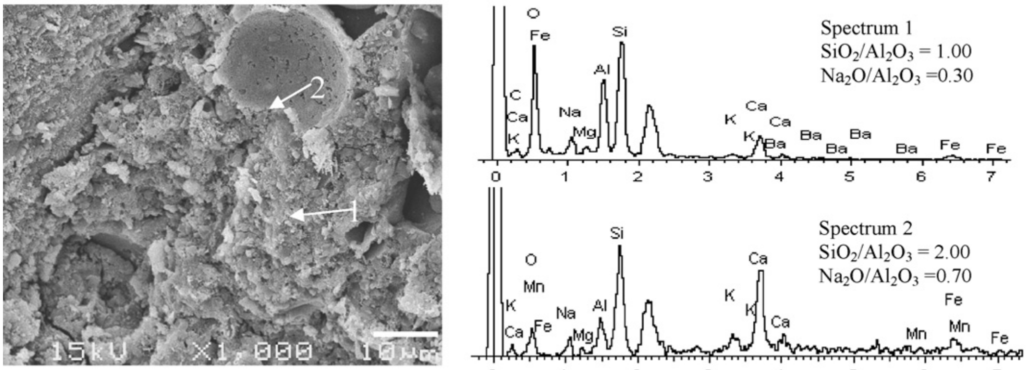
Fig. 9. Flow and strength of mortar with various 10 M NaOH or NSP/ash ratios.



(a) Control mix



(b) 10M NaOH/ash ratio of 0.09



(c) NSP/ash ratio of 0.09

Fig. 10. Microstructure of geopolymer mortars. (a) Control mix, (b) 10 M NaOH/ash ratio of 0.09 and (c) NSP/ash ratio of 0.09.

mortar with the addition of NaOH was due to the alkali roles for a proper charge balancing resulting in a stable and dense geopolymer networks. The samples of the matrix with SiO_2/Al_2O_3 ratios of 1.5 and 2.2 and Na_2O/Al_2O_3 ratios of 0.7 and 1.0 were identified in this system. The addition NaOH played a role in the attack of alumino-silicate material leading to lower values of SiO_2/Al_2O_3 and Na_2O/Al_2O_3 ratios than the control mix.

With the addition of NSP/ash ratio of 0.09, the matrix was less dense with noticeable pores in the geopolymer texture as shown in Fig. 10(c). The strength of the mortar was, therefore, affected and geopolymer mortar with lower strength of 40.0 MPa was obtained. In this case the samples of the matrix with reduced SiO_2/Al_2O_3 ratios of 1.0 and 2.0 and the significantly reduced Na_2O/Al_2O_3 ratios of 0.3 and 0.7 were identified. The results suggested the shifting from the normally molecular bonded geopolymers to the characteristic of particulate solid such as zeolite material [24].

4. Conclusions

Based on the obtained data, the BA can be used as a source material for making good geopolymer. The flow and strength of the geopolymer are improved with an increase in the fineness of BA. The grinding increases the surface area and the reactivity, and also destroys the pores in the as-received BA particles. Relatively good workability and strength are obtained using ground BA with 3% retained on sieve no. 325. The workability increases with the increase in the liquid alkaline/ash ratio owing to the larger interparticle distance and lower particle interference. The workable range of liquid alkaline/ash ratios is between 0.429 and 0.709. The workability, however, decreased with the increases in the sodium silicate/NaOH ratio and the NaOH concentration owing to the high viscosity of sodium silicate and NaOH. The workable ranges of sodium silicate/NaOH ratios and NaOH concentration are between 0.67–1.5 and 7.5–12.5 M, respectively.

The flow and strength are also affected by the additions of water, 10 M NaOH solution and NSP. The incorporations of these liquids increase the workability of the geopolymer mortars. The addition of water is found to be the most effective resulting in substantial increase in the flow with small reduction in strength. The incorporation of 10 M NaOH solution slightly improves the flow while maintaining the strength of the geopolymer mortar. Water and Na ions are an integral part of the geopolymer system and thus are required for the geopolymerization process. The incorporation of NSP is not effective in improving the flow of geopolymer mortars. The improvement in the workability is due mainly to the increase in the water content from the NSP solution.

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