

Influences of characteristics of the alkaline activator on the compressive strength and microstructure of the fly ash-based geopolymer pastes

Qingbo Tian^{a,*}, Daquan Sun^a, Zeyu Gu^a and Zhijie Lv^b

^aSchool of Materials Science and Engineering, Shandong Jianzhu University, Jinan, China

^bSchool of Mechanical and Electrical Engineering, Shandong Jianzhu University, Jinan, China

The effects of Na₂O content, water to binder (W/B) ratio, and the modulus (M_s) of alkaline activator solution on a fly ash-based geopolymer paste (FGP) were investigated. The microstructure of the pastes was observed and characterised by Scanning electron microscope and X-ray diffraction. The results showed that the increase of Na₂O content and M_s resulted in an increase in compressive strength: an increase in the W/B ratios led to a decreased compressive strength. It was found that the high compressive strength was achieved as M_s of the alkaline activator was 1.5, and the content of this activator was 8.0-12.0% by the proportion of Na₂O to fly ash. The compressive strength of these pastes was between 26.5-39.6 MPa at 28 days when they were cured at 85 °C for 1 h followed by curing under standard conditions. OH⁻ concentration, determined by Na₂O content, M_s , and water content, seemed to be a crucial parameter influencing the compressive strength. There was an optimal OH⁻ concentration range, in which the appropriate strength of geopolymer paste would be achieved. An increased alkaline content promoted the reaction between the mineral phases of the fly ash with the alkaline activator and the formation of an additional sodium aluminosilicate hydrate (N-A-S-H) gel.

Keywords: Fly ash-based geopolymer pastes (FGP), Geopolymers, Alkaline activation, Compressive strength, OH⁻ concentration.

Introduction

Geopolymers are synthesised using an aluminosilicate raw material and an alkaline activator solution (AAS) [1-6]. Compared to ordinary Portland cement (OPC), geopolymer materials are known for their excellent properties, such as comparable or better mechanical properties [3], thermal and chemical stability [4], and impact resistance [7], and are considered as potential alternatives to OPC [8]. In particular, waste materials that are not currently reused in other industrial sectors can be used as precursors to geopolymer materials. Therefore, geopolymers have received increasing attention among those trying to reduce the consumption of natural raw materials and their environmental impact.

Fly ash is a by-product derived from the combustion of coal powder in thermal power plants. It becomes an important material for geopolymeric precursor due to its containing suitable components of amorphous silica and alumina [4, 5]. Therefore, fly ash-based geopolymer products have attracted much interest and studies have been undertaken therewith in recent years [9-18]. In the fabrication of geopolymers, a strong alkaline medium is necessary to increase the dissolution of silica and alumina in precursor materials, in which, alkaline solutions such as sodium hydroxide, and sodium silicate, are most

commonly adopted. Fernández-Jiménez and Palomo [13] investigated the effects of NaOH content on the properties thereof: at NaOH contents of 5, 6, 7, 8, and 10% (by mass of binders), compressive strengths of 9 MPa, 21 MPa, 30 MPa, 31 MPa, and 50 MPa at 28 days ensued, respectively. Palomo et al. [14] also observed that the concentration of the activator affected the compressive strength and stiffness of fly ash-based geopolymers (other results also confirm this [15-17]): however, some authors reported that the ratios of SiO₂/Na₂O or Al₂O₃/Na₂O exerted greater influences on the strength than NaOH content alone [10, 13, 17]. Palomo et al. [14] investigated an alkaline activated fly ash mortar: the compressive strength reached almost 70 MPa at SiO₂/Al₂O₃ and Al₂O₃/Na₂O ratios of 3.95 and 2.62. As the SiO₂/Al₂O₃ and Al₂O₃/Na₂O ratios were changed to 3.87 and 3.13, the strength decreased to 54.5 MPa. Ryu et al. [18] also examined the effects of SiO₂/Al₂O₃ and Al₂O₃/Na₂O ratios in fly ash-based alkaline activated pastes. At SiO₂/Al₂O₃ ratio of 4.13, the compressive strength was reduced with the increase of Al₂O₃/Na₂O ratio.

Besides, the water content also played crucial roles in the reactivity and strength development of the FGPs [2, 13, 19-23]. As the W/B ratio was 0.47, the compressive strength was 50 MPa. When the ratio decreased to 0.42, 0.31 and 0.26, the strength increased to 63.5 MPa, 82.5 MPa and 96.0 MPa, respectively [3], indicating that the rises of water content greatly decreased the strength. Fang et al. [23] observed that a large portion

*Corresponding author:
Tel : +86 131 53128675
Fax: +86 531 86367282
E-mail: tqb11@sdjzu.edu.cn

of water was entrapped in discontinuous gel pores, this might be the cause to impair the strength of the paste. In addition, the effects of the curing processes on the properties of FGP has been generally recognised. Palomo et al. [14] studied a FGP cured at 65 °C and 85 °C respectively for 2-24 h. The compressive strength was 30 MPa in the binder cured at 65 °C for 5 h; while as cured at 85 °C for only 2 h, the compressive strength could arrived at 31.6 MPa. As the curing time attained 24 h, the strength of 68.7 MPa would reached in the binders cured at 85 °C. In another FGP, 28-day compressive strength was 24.4 MPa in the binder cured for 24 h at 40 °C; it increased by 96.7%, 104.1% and 145.8% as the curing temperature rose to 70 °C, 80 °C and 100 °C, respectively [16]. However, it was found that there was a crucial curing temperature and time in the geopolymerisation of FGPs and the optimal curing processes varied with different FGP systems [15, 24, 25]. In fact, all parameters, including the concentration of the activator, the SiO₂/Na₂O or Al₂O₃/Na₂O ratio, the water content, and pH, and the curing processes, seem to play roles in the synthesis of FGPs. However, the influences thereof are complex and it is difficult to predict which one is more important [13, 17, 21, 26]. In addition, the optimum conditions are different in fly ash based geopolymers due to the differences in fly ash resource and its characteristics [5, 20, 27-30]. Here, a fly ash with high iron content was used as the precursor to fabricate geopolymer pastes. The roles of the contents of the alkaline activator, the ratios of SiO₂/Al₂O₃, Al₂O₃/Na₂O, and W/B, especially the effects of OH⁻ concentration, on the properties and microstructure of FGPs are discussed.

Materials and Methods

Precursor material properties

The fly ash used in the study was supplied from Guizhou Alumina Corporation, Guiyang, China. The chemical composition of the fly ash, obtained by X-ray fluorescence (XRF) on a ZSX Primus II X-ray spectrometer, is summarised in Table 1. The reactive amounts in fly ash, which were estimated by a combined dissolution of fly ash in NaOH solution by following extraction with HCl solution [31], are also listed in Table 1. The fly ash was found to have 47.8% amorphous material, comprising 23.7% SiO₂, 13.2% Al₂O₃, 8.1% Fe₂O₃, 1.4% CaO, and 1.4% other elements.

Preparation of the AAS

Analytical sodium hydroxide pellets (99 wt %) were dissolved in distilled water, and then mixed with glass-

water solution (30.4% SiO₂, 9.6% Na₂O, and 60.0% H₂O by mass). Three different moduli (M_s) of SiO₂/Na₂O (mol) were adopted to explore the effect of M_s on the geopolymer pastes. The mix proportions of sodium hydroxide and water-glass solution for the AAS are summarised in Table 2.

Preparation of the FGPs

The pastes were prepared by stirring the fly ash and AAS together. The equivalent sodium oxide content in the AAS was set to 6, 8, 10, and 12% by mass of the fly ash binder, respectively. The appropriate amount of water was added to adjust the W/B ratios to 0.38, 0.49, and 0.57. After being stirred thoroughly, the pastes were cast in 40 mm × 40 mm × 160 mm metal moulds, vibratory compacted, wrapped in thin plastic film and cured for 24 h at 80 °C, then cured under standard curing conditions. The curing temperature of 80 °C was selected as it provided the required energy guaranteeing the reaction between the fly ash and activating solution [30,32]. The mix proportions of AAS, extra water, the molar ratios of SiO₂/Al₂O₃, Al₂O₃/Na₂O, and the OH⁻ concentration for preparing FGPs are summarised in Table 3.

Characteristic material properties

The compressive strength of FGPs was determined using a SANS machine (MTS CDT 1305-2) at a maximum load of 300 kN. The average of three measurements was calculated. The images of the FGPs were captured by scanning electron microscopy (SEM: Hitachi SU8010, Japan) method on a fractured section and a thin film of Au was sputter-coated onto the specimen surface before observation. The mineral phases of fly ash and alkaline activated binders were identified by X-ray diffraction (XRD: Bruker D8 ADVANCE, Germany) on powdered samples.

Results and Discussion

Fig. 1 shows XRD patterns of fly ash and geopolymer pastes developing at various Na₂O contents and values of M_s . The fly ash shows different mineral peaks

Table 2. Mix proportions of sodium hydroxide and water-glass for the AAS and its properties

	Sodium hydroxide/g	Water-glass/g	Water content/%	Density/ g·mL ⁻¹	[OH ⁻]/M
$M_s = 1.0$	28.0	100	52.2	1.38	10.21
$M_s = 1.25$	19.9	100	54.1	1.49	8.13
$M_s = 1.5$	14.6	100	55.7	1.53	7.20

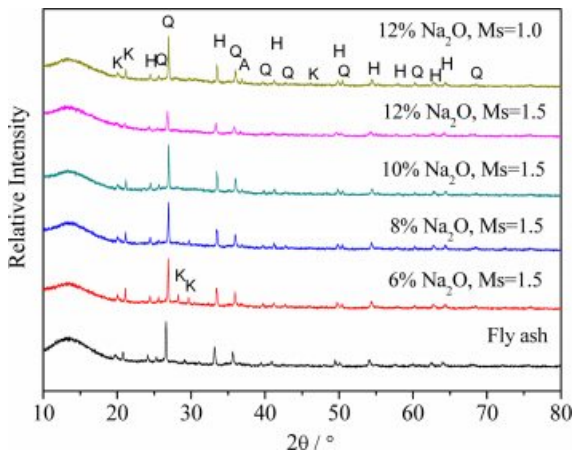
Table 1. Chemical composition of fly ash (wt. %).

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	TiO ₂	K ₂ O	MgO	Na ₂ O	Other	LOI	Total
Fly ash (raw)	38.3	20.7	20.9	1.6	2.2	1.7	0.7	0.7	1.2	12.0	100.0
Fly ash (amorphous)	23.7	13.2	8.1	1.4	-	-	-	-	1.4	-	47.8

Table 3. Mix proportions for fly ash-based binder pastes and their characteristics.

Na ₂ O content/ % ^{\$}	W*/B = 0.38				W/B = 0.47				W/B = 0.59			
	6	8	10	12	6	8	10	12	6	8	10	12
<i>M_s</i> = 1.0												
AAS/ % ^{\$}	24.7	32.9	41.2	49.4	24.7	32.9	41.2	49.4	24.7	32.9	41.2	49.4
EW [#] / % ^{\$}	25.1	20.8	16.5	12.2	34.1	29.8	25.5	21.2	46.1	41.8	37.5	33.2
SiO ₂ /Al ₂ O ₃	3.83	4.08	4.33	4.58	3.83	4.08	4.33	4.58	3.83	4.08	4.33	4.58
Al ₂ O ₃ /Na ₂ O	1.33	0.99	0.79	0.66	1.33	0.99	0.79	0.66	1.33	0.99	0.79	0.66
[OH ⁻]/ M ^{&}	4.30	4.78	6.57	7.57	3.51	4.42	5.60	5.17	2.79	4.08	4.56	5.27
<i>M_s</i> = 1.25												
AAS/ %	29.0	38.7	48.4	58.0	29.0	38.7	48.4	58.0	29.0	38.7	48.4	58.0
EW/ %	22.1	16.8	11.5	6.3	31.1	25.8	20.5	15.3	43.1	37.8	32.5	27.3
SiO ₂ /Al ₂ O ₃	4.02	4.34	4.66	4.97	4.02	4.34	4.66	4.97	4.02	4.34	4.66	4.97
Al ₂ O ₃ /Na ₂ O	1.33	0.99	0.79	0.66	1.33	0.99	0.79	0.66	1.33	0.99	0.79	0.66
[OH ⁻]/ M	3.89	4.95	6.07	7.53	3.26	4.15	4.99	5.88	2.65	3.43	4.13	4.80
<i>M_s</i> = 1.5												
AAS/ %	33.1	44.2	55.2	66.3	33.1	44.2	55.2	66.3	33.1	44.2	55.2	66.3
EW/ %	19.6	13.4	7.3	1.1	28.6	22.4	16.3	10.1	40.6	34.4	28.3	22.1
SiO ₂ /Al ₂ O ₃	4.07	4.58	4.95	5.33	4.07	4.58	4.95	5.33	4.07	4.58	4.95	5.33
Al ₂ O ₃ /Na ₂ O	1.33	0.99	0.79	0.66	1.33	0.99	0.79	0.66	1.33	0.99	0.79	0.66
[OH ⁻]/ M	3.98	4.61	6.10	6.82	3.36	4.28	4.24	5.26	2.78	3.72	4.12	4.93

Note: \$, percentage relative to fly ash; #, Extra water; *, Water includes water from water-glass and extra water; &, OH⁻ concentration was measured by acid-base titration.

**Fig. 1.** XRD patterns of fly ash and geopolymer pastes at various conditions.

related to the presence of quartz (Q, SiO₂), hematite (H, Fe₂O₃), and kyanite (K, Al₂SiO₅) mineral phases. Besides, the broad diffuse hump at about 10-20° indicates that it contains a certain amount of amorphous material [33]. After alkaline activation, the mineral phases in the pastes are not significantly altered from those found in the fly ash. The predominant phases in the fly ash are also found in all pastes, however, as the Na₂O content increased, the diffraction peaks of quartz, hematite, and kyanite gradually decreased in amplitude, indicating that the mineral phases present in the fly ash were reacting with AAS. In particular, diminished peak intensities in the XRD spectra are seen at a Na₂O content of 12% and *M_s* = 1.5. Meanwhile, an albite

phase (A, NaAlSi₃O₈) is gradually formed in the alkaline activated pastes. This albite phase is associated with the strong Na-Al-Si hydrated bonds which can be considered to play major effects to the strength of such geopolymers [34]. In addition, a slight shift in the peak for the binder was observed, implying that a new amorphous phase formed [19]. When alkaline activator solution was added, Na⁺ was accommodated within the cavities of -Si-O-Al- networks as a charge balancer to form sodium aluminosilicate gel (N-A-S-H). The formation of the gel also contributed to the development of compressive strength [35-38].

The effects of Na₂O percentage to binder, W/B ratios, and *M_s* on the compressive strength of FGPs are shown in Fig. 2. As *M_s* is 1.0 (Fig. 2(a)), the strength at 28 days is negligible in pastes with 6.0% Na₂O content (2.1 MPa) at a W/B ratio of 0.38. As the Na₂O content was increased, the strength gradually developed at 28 days for the pastes tested here. Compressive strengths of 10.1 MPa and 19.2 MPa in the paste samples with Na₂O contents of 8% and 10% were achieved (increased 3.8 and 8.1 times, respectively, compared with that at 6% Na₂O content). When the W/B ratios were increased to 0.47 and 0.59, a similar trend was observed. The compressive strength of pastes was low at low Na₂O content, and increased therewith; however, the difference was also observed in that the strength decreased with increasing W/B ratios at the same Na₂O content. At a Na₂O content of 8%, the strength decreased to 5.3 MPa and to almost zero from 10.1 MPa, when the W/B ratio was increased to 0.47 and 0.59 from 0.38. As the Na₂O content was 10%, the compressive strength of the paste

was decreased by 43.4% and 58.3% when the W/B ratio was increased to 0.47 and 0.59, respectively.

The alkaline activation of fly ashes leads to the formation of an alkaline aluminosilicate of amorphous nature[39]. Alkali activator solutions play an important role to dissolve Si and Al oxides in fly ash. In a low alkaline activator solution, the silica and alumina are filtered less [40] and a weak chemical reaction will occur [13, 26, 28, 39, 41, 42]. It is undoubtedly that a low compressive strength would be attained in a low-alkalinity solution. With the increase in Na₂O content, the higher reactivity of the fly ash would be expected [21]. Thus, a higher compressive strength was anticipated. It has been reported that there is a threshold alkaline activator content [7, 15, 20, 41]. Bakharev [15] reported that the strength developed rapidly at 8% Na₂O concentration in a geopolymer material. Cho et al. [16] observed that the strength at 28 days increased as the Na₂O content increased from 4.0% to 6.0%; however, when Na₂O content further increased beyond 6.0%, the rate of strength development decreased. Steveson et al. [35] found that the optimum compressive strength of

geopolymer materials could be achieved when the Na₂O content in AAS is between 7% and 9% by mass of fly ash. From our experimental results, an 8%-10% Na₂O content in the fly ash was a critical value range able to activate the fly ash efficiently. As the Na₂O content was less than 8%, regardless of any adjustment of M_s and W/B ratio, the compressive strength of the pastes did not exceed 10 MPa (Figs. 2(a)-(c)).

As the Na₂O content was further increased to 12%, no obvious increase in the strength, and even a decrease therein, was observed: this may have been caused by free OH⁻ present in the alkali-activated matrix [27] (as also observed elsewhere [26, 35, 41]). Table 3 also lists the OH⁻ concentration changes of the AAS at various W/B ratios, Na₂O contents, and M_s. The W/B ratios, Na₂O contents, and M_s exerted significant influences on the OH⁻ concentration. With the increase in W/B ratios, the water content increases and dilutes OH⁻ at the same M_s and Na₂O content so the OH⁻ concentration decreased accordingly. On the contrary, the OH⁻ concentration increased with increasing Na₂O content. The influences of M_s on the OH⁻ concentration are

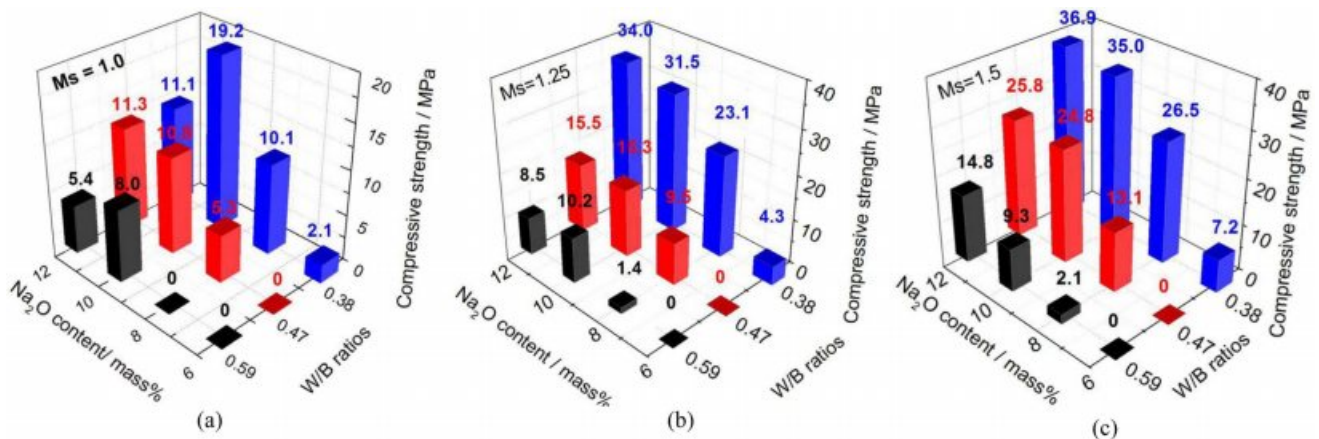


Fig. 2. Influences of Na₂O content, W/B ratio and M_s on compressive strength of FGAs (a) M_s = 1.0, (b) M_s = 1.25 and (c) M_s = 1.5.

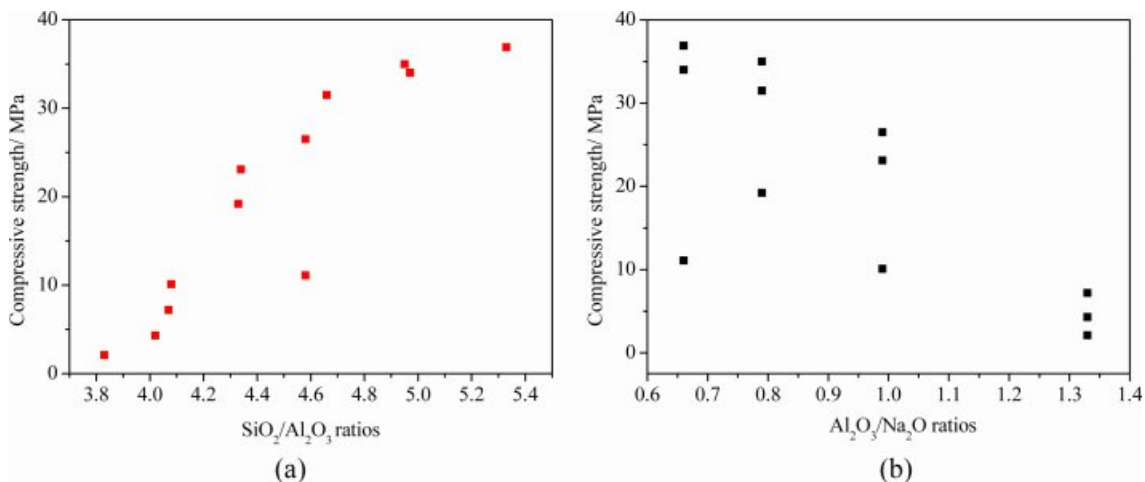


Fig. 3. Influence of SiO₂/Al₂O₃ and Al₂O₃/Na₂O ratios on the compressive strength at W/B=0.38.

mutual: on one hand, more NaOH was needed to obtain an AAS with lower M_s (Table 2), thus, with a larger M_s in the AAS, a lower NaOH content resulted, causing the decrease in OH^- concentration. On the other hand, as water-glass was mixed with NaOH, more NaOH was generated through the hydrolysis of sodium silicate, further increasing the OH^- concentration [13, 18, 43] (Table 3).

From the results (Fig. 2), the W/B ratios played an important role as described elsewhere [12-14, 42]. Palomo et al. [14] note that the role of the added water must be taken into account when investigating the properties of a geopolymer material. Firstly, enough water was necessary to ensure the workability of the mixtures [11], or a high strength was difficult to achieve: however, when excess water was added, there was sufficient mobility of ions in the mixtures for the solid to dissolve, causing a low level of unreacted material and a low strength [9]. This is attributed to the increase of porosity in the binder matrix [35].

At M_s of 1.25 or 1.5, similar trends in the compressive strength of the pastes along with the Na_2O content and W/B ratio were observed (Figs. 2(b) and 2(c)). That is: the compressive strength increased with increasing Na_2O content (relative to fly ash) and decreased with increasing W/B ratio. The compressive strength increased at larger M_s at the same Na_2O content and W/B ratio. At M_s of 1.25 and 1.5, the compressive strength increased to 31.5 MPa and 35.0 MPa from 19.2 MPa, respectively, with 10% Na_2O content and a W/B ratio of 0.38; while, the strength increased by 27.6% and 46.4%, respectively, with 8% Na_2O content and a W/B ratio of 0.38. The maximum strength of 36.9 MPa was achieved in that geopolymer paste with 12% Na_2O content at $M_s = 1.5$.

In fact, the changes in M_s and Na_2O content also changed the $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratios, which are important parameters in geopolymer synthesis [18]. Fig. 3 plots the results of the effects of the $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratios on the compressive strength of pastes at a W/B ratio of 0.38. The strength increased as the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio was increased from 3.83 to 5.33 (Fig. 3(a)): because the active SiO_2 content was relatively low (23.7%) in the fly ash used in the present study, the larger M_s , led to the higher SiO_2 content in the AAS and the binder matrix, thus increasing the compressive strength of fly ash-based geopolymers. The strength values were scattered with the change in $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratio, however, a high strength could be attained only with an $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratio of less than 1.0 (Fig. 3(b)). According to these results, the strength at 28 days is improved by more than 1.6 times at $\text{SiO}_2/\text{Al}_2\text{O}_3 = 4.58$, compared to that at $\text{SiO}_2/\text{Al}_2\text{O}_3 = 4.08$ at the same $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratio of 0.99; while, a strength greater than 30 MPa at 28 days could be achieved with an $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratio of less than 0.80. These indicated the roles of $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratios to strength development in the geopolymers.

The $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}$ ratios cannot reflect the role of water on the compressive strength, as they were kept constant as the W/B ratio was changed (Table 3). The OH^- concentration, which reflected the comprehensive effects of parameters such as the Na_2O content, W/B ratio, and M_s , represented parameter that could be used to characterise the compressive strength of FGA. From Table 3, it can be seen that OH^- concentration are influenced by the W/B ratio, Na_2O content, and M_s . The variation in OH^- concentration, which influences the chemical reaction of AAS with fly ash, would affect the final strength of these geopolymers as also demonstrated by our results. Considering the mutual effects of W/B ratio and Na_2O content, the influences of OH^- concentration on the compressive strength were as shown in Fig. 4. Regardless of the effects of M_s , the compressive strength could not be detected as the OH^- concentration was initially below its critical threshold value: it then generally increased at greater OH^- concentrations, and finally decreased at an excessive OH^- concentration. The critical values of OH^- concentration inducing the turning point in the compressive strength were similar, although the strength varied with M_s . When the OH^- concentration was less than 4.5 mol/L, it could not induce an efficient reaction, thus a low-strength product ensued. From the SEM image of the sample with $[\text{OH}^-]$ of 4.30, the unreacted fly ash particles were visible (Fig. 5). In addition, the presence of voids and cracks along the surface of fly ash particle was found, which may have impaired the compressive strength (Fig. 5(a)). At an OH^- concentration greater than 7.0 mol/L, a high strength could not be reached due to the free OH^- in the pastes, causing efflorescence from the binder [44]. An OH^- concentration of between 4.5-7.0 mol/L was able to induce the chemical reaction so as to produce a high strength product using the fly ash system adopted here [13, 18, 43]. From the microstructure as imaged

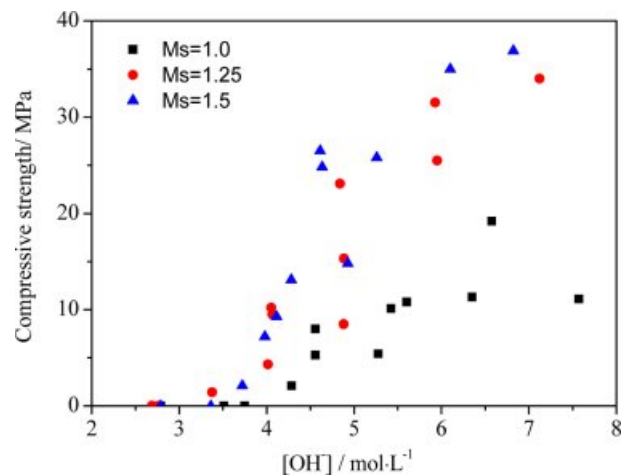
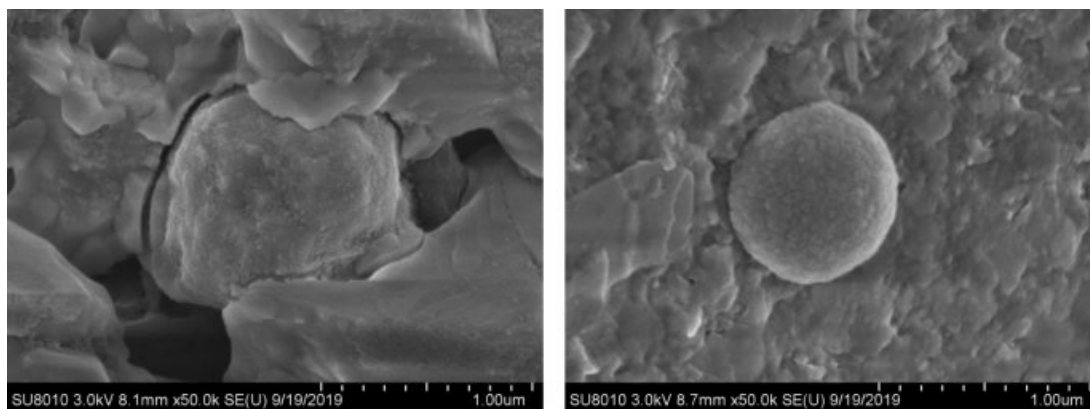


Fig. 4. Effects of $[\text{OH}^-]$ in AAS on the compressive strength of pastes.

(a) 6% Na₂O, W/B = 0.38, M_s = 1.0(b) 12% Na₂O, W/B = 0.38, M_s = 1.5**Fig. 5.** SEM image of paste with different [OH⁻] concentrations of (a) 4.30 and (b) 6.82.

under an SEM (Fig. 5(b)), the material was less void and more well development, compared with that seen in Fig. 5(a). This also indicated the higher degree of polymerisation and the presence of N-A-S-H gel [33], as evinced by XRD results. The compressive strength of geopolymer pastes at M_s = 1.5 was higher than that at M_s values of 1.25 and 1.0 at the same OH⁻ concentration: this may have been caused by the higher SiO₂/Al₂O₃ ratio (Table 3).

Conclusion

The influences of variables, such as Na₂O content, W/B ratio, and M_s of alkaline activator solution, on the compressive strength of a FGA were investigated. There was a critical value of the OH⁻ concentration of the alkaline activator required for optimal compressive strength development, as influenced by the W/B ratio, M_s, and Na₂O content. At an OH⁻ concentration of less than 4.5 mol/L, the compressive strength was less than 20 MPa due to the low OH⁻ concentration having induced a low reaction of fly ash with alkaline activator: however, at OH⁻ concentrations greater than 7.0 mol/L, free OH⁻ was present in the binder, and the compressive strength reached a plateau or decreased. Only at an OH⁻ concentration of between 4.5-7.0 mol/L, could the compressive strength have reached between 20.0-36.0 MPa. The ratios of SiO₂/Al₂O₃ and Al₂O₃/Na₂O affected the compressive strength. According to the results, the increase in SiO₂/Al₂O₃ values over the range of 3.83-5.33 promoted the increase of compressive strength; whilst, it was possible to achieve a high strength only when the Al₂O₃/Na₂O ratio was less than 1.0. As the alkali content was increased to 12% (relative to the mass to fly ash), the reaction of the mineral phases in the fly ash with the alkali activator was strengthened, which induced the formation of N-A-S-H gel, thus increasing the compressive strength.

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