

Effect of NaOH Concentration on the Strength of Non Sodium Silicate Fly Ash Geopolymer

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Abstract. The current work presents the effect of alkali concentration on fly ash based geopolymer material. In this work geopolymer material was prepared with a fixed Si/Al ratio (1.85: 1) and varied solid to water ratio and NaOH concentration. All samples were tested to establish the role of alkali on the strength. Samples prepared with 8M of NaOH showed the highest compressive strength. In this regards, Fourier transform infrared spectroscopy (FTIR) results support this observation where broad band becomes sharper and shifts towards lower wavenumbers. In XRD the broad hump slightly shifted towards higher values which indicate the formation of new product. These findings show that to obtain required processing time at desired mechanical strength for a specific application of geopolymer, the suitable concentration of alkali must be established.

Introduction

Geopolymer is a new materials with the microstructure of amorphous to semi-crystalline three dimensional aluminosilicate [1]. It is produced through a polymerization reaction between various aluminosilicate oxides with silicates solution or alkali hydroxide solution forming polymerized-Si – O – Al – O- bonds. The development of geopolymers offers green alternative material which acts as a binding agent in concrete or coating material. During polymerization process, the synthesis parameters such as Si/Al ratio, solid/water ratio, alkali concentration, curing temperature and curing time determine the final properties of geopolymer material [2].

There are three possible reaction mechanisms that may take place to produce silicate and aluminate species in the formation of geopolymer material. The polymerization mechanism consists of dissolution, hydrolysis and polycondensation steps. The first two steps, dissolution and hydrolysis, take place simultaneously in alkaline condition [3]. In dissolution, step silicate and aluminate convert into ions (Si^{+4} and Al^{+3}) and in hydrolysis these ionic species react with water. Then species react with each other and undergo a polycondensation reaction to form a polymer network [3]. So far the effects of NaOH concentration on the physical and mechanical properties of concrete are reported, where geopolymer is used only as a binder in the presence of sand and aggregates.

In this paper our interest is to examine the properties of different concentrations of NaOH although, so much work is done on the different concentration of NaOH, but this paper showed with a higher solid to water ratio as compared to the reported literature.

Experimental

Materials and Methods Fly ash was obtained from nearby coal power station and sodium hydroxide (Laboratory grade) 99 % pure, was supplied by R and M chemicals. The chemical composition of fly ash is given in Table 1.

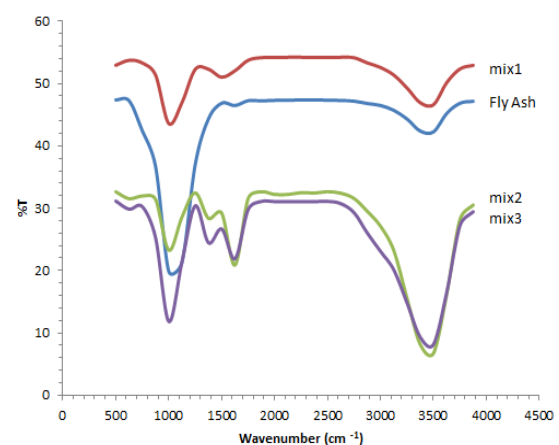
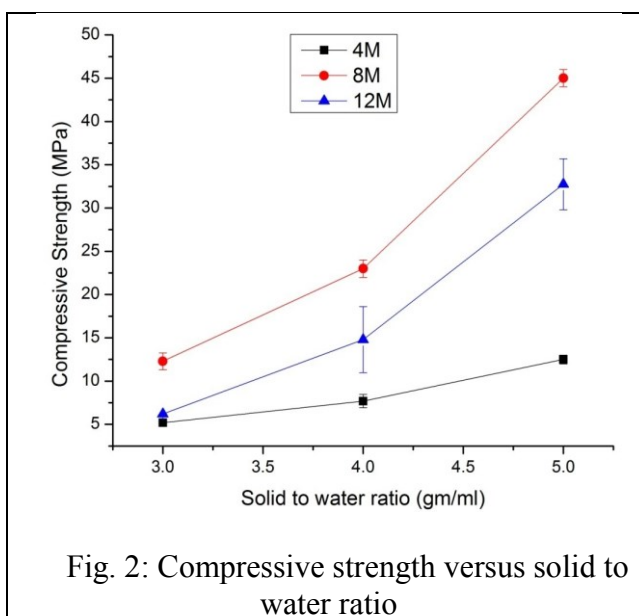
Table 1: XRF data analysis of fly ash

SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	CaO (%)	MgO (%)	K ₂ O (%)	SO ₃ (%)	Na ₂ O (%)	TiO ₂ (%)	P ₂ O ₅ (%)	BaO (%)	SrO (%)	MnO (%)
43.34	20.77	12.41	11.13	3.75	1.98	1.45	0.95	0.88	0.32	0.17	0.12	0.11

Method of mixing/geopolymer synthesis In the preparation of NaOH solution, NaOH pellets was dissolved in distilled water in a volumetric flask for 3 different concentration 4M, 8M, and 12M and the NaOH solution prepare at least before one day. The Si/Al ratio is fixed (1.85:1) for all experiments whereas solid to water ratio of 3, 4, 5 were maintained for geopolymer synthesis. Fly ash and NaOH were mixed in the mixer for 4 minutes. Then geopolymer paste was poured into a vacate mold, for setting time (ASTMC191), and the remaining paste was poured in 50 x 50 x 50 mm cube mold compressive strength (ASTM C109) and cured the mold at room temperature and 60 °C for 1 day, 7 days, and 28 days.

Result and Discussions:

Effects of NaOH concentration on compressive strength The compressive strength of synthesized geopolymer specimens at three different NaOH concentrations and three different solid/water ratios are shown in Fig. 2. The results of compressive strength (S/W 5:1) shows that 8M NaOH concentration gives high compressive strength (45 MPa) as compared to 4M (12.5MPa) and 12M (32.7MPa). At low concentration (4M) of NaOH, the dissolution was low, less leaching of Si⁺⁴ and Al⁺³ took place and produced a material which had low compressive strength, whereas the drop in compressive strength at high concentration (12M) could be due to the large amount of Na⁺ ion present in the cavities which prevent the formation of complete networks. Hence, the defect structure could be generated due the excess amount of Na⁺ ion. A similar observation is reported where the strength is decreased for the sample prepared above 10M NaOH [5]. The drop in strength is attributed to the lower degree of geopolymerization.



FTIR Results The FTIR results of fly ash and alkali activated fly ash after 28 days of curing at room temperature are shown in Fig. 5, the difference observed between FTIR spectra of fly ash and spectra of geopolymeric material concerning the band attributed to the asymmetric stretching

vibrations of Si – O – Si and Al – O – Si [6]. This band appeared in FTIR spectra of fly ash at 1082.36 cm^{-1} and becomes sharper and shifts to lower frequencies [7, 8], which indicate the formation of a new product (amorphous aluminosilicate gel phase), but the intensity of these peaks does not depend on the degree of crystallization [9]. The sample with 8M of NaOH (mix 2) shows largest shift in magnitude i.e. from 1082.36 to 985.19 cm^{-1} compared to samples of 4M (mix1) and 12M (mix3) 993 cm^{-1} and 995 cm^{-1} , respectively. These results also favor the compressive strength results which show that sample prepared at 8M NaOH gives the highest strength.

XRD Results The XRD analysis was used to confirm the crystalline and amorphous character of the phases formed during the alkali activation. Fig 4a and 4b shows the XRD patterns of original fly ash and geopolymeric material (mix1, mix2 and mix3). It can be seen that the major crystalline peaks originally existing in fly ash are also present in the synthesized geopolymer which indicate that there is a presence of undissolved fly ash particles in the geopolymeric material and also indicating that the crystalline phases are also not involved in the geopolymerization, but present in the form of inactive fillers [10].

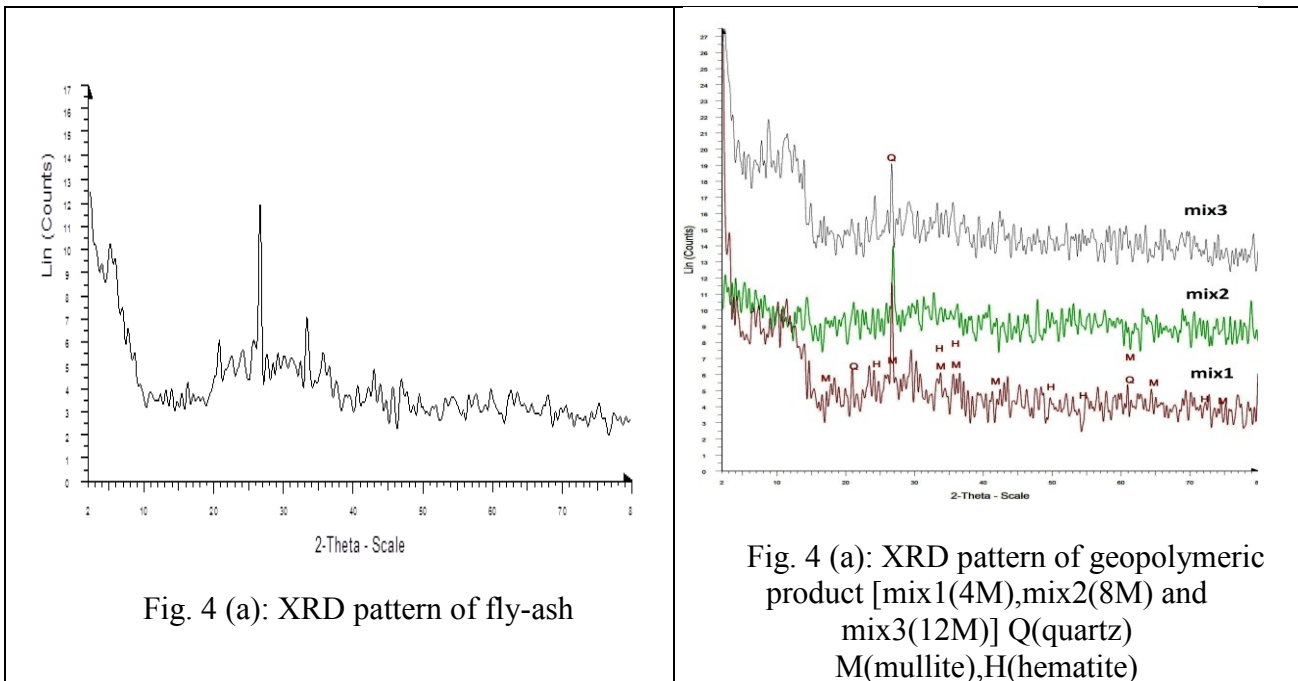


Fig. 4 (a): XRD pattern of geopolymeric product [mix1(4M),mix2(8M) and mix3(12M)] Q(quartz) M(mullite),H(hematite)

Amorphous phases in raw material are reactive and involved in geopolymerization reactions [11, 12]. Fly ash shows a broad hump with a few sharp peaks indicating that amorphous phases (alumina) are dominant at large quantities and crystalline phases (silica) are found in small quantities. By comparing the XRD pattern of fly ash and geopolymeric material, the broad hump registered between $2\Theta=20^\circ$ and 30° attributed to the amorphous phase of fly ash, is slightly shifted towards higher values ($2\Theta=25^\circ$ - 35°) indicating the dissolution of fly ash and the formation of new amorphous phase [13, 14].

Conclusion Alkali concentration is proved to be crucial parameter. In this work, sample prepared with 8M of NaOH showed the highest compressive strength. FTIR results support this observation where broad band become sharper and shift towards lower wavenumber. The changes in wavenumber suggest that the formations of more order aluminosilicate networks are formed. From this study the slightly shifting of peak is detected as the 2Θ change from 25° to 35° which shows the formation of new amorphous phase.

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References

- [1] J. Davidovits and D. Comrie, "Long term durability of hazardous toxic and nuclear waste disposals," in 1st European Conference on Soft Mineralogy, Compiègne, France, 1988, pp. 125–134.
- [2] D. Khale and R. Chaudhary, "Mechanism of geopolymerization and factors influencing its development: a review," *Journal of Materials Science*, vol. 42, no. 3, pp. 729–746, Jan. 2007.
- [3] Van Jaarsveld, J. G. S., J. S. J. Van Deventer, and L. Lorenzen. "Factors affecting the immobilization of metals in geopolymerized fly ash." *Metallurgical and materials transactions B* 29.1 (1998): 283-291.
- [4] Perera, D. S., Uchida, O., Vance, E. R., & Finnie, K. S. "Influence of curing schedule on the integrity of geopolymers." *Journal of materials science* 42.9 (2007): 3099-3106.
- [5] Alonso, S., and A. Palomo. "Alkaline activation of metakaolin and calcium hydroxide mixtures: influence of temperature, activator concentration and solids ratio." *Materials Letters* 47.1 (2001): 55-62.
- [6] Oh, J. E., Monteiro, P. J., Jun, S. S., Choi, S., & Clark, S. M. "The evolution of strength and crystalline phases for alkali-activated ground blast furnace slag and fly ash-based geopolymers." *Cement and Concrete Research* 40.2 (2010): 189-196.
- [7] Kovalchuk, G., Ana Fernández-Jiménez, and A. Palomo. "Alkali-activated fly ash: Effect of thermal curing conditions on mechanical and microstructural development—Part II." *Fuel* 86.3 (2007): 315-322.
- [8] Swanepoel, J. C., and C. A. Strydom. "Utilisation of fly ash in a geopolymeric material." *Applied Geochemistry* 17.8 (2002): 1143-1148.
- [9] Bakharev, T. "Geopolymeric materials prepared using Class F fly ash and elevated temperature curing." *Cement and Concrete Research* 35.6 (2005): 1224-1232.
- [10] He, Jian, et al. "The strength and microstructure of two geopolymers derived from metakaolin and red mud-fly ash admixture: A comparative study." *Construction and Building Materials* 30 (2012): 80-91.
- [11] Zhang, Guoping, Jian He, and Robert P. Gambrell. "Synthesis, characterization, and mechanical properties of red mud-based geopolymers." *Transportation Research Record: Journal of the Transportation Research Board* 2167.1 (2010): 1-9.
- [12] Zhang, Shuzheng, Kecheng Gong, and Jianwen Lu. "Novel modification method for inorganic geopolymer by using water soluble organic polymers." *Materials Letters* 58.7 (2004): 1292-1296.
- [13] Fernández-Jiménez, Ana, and A. Palomo. "Composition and microstructure of alkali activated fly ash binder: effect of the activator." *Cement and Concrete Research* 35.10 (2005): 1984-1992.
- [14] Bakharev, T. "Geopolymeric materials prepared using Class F fly ash and elevated temperature curing." *Cement and Concrete Research* 35.6 (2005): 1224-1232.