Research Article

Influence of some additives on the properties of fly ash based geopolymer cement mortars

Mukesh Kumar[1] · **Sunil Kumar Saxena**[1,2] · **Nakshatra Bahadur Singh**[2,3]

Abstract
In order to minimize CO_2 emission due to manufacture of Portland cement, researches have been focused on alternative construction materials such as geopolymer cement. Geopolymer cement is made from waste materials such as fly ash by alkali-activation. This paper reports the properties of fly ash based geopolymer mortars activated by sodium hydroxide/potassium hydroxide and sodium silicate/lithium silicate. Alccofine powder, aluminum powder and calcined clay were added during geopolymerization. Curing was done at 80 °C. Compressive strength of geopolymer mortar was found maximum in the presence of potassium hydroxide–lithium silicate—5% alccofine powder—10% calcined clay. Durability of cubes in sulphuric acid was studied. Fire resistant properties of some of the mortars at 600, 800 and 1000 °C were also studied.

Keywords Geopolymer cement · Aluminum powder · Compressive strength · Alccofine powder · Calcined clay

1 Introduction

Nowadays, people are trying to minimize the use of cement since its production is energy intensive and its waste gases from cement production cause significant environmental problems, including large amount of CO_2 production. Geopolymer based cement and concrete may be a better alternative for sustainable concrete usage and can reduce CO_2 emissions.

Geopolymer binders can be produced from a variety of natural materials and industrial by-products like metakaolin, fly ash (FA), ground granulated blast-furnace slag, red mud, mine waste, etc. Davidovits discussed the origin and basic principles involved in the geopolymerization [1]. Singh et al. [2] and Zhang et al. [3] presented a comprehensive review of literature on gepolymers. Bignozzi et al. [4] described in detail the recycling of industrial wastes for the manufacture of binding materials. The combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) is generally used as an activator for high strength geopolymer cements [5, 6]. FA is one of the waste materials of thermal power plants and can easily be converted to geopolymer at moderate temperature [7]. About 200 Mt FA are produced in India every year with only 50% being utilized [8]. There is an urgent need to dispose of this FA so that the environment can be protected from pollution. One of the suitable ways is to develop geopolymer cement binder which can partially replace Portland cement. Further, the use of FA in the geopolymer preparation is important for economic reasons [9]. There are number of factors which affect the properties of geopolymer cements [10]. Alkali cations supplied by the alkaline solution influence the first stage of geopolymerization. This in turn affects the mechanical performance [11]. Provis [12] reported that sodium and potassium silicates and hydroxides are the most commonly used activating solutions. The effect of different combinations of alkali metal hydroxides and alkali metal silicates on the properties of geopolymer

✉ Nakshatra Bahadur Singh, nbsingh43@gmail.com | [1]Research and Development Centre, JK Lakshmi Cement Ltd., Jhajjar 124103, India. [2]Chemistry Department, School of Basic Sciences, Sharda University, Greater Noida 201306, India. [3]Research and Technology Development Centre, Sharda University, Greater Noida 201306, India.

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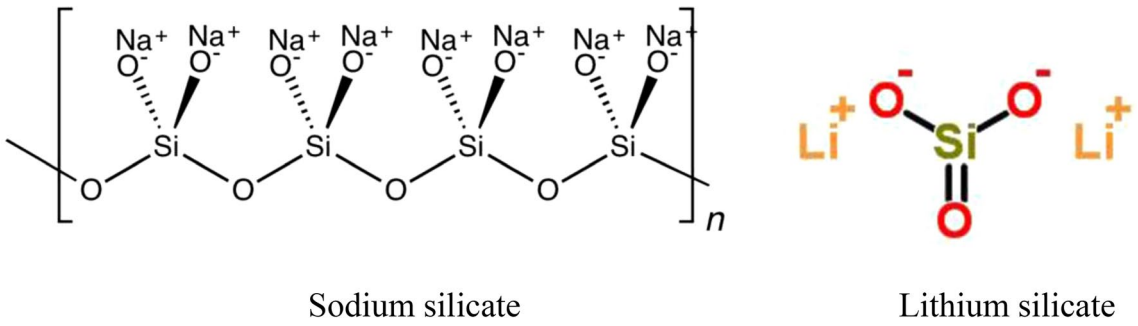


Fig. 1 Structures of sodium silicate and lithium silicate

Table 1 Physical properties

S. no.	Material	Sp. gravity	Blain surface area (m^2/kg)
1	OPC-53	3.5	305
2	Clay	2.84	591
3	Fly Ash	2.12	290

cements and mortars have not been studied in detail. The combination of different alkalis and silicates influencing the process of geopolymerization leading to changes in properties have not been understood well. In this paper FA-based geopolymer cement mortars have been prepared by using sodium silicate/lithium silicate in combination with sodium hydroxide/potassium hydroxide activators and the properties were studied in the presence of alccofine powder (AFP), aluminum powder (AP) and calcined clay (CC). Effect of high temperatures on compressive strength and durability in sulphuric acid (H_2SO_4) was examined.

2 Experimental details

2.1 Materials

N type Ordinary Portland cement-53 grade (OPC), FA, AFP (micromaterial, Ambuja Cement Ltd., Mumbai, India), AP and CC were used. NaOH, KOH, Na_2SiO_3 and lithium silicate (Li_2SiO_3) were used as alkali activators. The structures of sodium silicate and lithium silicate are given in Fig. 1. Polycarboxylate type superplasticizer (ATPL-401 PC) was used as an admixture. The physical properties and chemical composition of OPC and FA are given in Tables 1 and 2 respectively.

2.2 Experimental methods

2.2.1 Proportions in geopolymer cement mortar and casting

FA and fine aggregate (silica sand) mixed in the ratio of 1:2 (350 g:700 g) and a number of mixtures were made at room temperature. The mixture of Fly ash and fine aggregate was homogenized in Hobart mixer for 2 min. The dry homogenized mixture was then mixed with different concentrations of alkaline activator (NaOH/KOH (8, 10, 12 and 14 M) solutions along with Na_2SiO_3 and Li_2SiO_3 and 8% ATPL-401 PC type superplasticiser for another 1 min. The mixtures were put into moulds, which were then kept on Vibrating mortar machine at RPM 12,000 ± 400 for 2 min. Mixtures (Table 3) were transferred to $7.5 \times 7.5 \times 7.5$ cm^3 mould as per IS code 4031: 2014. Six mortar cubes were cast for each mix.

Table 2 Chemical composition of all the materials (mass %)

Constituents	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	IR*	LOI**
Portland cement	20.50	5.05	2.99	62.0	2.07	0.48	0.09	2.40	–	3.10
Fly Ash	61.39	24.42	4.42	3.75	1.05	0.21	0.74	0.08	–	1.05
Alccofine powder	32.84	22.00	2.50	36.10	4.00	0.34	0.74	0.30	0.69	0.49
Calcined Clay	61.93	22.95	3.22	10.12	0.00	0.11	0.01	0.02	0.44	1.20
Silica Sand (Course)	89.77	3.10	1.02	1.21	0.48	0.12	0.32	0.005	–	–

IR Insoluble residue, ***LOI* Loss on ignition

Table 3 Mix design of all the mortars (amount in grams)

Mix no	OPC	FA	Silica sand	NaOH	KOH	Sodium silicate	Lithium silicate	Admixture (SP) (%)	AFP	AP, CC
1	700	0	350	0	0		0	0.8	0	0
2	0	700	350	40	0	200	0	0.8	0	0
3	0	700	350	40	0	0	200	0.8	0	0
4	0	700	350	0	40	200	0	0.8	0	0
5	0	700	350	0	40	0	200	0.8	0	0
6	0	700	350	40	0	300	0	0.8	0	0
7	0	700	350	40	0	0	300	0.8	0	0
8	0	700	350	0	40	300	0	0.8	0	0
9	0	700	350	0	40	0	300	0.8	0	0
10	0	700	350	40	0	300	0	0.8	35 (5%)	
11	0	700	350	40	0	0	300	0.8	35 (5%)	
12	0	700	350	0	40	300	0	0.8	35 (5%)	
13	0	700	350	0	40	0	300	0.8	35 (5%)	
14	0	700	350	0	40	0	300	0.8	35 (5%)	35 (5% AP)
15	0	700	350	0	40	0	300	0.8	35 (5%)	70 (10% CC)
16	0	700	350	0	40	0	300	0.8	35 (5%)	35 (5% AP) + 70 (10% CC)

Fig. 2 Slump test apparatus

2.2.2 Workability test

The slump test measures the consistency and workability of fresh mortars. The slump cone (Fig. 2) is made of steel and plastic. The diameter of the base opening is 20 cm with top opening 10 cm. Slump cone test was done to determine the workability of the fresh mortars (BS EN 12350-2:2000 standard).

2.2.3 Curing and testing of geopolymer mortars

Demoulded mortar cubes (Mix 1–Mix 13) were cured at room temperature (32 °C), 60 and 80 °C for 12 h and the compressive strengths were determined with a compressive strength testing machine (Fig. 3).

The cubes (Mix 14–Mix 16) were heated at 600, 800 and 1000 °C for 2 h and after cooling at room temperature, compressive strengths were determined. Weight losses were also recorded.

2.2.4 Determination of density

Densities of Mix 14, 15 and 16 after heating at 80, 600, 800 and 1000 °C for 2 h were determined as per IS 4031 (Part 11)-1998 RA 2014 using Le- Chatelier Flask Method.

2.2.5 Scanning electron microscope (SEM)

SEM photographs of FA based geopolymer mortar (Mix 6) cured at room temperature and 80 °C were recorded. SEM pictures of others (Mix 14, 15 and 16) cured at 80 °C were also recorded.

Fig. 3 Compression testing machine

2.2.6 Durability in sulphuric acid

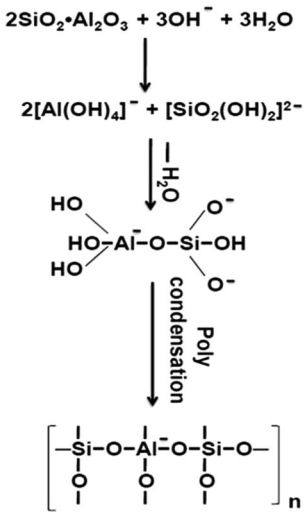
Cubes of Mix 14, 15 and 16 were immersed in 5% sulphuric acid for 24 h. Weight changes and compressive strengths were recorded.

3 Results and discussion

3.1 Fly ash activation and geopolymerization

The fly ash contains aluminosilicate which upon activation by alkalis is converted to geopolymer as shown in the following Scheme 1 [13, 14].

A model for geopolerization in the presence AFP, CC and AP can be represented by Fig. 4. FA in the dry state was mixed thoroughly with sand and then mixed with alkali hydroxide (NaOH/KOH) and silicate solution (Na_2SiO_3/Li_2SiO_3) and homogenized. The mixtures were cured at room temperature after adding (i) AFP, (ii) AFP + CC, (iii) AFP + AP and (iv) AFP + AP + CC. Geopolymer mortars of different properties were obtained.



Scheme 1 Geopolymerisation reaction

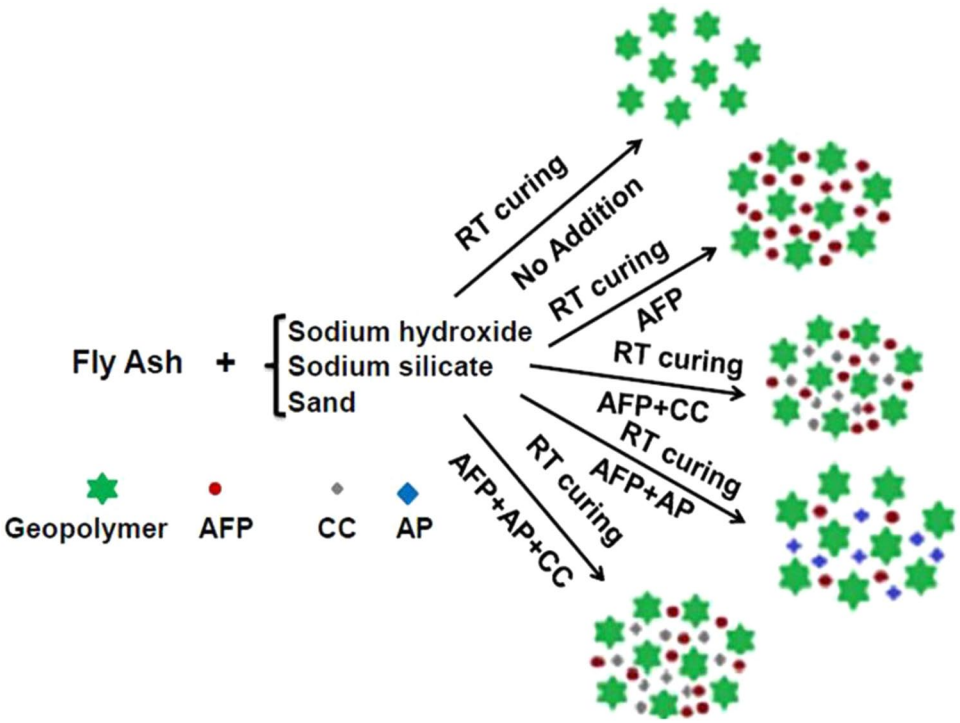


Fig. 4 Model for geopolerization

Table 4 Compressive strength of mortars cured at different temperatures (MPa)

Mix no	Compositions	Temperature (°C)					
		32	60	80	600	800	1000
1	OPC + silica sand	12.5	13.9	14.6			
2	FA + silica sand + 200 Na_2SiO_3 + 40 NaOH	7.4	10.4	10.5			
3	FA + silica sand + 200 Li_2SiO_3 + 40 NaOH	7.5	10.7	10.8			
4	FA + silica sand + 200 Na_2SiO_3 + 40 KOH	7.9	11.0	11.2			
5	FA + silica sand + 200 Li_2SiO_3 + 40 KOH	7.9	11.5	11.6			
6	FA + silica sand + 300 Na_2SiO_3 + 40 NaOH	9.5	21.0	22.5			
7	FA + silica sand + 300 Li_2SiO_3 + 40 NaOH	9.5	21.3	22.8			
8	FA + silica sand + 300 Na_2SiO_3 + 40 KOH	9.8	21.9	22.9			
9	FA + silica sand + 300 Li_2SiO_3 + 40 KOH	9.7	22.1	23.2			
10	FA + silica sand + 300 Na_2SiO_3 + 40NaOH + 5% AFP	10.4	28.6	29.9			
11	FA + silica sand + 300 Li_2SiO_3 + 40 NaOH + 5% AFP	10.5	29.3	32.6			
12	FA + silica sand + 300 Na_2SiO_3 + 40 KOH + 5% AFP	10.5	29.8	35.1			
13	FA + silica sand + 300 Li_2SiO_3 + 40 KOH + 5% AFP	11.3	32.8	38.7			
14	FA + silica sand + 300 Li_2SiO_3 + 40 KOH + 5% AFP + 5%AP	8.6	29.8	36.7	36.9	35.8	22.7 (Cracked)
15	FA + silica sand + 300 Li_2SiO_3 + 40 KOH + 5% AFP + 10%CC	13.1	32.4	39.7	40.8	40.7	40.1
16	FA + silica sand + 300 Li_2SiO_3 + 40 KOH + 5% AFP + 5% AP + 10%CC	9.9	29.2	31.5	31.8	29.9	28.9 (Cracked)

3.2 Compressive strength

The compressive strengths of different geopolymer mortars after 12 h curing at different temperatures (32, 60 and 80 °C) are given in Table 4. In order to know the fire resistance of the mixes, the Mixes 14–16 were also heated for 2 h at high temperatures i.e. at 600, 800 and 1000 °C as required for AAC blocks application and the compressive strengths are given in Table 4.

The compressive strengths of different geopolymer mortars containing 200 g Na_2SiO_3/Li_2SiO_3 and 14 M NaOH/KOH and the control at different temperatures (32, 60 and 80 °C) are shown in Fig. 5a. The results showed that the compressive strengths of the geopolymer mortars (Mix

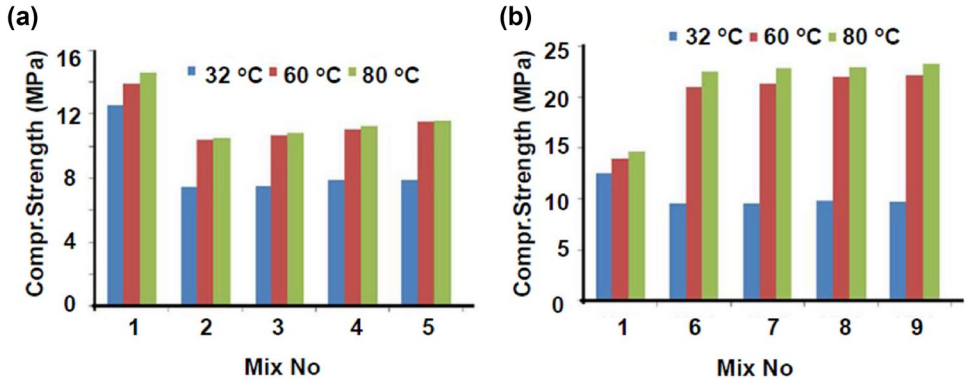


Fig. 5 **a** and **b** Compressive strength of control (Mix 1) and fly ash based geopolymer cement mortar Mixes at different temperatures after 12 h

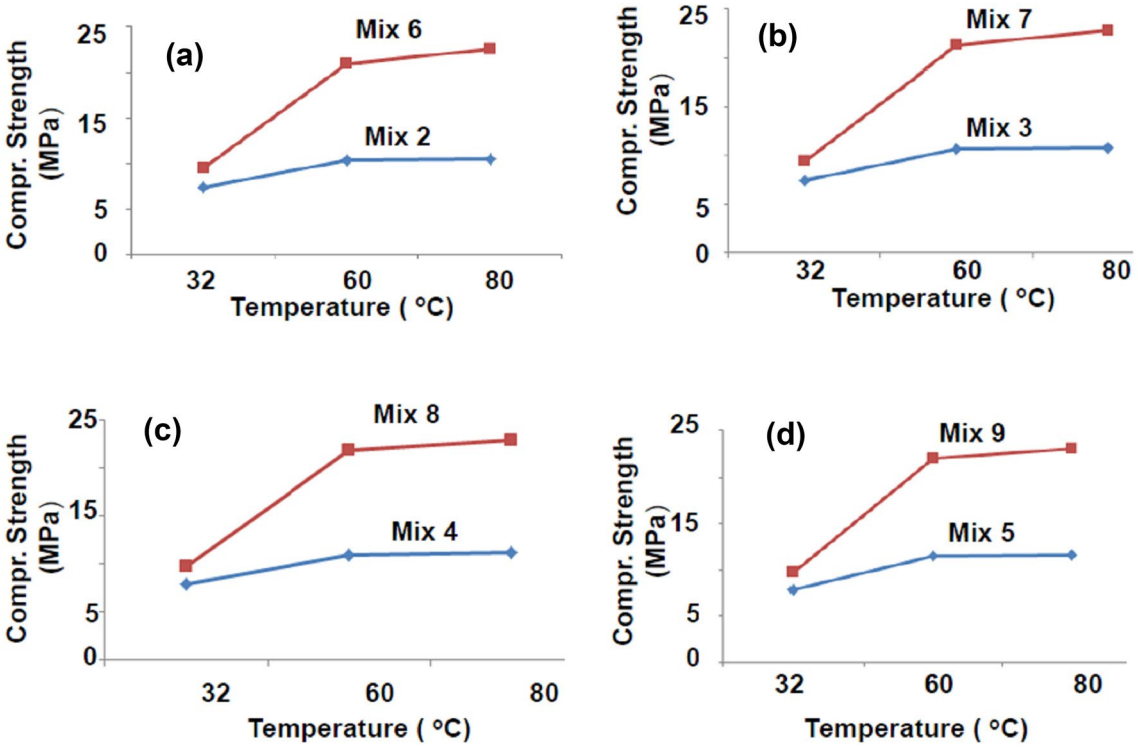


Fig. 6 Compressive strength of different geopolymer cement mortars at different temperatures

2–5) were lower than that of the control at all the curing temperatures. It did not matter whether silicate used was Na_2SiO_3 or Li_2SiO_3 and the alkali used was NaOH or KOH. The compressive strength of geopolymer mortars (Mix 6–9) containing 300 g Na_2SiO_3/Li_2SiO_3 and 14 M NaOH/KOH were higher than that of the control at 60 and 80 °C (Fig. 5b). It appears that at room temperature curing and in the presence of 200 g silicate solution, the geopolymerization process was incomplete and the strengths in all the cases were lower as compared to that of the control. Haidi et al. [15] reported that lower amounts of Si^{4+} and Na^+ affect adversely the formation of the coherent structure that consequently reduces the compressive strength. Even if the amount of silicate was higher (300 g), the geopolymerization was not complete at room temperature in 12 h. Thus with the increase of silicate concentration and curing temperature, the compressive strength increased in all the cases (Fig. 6). From Table 4 and Fig. 5b, it is apparent that the compressive strengths of the mortars are in the following sequence.

$$\begin{aligned}&\text{FA} + 300\ Li_2SiO_3 + 40\ \text{KOH} > \text{FA}\\&\quad + 300\ Na_2SiO_3 + 40\ \text{KOH} > \text{FA}\\&\quad + 300\ Li_2SiO_3 + 40\ \text{NaOH} > \text{FA}\\&\quad + 300\ Na_2SiO_3 + 40\ \text{NaOH}\end{aligned}$$

Lithium silicate has edge over sodium silicate in all the cases and combination of Li_2SiO_3 with KOH gave

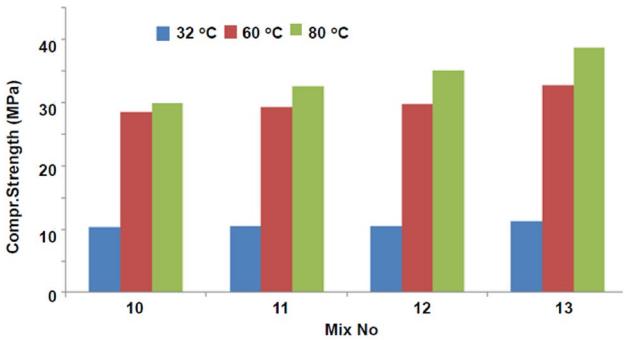


Fig. 7 Compressive strength of different Mixes in presence of alccofine powder at 32, 60 and 80 °C

comparatively higher compressive strength. With 5% addition of AFP in the geopolymer mortars, the compressive strengths increased with temperature and the value was quite high for Mix 13 at 80 °C (Fig. 7). Preliminary experiments in the presence of AFP have been done but the mechanism is not understood [16]. In the presence of silica fume, the compressive strengths are also increased [17] but AFP with lower cost yielded comparable compressive strength and can be used in place of silica fume.

pH in the range of 13–14 is most suitable for the formation of the geopolymers with better mechanical strength [18]. Generally, NaOH/KOH and Na_2SiO_3 have been used as alkali activator. It is reported that KOH because of larger size of K^+ favours the formation of geopolymers [19]. Lithium silicate solution with low viscosity increases the ionization of KOH giving more alkaline character. Thus Li_2SiO_3 in combination to KOH may enhance geopolymerization as in the present case. Further, it is already reported that AFP in geopolymer mortars enhances the compressive strength by increasing geopolymerization and partly entering into the pores [20]. Thus geopolymer mortar made from Li_2SiO_3 combined with KOH in the presence of AFP gives maximum strength.

The better performance of Li_2SiO_3 over Na_2SiO_3 may be due to smaller size of lithium ion or low solubility of Li_2SiO_3 or both. It is already reported that the use of several alkali ions (Na, K, and Cs) differing by their size and by their kosmotropic or chaotropic properties showed rapid dissolution of metakaolin and the rapid appearance of a rigid percolating network with a small alkali activator [21]. However, to understand the detailed role of different alkali metal ions during geopolymerization, a separate and detailed investigation is needed.

3.3 SEM studies

SEM picture of geopolymer (Mix 6) (Fig. 8a) cured at room temperature shows the presence of unreacted FA indicating incomplete geopolymerization. However, at 80 °C, probably higher degree of geopolymerization has taken place (Fig. 8b) and many fibrous materials are formed. Figure 8c shows the formation of fibrous needle shaped products. The size of the needles is about 35 nm. This situation arose because of higher curing temperature and higher concentration of silicate. The results showed that nanostructures are formed during geopolymerization.

3.4 Durability studies

Photos of geopolymer mortar cubes cured with wrap cover and without wrap cover are shown in Fig. 9. Sample (a) was cured at 80 °C for 12 h without polythene wrap, whereas sample (b) was cured with polythene wrap. In the sample (a), cracks were found and in sample (b), no crack was seen. The cubes when heated without polythene wrap, water came out and the cubes cracked. However, when the cubes were covered with polythene wrap and heated, no water could come out. As a result, no cracking occurred (Fig. 9b).

Since cube (FA + silica sand + 300 Li_2SiO_3 + 40 KOH + 5% AFP) (Mix 13) gave maximum strength after 12 h curing at

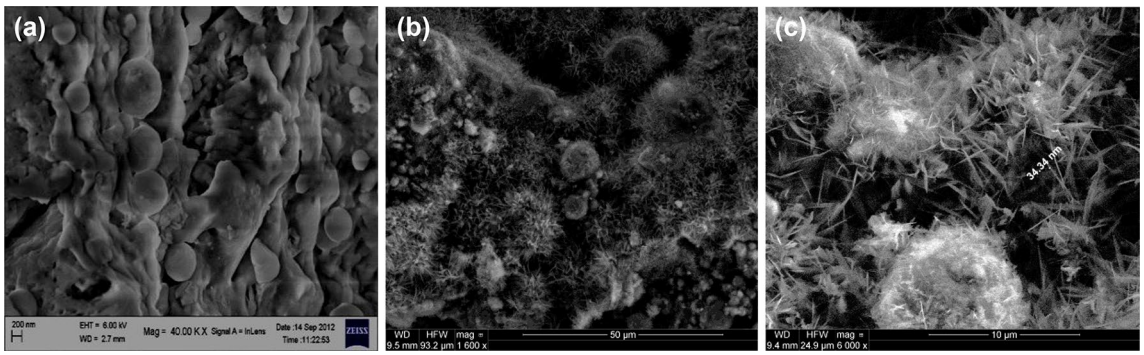


Fig. 8 SEM picture of Mix 6 cured at **a** room temperature (32 °C) and **b**, **c** 80 °C

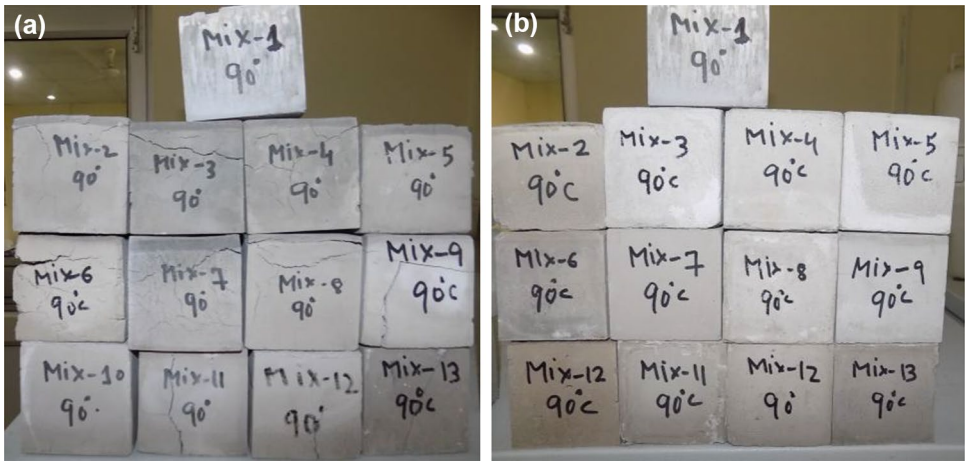


Fig. 9 Mix 1 to Mix 13 of sample **a** (without wrap) and sample **b** (with wrap) were cured in oven for 80 °C

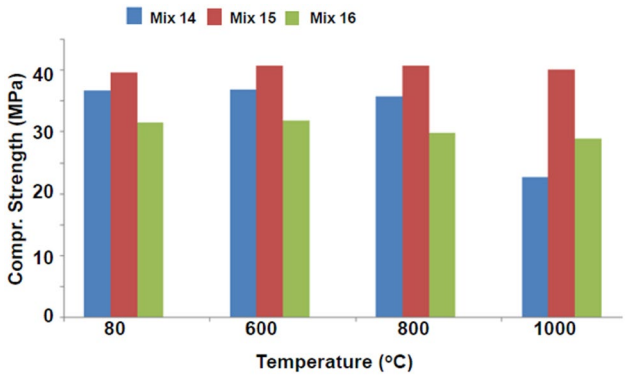


Fig. 10 Effect of different temperatures on compressive strength of Mix 14, Mix 15 and Mix 16

80 °C; effect of 5% AP, 10% CC and 5% AP + 10% CC (Mix 14, Mix 15 and Mix 16, respectively) were also examined. Compressive strengths were determined at 600, 800 and 1000 °C also (43, Fig. 10). On heating at different temperatures for 2 h, there was a loss in weight due to removal of water (Table 5).

The sharp reductions of compressive strength upon heating at 600, 800 and 1000 °C are probably caused by the loss of structural water as well as development of micro cracks [22, 23]. In the presence of 5% AP (Mix 14), the compressive strength was found to be lower as compared to that without AP (Mix 13) (Table 4). It has been reported that when aluminum powder is added, it reacts with alkalies in the following way liberating H_2 gas [24].

$$Al(s) + 6H_2O(l) + 2NaOH(aq) \rightarrow 2NaAl(OH)_4(aq) + 3\ H_2(g)$$
$$2NaAl(OH)_4 \rightarrow NaOH + Al(OH)_3$$
$$2Al + 6H_2O(aq) \rightarrow 2Al(OH)_3 + 3\ H_2(g)$$

Hydrogen gas was entrapped in the structure in a random fashion creating voids. Porous structure of Mix 14 is indicated by SEM (Fig. 11). The density was decreased (Table 6) resulting in decreased compressive strength and at high temperature the cubes were cracked. In the presence of CC, the compressive strength was increased because of enhanced dissolution/hydrolysis of fly ash via heat release [25]. This increased the process of geopolymerization. Since there were less voids, it did not crack at 1000 °C. The combination of AP and CC gave density in between that of the cubes containing AP and CC alone. However, the compressive strength of Mix 16 containing AP and CC was lower even with that containing AP. It appears that in the presence of AP, pores were created and CC entered into the pores and could not get an opportunity in assisting geopolymerization process and as a result the strength was lower. This could be supported by SEM structure (Fig. 11). In general, the density of the cubes decreased with increase of temperature.

Table 5 Weight losses of mortars after heating at different temperatures

Mix no.	Mix composition	Weight before heating (g)	Weight at 600 °C after 2 h (g)	Weight at 800 °C after 2 h (g)	Weight at 1000 °C after 2 h (g)
14	FA + Silica Sand + KOH + Li_2SiO_3 + 5% AFP + 5% AP	730	630	616	615
15	FA + Silica Sand + KOH + Li_2SiO_3 + 5% AFP + 10% CC	749	651	642	637
16	FA + Silica Sand + KOH + Li_2SiO_3 + 5% AP + 5% AFP + 10% CC	741	648	637	629

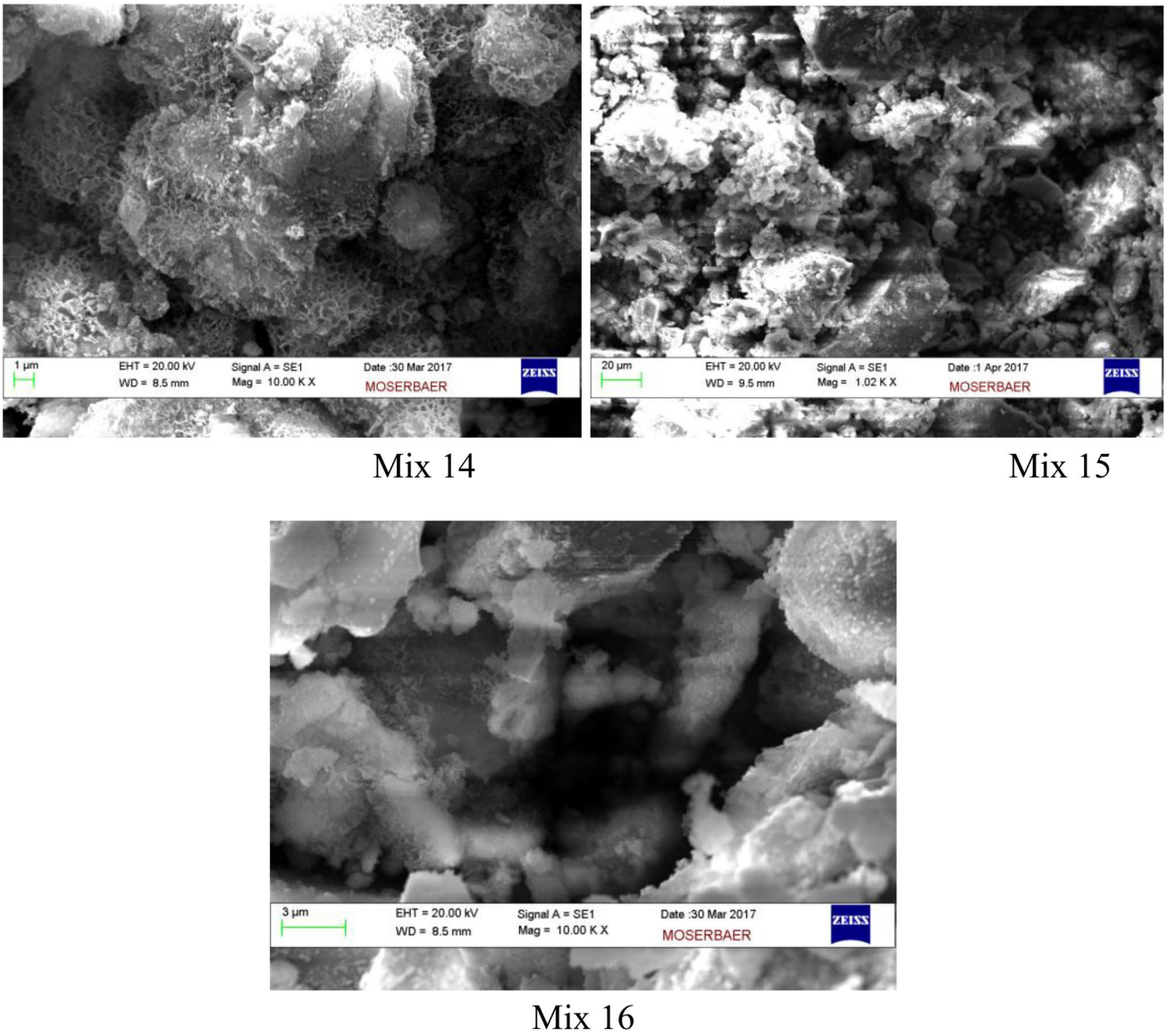


Fig. 11 SEM pictures of Mix 14, Mix 15 and Mix 16

Table 6 Density of mortars after heating at different temperatures for 2 h

Mix No.	Detail Mix (after 2 h curing at)	Specific gravity (g/mL) at different temperatures on heating for 2 h			
		80 °C	600 °C	800 °C	1000 °C
14	FA + Silica Sand + KOH + Li_2SiO_3 + 5% AFP + 5% AP	2.27	2.25	2.25	2.23
15	FA + Silica Sand + KOH + Li_2SiO_3 + 5% AFP + 10% CC	2.86	2.81	2.76	2.68
16	FA + Silica Sand + KOH + Li_2SiO_3 + 5% AFP + 5% AP + 10% CC	2.31	2.30	2.29	2.29

When cubes of Mix 14, Mix 15 and Mix 16 cured at 80 °C for 12 h, immersed in 5% H_2SO_4 for 24 h (3/4th in acid and 1/4th above the acid solution), some changes in appearance (Fig. 12) and compressive strength (Fig. 13) occurred. When the mixes were dipped in H_2SO_4, colour of the acid became turbid in Mix 15 and the acid percolated up to top whereas in Mix 14 and 16, the acid almost did not percolate. There was a very little change in the

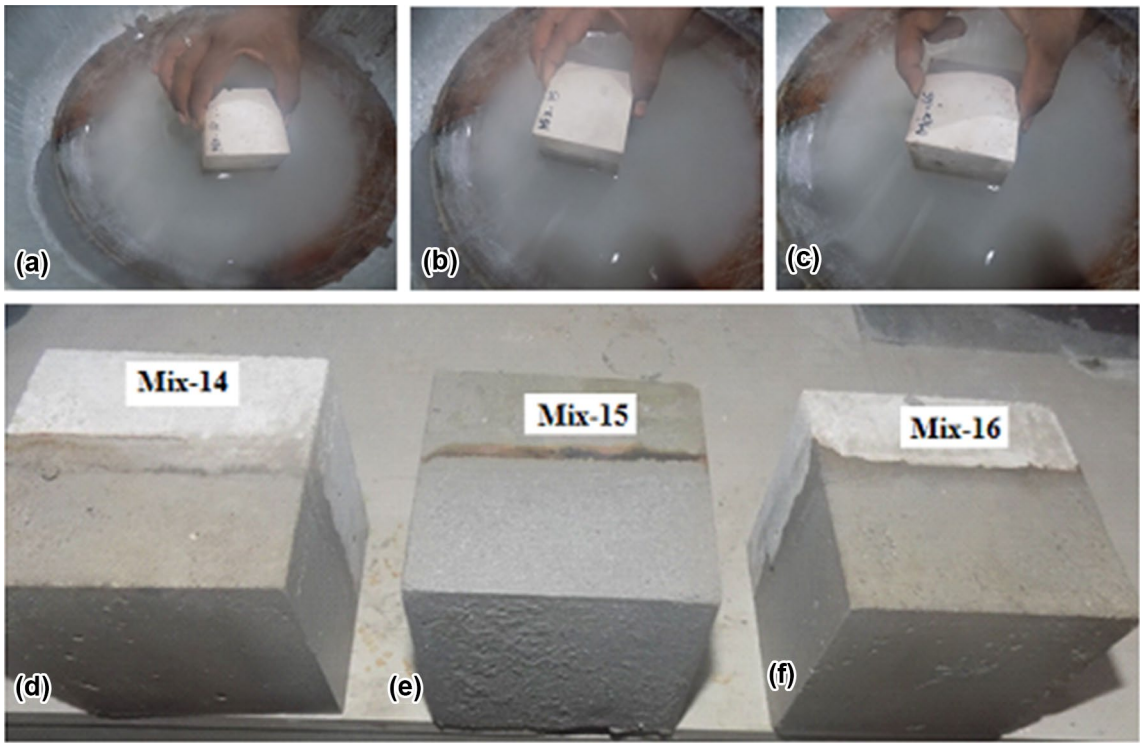


Fig. 12 A-Mix 14, B-Mix 15 and C-Mix 16 just immersing into 5% H_2SO_4; D-Mix 14, E-Mix 15 and F-Mix 16 after immersing into 5% H_2SO_4 for 24 h

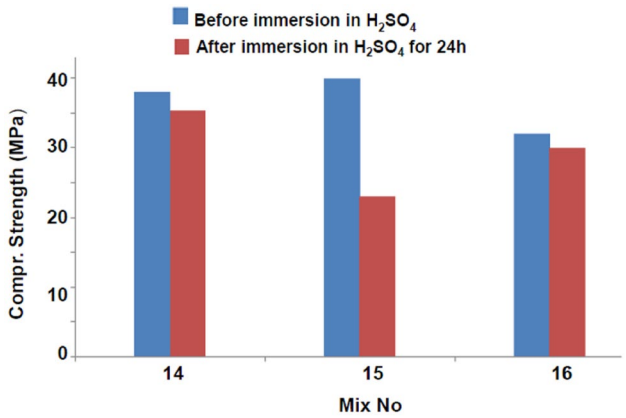


Fig. 13 Change in compressive strength when immersed in 5% H_2SO_4 for 24 h

compressive strength in the case of Mix 14 and 16 when immersed in 5% H_2SO_4 for 24 h. Some reduction in compressive strength occurred when Mix 15 was immersed in 5% H_2SO_4 for 24 h. The reaction between AP and alkali activator was fast while geopolymerization reaction required longer time for completion. As a result, stoichiometry of alkali activator was disturbed. This as well as generation of pores due to liberation of H_2 gas resulted in deceased density and compressive strength. Further, the pores were not interconnected and as a result the damage by sulphuric acid was low. Mix 15 containing CC deteriorated much faster. The deterioration of geopolymer in acidic media may be due to depolymerisation. It appears that depolymerisation occurred in Mix 15. However, for the Mix 16, the deterioration effect was much lower, may be due to presence of AP.

Cubes were heated at different temperatures (600–1000 °C) in a furnace. Cubes containing CC (Mix 15) became red when heated at different temperatures (Fig. 14). However, when Mix 14 containing AP was heated, it did not change its colour (Fig. 14). Since in the presence of AP, the cubes became porous, heat was dissipated in different regions of the cube. In the case of Mix 16, the colour became red but much less than that of Mix 14. Mix 16 also contained AP but the pores were lesser than that of Mix 14.

4 Conclusions

FA based geopolymer mortar using silica sand was made by activating with NaOH/KOH–Na_2SiO_3/Li_2SiO_3 and curing from room temperature to 80 °C. Lithium silicate

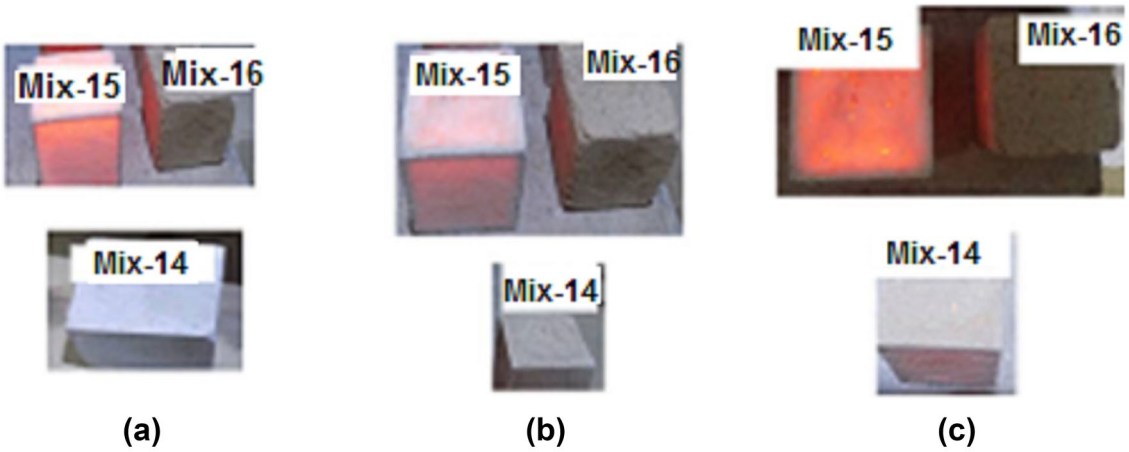


Fig. 14 Curing in muffle furnace at **a** 1000 °C, **b** 800 °C and **c** 600 °C for 2

(300 g) in combination to KOH (14 M) in presence of AP enhanced geopolymerization leading to highest compressive strength of the mortar cured at 80 °C. Inclusion of AFP and CC increased the compressive strength whereas AP decreased the strength. Durability of the mortar containing CC in the presence of 5% H_2SO_4 was poor. Compressive strength of Mix 14 and 15 increased up to 800 °C, but decreased at 1000 °C, whereas for Mix 16, compressive strength decreased after 600 °C. The mortars containing AP cracked at 1000 °C whereas, the mortar containing CC did not crack. To optimize the fire resistant properties with high compressive strength, a detailed study of various combinations of silicate/hydroxides and additives in FA based geoplymer mortars is needed.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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