



Enhancement of the Thermal Durability of Fly Ash-Based Geopolymer Paste by Incorporating Potassium Feldspar

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Abstract The aim of this research is to study the effect of different percentages of potassium feldspar (KF) (0%, 5%, 10% and 20% by weight) on the proficiency of fly ash (FA)-based geopolymer paste when exposed to elevated temperatures of 300 °C, 600 °C and 900 °C. The percentages (by weight of the source material) of both Na₂O and SiO₂ were fixed at 8% each. The behaviour was evaluated on the behalf of residual strength, weight loss, volumetric shrinkage and physical changes at various temperatures. To observe the changes in microstructure and mineralogy during the thermal exposure, scanning electron microscopy (SEM) images of the samples were studied. After observing the results, good durability was revealed at elevated temperatures when KF was incorporated in FA-based geopolymer paste. At 900 °C, the residual compressive strengths of adding of KF @ 0%, 5%, 10% and 20% by weight were 22%, 25%, 29% and 42% of those at room temperature, respectively. At 900 °C, the geopolymer matrix was observed to be most densified, which imparts superior durability to the specimens.

Keywords Geopolymer · Fly ash · Potassium feldspar · SEM

Introduction

Primarily, the term geopolymer was coined by Davidovits. He pioneered its manufacturing from aluminosilicate source material, hardened by various alkaline solutions. The high strength properties of geopolymer, its durability against acids and sulphate, the absence of hazardous alkali-aggregate reaction during the manufacturing, its ability to withstand high temperature were reported. During a quest of finding a fire-resistant material, more focus was directed towards geopolymer [1]. Investigation on factors affecting the geopolymerization of sodium feldspar and potassium feldspar was done. The resultant geopolymers show a high compressive strength [2]. Clay and other natural minerals such as feldspar can be treated as aluminosilicate source material for geopolymers [3]. Nearly 1000 °C temperature is necessary to calcine feldspar with sodium hydroxide as an activator to achieve desirable compressive strength [4]. In test structures exposed to fire, it was observed that temperature increased rapidly and reached up to 800 °C during the first 30 min. It took 2 to 2.5 h to reach the 1100 °C mark; gradually, there were disintegrations where the stability of structure was affected and as a result of which the structure collapsed. Compilation of all the facts indicated that decomposition of Portland cement paste hydration was responsible for such behavioural change in concrete. While in the case of geopolymer concrete, partial sintering of components was occurred due to its exposure to elevated temperature, which affected the mechanical strength [5–8]. Thermal behaviour of metakaolin with sodium hydroxide and sodium silicate, excellent fire resistance was observed up to 1200 °C [1, 5]. After exposure to elevated temperature on sintering reaction of unreacted fly ash particle, the performance of fly ash-based geopolymer was observed to be better than that of

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metakaolin-based geopolymer, Bakharev et al. [9]. It was found that higher concentration of sodium hydroxide in activating solution leads to higher rates of filtration of silica and alumina, resulting in increased compressive strength, Chindaprasirt et al. [10]. Good performance of limestone aggregate concrete exposed to elevated temperatures of 110, 210 and 310 °C was observed where the water loss was significant, Noumowe et al. [11]. It was observed that FA geopolymer paste has superior fire resistance over OPC paste at temperature above 100 °C, Sanjayan and Kong [12]. It was reported that with the increase in content of FA, the compressive strength of self-compacting concrete improves at 200–300 °C, while the corresponding split tensile strength reduces from 20 to 300 °C, Pathak and Siddique [13]. It was indicated that for designing nuclear structures, up to 20% GGBS could be used in concrete, Siddique and Kaur [14]. It was reported that the use of GGBS is beneficial in terms of hydration of cement, denser structure, increase in compressive, split tensile and flexural strength, improves permeability and sulphate resistance of concrete, Siddique [15]. It was reported that a higher concentration of sodium sulphate and finer slag particles in slag-based geopolymer leads to a good temperature-resistant material by considering elevated temperatures up to 800 °C, Rashad et al. [16].

While formulating geopolymer with metakaolin, iron oxide and red mud mixture activated by sodium silicate and sodium hydroxide solution, it was observed that silica present in the sodium silicate is more reactive than silica present in metakaolin and mechanical strength depends on porosity and non-reacted source materials, Hajjaji et al. [17]. After studying the influence of elevated temperature on geopolymer paste and mortar at 400, 600 and 800 °C, it was concluded that dehydration and dehydroxylation processes were responsible for the strength deterioration at 400 and 600 °C, while sintering and densification were responsible for the failure at 800 °C, Abdulkareem et al. [18]. It was observed that at elevated temperatures particle size, shape of binders and different activation of different binders affect the compressive and flexural strength of GGBS and coarse FA-based geopolymer mortar. Keeping in view the physical changes and compressive strengths, it was quoted that 400 °C and 600 °C were critical temperatures, Kürklü [19]. It was observed that the dosages of activators play a vital role in strength, and GGBS reduces the curing temperature of geopolymer, Soutsos et al. [20]. It was stated that curing temperature and curing time of 60 °C and 2 h, respectively, are sufficient enough for production of FA and metakaolin-based geopolymer paste with a compressive strength of 25.1 MPa, Gorhan et al. [21]. It was reported that the residual strength of geopolymer made with 80:20 ratio of FA/GGBS at 700 °C

was 26% higher than that of FA/OPC, Saavedra and Gutierrez [22].

From the above-mentioned previous literature works, to the best of the author's knowledge, it can be affirmed that there has been lack of experiments on geopolymer with KF. For the present study, experiments were carried out to evaluate the effect of KF on performance of alkali-activated FA-based geopolymer paste at elevated temperature. The amounts of FA were replaced with four different percentages of KF (0%, 5%, 10% and 20% by weight). The percentages of Na₂O and SiO₂ were kept constant at 8%. The geopolymer specimens were exposed at 300, 600 and 900 °C. The performance of the specimens was evaluated in terms of weight loss, volumetric change, compressive strength and physical appearance recorded after exposure to elevated temperature. Microstructural analysis was done using SEM to study the effect of elevated temperature on the specimens.

Experimental Procedure

Source Materials

For the present experimental programme, low calcium F Class fly ash was procured from Kolaghat Thermal Power Plant, Kolkata and KF was purchased from Ceramique Industries, Kolkata. Chemical compositions of FA and KF are shown in Table 1. Mineralogical compositions of KF and FA, respectively, are shown in Fig. 1. More than 90% of the particles were finer than 45 microns of FA. Sodium hydroxide pellets (98% purity) of laboratory grade and sodium silicate solution (SiO₂ = 26.5%, Na₂O = 8% and 65.5% water) with silicate modulus of 3.30 (approx.) and bulk density of 1410 kg/m³ were procured from Sharma Bros, Imphal, India. The percentages of Na₂O = 8% and SiO₂ = 8% by weight of source material were fixed in the activator solution, which was prepared 1 day before each manufacturing of the geopolymer paste. Normal tap water was used.

X-ray diffractogram (XRD) for raw materials KF and FA, respectively, is presented in Fig. 1. In the FA, the presence of quartz (Q), mullite (M) and haematite (H) was observed, while in potassium feldspar, microcline (ML) and quartz (Q) were observed.

Preparation of Specimens

To produce a homogeneous paste, required quantity of sodium hydroxide pellets was mixed directly with sodium silicate solution. The obtained alkaline solution has 8% Na₂O and 8% SiO₂ by weight of FA. Water to FA material was 0.33. The alkaline solution was kept for overnight at

Table 1 Chemical composition of FA and KF

Chemical composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	K ₂ O	TiO ₂	MgO	P ₂ O ₅	MnO
Fly ash	41.72	27.34	5.111	1.145	0.07	1.418	1.989	0.567	0.722	0.046
Potassium feldspar	63.22	15.15	0.134	0.197	3.0	10.86	0.017	0.299	0.016	0.003

room temperature for manufacturing of geopolymer paste. The aluminosilicate source material and alkaline activator solution were mixed in Hobart mixer for 5 min. The geopolymer paste was of grey colour, sticky in nature and of normal workability. With the increase in the percentage of potassium feldspar, the workability of paste also increased. The mix was poured into cube moulds of 50 mm x 50 mm x 50 mm and vibrated for 10 min on vibration table to expel the entrapped air.

Curing Regime

The specimens were wrapped with polyethylene and heat cured along with the moulds in an oven for a period of 24 h at 80 °C and kept within the oven before being removed at room temperature. Table 2 shows the nomenclature of specimen ID and mechanical strength of geopolymer cube specimen cast with 0%, 5%, 10% and 20% KF, respectively, at 7 days. In specimen GPKF0, GP represents geopolymer and KF represents the potassium feldspar and

the numerical value represents the percentage of FA replaced by KF.

Elevated Temperature Exposure

The geopolymer paste specimens prepared of series GPKF0, GPKF5, GPKF10 and GPKF20 were exposed to elevated temperature of 300 °C, 600 °C and 900 °C at the age of 7 days. The cast specimens were kept in the muffle furnace and heated at a fixed heating rate of 5 °C/min till the desired temperature is achieved and seized at this temperature for a period of 1 h. After that, the furnace was shut down and the specimens were allowed to cool down inside the muffle furnace to room temperature before removing them for further tests. Measurement of weight and dimension shrinkage was done immediately with weight machine and vernier calliper, respectively. Hence, the reduction in weight loss and volumetric shrinkage was obtained. SEM was conducted on powdered sample.

Fig. 1 Mineralogical composition of **a** KF and **b** FA (XRD)

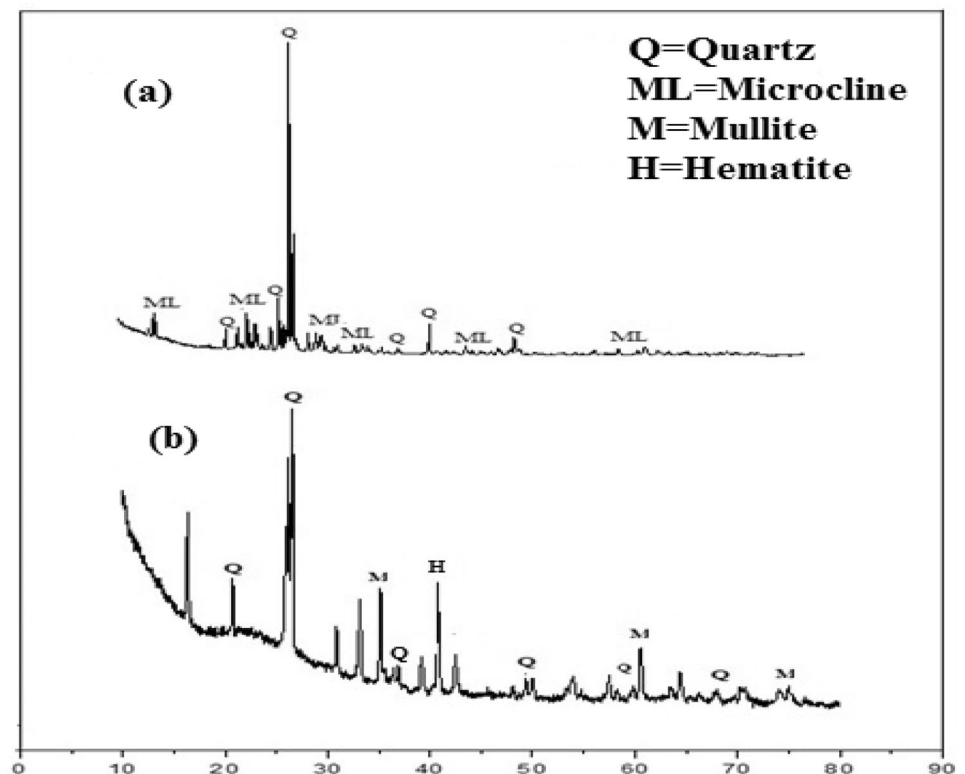


Table 2 Details of experimental test specimens

Specimen ID	Potassium feldspar (%)	Fly ash (%)	Na ₂ O (%)	SiO ₂ (%)	Water to source material ratio	Curing temperature and duration	7 days compressive strength (MPa)
GPKF0	0	100	8	8	0.33	80 °C and 24 h	54
GPKF5	5	95					43
GPKF10	10	90					39.2
GPKF20	20	80					28

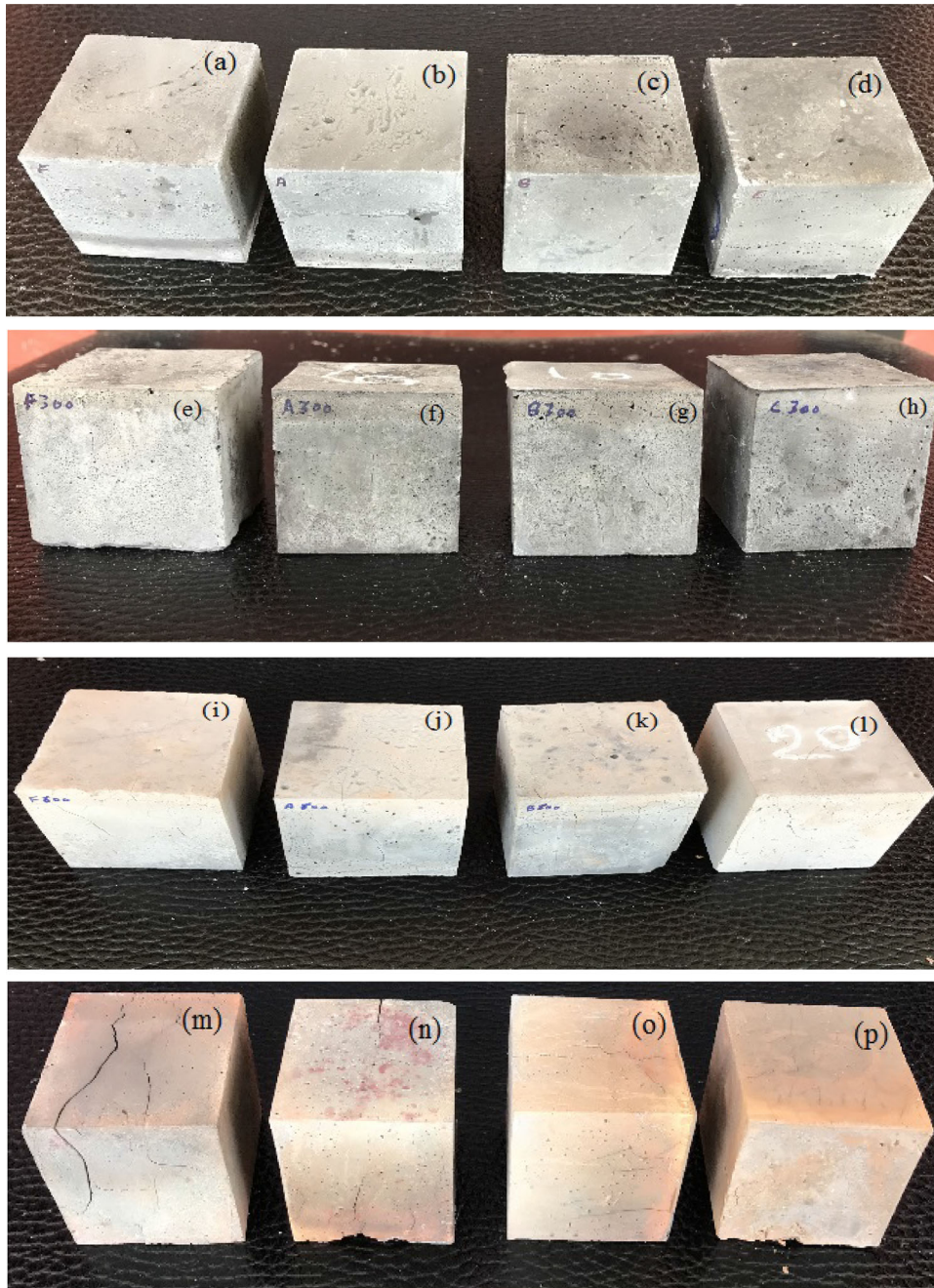


Fig. 2 Unexposed geopolymer paste samples, **a** GPKF0, **b** GPKF5, **c** GPKF10, **d** GPKF 20. Geopolymer paste samples exposed to 300 °C, **e** GPKF0, **f** GPKF5, **g** GPKF10, **h** GPKF20. Geopolymer paste samples exposed to 600 °C, **i** GPKF0, **j** GPKF5, **k** GPKF10, **l** GPKF 20. Geopolymer paste samples exposed to 900 °C, **m** GPKF0, **n** GPKF5, **o** GPKF10, **p** GPKF20

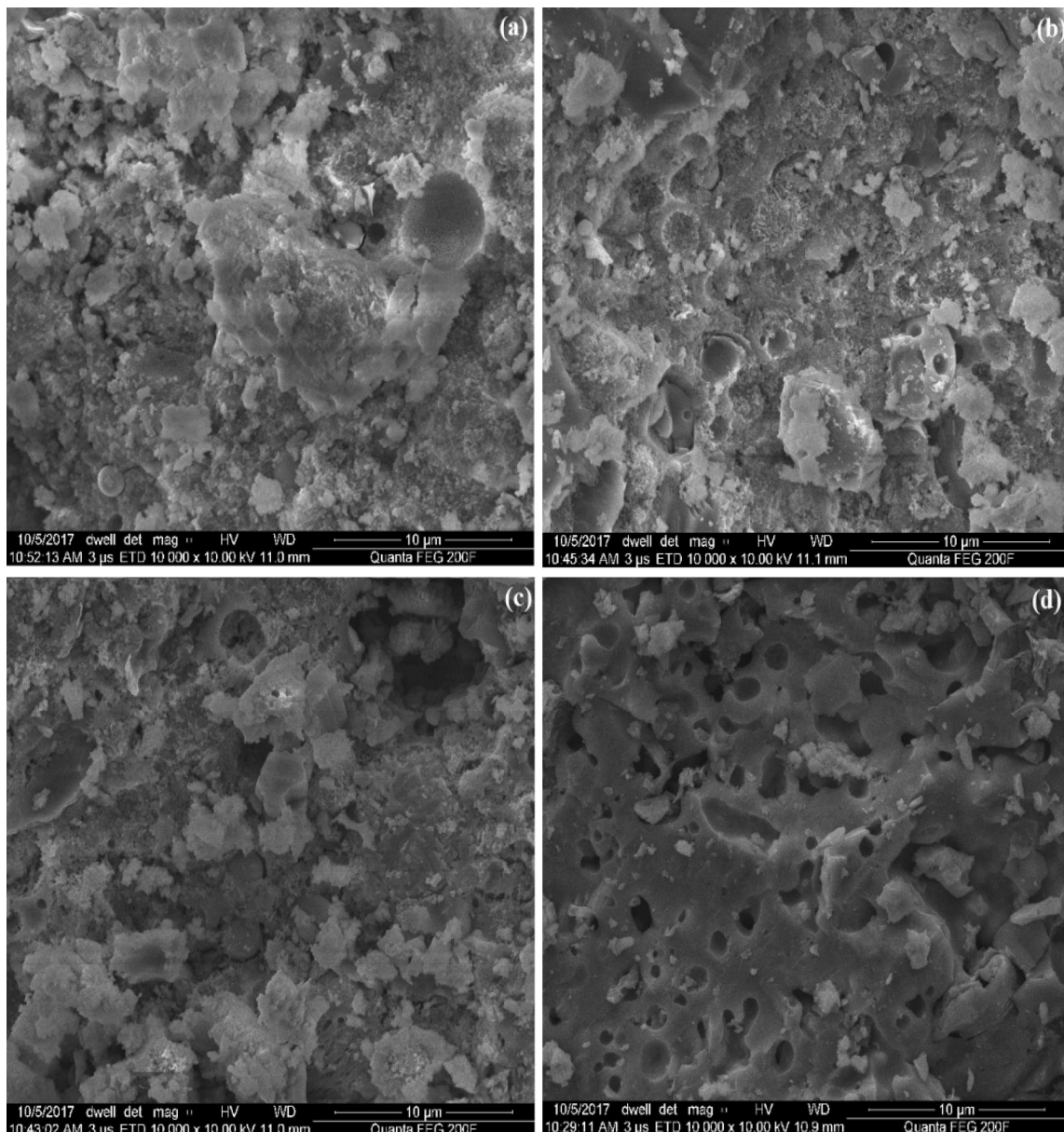


Fig. 3 SEM micrograph of GPKF10 at various temperature. a Unexposed, b 300 °C, c 600 °C, d 900 °C

Results and Discussion

Physical Appearance

Figure 2 shows the variation in physical appearance with respect to change in colour, at unexposed and exposed conditions. The colour of all specimens is grey at room temperature. As the percentage of potassium feldspar

increases, the colour darkens. Figure 2a–d represents the geopolymer specimens GPKF0, GPKF5, GPKF10 and GPKF20, respectively, at unexposed condition. Figure 2e–h shows the samples GPKF0, GPKF5, GPKF10 and GPKF20, respectively, exposed to 300 °C, which shows that samples' colour changes to dark grey. Figure 2i–l shows the specimens GPKF0, GPKF5, GPKF10 and GPKF20, respectively, exposed to 600 °C and indicates

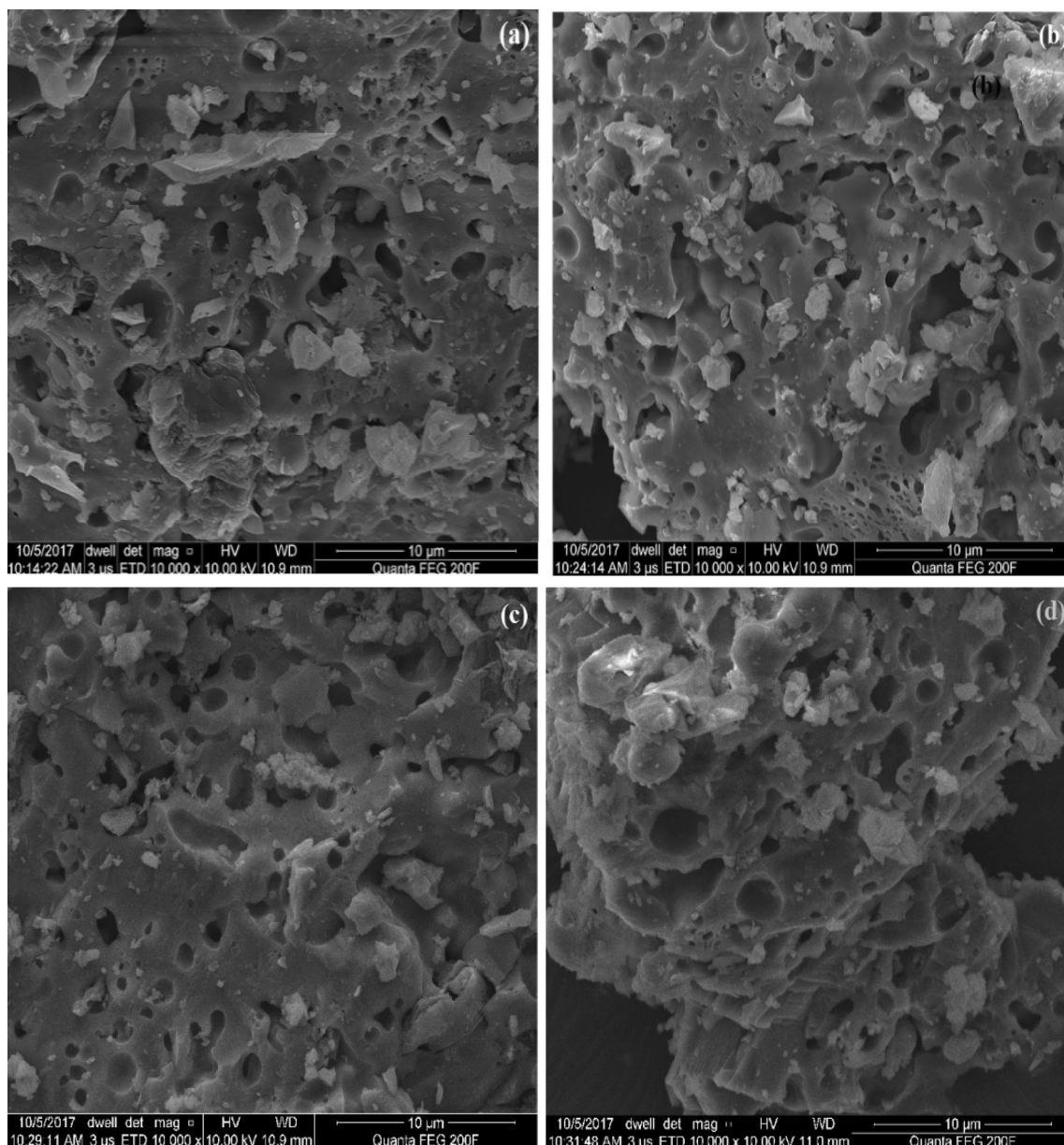


Fig. 4 SEM micrograph of geopolymer paste at 900 °C, **a** GPKF0, **b** GPKF5, **c** GPKF10, **d** GPKF20

that the colour of specimen turns to light yellowish. Figure 2m–p shows the specimens GPKF0, GPKF5, GPKF10 and GPKF20, respectively, at 900 °C which reflects that colour of specimen turns to light reddish.

This colour change is due to oxidation change in the Fe_2O_3 in the geopolymer specimens. Similar phenomenon was also reported by Temuujin and van Riessen, Mandal et al. [23, 24]. No cracks were observed in all specimens at 300 °C. The cracks gradually increase with the rise in temperature. Hence, deterioration in the structure of geopolymer paste specimens increases with increase in temperature. Notable cracks are observed in Fig. 2m of GPKF0 exposed to 900 °C. As the percentage of KF

increases, the crack formation is decreased. Figure 2p shows the microcracks of GPKF20 compared with that of GPKF10, GPKF 5 and GPKF0. Hence, the addition of potassium feldspar to geopolymer increases the ability of resistance to elevated temperature. This may be due to dense matrix formation of the geopolymer at elevated temperature.

Scanning Electron Microscopy (SEM)

For SEM, the specimens were collected from near the surface (0–3 mm depth). SEM micrographs of GPKF10 before and after exposure to different elevated

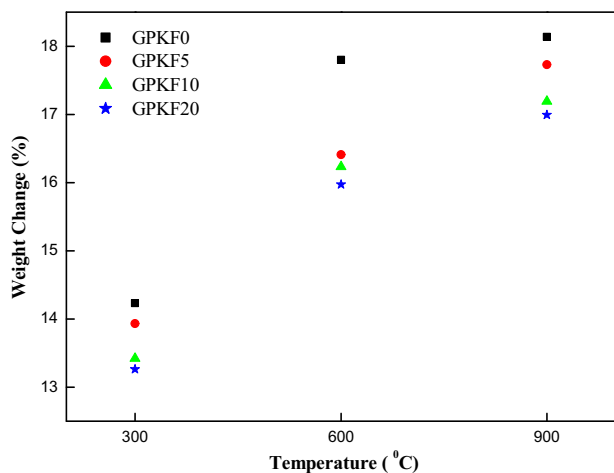


Fig. 5 Variation in weight change with temperature

temperatures are shown in Fig. 3. The mechanism of formation of silico-aluminate gel to be crystalline in species with ratio Si/Al > 2 needs higher temperature, about 175 °C and in some case up to 300 °C. It links the chemical reaction of geopolymeric precursors such as aluminosilicate oxide (Al^{3+} in fourfold co-ordination) with alkali polysilicates yielding polymeric Si–O–Al bonds [3]. Micrographs at 600 °C show better microstructure than those at 300 °C. Some phase change has been taking place due to sintering. Notable changes take place in the specimen exposed at elevated temperature of 900 °C. The micrographs of GPKF0, GPKF5, GPKF10 and GPKF20 after exposure at 900 °C are presented in Fig. 4. With the increase in the KF percentage in FA-based geopolymer, the structure becomes denser with less porous structure and due to which its performance increases at elevated temperature.

Variation in Weight Loss

Figure 5 shows the weight loss recorded for all specimens at elevated temperatures. A rapid weight loss was observed in all specimens of geopolymer paste up to 300 °C. At 300 °C, GPKF0 specimen exhibits weight loss of 14.23%, which is quite high. But further heating up to 900 °C results in a loss of 18.14%, showing very major increase in weight loss over that of 300 °C. Although weight loss was detected in all specimens, the specimens having a higher percentage of KF in FA-based geopolymer recorded minimum losses. Weight loss is mainly due to the evaporation of moisture at lower temperature and chemical bonded water at higher temperatures. Davidovits told that the hardened geopolymer specimen contains three types of water, i.e. physically bonded water, chemically bonded water and hydroxyl group. This water is evaporated during

the heating of geopolymer above 100 °C. Each type of water is evaporated at a definite range of temperature. The free water is evaporated up to 100 °C. By further heating the geopolymer paste specimens up to 300 °C, chemically bonded water is evaporated beyond 300 °C, and dehydroxylation takes place [18, 25].

Variation in Volumetric Shrinkage

The evaporation of water leads to mass loss which results in volumetric shrinkage and causes crack formation. The variation in volumetric shrinkage with respect to temperature for the specimens GPKF0, GPKF5, GPKF10 and GPKF20 is represented in Fig. 6. At 300 °C, the volumetric change was a maximum of 5.62% for GPKF0, 4.59% for GPKF5, 4.07% for GPKF10 and 3.69% for GPKF20. At 600 °C, it was 9.67% for GPKF0, 6.77% for GPKF5, 5.88% for GPKF10 and 4.4% for GPKF20. Therefore, no substantial change in volumetric shrinkage up to 600 °C was observed. Further increase in temperature to 900 °C results in brisk rise in volumetric strain up to 14.67% for GPKF0, 14.05% for GPKF5, 12.55% for GPKF10 and 10.75% for GPKF20. Increase in volumetric shrinkage showed a relation with a percentage of KF in the mix. Geopolymer paste specimen with higher amount of KF results in lower shrinkage at all exposure temperatures.

Residual Compressive Strength

Figure 7 represents the initial compressive strength of unexposed specimens and residual compressive strength of specimens exposed to elevated temperature up to 900 °C of series GPKF0, GPKF5, GPKF10 and GPKF20. It illustrates that as the elevated temperature increases, the compressive

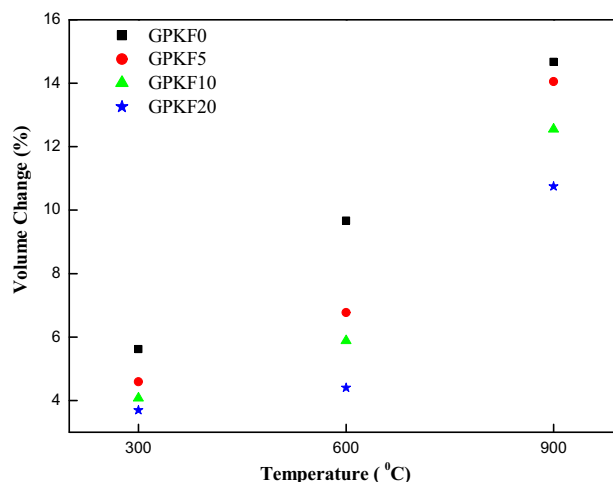


Fig. 6 Variation in volume change with temperature

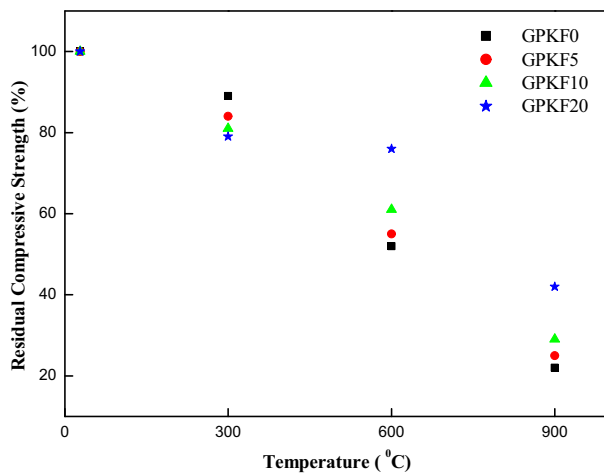


Fig. 7 Variation in residual compressive strength with temperature

strength of all geopolymer specimens decreases. At 300 °C, the residual compressive strength of GPKF0, GPKF5, GPKF10 and GPKF20 was 89%, 84%, 81% and 79%. At 600 °C, the residual strength of GPKF0, GPKF5, GPKF10 and GPKF20 was 52%, 55%, 61% and 76%, respectively. At 900 °C, the residual compressive strength of GPKF0, GPKF5, GPKF10 and GPKF20 was 22%, 25%, 29% and 42%, respectively. Figure 5 shows that at 300 °C, GPKF0 has minimum weight loss, therefore its residual strength was maximum among all. The residual compressive strength of GPKF20 was minimum at 300 °C because the formation of crystalline species in source material with a ratio of $\text{Si}/\text{Al} > 2$ needs a higher temperature up to 300 °C. Also, the time required varies from hours to several days [18]. Beyond 300 °C, crystalline phase takes place which imparts the strength to specimens and pattern of residual compressive strength changes. As the percentage of KF increases in FA-based geopolymer, the residual strength increases. As the temperature rises, weight change increases, resulting in continuous loss in strength due to the vapour effect [20]. According to this phenomenon, increase in thermal shrinkage due to water evaporation from geopolymer structure is responsible for the fall in compressive strength with increase in temperature.

Conclusion

This paper shows the characteristics of FA-based geopolymer paste with incorporation of different percentage of KF, before and after exposure to elevated temperatures of 300 °C, 600 °C and 900 °C. The parameters opted to study were physical appearance, volumetric shrinkage, weight loss and residual compressive strength. Experimental data show substantial variation in

microstructure, performance and properties after exposure to elevated temperature. A summary of the present study is given as follows:

- Geopolymer paste specimen with a higher percentage of KF remains physically stable without any structural disintegration at elevated temperature, but gradual change in colour was identified.
- A rapid weight loss was recorded up to 300 °C in all the specimens. A sharp increase in volume change was observed in all specimen's exposure beyond 600 °C. The phenomenon of sintering was observed at high temperatures, although behaviour of specimens with higher percentage of KF shows better resistance at elevated temperatures.
- Geopolymer specimens containing KF show better microstructure when exposed to elevated temperature.

The results have shown the potentiality of developing a construction material, resistant towards elevated temperature by the partial replacement of FA with KF in geopolymer specimens.

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