

Reactivity, workability and strength of potassium versus sodium-activated high volume fly ash-based geopolymers

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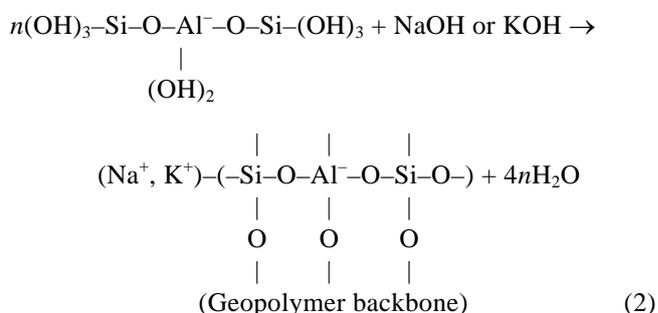
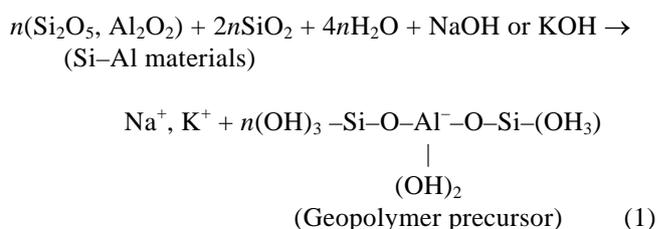
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Geopolymer is the product resulting from a geosynthetic reaction of aluminosilicate minerals in the presence of strong alkalis. This communication reports a comparative study on the utility of lower concentration of sodium and potassium hydroxides and alkali silicates at room temperature. The study reveals that the liquid demand is lower for geopolymers with potassium activators. The test results show that potassium activators lower the initial setting time, enhance the reactivity of the components and thereby improve the compressive strength. Sucrose and sucrose with tartrate are attempted as workability and set control aids. Sucrose provided workability in potassium geopolymer but decreased the strength.

Keywords. Alkali-activated solution, geopolymers, reactivity, sucrose, workability.

ORDINARY Portland cement (OPC) is the most commonly used binder in concrete. Besides serious limitations with respect to durability in aggressive environments, OPC happens to be a highly energy-intensive material with the highest carbon dioxide emission among building materials. Therefore, there is a strong need for a more energy-efficient and green-binder system for concrete. Davidovitis¹ introduced an alternate binder made of silica and alumina activated by a highly alkaline solution. He used calcined clays as the source material and showed that with alkaline activators, a hardened ceramic-like product could be produced at room temperature. The resulting product was identified as an inorganic polymer resulting from polycondensation reaction and was termed as a geopolymer. Normally, the geopolymerization of aluminosilicate minerals from industrial wastes, such as fly ash and ground granulated blast furnace slag (GGBS) is brought about by using a highly concentrated alkaline activator solution (AAS) at high temperature. Sodium-based compounds have been utilized for this purpose due to their easy availability.

Geopolymers possess excellent mechanical properties, fire resistance and acid resistance². The polymerization process involves a substantially rapid chemical reaction under alkaline condition on silicon (Si)-aluminium (Al) minerals that result in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds.



The nature of AAS plays a pivotal role in controlling the kinetics of the geopolymerization process and it is one of the principal factors that influences the structure and composition of the gel initially formed in addition to Si/Al ratio³ and the curing time⁴. Fly ash-based geopolymer systems have been developed and studied^{5,6}. The main disadvantage of this system is the utility of non-user-friendly, high-molarity activator systems and the necessity of supplementary activation by heat curing. Some workers have attempted to synthesize geopolymers at ambient temperature using certain combination of mineral additives and more user-friendly activators⁷⁻¹⁴.

Rheological properties of the geopolymers are different from those of OPC-based systems. Both the static and dynamic viscosity of geopolymers is substantially higher and treatment by vibration or by other techniques has

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been used to overcome the problem^{13,14}. Another drawback is the pronounced adhesion ability of the fresh composition to the mixer and mixing tools¹⁵. Therefore, the coarse aggregate volume and gradation must be selected with great care. Settlement of coarse aggregates and movement of paste/mortar on the surface may also occur, although this tendency is much less compared to OPC concrete. Therefore, improvement of workability of geopolymer systems and the effect of admixtures used for this purpose on the mechanical properties has been a topic of considerable interest to extend the range of applicability of geopolymers. Although several admixtures are available for the workability enhancement of Portland cement concrete, there is lack of information on geopolymers. Fresh geopolymer concrete has been reported to be highly viscous and cohesive with low workability. Using calcined kaolin as the source material, Teixeira-Pinto *et al.*⁹ reported that the fresh geopolymer concrete became stiff in a short time, thus implying a short initial setting time.

Chareerat *et al.*¹⁰ studied the workability and strength of a high-calcium fly ash-based geopolymer. Their study revealed that the workable flow of the geopolymer mortar relied on the ratio by mass of sodium silicate (Na_2SiO_3) (SS) to sodium hydroxide (NaOH) (SH) and as the concentration of SH solution increased, the workability decreased.

Sathonsaowaphaka *et al.*¹³ studied the workability and strength of lignite bottom ash geopolymer mortar with fineness as the parameter. The flow and compressive strength increased with decrease in fineness. It was also observed that the flow and strength increased with the increase in liquid to binder ratio. They also reported increase in workability with the reduction of SH concentration while the strength remained constant.

The addition of naphthalene-sulfonate-based superplasticizer, which is used as a workability aid in conventional concrete, up to 4% of fly ash by mass, improves the workability of fresh geopolymer cement (GPC) concrete^{6,14,15}. However, there is a slight degradation in the higher compressive strength of geopolymer concrete when the superplasticizer dosage is greater than 2%. There is a need to study the effect of other chemical admixtures in order to enhance the workability of geopolymer concrete without sacrificing the strength.

The setting time of the geopolymer paste was affected by the curing temperature, type of alkaline activator and the composition of the source material^{16,17}. The setting time of the geopolymer paste was between 15 and 45 min at 60°C. However, Hardjito *et al.*¹⁵ reported a working time of 120 min for GPC concrete. It is believed that the presence of compounds other than Al_2O_3 and SiO_2 in the source material may delay the setting. Although the setting may be delayed, the working time of the mixes is rather limited and therefore, there is a need to identify suitable chemical admixtures, which can extend the working time.

Research significance

Although sodium compounds are widely used for the preparation of AAS in GPC production and majority of the studies reported on GPC are based on sodium compounds, potassium-based compounds are also claimed to impart better workability and improved characteristics to GPC¹⁸. Some contrasting results have been reported with regard to the relative efficiency of sodium and potassium compounds. Therefore, in the present study an attempt has been made to assess the workability, setting time and compressive strength of potassium and sodium-based GPC composites. As the physical and mechanical properties of a composite are a consequence of the physical and chemical processes occurring in a mixture after blending of the ingredients, the chemical parameter which influences the geopolymerization process, viz. pH has been determined and some of the important oxide molar ratios have been computed to analyse their effect on the properties. To assess the extent of effectiveness of the different activators, two significant parameters, the degree of reactivity and the compressive strength were determined for the GPC specimens.

The addition of inorganic salts like chlorides, sucrose and organic chemical admixtures has been reported in the literature. Problems of long delay in setting, loss of strength, appearance of efflorescence on the surface and requirements of excessive dosages with concomitant problems in setting time and strength have occurred in such cases. In the present study, some of the selected admixtures are studied for their effect on workability, setting time and strength.

Methodology

Reacting materials

Low-calcium class F-type fly ash obtained from Ennore Thermal Power Station, Chennai and granulated blast furnace slag powder procured from Andhra Cements, Visakhapatnam were the primary aluminosilicate sources used in this study. The properties of the materials are given in Tables 1 and 2. Commercial-grade SH and potassium hydroxide (KOH) (PH) flakes were dissolved in distilled water to obtain the respective alkali solutions one day prior to GPC preparation to avoid excessive heat resulting from the exothermic reaction. SS and potassium silicate (K_2SiO_3) (PS) solutions were used as the alkaline activators and their properties are given in Table 3.

Geopolymer paste synthesis

The geopolymer paste was prepared in a Hobart mixture by dry-mixing the binder material comprising 80% fly ash and 20% GGBS for 1 min followed by the addition

Table 1. Chemical composition of fly ash (% by mass)

Loss of ignition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	Mn ₂ O ₃	SO ₃	Specific gravity
0.76	62.10	27.44	4.57	0.83	0.55	0.04	1.17	1.09	0.04	0.40	2.2

Table 2. Chemical composition of slag (% by mass)

Specific gravity	Chemical composition						Loss of ignition
	CaO	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	MgO	
2.91	40.3	43.4	12.5	0.9	0.6	1.5	2.1

Table 3. Properties of silicate

Parameter	Potassium silicate	Sodium silicate
Specific gravity	1.38	1.50
K ₂ O	12.50	–
Na ₂ O	–	14.20
SiO ₂	26.30	31.26
Total solids (%)	38.8	45.46
Hardness (Baume)	40	50

of AAS consisting of SH with SS or PH with PS, such that the liquid/binder (l/b) is 0.6 and 0.5 respectively. After mixing for 5 min, a mix of uniform colour and consistency was obtained. The fresh slurry was placed in a 50 × 100 mm cylinder mould. The specimens were compacted in three layers and each layer vibrated for 10 sec in the vibrating table. Demoulding of the specimens was carried out depending on their final setting time and the hardened specimens were kept at room temperature, wrapped in wet gunny bags until the age of testing. The compressive strength test was performed at the age of 7 and 28 days. The reported strengths are the average of three test specimens.

Testing

Workability

The fresh geopolymer paste made using SH with SS was too stiff. As in the case of OPC paste, the liquid content of the mix influenced the workability of the geopolymer paste. In the present study, the workability of the geopolymer pastes was determined by varying AAS and chemical admixtures to find the flow characteristics of various geopolymer pastes. The flow of fresh slurry was determined with the help of mortar flow table conforming to IS: 5512–1983 (ref. 19). In order to improve the workability of fresh paste, attempts were made using chemical admixtures such as sucrose and tartrate. The chemical admixtures were added in selected percentages of the binder mass.

Setting time

Another important characteristic of fresh geopolymer paste is the setting time. The setting time of the geopolymer paste also varied depending on the composition of AAS and chemical admixture used. The initial setting time was determined using a penetrometer conforming to IS: 8142–1976 (ref. 20) and the setting times of the different pastes are given in Table 4.

Chemical admixture

The earliest use of chemical admixtures in GPC was in Pyrament cement comprising 80% Portland cement and 20% geopolymeric materials activated by potassium carbonate having citric acid as retarder². Cheng *et al.*¹¹ studied alkali-activated slag and reported a substantial increase in mechanical strength when 1.9–3.4% of Ca(OH)₂ was used. When the alkaline activator concentration was 10 M or higher, dissolution of Ca(OH)₂ was found to be difficult. This is due to the presence of hydroxides (OH[–]) and there will not be enough Ca²⁺ for the formation of the CSH gel; instead sodium-based aluminosilicate is formed. When this kind of silicate is formed, it attracts (OH[–]) to its structure, lowering the amount and allowing the formation of the CSH gel as a secondary reaction product. Lee and Deventer²¹ studied the influence of inorganic salts in alkali-activated mixtures of fly ash and kaolin. They reported that the strength and durability are negatively affected by chloride salts, because they formed crystals inside the structure lowering its strength. They also found that carbonate salts are beneficial because they diminish the amount of dissolved water preventing the hydrolytic attack.

The addition of sucrose as a chemical admixture to GPC in order to delay the setting time and extend the working period of GPC was attempted by Nuruddin *et al.*²². This resulted in efflorescence formation on the surface. To overcome this problem, sucrose and tartrate were used as chemical admixtures to enhance the working time and setting time of GPC pastes.

pH

The AAS provides a conducive environment for geopolymerization²³ by dissolving the alumina and silica phases in the raw materials and acts as a liquid medium facilitating the condensation polymerization to take place.

Table 4. Test parameters and results

Mix id	l/b	Alkaline activator solution (AAS) composition	Chemical admixture	Flow (mm)	Workability	Initial setting time (IST) (h)	pH	Degree of reactivity	Compressive strength (MPa)	
									7th Day	28th Day
SHSS	0.6	SH + SS	Nil	190	Quite stiff	4	13.5	55.4	12.9	13.7
PHPS	0.5	PH + PS	Nil	220	Easily workable	3	13.2	64.1	19.9	43.0
SSS	0.6	SH + SS	2% Sucrose	130	Easily workable	4	13.6	60.7	11.9	12.0
SSST	0.6	SH + SS	2% Sucrose + 0.5% tartrate	105	Stiff	0.5	13.6	53.7	4.5	11.5
PPS	0.5	PH + PS	2% Sucrose	230	Easily workable	3	13.2	56.0	17.8	25.8

According to Fernandez-Jimenez *et al.*¹², the most relevant parameters that influence the mechanical strength of alkali-activated GPC mortars are: nature of the alkaline activator, its concentration, curing temperature and optimum concentration of the alkaline activator to the percentage of Na₂O of the slag mass. Using Na₂O in excess causes cost-inefficient mixtures and shows efflorescence. Misra *et al.*²⁴ reported that greater the concentration of alkaline materials, higher is the compressive strength. However, for the alkali-activation of fly ash, Palomo *et al.*²⁵ reported that an activator with a 12 M concentration leads to better results than a 18 M concentration. Some authors used free water glass as alkaline activator and reported lower mechanical performance^{11,12}. According to Palomo *et al.*²⁵, the alkaline activator plays a crucial role in the polymerization reaction, behaving more swiftly when the soluble silica is present. Water glass favours the polymerization process leading to a reaction product with more Si and more mechanical strength. Jaarsveld *et al.*^{26,27} claim that the H₂O/SiO₂ molar ratio is important in the study of alkali-activated mixtures.

Another quantitative measure of strength of alkalinity of AAS is pH or molarity, as the breaking down of aluminum and silicate bonds in the source material occurs only beyond a certain threshold pH. Therefore, in the present study, in the absence of a standard procedure, the pH of the paste samples was determined at 1:5 dilution as adopted by ISO 10390: 1994 (ref. 28). The filtrate was titrated against 0.1 M HCl using methyl red as indicator.

Degree of reactivity

Geopolymerization of fly ash occurs via an alkaline reaction followed by polycondensation of the largely amorphous aluminosilicate phase. Degree of reactivity of the geopolymer is related to the rate of fly ash dissolution and how much of the total amorphous aluminosilicate material is available for geopolymerization. The amount of crystalline and glassy phases in the fly ash depends largely on the combustion and gasification process used at the thermal power plant. When the maximum temperature of the combustion process is above 1200°C and the cooling time is short, the ash produced is mostly a glassy

phase material²⁹. Reactive fly ash particles are a mix of crystalline phases embedded within an amorphous aluminosilicate sphere and fly ash spheres with crystalline phases encapsulated within a hollow amorphous aluminosilicate spherical shell. The limiting factor, which has hindered the use of fly ash in geopolymers, is its low reactivity, which often leads to slow setting and strength development. In many cases, the dissolution of fly ash is not complete before the final hardened structure is formed²⁵. In order to improve the reactivity of fly ash and to increase the compactness of the microstructure and hence the compressive strength, the slag was added in the present study. Xu and Van Deventer^{30,31} observed that generally the NaOH solution caused a higher extent of dissolution of minerals than the KOH solution. In the present study an attempt has been made to assess the degree of reactivity of GPCs activated by Na and K compounds. The degree of reactivity is a measure of the portion that does not go into the insoluble residue. The samples for degree of reactivity test were prepared by breaking hardened paste samples, grinding the fragments in agate mortar with pestle and sieving through 75 µm sieve. The hardened pastes were treated with 1:20 HCl (by volume) to determine the percentage of reaction products generated at the respective reaction times. This procedure separates the reaction products (sodium aluminosilicate gel and zeolites, if any) which are dissolved in the acid, from the unreacted ash, which remains in the insoluble residue. The dissolved fraction is a parameter that provides information on how far the reaction has progressed. In order to select a suitable procedure for the determination of insoluble residue in GPC, the insoluble residue was determined by three methods, namely the procedure of Criado *et al.*¹⁸, Pipat Termkhajornkita *et al.*³² and a combination of procedure proposed by Ohsawa *et al.*^{33,34} and ASTM³⁵. The purpose of attempting various methods is to find a suitable method for precisely determining the degree of reactivity of the geopolymer. The procedure of Criado *et al.*¹⁸ and Termkhajornkita *et al.*³² did not yield appropriate results for GPC and was found to be overestimating the degree of reactivity because of the insufficient digestion and chance of residue to be carried away into solution. The results of the combination procedure are presented in this work.

Table 5. Molar computation

	Units	SHSS	SSS	SSST	PHPS	PPS
Fly ash	Grams	370.28	370.28	370.28	370.28	370.28
GGBS	Grams	22.068	22.068	22.068	22.068	22.068
AAS	Grams	310.10	310.10	310.10	310.10	310.10
Na ₂ O	Moles	2157.49	2157.49	2157.49	4.51	4.51
SiO ₂	Moles	4262.92	4262.92	4262.92	4262.92	4262.92
Al ₂ O ₃	Moles	1665.81	1665.81	1665.81	1665.81	1665.81
H ₂ O	Moles	298.91	298.91	298.91	306.50	306.50
H ₂ O/Na ₂ O	Moles	0.138	0.138	0.138	67.96	67.96
SiO ₂ /Na ₂ O	Moles	1.97	1.97	1.97	945.21	945.21
SiO ₂ /Al ₂ O ₃	Moles	2.55	2.55	2.55	2.55	2.55
Water/GPS	Moles	0.76	0.76	0.76	0.77	0.77
Na ₂ O/(Al ₂ O ₃ + SiO ₂)	Moles	0.43	0.43	0.43	0.0007	0.0007
K ₂ O	Moles	47.48	47.48	47.48	2111.10	2111.10
H ₂ O/K ₂ O	Moles	6.29	6.29	6.29	0.14	0.14
SiO ₂ /K ₂ O	Moles	89.78	89.78	89.78	2.01	2.01
K ₂ O/(Al ₂ O ₃ + SiO ₂)	Moles	0.008	0.008	0.008	0.35	0.35

Table 6. Cost estimation of geopolymers

	Cost in Rs for the formulation					
	Cost in Rs/kg	SHSS	SSS	SSST	PHPS	PPS
Fly ash	2	0.74	0.74	0.74	0.74	0.74
GGBS	3	0.07	0.07	0.07	0.07	0.07
Sodium hydroxide	64	3.28	3.28	3.28	–	–
Potassium hydroxide	125	–	–	–	6.54	6.54
Sodium silicate solution	11	1.13	1.13	1.13	–	–
Potassium silicate solution	19	–	–	–	1.99	1.99
Water	0.05	0.004	0.004	0.004	0.002	0.002
AAS	–	4.414	4.414	4.414	8.529	8.529
Sucrose	32	–	–	–	–	–
2% Sucrose solution	–	–	0.064	–	–	0.064
Tartrate	1920	–	–	–	–	–
2% sucrose + 0.5% tartrate solution	–	–	–	9.664	–	–
Total cost in Rs	–	5.22	5.29	14.89	9.34	9.40

Results and discussion

The mix composition and the molar computation of the ingredients of GPC are given in Table 5. Table 4 gives the properties of geopolymer pastes produced. Table 6 provides the cost estimation of the geopolymer pastes.

Workability

The workability of the geopolymer paste was found to depend on the ratio of AAS liquid to binder, composition of AAS and the type and dosage of the chemical admixture used. As seen from Table 4 and Figure 1, the spread was 190 mm for the mix SHSS involving a combination of SH and SS at a liquid binder ratio *l/b* of 0.6, but the mix was difficult to handle due to sticky gel-like consistency. However, the combination of PH and PS in mix PHPS imparted a better spread of 220 mm (flow 120%) and the handling of the mix with a trowel was smooth

even though the *l/b* was reduced to 0.5. This is attributed to the higher water content of PS and its higher water retention (due to slower rate of gelling), besides lower viscosity compared to SS. The use of potassium-based compounds in GPC in lieu of conventional sodium compounds is claimed to impart better workability and improved characteristics to geopolymers by other reporters as well^{30,36}.

The flow of geopolymer paste with sodium activators decreased when sucrose was used as an admixture, while the increase was minimal in the case of PH + PS admixture by a huge margin of 90 mm (flow 30%). Further, the addition of another admixture, tartrate, brought it down to almost zero flow (spread 105 mm) in case of SH + SS. On the other hand, the geopolymer prepared with PH and PS showed increase in workability and the flow showed a marginal increase with the addition of sucrose. The increased solubility and low viscosity of PS compared to SS are believed to be the reasons for the increase in workability due to sucrose.

Setting time

The initial setting time (IST) of the mixes studied is represented in Figure 2. The setting time of the geopolymer paste was found to be strongly dependent on the AAS composition and chemical admixture used. While the SHSS system recorded the highest IST, the PHPS system showed relatively lower IST. The use of sucrose as an admixture decreased the IST and decreased the final setting time. Further addition of tartrate reduced IST even more, almost leading to flash setting interference with geopolymerization due to sucrose.

Compressive strength

The results of the compressive strength tests on geopolymer cement pastes for various mixes at the 7th and 28th day are plotted in Figure 3. The potassium compounds imparted higher compressive strength at both 7 and 28 days compared to sodium compounds. The higher water/alkali metal ratio (H_2O/M_2O) and lower alkali metal to geopolymer solids ratio (M_2O/GPS) and the

better solubility of potassium compounds are the possible causes for the relatively lower strength development of sodium compound activator systems³⁷.

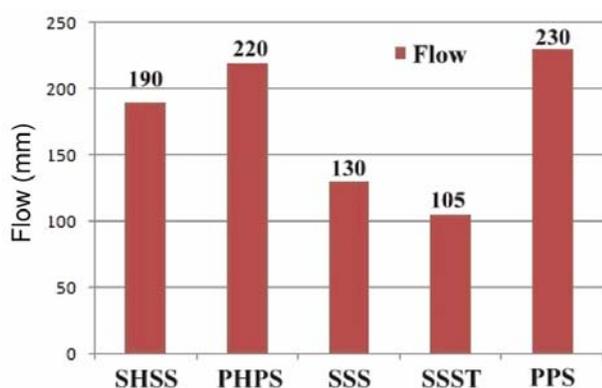
Both tartrate and sucrose are found to interfere with dissolution and polycondensation process and therefore lead to reduced compressive strength. Further, addition of sucrose leads to the formation of surface efflorescence due to free unreacted alkalis oozing to the surface in the relatively porous system (Figure 4).

pH

The pH of the geopolymer was in a narrow range 13.2–13.6 and therefore had a negligible role in influencing the property differences obtained in the study.

Degree of reactivity and compressive strength

The degree of reactivity (DOR) and compressive strength of geopolymer pastes are presented in Figure 4 and the correlation between them is shown in Figure 5. It is



Note: SHSS = SH + SS, PHSS = PH + PS, SSS = SH + SS + S, SST = SH + SS + T, PPS = PH + PS + S.

Figure 1. Effect of admixtures on flow.

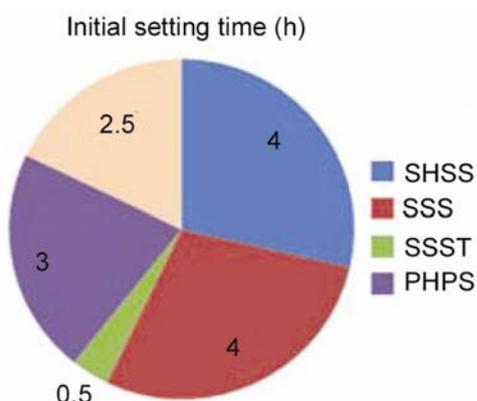


Figure 2. Setting time of various mixes.

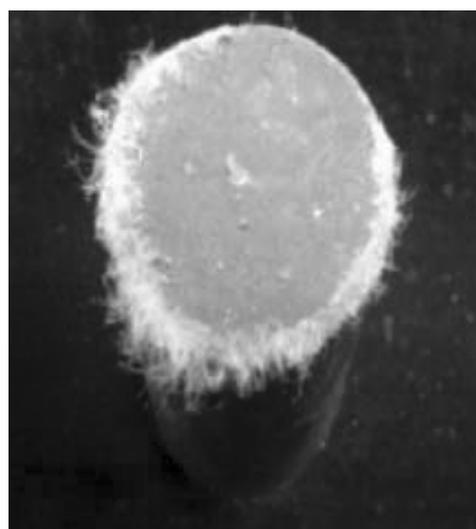


Figure 3. Efflorescence in PPS.

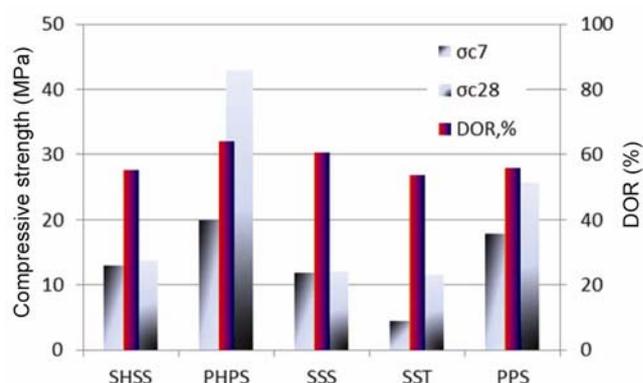


Figure 4. Compressive strength and degree of reactivity.

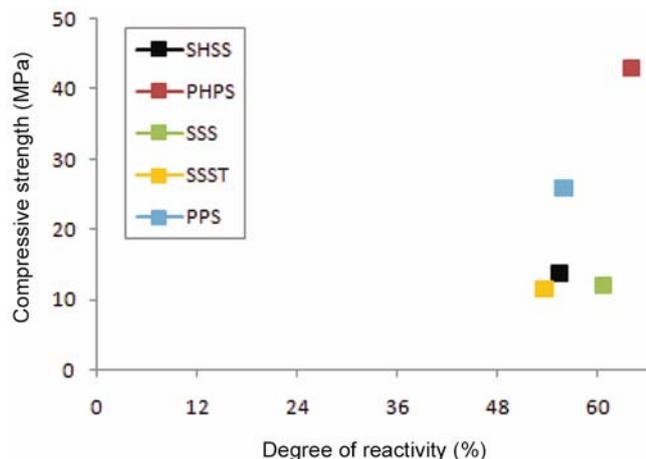


Figure 5. Correlation between degree of reactivity and compressive strength.

observed that higher the degree of reactivity, higher is the compressive strength, except for SSS. More crystalline compound may be formed when sucrose is the admixture which interferes with the amorphous phase and this may be the reason for the decrease in compressive strength of geopolymer paste and efflorescence is an indication of the presence of crystalline compound. Disodium tartrate was added due to its action as a binding agent, but the action of tartrate was nullified in the presence of sucrose.

Choice of PS as an activator provides higher degree of reactivity and increased strength, but its counterpart SS provides less strength with lesser degree of reactivity. This is mainly due to the higher basicity of potassium giving rise to dense polycondensation favouring greater overall network and hence increased strength. The results of the present work are in the range 53.6–64.1% at the age of 28 days with fly ash and slag as raw materials. The results of Fernandez-Jimenez *et al.*³⁸ exhibit the degree of reactivity in the range 60–90%, and the temperature range 85–200°C with the alkaline activation of metakaolin–fly ash mixtures at the age of 1 day. The degree of reactivity (%) was greater in the metakaolin samples than in the fly ash with metakaolin in any of the tested temperatures³⁹. As the content of glassy phase in the ash used in the study was about 76%, the maximum attainable degree of reaction for any fly ash with metakaolin sample lies between about 80% and 85%. As the present work was carried out at room temperature and has utilized 75% fly ash with 25% slag as against 50% fly ash with 50% metakaolin, the results are convincing mainly because metakaolinite reacts almost in its entirety, whereas in the fly ash, only the glassy phase reacts and may not do so fully; the other phases present in the fly ash (quartz, mullite, etc.) are practically unaltered at the activation conditions used. Besides that, the glassy phase of the fly ash used in this study is much less than what was used by the above researchers.

The DOR determined from the content of acid-insoluble residue in the GPC paste was found to be poorly correlated to the compressive strength, clearly indicating that DOR is not a function of insoluble residue content alone and that other factors such as residual alkali content, glass content of source materials and concentration of solutions have a role to play in fixing the compressive strength.

The chemical admixtures play a negative role on the compressive strength of GPC, but the DOR is only marginally affected.

Economic analysis

A comparison of the cost of the different formulations studied as seen from Table 6, indicates that better performance of potassium activators comes with a much higher investment. It can be seen that the cost of PH is almost double that of SH. Using the combination of SH and PS for making AAS, the cost may be brought down.

Conclusions

The potassium compounds imparted the maximum compressive strength as well as workability for the same binder content and for the same alkali to silicate ratio. Potassium compounds are costlier compared to sodium compounds. The liquid demand for potassium-based geopolymer is less compared to sodium-based activators. The potassium-based activators can be used without chemical admixture, whereas an admixture or a higher liquid/paste ratio is necessary for sodium-based activators. However, the use of chemical admixtures in a mix such as sucrose and sucrose with tartrate with sodium-based activators did not improve the strength. Contrary to expectations, sucrose and tartrate did not function synergistically. Sucrose decreased the strength in the case of potassium-based activators.

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