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Fresh and Hardened Properties of Fly Ash–Slag Blended Geopolymer Paste and Mortar

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Abstract

Huge amounts of by-products are generated from industrial processes which affect the environment adversely. Production process of conventional cement is energy demanding and it also emits enormous amounts of greenhouse gases. Geopolymers are the new generation green material that has a great potential of replacing the conventional cementitious materials. The fresh and hardened properties of sodium hydroxide activated binary blends of slag and fly ash based geopolymer paste and mortars are reported in this paper. Experimental outcomes on fresh and hardened properties such as normal consistency, flow value, setting time, drying shrinkage, soundness, and compressive strengths of the geopolymer binders are presented. Additionally, the chemical products, bonding and microstructural changes occurring during the setting and hardening course are examined. The experimental outcomes showed that the physical and mechanical properties of the binders are very much akin to that of conventional cement and the same is significantly influenced by the chemical composition of the source materials, concentration of the activator and the processing environment. The consistency and setting times of geopolymers are found to be within the ranges that are prescribed for ordinary Portland cement. Highest compressive strength of around 44 MPa is obtained for slag based geopolymer mortar that is activated using 8 M sodium hydroxide solution. Fly ash and slag geopolymers exhibited excellent stability against expansion and shrinkage. Raw materials are optimized by design of experiment and the fitted model shows a good relation with the experimental data.

Keywords: geopolymer, fly ash, slag, physical and mechanical properties, optimization technique

1 Introduction

A huge amount of industrial by-products are generated due to the industrial activities, which affects the environment adversely. A large amount of CO_2 is emitted during the conventional cement production processes, which are energy intensive. Geopolymers are an alternative to the traditional cementitious binders which are manufactured by activating the industrial by-products rich in alumino-silicates which include slag, fly ash, red mud, etc. with alkaline solution (Duxson et al. 2007; He and Zhang 2011). Geopolymerization reaction occurs when a suitable alkali activator solution is used to activate alumino-silicate source materials. The geopolymerization reaction which occurs depends on the nature of

the source materials, activator type and the processing conditions (Duxson et al. 2007). Aluminium and silicon–oxygen tetrahedrons form a three-dimensional polymeric structure where alkali cation acts as a charge balancing role for aluminium (Allahverdi et al. 2017). Geopolymers exhibits high compressive strength, low shrinkage, fast/slow setting time with excellent acid and fire resistance (Duxson et al. 2007). The manufacturing process requires less energy and emits about 80 to 90% less greenhouse gas. Considering the economic and environmental aspects, the manufacturing process is beneficial (Deb et al. 2014).

The fresh and hardened properties of cementitious materials are important to use them in construction dealings. The comprehensive properties of cement like consistency and workability are imperative as ease for construction with greater homogeneity. The setting properties of the cementitious binder are also equally critical as those controls the transportation time, placement,

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finishing and other such operations. Also, the mechanical properties such as compressive strength are an important parameter. The composition of raw material, placing and curing condition greatly influences these properties (Samantasinghar and Singh 2018). When deciding the stability aspect, an assessment on soundness and shrinkage behavior of cementitious binder is necessary. The geopolymers are eco-friendly new generation cementitious binders which possess physical and mechanical properties similar to that of an ordinary Portland cement. Fly ash being a pozzolanic material is very often used to enhance the physical and mechanical properties of cement and concrete. Whereas, blast-furnace slag cement is used due to its low heat of hydration and high sulphate and sea water resistance. Researches indicate that fly ash geopolymers show delayed setting under ambient environment. However, slag is relatively faster at setting thus showing a higher strength at an early age. Therefore, addition of slag to the fly ash may substantially influence the strength and setting aspects of the geopolymer, comparable to mechanical and physical properties to that of ordinary Portland cement.

2 Literature Review

Available literatures on geopolymer cement are summarised and discussed. Puertas et al. (2000) concluded that the activator concentration and the slag content showed significant impact on strength development, whereas the effect of curing temperature is minimal while assessing the mechanical behaviour of sodium hydroxide activated fly ash/slag paste. Temuujin et al. (2009a, b) assessed the effect of mechanical activation of fly ash on compressive strength of geopolymer. The milled fly ash resulted in the change in particle size, morphology and an increased reactivity. Songpiriyakij et al. (2010) made fly ash geopolymer using rice husk and bark ash. The reactivity of source material and the quality of matrix formed contributed to the enhanced strength. Nath and Sarker (2014) reported the setting, workability and strength properties of fly ash geopolymer. Fly ash when replaced by a small amount of slag is studied under an ambient condition, where a mixture of sodium silicate and sodium hydroxide is used to activate the material. The workability of geopolymer is not representative of cement due to the high viscosity of the alkaline solution. Increase in the slag content resulted in the reduced workability and setting time and an increased compressive strength. Topark-Ngarm et al. (2014) opined that the presence of high calcium content in fly ash resulted in producing high strength geopolymer concrete with setting time of 28–58 min. Cihangir et al. (2015) studied the effect of activator type, concentration and slag composition on strength

and stability of alkali-activated slag. In short-term curing, sodium hydroxide resulted in higher unconfined compressive strength while sodium silicate resulted in higher unconfined compressive strength during long-term curing. Junaid et al. (2015) considered various parameters for the development of fly ash geopolymer and stated that the alkali concentration, composition of sodium silicate, silicate to hydroxide ratio, temperature and curing time were the influencing parameters. Rao and Rao (2015) studied the setting time and compressive strength of fly ash and slag based geopolymers activated with alkali solution (combination of sodium hydroxide and sodium silicate). Increased alkali concentration resulted an increased setting time while slag addition decreased the setting time.

In this paper, fresh and hardened properties of geopolymer paste and mortar prepared from the fly ash–slag blends activated with sodium hydroxide solution under ambient condition were emphasised. The source material proportion and the activator concentration were the parameters considered in this study. The mineralogical, chemical and microstructural analysis was also carried out to substantiate the physical and mechanical behaviour of fly ash–slag geopolymer pastes and mortars.

3 Materials and Methodology
3.1 Raw Materials

Granulated blast furnace slag (GBS) and class-F fly ash (FA) are used for the preparation of the geopolymer (GP) as the mineral precursor. Standard sand conforming IS: 650 (1991) is used for preparing geopolymer mortar specimen. Slag and the fly ash are collected from slag granulation and captive thermal power plants of the Rourkela Steel Plant (RSP), Odisha, India respectively. These raw materials are dried under sun and thoroughly mixed to achieve homogeneity of the sample. These materials are further oven dried for 24 h at a temperature of 105 °C to 110 °C prior to use. Pulverizing GBS in a ball mill obtained Ground granulated blast furnace slag (GGBS). The Brunauer–Emmett–Teller (BET) specific surface values for FA and GGBS are found to be 28.07 and 22.73 m^2/g respectively. Sodium hydroxide (NaOH) flakes with 98% purity are used as an alkali activator, procured from Loba Chemie Pvt. Ltd., Mumbai, India. Different concentrations of NaOH solutions are prepared a day before its use by dissolving NaOH flakes in distilled water accordingly. X-ray fluorescence (XRF) spectroscopy was used to analyse the oxide composition of raw materials and are summarized in Table 1. Silicon dioxide, aluminium oxide, calcium oxide, and magnesium oxide are among the major constituents found. However, fly ash consist oxides of silicon, aluminium and iron.

Table 1 Chemical composition (wt.%) of raw materials.

Compounds	FA	GGBS	Compounds	FA	GGBS
MgO	0.542	9.700	Fe_2O_3	5.450	0.60
Al_2O_3	28.497	18.00	Na_2O	0.600	0.088
SiO_2	55.706	29.31	MnO	0.430	0.14
K_2O	1.264	1.04	TiO_2	0.611	1.04
P_2O_5	0.605	–	SO_3	0.903	1.40
CaO	2.143	37.2	Loss on ignition	2.549	0.81

3.2 Experimental Program

Vicat's apparatus in accordance with IS: 4031 (Part 4)-1988 (1988) and IS: 4031 (Part 5)-1988 (1988) was used to ascertain the normal consistency and setting times of fresh geopolymer paste respectively. Soundness was obtained according to IS: 4031 (Part 3)-1988 (1988). Drying shrinkage value of FA and GGBS based mortar specimen, activated with different concentrations of NaOH are obtained as per IS: 4031 (Part 10)-1988 (1988). The flow of fresh geopolymer mortar is determined in terms of mortar flow using flow table as prescribed in IS: 5512-1983 (1983). The compressive strength of geopolymer mortar samples at the age of 3, 7 and 28 days are evaluated according to IS: 4031 (Part 6)-1988 (1988). For which, one part of binder was mixed with three parts of Indian standard sand to prepare the mortar specimen. The binder includes fly ash and GGBS mixed in different proportions. The percentage of GGBS in the mix was varied from 0 to 100% at 20% increment each. These are referred as S0, S20, S40, S60, S80 and S100 respectively. The concentrations of NaOH solutions taken are 2 M, 4 M, 8 M, 12 M and 16 M (where 'M' represents Molarity). Specimens for the above mentioned tests are prepared according to the codal provisions except the replacement of water with alkaline solution. All the specimens are prepared, cured, and tested under an average temperature of 29 °C and at a relative humidity of 63%.

The reaction products, chemical bonds and microstructure of NaOH activated fly ash and slag pastes at initial and final setting periods are studied by X-ray diffraction (XRD), Fourier transform infrared (FTIR) and scanning electron microscopy (SEM) analysis respectively. The broken pieces of geopolymer materials after their initial and final setting are collected and soaked in ethanol to discontinue further reactions, if any. These representative samples were finely ground to a size less than 75 μm using mortar and pestle, and then homogenized. The mineralogical analysis (XRD analysis) are done using Rigaku Japan/Ultima-IV model diffractometer with Cu Kα radiation at 40 kV and 40 mA over the range of 5° to 70° 2θ at a scanning rate of 5°/min with step size of 0.05°. X'Pert HighScore software was used to identify the peaks. The secondary analysis was done through the molecular fingerprint resulted from the spectrums obtained by FTIR analysis using PerkinElmer Spectrum version 10.4.00 with a LiTa03 detector for the frequency range of 400–4000 cm^{-1}.

Response model is developed to optimize raw materials by response surface methodology (RSM). RSM is an effective technique which is a collection of both mathematical and statistical modelling. From historical data design interface in the response surface tab, the experimental data is imported and analysed. The predicted values of response (compressive strength) are obtained by model fitting technique. Kumar and Mishra (2015) followed the similar technique for optimization of additives to obtain a best composite material with the highest response. The GGBS content is varied by 10% of FA-GGBS mix and 5 numbers of NaOH concentrations are considered for the design of experiment. Thus, an experimental data set of 55 numbers is used.

4 Results and Discussion

4.1 Normal Consistency

Normal consistency values of various GGBS-FA mixes are found to vary from 26 to 39% (Table 2). These conforms the range recommended for OPC. An increase of GGBS fraction in the mix, results in a reduction of the normal consistency value. The specific surface of fly ash and GGBS are 28.07 and 22.73 m^2/g respectively and both these materials are non-plastic in nature. Owing to greater specific surface area of fly ash, a mix containing higher fraction of fly ash, requires more amount of alkaline solution to lubricate the particle surface and thus it exhibits greater consistency value. However, this finding contests the results reported by Rao and Rao (2015). Further, it is noticed that the consistency values also depend on the concentration of NaOH solution. The consistency values of the mixes are increased with an increase of the concentration of the alkali solution. The viscosity of the NaOH solutions depends on

Table 2 Normal consistency values (%) of FA-GGBS geopolymers.

FA-GGBS mixes	NaOH concentration (M)				
	2	4	8	12	16
S0	30	30	31	33	39
S20	29	29	30	32	38
S40	28	29	30	31	36
S60	27	28	29	31	34
S80	26	28	29	30	32
S100	26	28	29	30	31

its concentration. A highly viscous solution makes the geopolymer paste plastic in nature, which in turn during testing limits the sinking of plunger attached to the Vicat apparatus into the paste. Therefore, the consistency value increases. This effect is more clearly visible in fly ash rich mixtures compared to slag rich mixes. Thus, it can be concluded that the normal consistency values of binary blended FA-GGBS geopolymer mixes are the function of chemical composition, fineness of source materials and the concentration of the alkaline activator solution.

4.2 Setting Time

The initial setting (IS) and final setting (FS) of FA-GGBS mixes are presented in Fig. 1. The IS times of the mixes are found to vary from 25 to 300 min whereas the FS times vary between 40 and 490 min except the mix S0. The S0 (100% fly ash) sample displayed an IS time of 12 h and the FS time exceeds 24 h. This is owing to sluggish chemical reaction of fly ash with time under an ambient room temperature. A similar remark is also made by Temuujin et al. (2009a, b). Further, it is observed that an increase of the concentration of the alkaline activator results in a decrease of setting time. However, no significant reduction in setting times is noticed beyond 8 M concentration. Allahverdi et al. (2010) observed similar trend for slag activated by sodium hydroxide solutions. A significant decrease of setting time is noted with an increase of GGBS content in FA-GGBS mix. This tendency clearly indicates that at an ambient temperature slag rapidly reacts with NaOH solution in comparison to fly ash. The release of Ca^{2+} ions happen beside release of Si^{4+} and Al^{3+} ions from the slag particles in aqueous solution of NaOH. Higher calcium content in slag supports the pozzolanic reaction besides geopolymerization

reaction. These supplementary hydration products along with geopolymeric reaction products decrease the setting times.

4.2.1 Hydration Products, Chemical Bonds and Microstructure

The alumino-silicate phase of slag and fly ash undergoes polymerization process when activated by the alkali solution. GGBS is a rich source of alumino-silicates and also calcium compounds. It undergoes hydration reaction besides geopolymerization process. The reaction compounds formed by the activation of fly ash and slag with 8 M NaOH solution at different periods are examined primarily by XRD analysis. Further, the developments of

Fig. 2 XRD pattern of raw materials and geopolymers at setting periods. 1: Hematite, 2: Mullite, 3: Quartz, 4: Corundum, 5: Ettringite, 6: N-A-S, 7: N-C-A-S, 8: C-A-S-H, 9: C-S-H.

Fig. 1 Setting behaviour of FA-GGBS geopolymers.
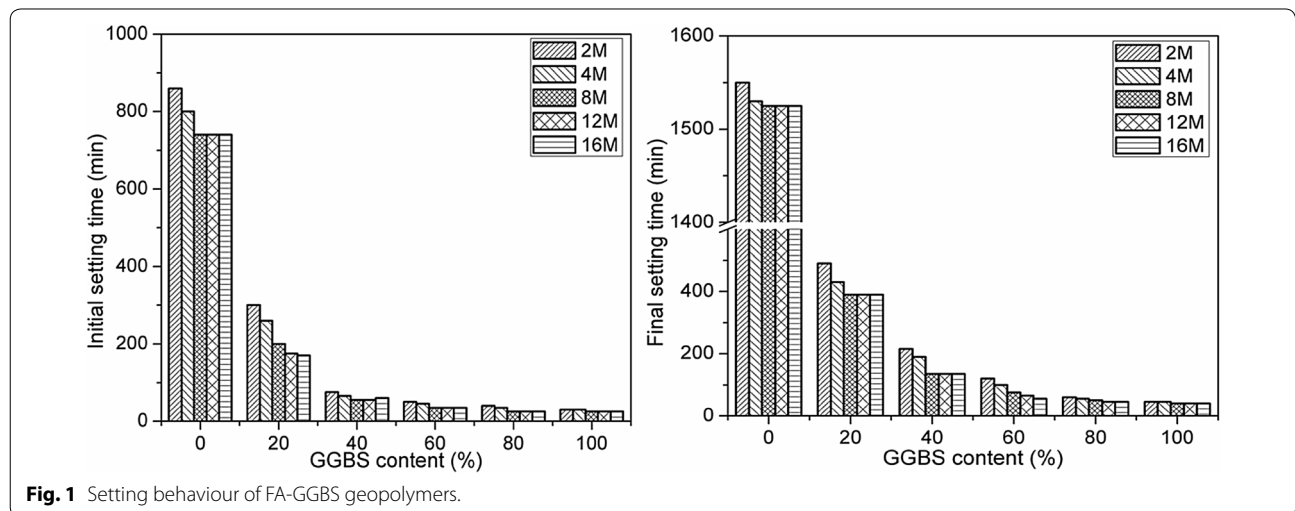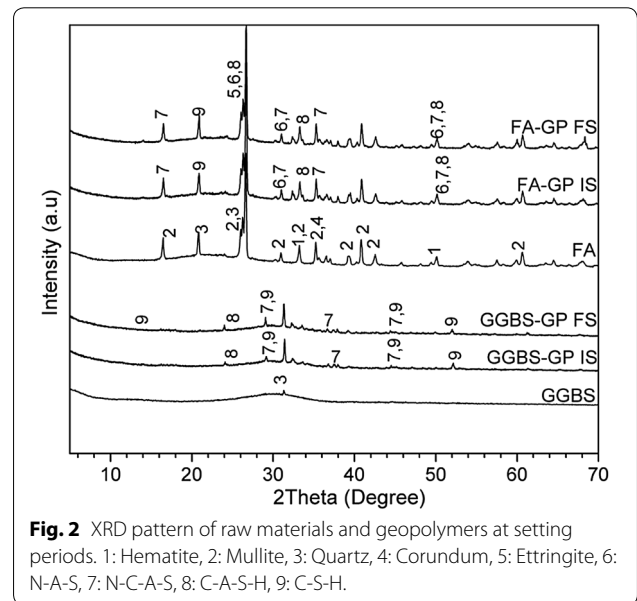
chemical bonds are investigated by FTIR technique. Also, the microstructures are studied from the SEM images.

The XRD patterns of the raw materials are different as the slag is purely a glassy material whereas fly ash encompasses peaks of quartz, hematite, mullite and corundum (Fig. 2). At both the setting periods of FA geopolymer, the peaks of mullite and quartz remain unaltered as in the virgin fly ash specimen. Sodium alumino-silicate (N-A-S) compounds of altisite phase along with less intense peaks representing semi-crystalline sodium-calcium alumino-silicate (N-C-A-S) compounds of liottite, hauyne and franzinite are found at the setting periods of fly ash geopolymer. Further, few peaks of calcium alumino-silicate hydrates (C-A-S-H) and calcium silicate hydrates (C-S-H) are also observed. As the reaction duration increased, these peaks are magnified. XRD micrograph of GGBS geopolymer is quite different from the fly ash based geopolymer. It displayed X-ray amorphous patterns along with humps representing the calcium based hydration products and calcium–sodium alumino-silicate compounds. Calcium silicate hydrate and calcium alumino-silicate hydrate are detected at both the initial and final setting periods. The intensities of these peaks are more prominent at the final setting period. Also, zeolitic hybrid reaction products of hauyne, lazurite, liottite, franzinite are found. The more calcium based products majorly contribute to rapid hardening of the paste which causes early setting and strength gain in slag based geopolymer.

The main characteristic bands of both raw materials and geopolymers after setting are shown in Fig. 3. The peak at wave number 1491 cm^{-1} indicates the presence of calcium in GGBS. Similarly, the peak at wave number 959 cm^{-1} specifies the existence of the siliceous
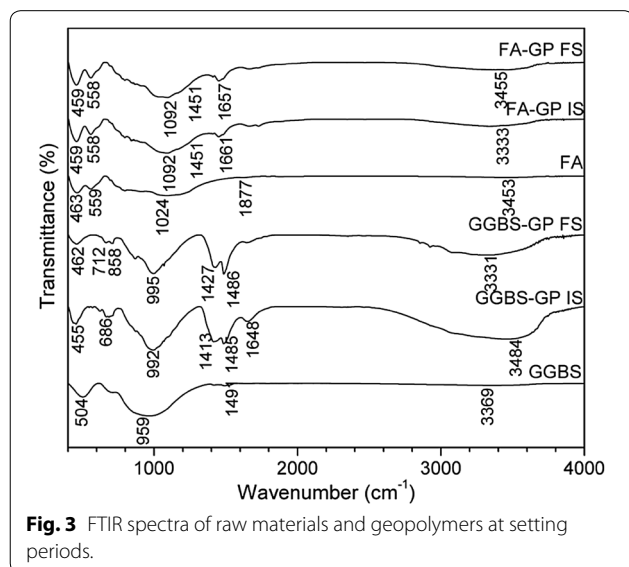
material. This indicates that the slag contains silicate glass. Band width of 463–559 cm^{-1} specifies the existence of silica both in slag and ash specimens. In fly ash sample, the mullite spectra are observed at the main band of 1024 cm^{-1} along with a trivial peak at 797 cm^{-1}. The peak at wave number 1877 cm^{-1} is attributed to the presence of water molecule in fly ash specimen. The low intensity peaks observed at 3453 and 3369 cm^{-1} are due to water molecules in fly ash and slag respectively. After setting, the spectrum of activated FA shows the bending and stretching vibration bands of Si–O which are peaked at wave numbers 459 cm^{-1} and 1092 cm^{-1} respectively. The symmetric stretching vibration bond of T–O–Si (T=Si or Al) is noticed at wave number 558 cm^{-1} for the activated FA specimen. The peak at wave number 1451 cm^{-1} denotes O–C–O stretching vibration band, indicating the absorption of atmospheric carbon dioxide by the specimens. Peaks at 1657 and 1661 cm^{-1} represent S–O bonds. The O–H bands at 3332 cm^{-1} and 3455 cm^{-1} signify the presence of alkali hydroxides. The Si–O and O–H bonds are found to change their wavenumber with an increase in the setting period, indicating greater alkali-silicate hydration products. The peaks observed at wave numbers 455 and 462 cm^{-1} for activated GGBS samples represents the bending vibration bands of Si–O. The symmetric stretching vibration bond of T–O–Si is peaked at 571–712 cm^{-1} at the setting period. The asymmetric stretching vibration bonds of T–O–Si are noticed at wave numbers of 992 and 995 cm^{-1}. The stretching vibration bond of O–C–O is peaked at 1413–1486 cm^{-1}. The broad bands that are noticed at wave numbers 3331 and 3464 cm^{-1} signifies the stretching of O–H groups. The shifting of T–O–Si and Si–O bonds that observed at time specifies the formation of higher amounts of alumino-silicate and silicate hydration products. Similar chemical bonds at their respective wave numbers are reported by Zhang et al. (2014), Abdel-Gawwad and Abo-El-Enein (2016) and Murmu and Singh (2014).

The SEM micrographs with EDX map of samples at their IS and FS times (Fig. 4) indicate the formation of some new structures. Needle-shaped ettringite crystals and amorphous sodium alumino-silicate products are found in NaOH activated ash samples at its initial setting. Partially reacted ash particles are also noticed. However, at its final setting time, these crystals practically vanished and sodium based gel-like products are appeared along with some platy structure. In case of slag geopolymer, both sodium and calcium based reaction products are noticed at initial setting time itself. A solid compact structure is noticed at the final setting time of the slag based geopolymer sample. The more alumino-silicate gel-like substance is observed at the later setting period as more aluminium and silicon are found form the EDX result.
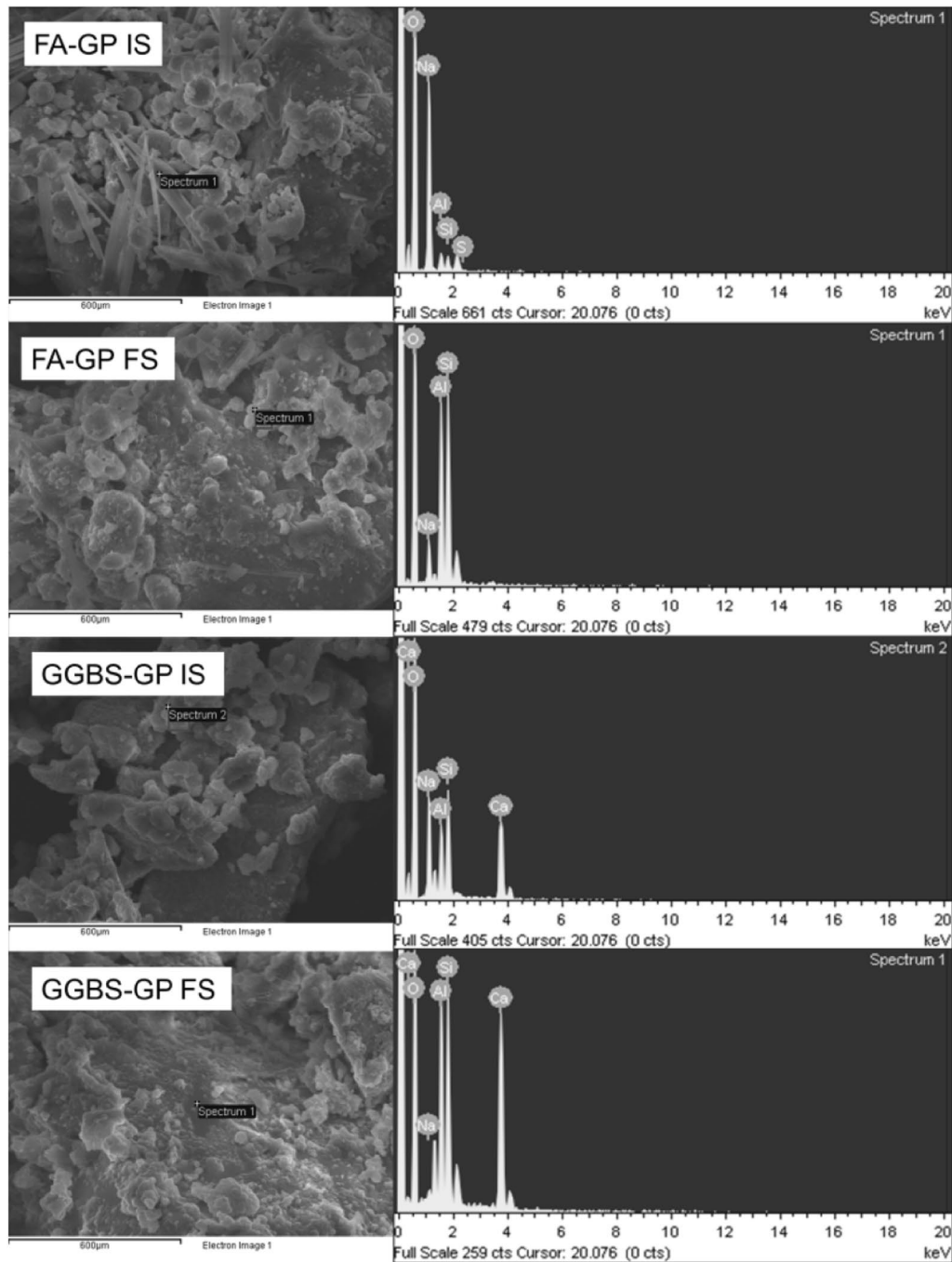

Fig. 3 FTIR spectra of raw materials and geopolymers at setting periods.

Fig. 4 SEM images with EDX map of geopolymers at setting periods.

4.3 Soundness

The occurrence of free lime and magnesia makes the geopolymer binder unsound as they may cause expansion in the hardened binder. The soundness values of geopolymer specimens synthesized by activating fly ash and slag with different concentration of NaOH solutions are assessed. The FA geopolymer exhibited zero

expansion value whereas; an increased expansion of about 1 mm is observed in case of GGBS geopolymer. Commonly, unsoundness of ordinary Portland cement is caused owing to the existence of greater proportion of free lime, magnesia and calcium sulphate. XRF analysis of slag indicates that it contains about 10% of magnesium oxide (Table 1). The expansion observed in S100 mix is

Table 3 Drying shrinkage and flow diameter of FA and GGBS geopolymer.

NaOH concentration (M)	Drying shrinkage (micro-strain)		Flow diameter (mm)	
	S0	S100	S0	S100
2	500	1050	150	137
4	440	900	144	124
8	400	800	130	116
12	360	770	124	104
16	320	750	112	101

attributed to the occurrence of free lime and magnesia present in slag specimen. Also, the development of reaction compounds such as ettringite and gypsum might have caused expansion. However, due to higher pozzolana content of raw materials, the available lime and magnesia get consumed in chemical reactions thus make the binder sound. As per Indian standards, the soundness of cementitious materials should not be more than 10 mm. Therefore, as per the soundness requirements of the cementitious materials the FA-GGBS based geopolymers satisfies the same.

4.4 Drying Shrinkage

The drying shrinkage behaviour of slag and fly ash mortar specimens, prepared using NaOH solutions of varied concentrations, are determined by performing shrinkage tests after curing periods of 28 days. The experimental findings are presented in Table 3. It is noticed that slag specimens exhibited greater shrinkage values than specimens prepared from fly ash. This is attributed to the decrease in hydral volume during the hydration reaction of slag at later stage of curing. Additionally, the concentrations of alkali solution are found to influence the shrinkage values of specimens. A higher concentration solution reduces the shrinkage value. This is owing to the loss of higher amount of capillary water that is available in the samples prepared at low molarity of NaOH solution. The shrinkage values of slag geopolymers reduced remarkably up to 8 M of NaOH; thereafter an insignificant reduction is observed with increase of alkali concentration. At lower concentration of NaOH, leaching of alumino-silicates are comparatively sluggish whereas slag specimens activated with NaOH solutions of 8 M or higher concentration develops the skeletal matrix of geopolymer quickly which restricts further shrinkage.

4.5 Flow

The workability of the FA and GGBS based geopolymer mortars; that are activated by NaOH solutions of various concentrations; are studied by measuring the flow

value of the binder. The flow table test is adopted as water comes out form the viscous geopolymer mortar during geopolymerization process. The flow value of the geopolymer mortars are determined by measuring the flow diameter of the mortar. These are presented in Table 3. The flow varied between 101 and 150 mm, depending on the raw material's property and alkali concentration. Fly ash geopolymer mortar displayed higher flow value than slag based geopolymer irrespective of the concentration of the activating solution. This is related to arrangement of the particles. More spherical particles of fly ash encourage the free flow of the mortar while the angular slag particles hinder the flow due to higher interlocking between particles. Furthermore, the faster dissolution of alumino-silicates of slag renders the alkali solution more viscous. It is noticed that higher the concentration of alkali solution, lesser is the flow. The mortar became stiff and the flow reduces as the viscosity of NaOH solution increases. At lower NaOH concentration, the flow is more due to lubricating effect of alkaline solution. But highly concentrated alkaline solution increases the cohesiveness of the pore fluid that restricts the flow and hence decreases the flow diameter.

4.6 Compressive Strength

4.6.1 Effects of Alkali Concentration

Figure 5 presents the variation of compressive strength of 28 days cured FA-GGBS mortar specimens with the strength of NaOH solution. It is observed that the concentration of alkali solution played a key role in strength gain. With the increase of the concentration from 2 to 8 M, the strength increased significantly. Thereafter, the strength reduces as the concentration of NaOH solution increased further from 8 to 12 M. The improvement in strength is mostly governed by the quantity of leached
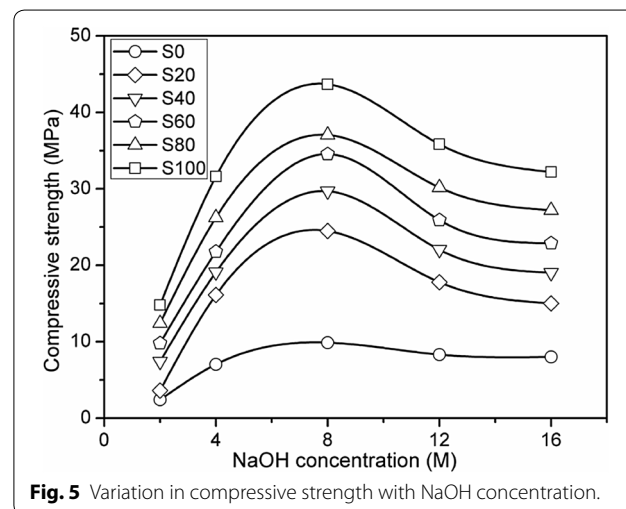
Fig. 5 Variation in compressive strength with NaOH concentration.

alumino-silicates from the source materials. In the polymer structure, the OH^- ion from the alkaline solution plays as catalyst in the reaction process and stimulates the Si^{4+} and Al^{3+} ions dissolution from the source material. Further, the Na^+ cation balances the charge deficit of the matrix (Marjanovic et al. 2015). Leaching is slow at low alkaline concentration. This results in weaker polymeric structure with inferior strength. At a high base condition the leaching is comparatively higher. However, beyond an optimum concentration of alkali, the excess leaching of silica delays the geopolymerization process by congealing particle surfaces (Rattanasak and Chindaprasirt 2009). This inhibits further leaching of ions resulting in a reduction of the strength. Further, the extent of Na^+ ion needed to form a firm geopolymeric matrix may be fulfilled at 8 M NaOH concentration. More than this concentrations, an excess hydroxide ion (OH^-) although increases the rate of dissolution but lowers the polycondensation process by precipitation of alumino-silicate gel (Kotwal et al. 2015). This reduces the compressive strength. Further, it is noticed that the strength of the geopolymer binders is considerably influenced by the GGBS content in the mixture. A higher solubility of slag in alkaline medium increases the concentration of Si^{4+} and Al^{3+} ions in aqueous phase. This promotes faster polycondensation and thus aids stronger polymeric matrix. Thus increased GGBS content in the mix resulted in enhanced strength. This is because of the presence of abundant reactive alumino-silicates in slag that easily dissolves in alkali solution. Additionally, the presence of CaO in GGBS adds to the strength owing to the development of calcium based compounds like C-S-H and C-A-S-H gel. An extremely soluble alumino-silicate source material in alkaline phase increases the Si^{4+} and Al^{3+} ions content in the aqueous phase. This enhances the polycondensation process and consequently, a stronger material is produced. This finding is similar to the observations reported by Deb et al. (2014) and Degirmenci (2016).

4.6.2 Effect of Curing Period

Figure 6 presents the compressive strength of fly ash and slag mortars activated with 8 M NaOH solution for different curing periods. It is noticed that with the increase of curing time, the compressive strength increases. GGBS mortar specimens attained 59% and 77% of its 28 days strength at curing periods of 3 and 7 days respectively. However, fly ash specimens achieved only 23% and 51% of 28 days strength. The strength gain with time is very quick for slag based geopolymer than the fly ash based geopolymer specimens. This is due to the slow leaching
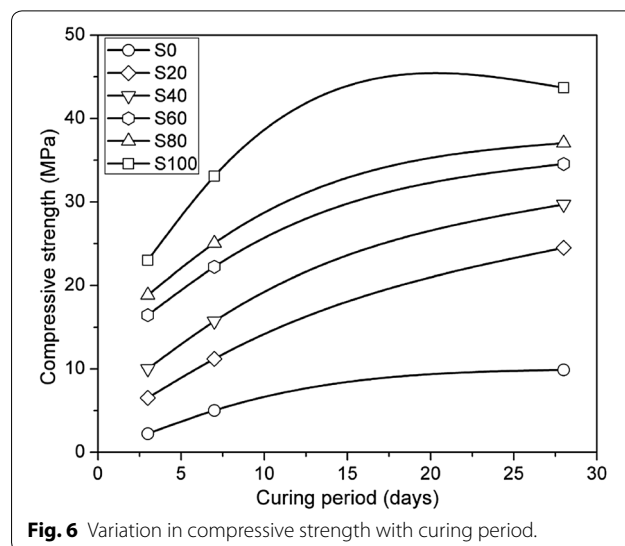

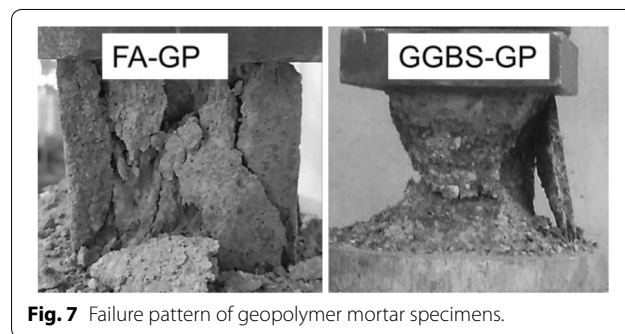**Fig. 6** Variation in compressive strength with curing period.

Fig. 7 Failure pattern of geopolymer mortar specimens.

of fly ash samples at ambient curing condition (Li et al. 2013). Further, it is noted that the compressive strength of S100 specimen almost stabilizes within 28 days of curing. However, fly ash rich specimens show an increasing trend up to 28 days of curing. As slag contains compounds of calcium, it promotes quick setting and also early strength gain. Previous studies on slag based geopolymers have identified the similar properties (Li et al. 2013; Nath and Sarker 2014; Samantasinghar and Singh 2018).

The failure pattern of the S0 and S100 geopolymer specimens; during the compressive strength test is shown in Fig. 7. Cracks developed on four exposed surfaces of the cubical specimens without much damage to the surfaces in touch with the bottom and top plates of the equipment. Fly ash specimens displayed distributed cracks representing quasi-brittle nature of the failure. The developed failure planes are vertical in nature with zigzag shape representing non-explosive failure type whereas, GGBS specimen displayed cleavage cracking
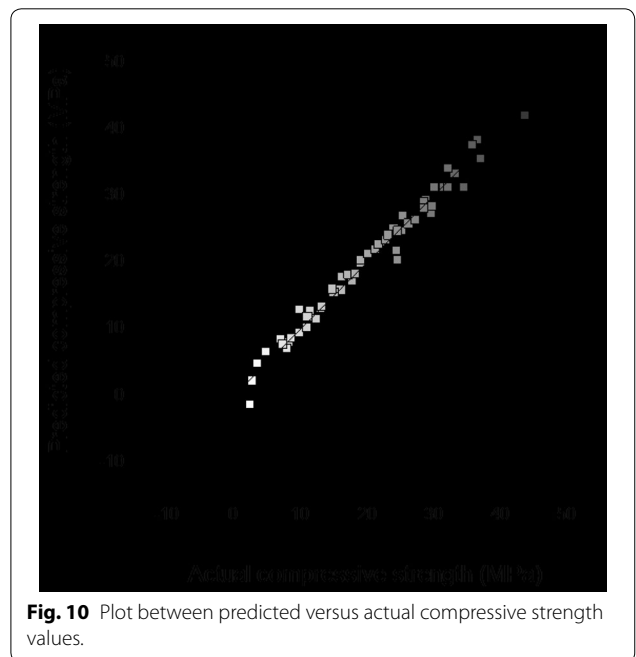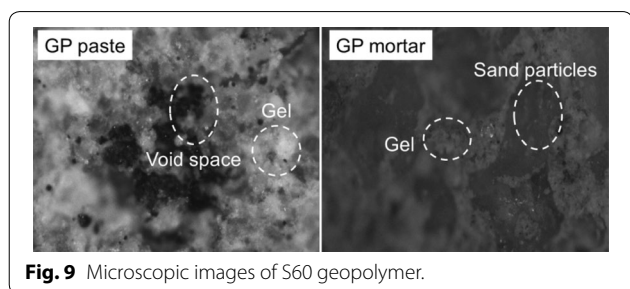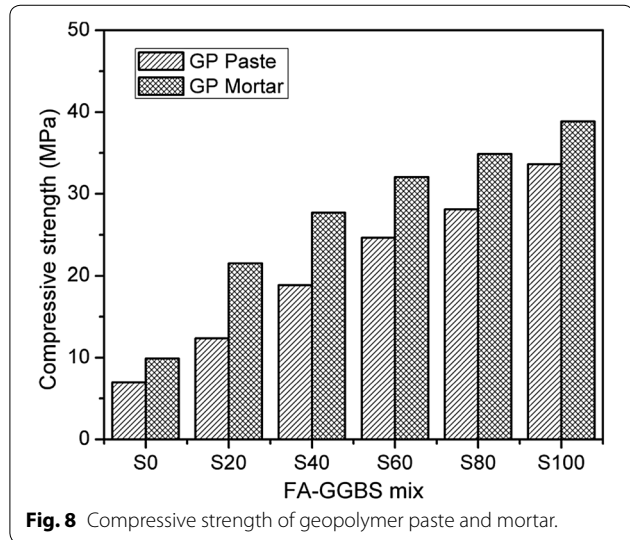
with a semi-explosive failure type. The different kinds of failures are described in BS EN 12390-3: 2009. Such kind of failure in slag based geopolymer is due to prevalence of high cohesive force due to development of a greater amount of reaction compounds as compared to fly ash based geopolymer.

The 28 days compressive strength of FA-GGBS geopolymer mortar and paste specimens made with NaOH solution of 8 M concentration are presented in Fig. 8. The geopolymer mortar specimens yielded greater compressive strength in comparison to geopolymer paste specimens since the strength of sand is generally higher than the cement paste. However, a similar trend in the rate of strength gain is observed for both geopolymer mortar and paste specimens. When the raw materials are activated with alkali solution, the leaching of alumino-silicates along with other minerals started in an aqueous phase followed by polycondensation process. The reaction compounds bind unreacted particles and thus a dense solid structure is formed. In geopolymer mortar specimens, the voids may be filled with fine unreacted materials whereas, micro voids may possibly be created in the geopolymer paste specimens. Figure 9 displays the magnified image of S60 geopolymer mortar and paste specimens. A solid

non-porous structure is noticed in the geopolymer mortar specimen whereas large numbers of fine pores are observed in geopolymer paste specimen. It is understood that during polycondensation process of the binder, water molecules are removed out of the geopolymer matrix, thereby, forming numerous micro-voids. Incorporation of sand forms a compact skeletal framework with well-connected sand particles and the geopolymeric gel fills up the matrix with the inter-particle voids space between the coarse grained sand structures being closed. An interfacial bonding between the reaction products and the sand particles greatly influences the compressive strength. Therefore, a dense and compact matrix is formed, that possesses a relatively greater compressive strength than the hardened geopolymer pastes.

4.7 Optimization

The optimization of GGBS content (A) and NaOH concentration (B) giving the maximum compressive is done by RSM. The experimental values of response for the input parameters are assembled and built-in with the non-linear cubic polynomial model as it showed a good coefficient of determination with a lower standard error. The scattering of predicted responses with the observed values of compressive strength for 28 days cured specimens are presented in Fig. 10, which indicates a better correlation between the predicted and actual values of response with co-efficient of determination (R^2) of 0.9793. The statistical model expressing the 28 days compressive strength is given below:

Fig. 8 Compressive strength of geopolymer paste and mortar.

Fig. 9 Microscopic images of S60 geopolymer.

Fig. 10 Plot between predicted versus actual compressive strength values.

$$UCS_{28days} = 28.85 + 10.13A - 5.28B + 1.72AB$$
$$- 1.99A^2 - 13.66B^2 - 0.72A2B - 4.51AB^2$$
$$+ 4.77A^3 + 11.94B^3 \left(R^2 = 0.9793\right)$$

The 3-dimensional surface plot of the predicted output response is shown in Fig. 11. It indicates that the UCS response increases with GGBS content but in case of NaOH concentration UCS value increases up to a certain limit. To optimize raw materials, 60% GGBS content is targeted and NaOH concentration is selected in range to get maximum compressive strength. This GGBS content is so targeted to satisfy the setting aspect too. The resulted contour plot of desirability and compressive strength (Fig. 12) indicates a maximum compressive

Fig. 11 Response surface plot of predicted compressive strength.

strength of 31 MPa with 0.83 desirability for 60% GGBS content and 8 M NaOH concentration.

5 Conclusions
Consistency, flow, setting, shrinkage, soundness, and compressive strength values of slag-fly ash blended geopolymers are investigated. The chemical composition and microstructure changes associated to physical and mechanical properties are correlated. Based on experimental outcomes, the following conclusions are drawn:

1. FA-GGBS mixes activated by alkali showed normal consistency values ranging from 26 to 39%. The consistency value decreases with an increase in the slag content. Concentration of NaOH solution has nominal influence on the consistency values of the slag rich mixes than the mixes rich in fly ash.
2. Slag rich mix exhibits quicker setting than mixes rich in fly ash. The concentration of NaOH solution accelerates the setting. The setting times of blended fly ash–slag geopolymer except 100% FA fall in the range of setting time for cement. Hybrid sodium-calcium alumino-silicate geopolymeric compounds along with calcium based hydration compounds are the major reaction products observed in FA-GGBS geopolymer after setting and hardening of the paste.
3. Both the fly ash and slag geopolymer show good stability by exhibiting low soundness and drying shrinkage values regardless of the concentrations of the NaOH solutions. The chemical composition, fineness, and shape of these source materials and the concentration of alkali solution control the flow value of these mortars.

Fig. 12 Contour plot of desirability and compressive strength.
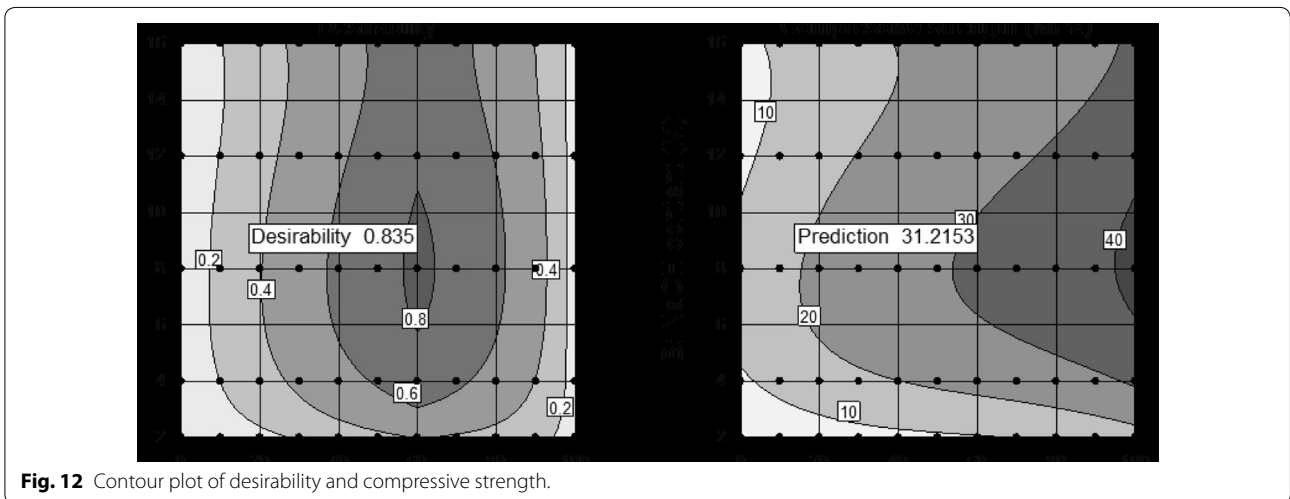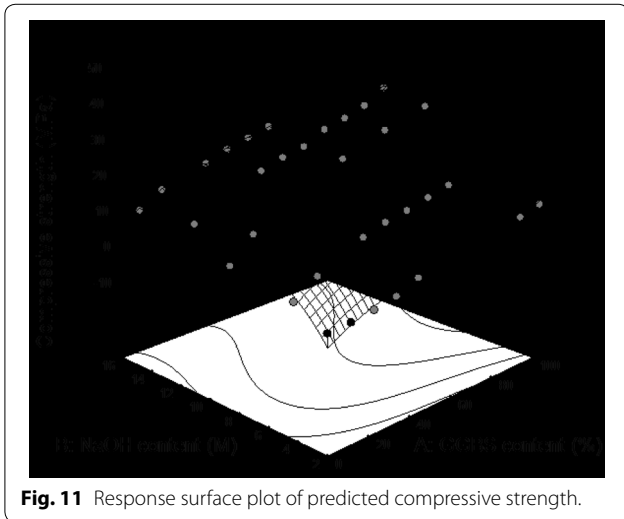
4. The compressive strength of mortar specimens increases with an increase of slag content in the mix. The optimum concentration of the activator solution is found to be 8 M. Highest compressive strength of 44 MPa is recorded at this concentration. Geopolymer mortars show comparatively higher compressive strength than that of geopolymer pastes. Calcium based alumino-silicate hydration products and sodium based alumino-silicate complexes are the main reaction compounds accountable for the strength gain in slag and fly ash based geopolymers respectively.

5. The optimized result showed 60% GGBS content and 8 M NaOH content gives a maximum UCS of 31 MPa with a desirability value of 0.83.

The concentration of alkali and slag content are the influential parameters governing the physico-mechanical property of FA-GGBS blended binary geopolymer. The consistency, setting, soundness and strength properties are greatly influenced by these parameters. Finally, it is concluded that the use of industrial waste products such as slag and fly ash could be utilized for production of a sustainable, eco-friendly cementing material comparable to conventional cementitious materials.

Authors' contributions
The experimental design was defined by SPS. All experiments was carried out by SS. The experimental results and discussions were made by SS and SPS. The paper was wrote by SS and corrected by SPS. Both authors read and approved the final manuscript.

Competing Interests
The authors declare that they have no competing interests.

References
Abdel-Gawwad, H. A., & Abo-El-Enein, S. A. (2016). A novel method to produce dry geopolymer cement powder. *HBRC Journal, 12*(1), 13–24.

Allahverdi, A., Kani, E. N., & Shaverdi, B. (2017). Carbonation versus efflorescence in alkali-activated blast-furnace slag in relation with chemical composition of activator. *International Journal of Civil Engineering, 15*(4), 565–573.

Allahverdi, A., Shaverdi, B., & Kani, E. N. (2010). Influence of sodium oxide on properties of fresh and hardened paste of alkali-activated blast-furnace slag. *International Journal of Civil Engineering, 8*(4), 304–314.

BS EN 12390-3. (2009). *Testing hardened concrete. Compressive strength of test specimens.* London: British Standards Institution.

Cihangir, F., Ercikdi, B., Kesimal, A., Deveci, H., & Erdemir, F. (2015). Paste backfill of high-sulphide mill tailings using alkali-activated blast furnace slag: Effect of activator nature, concentration and slag properties. *Minerals Engineering, 83,* 117–127.

Deb, P. S., Nath, P., & Sarker, P. K. (2014). The effects of ground granulated blast-furnace slag blending with fly ash and activator content on the workability and strength properties of geopolymer concrete cured at ambient temperature. *Materials and Design (1980–2015), 62,* 32–39.

Degirmenci, F. N. (2016). Utilization of natural and waste pozzolans as an alternative resource of geopolymer mortar. *International Journal of Civil Engineering,* 1–10.

Duxson, P., Fernandez-Jimenez, A., Provis, J. L., Lukey, G. C., Palomo, A., & Van Deventer, J. S. J. (2007). Geopolymer technology: The current state of the art. *Journal of Materials Science, 42*(9), 2917–2933.

He, J., & Zhang, G. (2011). Geopolymerization of red mud and rice husk ash and potentials of the resulting geopolymeric products for civil infrastructure applications. *Developments in Strategic Materials and Computational Design II: Ceramic Engineering and Science Proceedings, 32,* 45–52.

IS: 4031 (Part 10)-1988. (1988). *Methods of physical tests for hydraulic cement. Determination of drying shrinkage.* New Delhi: Bureau of Indian standards.

IS: 4031 (Part 3)-1988. (1988). *Methods of physical tests for hydraulic cement. Determination of soundness.* New Delhi: Bureau of Indian standards.

IS: 4031 (Part 4)-1988. (1988). *Methods of physical tests for hydraulic cement. Determination of consistency of standard cement paste.* New Delhi: Bureau of Indian standards.

IS: 4031 (Part 5)-1988. (1988). *Methods of physical tests for hydraulic cement. Determination of initial and final setting times.* New Delhi: Bureau of Indian standards.

IS: 4031 (Part 6)-1988. (1988). *Methods of physical tests for hydraulic cement. Determination of compressive strength of hydraulic cement other than masonry cement.* New Delhi: Bureau of Indian standards.

IS: 5512–1983. (1983). *Specification for flow table for use in tests of hydraulic cements and pozzolanic materials.* New Delhi: Bureau of Indian standards.

IS: 650. (1991). *Standard sand for testing cement-specification.* New Delhi: Bureau of Indian standards.

Junaid, M. T., Kayali, O., Khennane, A., & Black, J. (2015). A mix design procedure for low calcium alkali activated fly ash-based concretes. *Construction and Building Materials, 79,* 301–310.

Kotwal, A. R., Kim, Y. J., Hu, J., & Sriraman, V. (2015). Characterization and early age physical properties of ambient cured geopolymer mortar based on class C fly ash. *International Journal of Concrete Structures and Materials, 9*(1), 35–43.

Kumar, H., & Mishra, M. K. (2015). Optimization and evaluation of fly ash composite properties for geotechnical application. *Arabian Journal of Geosciences, 8*(6), 3713–3726.

Li, X., Wang, Z., & Jiao, Z. (2013). Influence of curing on the strength development of calcium-containing geopolymer mortar. *Materials, 6*(11), 5069–5076.

Marjanovic, N., Komljenovic, M., Baacarević, Z., Nikolic, V., & Petrovic, R. (2015). Physical–mechanical and microstructural properties of alkali-activated fly ash–blast furnace slag blends. *Ceramics International, 41*(1), 1421–1435.

Murmu, M., & Singh, S. P. (2014). Hydration products, morphology and microstructure of activated slag cement. *International Journal of Concrete Structures and Materials, 8*(1), 61–68.

Nath, P., & Sarker, P. K. (2014). Effect of GGBFS on setting, workability and early strength properties of fly ash geopolymer concrete cured in ambient condition. *Construction and Building Materials, 66,* 163–171.

Puertas, F., Martinez-Ramirez, S., Alonso, S., & Vazquez, T. (2000). Alkali-activated fly ash/slag cements: Strength behaviour and hydration products. *Cement and Concrete Research, 30*(10), 1625–1632.

Rao, G. M., & Rao, T. G. (2015). Final setting time and compressive strength of fly ash and GGBS-based geopolymer paste and mortar. *Arabian Journal for Science and Engineering, 40*(11), 3067–3074.

Rattanasak, U., & Chindaprasirt, P. (2009). Influence of NaOH solution on the synthesis of fly ash geopolymer. *Minerals Engineering, 22*(12), 1073–1078.

Samantasinghar, S., & Singh, S. P. (2018). Effect of synthesis parameters on compressive strength of fly ash–slag blended geopolymer. *Construction and Building Materials, 170,* 225–234.

Songpiriyakij, S., Kubprasit, T., Jaturapitakkul, C., & Chindaprasirt, P. (2010). Compressive strength and degree of reaction of biomass-and fly ash-based geopolymer. *Construction and Building Materials, 24*(3), 236–240.

Temuujin, J. V., Van Riessen, A., & Williams, R. (2009a). Influence of calcium compounds on the mechanical properties of fly ash geopolymer pastes. *Journal of Hazardous Materials, 167*(1), 82–88.

Temuujin, J., Williams, R. P., & Van Riessen, A. (2009b). Effect of mechanical activation of fly ash on the properties of geopolymer cured at ambient

temperature. *Journal of Materials Processing Technology, 209*(12), 5276–5280.

Topark-Ngarm, P., Chindaprasirt, P., & Sata, V. (2014). Setting time, strength, and bond of high-calcium fly ash geopolymer concrete. *Journal of Materials in Civil Engineering, 27*(7), 04014198.

Zhang, M., El-Korchi, T., Zhang, G., Liang, J., & Tao, M. (2014). Synthesis factors affecting mechanical properties, microstructure, and chemical composition of red mud–fly ash based geopolymers. *Fuel, 134,* 315–325.

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