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# Engineering properties of geopolymer prepared by mild chemical activation of ground granulated blast furnace slag

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In this research article engineering properties of solely ground granulated blast furnace (GGBS) slag geopolymers have been investigated at varying proportions of alkali activators. Combination of sodium hydroxide (SH) and sodium meta silicate (SMS) powder has been used as alkali activators. Consistency, setting time and compressive strength have been determined at different concentration of SH and ratio of SMS/SH for GGBS geopolymer. Maximum compressive strength of 87.76 MPa has been achieved at 2 molar concentration of SH and SMS/SH = 2.0 after 28 days of curing at ambient temperature. The consistency of mix attaining maximum compressive strength is 27.5%. The initial and final setting time of 55 and 105 minutes has been observed. The soundness of geopolymer paste is within permissible limits. The micro-structural analysis of GGBS and geopolymer paste has been performed by XRD, FTIR, FESEM and EDAX. The co-existence of calcium silicate hydrate gel and geopolymeric gel may be responsible for achieving high compressive strength at low concentration of activators under ambient temperature curing.

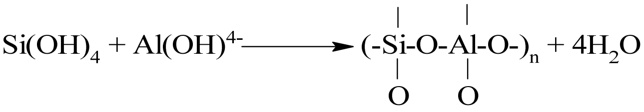
**Keywords:** Geopolymer, Fly ash, Ground granulated blast furnace slag

## 1 Introduction

Increasing population, urbanization and industrialization has lead to rapid growth in construction activities *e.g.* buildings, road networks, bridges and dams *etc.* thereby accelerating demand of portland cement (PC) production. PC is ranked second world wide in material consumption after water. It is most commonly used binding agent for making concrete structures. Production of PC releases large amount of carbon dioxide in environment due to calcination of limestone and heating of raw material in rotary kiln at 1450 °C temperature contributing approximately 5-7% of global anthropogenic carbon dioxide emissions[1]. Manufacturing of PC leads to excessive consumption of natural resources and huge amount of energy further enhancing carbon dioxide emissions. Hence, production of PC, being detrimental to environment, is required to be minimized and suitable alternative binder with low carbon footprints needs to be developed[2].

Davidovits has developed Geopolymer (GP) binder as an eco-friendly solution to carbon dioxide emissions caused by cement production[3]. Generally, GP is synthesized by reaction of materials rich in alumina and silica, like fly ash (FA), metakaoline, ground granulated blast furnace slag (GGBS) *etc.* in the presence of highly alkaline hydroxide or silicate solution. During geopolymerization process, under high alkaline conditions dissolution and hydrolysis reactions of alumino-silicate result in formation of silicate and aluminate tetrahedral units[4]. The tetrahedral units alternatively link to polymeric precursor by sharing oxygen atom, thus, forming polymeric Si–O–Al–O bonds[5]. Three types of structures poly (sialate) (–Si–O–Al–O), poly (sialate-siloxo) (Si–O–Al–O–Si–O) and poly (sialate-disiloxo) (Si–O–Al–O–Si–O–Si–O) are mainly recognized in 3-D alumino-silicate stable network[6]. The schematic formation of geopolymer material is described below in equation (1) and (2) [6]:

$$\mathrm{(Si_2O_5.Al_2O_2)_n + H_2O + OH^- \longrightarrow Si(OH)_4 + Al(OH)^{4-}} \quad \dots (1)$$



$$\mathrm{Si(OH)_4 + Al(OH)^{4-} \longrightarrow (-\underset{\underset{O}{|}}{\overset{|}{Si}}-O-\underset{\underset{O}{|}}{\overset{|}{Al}}-O-)_n + 4H_2O} \quad \dots (2)$$

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These dissolved aluminate and silicate species restructure and condense among themselves to form Si-O-Si and Si-O-Al framework[7]. Denser geopolymeric gel enhances degree of geopolymerization, which makes it suitable as construction binder for infrastructural development. Due to these unique properties, geopolymeric binders possess excellent mechanical strength at early age, higher resistance to elevated temperature, chemicals, like sulphates, acids *etc*[8]. Such improved characteristics has encouraged researchers to focus on alternative binders to reduce use of cement for infrastructural development, as GP demonstrates similar or/and enhanced mechanical and durability properties as compared to conventional cement concrete[9].

Geopolymerization of FA (a thermal power plant waste) takes place, when it is subjected to thermal curing at high temperatures. Recent studies indicate that FA based GP has initial setting time (IST) ~ 1535 minutes and final setting time (FST) ~ 2220 minutes when activated by sodium hydroxide (SH) and sodium silicate (SS) solution having ratio of SS/SH = 2[10]. FA based GP are generally prepared using high concentration of alkali activator ~ 10 molar (M) SH solution and SS solution with SS/SH = 1 and requires high curing temperature ~65°C for a period of 48 hours[11]. Attempts have been made to reduce time required for high temperature curing by adding metakaoline to FA based GP and it is revealed that ~9 M SH and SS solution with SS/SH = 2 can be used to reduce curing period to 2 hours at 60°C to achieve GP paste with strength of 25 MPa[12]. FA based GP attains desired compressive strength when cured at elevated temperature in the range of 40 to 85°C, as it comprises of large volume of crystalline phase, and elevated temperature curing catalyses the geopolymerization reaction[13]. In-situ field applications of FA based GP becomes impracticable as it requires thermal curing at high temperature, thus, limiting its usage to pre-cast units only.

Such limitations have been overcome by partial replacement of FA by additives like, metakaolin, nano-silica, OPC, GGBS, *etc.* in FA based GP binder. It is reported that addition of certain amount of GGBS in FA based GP achieves good compressive strength at ambient temperature curing[14]. A compressive strength of 78.2 MPa with 56 days curing at ambient temperature is achieved for GP prepared by FA (50%) and GGBS (50%) activated with 16 M SH and liquid SS having SS/SH = 1[15].

FA based GP binder with additives, have also shown improved engineering properties e.g. compressive strength, setting time *etc.* The setting times of GGBS blended FA based GP (upto 15%) are comparable to that of conventional OPC[10]. Though, compressive strength is achieved at ambient curing temperature, eliminating need for accelerated curing by incorporation of slag in FA based GP activated with higher concentration of SH solution, however, leaching problem is observed on account of higher concentration of SH solution.

Different routes were tried to synthesize GP by blending alumino-silicate source material (SM) with activators, that is, SH and SS in solid/liquid phase and procedure followed for their preparation can be well understood in Fig. 1[16].

Blast furnace slag (BFS) is an industrial by-product obtained during extraction of pig iron from its ore[17]. Around 300 - 540 kg BFS is generated per tonne of pig iron production[17]. BFS is ground finely to produce GGBS. Its dumping on landfills poses threat to environment. Being rich in oxides of Si, Al and Ca in amorphous phase, GGBS has higher activation potential thus, it is suitable as source material for synthesis of GP.

Although a lot of research had been done on strength and durability development of GP with blending of various source materials, only handful studies were attempted for preparing solely GGBS based GP by researchers, therefore there arises a necessity to investigate the effects of varying ratios of alkaline activator on mechanical properties of GGBS based geoploymer, in particular[9,18,19]. GP concrete prepared using 8 M and 12 M SH in addition to SS

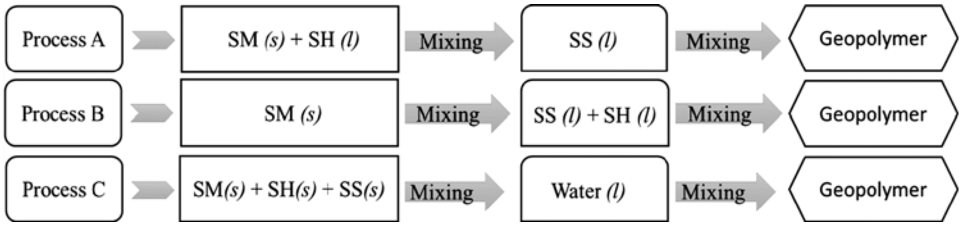


Fig. 1 — Different methods of preparing geopolymer[16].

attained desired strength under 75°C steam curing and 80°C hot air oven curing respectively[20, 21]. Paste and mortar were prepared using SS solution along with 10M and 9M SH solution, cured under ambient temperature to achieve 28 days compressive strength of 168.7 MPa and 65 MPa respectively[19, 22]. Even though the strength achieved is significant, but, use of higher concentration of alkali activator releases large amount of heat, which poses high risk and handling trouble during its mixing while making GP. Furthermore, higher concentration of alkali solution increases cost of GP synthesis, which is uneconomical. Scarcely any study is available on exploring GGBS for producing GP at low concentration without compromising its engineering properties. Thus, further research is desired to focus on GP at lower concentration of alkali activator with appropriate engineering properties for making it economically viable and easy to handle while working with it, thus, making it popular in general construction field practices.

Present research work focuses on sustainable utilization of solely GGBS for synthesis of GP paste with low alkali concentration cured at ambient temperature by utilising the higher activation potential of GGBS and making it further reactive through mechanochemical activation achieved through grinding of sodium metasilicate (SMS) with GGBS forming geopolymeric precursor. GGBS based GP paste is prepared using SMS powder and SH solution as alkali activators. GGBS and GGBS based GP paste are characterized by various techniques such as powder X-ray diffraction (XRD), field emission scanning electron microscope (FESEM) with energy dispersive x-ray analysis (EDAX) and fourier-transform infrared spectroscopy (FTIR) for microstructural analysis. The effect of alkali concentration (SH) and proportioning of alkali activators (SMS/SH) on different engineering properties of GGBS paste, like normal consistency, soundness, setting time and compressive strength at different ages is investigated. Experimental investigations are performed for achieving appropriate compressive strength at low concentration of alkali activators to achieve economy and feasibility for field applications.

## 2 Experimental Details

### 2.1 Materials

In this study GGBS is used as source material to synthesize geopolymers. It is procured from Jindal Steel and Power Limited, Raigarh, India and its chemical composition is enumarated in Table 1. Primarily, GGBS consists of oxides of calcium, silicon and aluminium. The maximum CaO content (36.94%) in GGBS is due to usage of limestone during extraction of iron from its ore to discard impurities. Its specific gravity is 2.73 and specific surface area is 369 $m^2$/kg. The residue obtained after wet sieve analysis of GGBS on 45 µm IS sieve is 1.93%.

GP prepared using alkali hydroxide or alkali silicate alone attains lower strength as compared to combination of alkali hydroxide and alkali silicate[23]. Thus, commonly used alkali activators for making geopolymer are SH and SS solution. Addition of SS solution along with alkali hydroxide enhances development of geopolymer precursors[24]. It also encourages gelation and precipitation of silicates during geopolymerization[25]. However, high viscosity of SS solution used for making geopolymer results in rapid setting time[26]. To overcome this issue of rapid setting combination of SMS ($Na_2SiO_3.9H_2O$) powder and SH (NaOH) solution are used as alkali activator for synthesis of GP in this study. The purity of SH pellets is 97%, purchased from Avantor Performance Materials India Ltd. Molecular weight of SMS is 284.20 and its grade is extra pure procured from Loba Chemie Pvt. Ltd.

### 2.2 Mixing and sample preparation

#### *2.2.1 Mechanochemical activation*

Mechanical activation of FA demonstrates high reactivity and improved geopolymerization rate[27]. Decrease in particle size due to milling process, enhances the surface reactivity of solids through physicochemical changes without altering the overall chemistry of material thereby improving its strength and setting properties[28,29]. Based on such previous researches, gepolymeric precursor is prepared in the

Table 1 — Chemical composition of GGBS.

| S. No. | Component | Percentage (%) |
|---|---|---|
| 1. | Calcium Oxide (CaO) | 36.94 |
| 2. | Silicon Dioxide ($SiO_2$) | 33.89 |
| 3. | Aluminium Oxide ($Al_2O_3$) | 20.43 |
| 4. | Magnesium Oxide (MgO) | 6.58 |
| 5. | Sulphur (S) | 0.55 |
| 6. | Sulphuric Anhydride ($SO_3$) | 0.28 |
| 7. | Iron (III) Oxide ($Fe_2O_3$) | 0.18 |
| 8. | Chloride (Cl) | 0.016 |
| 9. | Insoluble Residue (by mass) | 0.36 |
| 10. | Loss on Ignition (by mass) | 0.774 |

present study, by mixing GGBS with SMS powder in a laboratory ball mill of 10 kg capacity. For homogeneous mixing the ball mill is loaded with GGBS + SMS and grinding media consisting of steel balls whose diameter ranges from 20-70 mm. Total weight of grinding media added to ball mill is adjusted to 30 kg and the GGBS + SMS is ground in ball mill at the rate of 1000 rpm for a period of 30 minutes for their mechanochemical activation. The fineness of precursor is maintained such that not more than 2% of precursor is retained in 45 µm IS sieve. This divergent route, unlike previously indicated in Fig. 1 is adopted for making GGBS highly reactive for geopolymerization.

#### *2.2.2 Mix proportioning and preparation of GP paste*

The geopolymeric precursor (GGBS + SMS) obtained, is activated by SH solution to prepare GP paste. The systematic methodology adopted for preparing GP paste is depicted in Fig. 2. Alkali solution of 1 M, 1.5 M, 2 M and 4 M SH are prepared by dissolving desired quantity of SH pellets in distilled water and cooled down at room temperature 24 hours prior to casting of GP paste cubes. Normal consistency and setting time of geopolymeric precursor (GGBS + SMS) is calculated with SH solution. The quantity of SMS added to GGBS is ascertained by concentration of SH solution. The amount of SMS calculated so as to maintain proportioning of SMS/SH (denoted by R-Ratio) is in the range of 0.5 to 4.0 in order to obtain desired normal consistency. Various parameters considered to study the engineering properties include, concentration of SH solution measured in molarity (M) 1 M, 1.5 M, 2 M, 4 M and R ratios of 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0. In total 32 mixes of GP paste are prepared and their mix proportioning in described in Table 2. Samples are denoted as ${}_{M}GP^{R}$ where M refers to concentration of SH solution in molarity and R refers to R-Ratio.

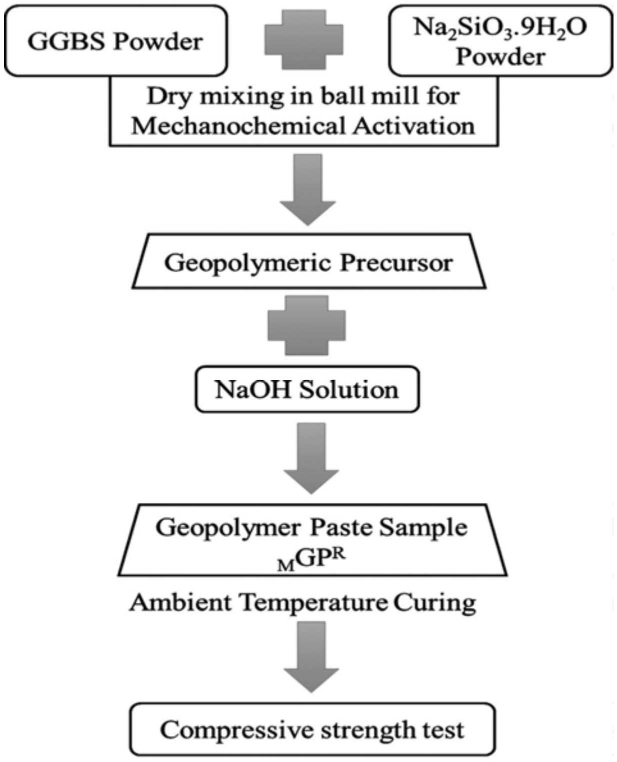


Fig. 2 — Schematic methodology adopted for preparing GGBS GP.

Table 2 — Mix proportioning of GP paste for preparing a batch of 12 cubes.

| ${}_{M}GP^{R}$ | Normal Consistency (%) | GGBS (gm) | SH Solution (ml) | SH(gm) | SS(gm) |
|---|---|---|---|---|---|
| ${}_{1.0}GP^{0.5}$ | 28.5 | 9600 | 2736 | 112.82 | 56.41 |
| ${}_{1.0}GP^{1.0}$ | 28.0 | 9600 | 2688 | 110.85 | 110.85 |
| ${}_{1.0}GP^{1.5}$ | 27.5 | 9600 | 2640 | 108.87 | 163.30 |
| ${}_{1.0}GP^{2.0}$ | 27.0 | 9600 | 2592 | 106.89 | 213.77 |
| ${}_{1.0}GP^{2.5}$ | 27.5 | 9600 | 2640 | 108.87 | 272.16 |
| ${}_{1.0}GP^{3.0}$ | 26.5 | 9600 | 2544 | 104.91 | 314.72 |
| ${}_{1.0}GP^{3.5}$ | 25.5 | 9600 | 2448 | 100.95 | 353.32 |
| ${}_{1.0}GP^{4.0}$ | 25.5 | 9600 | 2448 | 100.95 | 403.79 |
| ${}_{1.5}GP^{0.5}$ | 29.0 | 9600 | 2784 | 114.80 | 57.40 |
| ${}_{1.5}GP^{1.0}$ | 28.5 | 9600 | 2736 | 112.82 | 112.82 |
| ${}_{1.5}GP^{1.5}$ | 27.7 | 9600 | 2659 | 109.66 | 164.49 |
| ${}_{1.5}GP^{2.0}$ | 27.0 | 9600 | 2592 | 106.89 | 213.77 |
| ${}_{1.5}GP^{2.5}$ | 26.5 | 9600 | 2544 | 104.91 | 262.27 |
| ${}_{1.5}GP^{3.0}$ | 26.0 | 9600 | 2496 | 102.93 | 308.78 |
| ${}_{1.5}GP^{3.5}$ | 25.5 | 9600 | 2448 | 100.95 | 353.32 |
| ${}_{1.5}GP^{4.0}$ | 25.0 | 9600 | 2400 | 98.97 | 395.88 |
| ${}_{2.0}GP^{0.5}$ | 30.0 | 9600 | 2880 | 118.76 | 59.38 |
| ${}_{2.0}GP^{1.0}$ | 28.5 | 9600 | 2736 | 112.82 | 112.82 |
| ${}_{2.0}GP^{1.5}$ | 28.0 | 9600 | 2688 | 110.85 | 166.27 |
| ${}_{2.0}GP^{2.0}$ | 27.5 | 9600 | 2640 | 108.87 | 217.73 |
| ${}_{2.0}GP^{2.5}$ | 27.0 | 9600 | 2592 | 106.89 | 267.22 |
| ${}_{2.0}GP^{3.0}$ | 26.5 | 9600 | 2544 | 104.91 | 314.72 |
| ${}_{2.0}GP^{3.5}$ | 26.0 | 9600 | 2496 | 102.93 | 360.25 |
| ${}_{2.0}GP^{4.0}$ | 25.5 | 9600 | 2448 | 100.95 | 403.79 |
| ${}_{4.0}GP^{0.5}$ | 31.5 | 9600 | 3024 | 124.70 | 62.35 |
| ${}_{4.0}GP^{1.0}$ | 30.0 | 9600 | 2880 | 118.76 | 118.76 |
| ${}_{4.0}GP^{1.5}$ | 28.0 | 9600 | 2688 | 110.85 | 166.27 |
| ${}_{4.0}GP^{2.0}$ | 27.5 | 9600 | 2640 | 108.87 | 217.73 |
| ${}_{4.0}GP^{2.5}$ | 27.0 | 9600 | 2592 | 106.89 | 267.22 |
| ${}_{4.0}GP^{3.0}$ | 26.0 | 9600 | 2496 | 102.93 | 308.78 |
| ${}_{4.0}GP^{3.5}$ | 25.5 | 9600 | 2448 | 100.95 | 353.32 |
| ${}_{4.0}GP^{4.0}$ | 25.0 | 9600 | 2400 | 98.97 | 395.88 |

Twelve GP paste cubes for each mix proportions are mixed and casted in cube moulds. Cubes are demoulded after 24 hours of casting and cured at 27$^{o}$C +/- 2 and relative humidity of 65 +/- 5% until 1, 3, 7 and 28 days age till testing. After testing samples for 28 days compressive strength test, the broken cubes of samples achieving maximum compressive strength (*i.e.* $_{2.0}$GP$^{2.0}$) are crushed and ground to prepare fine powdered sample and dried in oven at 105$^{o}$C for 24 hours for microstructural testing on XRD, FTIR and FESEM with EDAX.

### 2.3 Characterization and experimental test

#### *2.3.1 X-Ray diffraction (XRD) analysis*

The mineralogical phases are monitored using Bruker D8 Advance X-ray diffractometer. The samples for analysis are prepared in powdered form. The x-rays are produced using a sealed tube using Cu Kα radiation ($\lambda$ = 0.154 nm). The x-rays are detected using Bruker LynxEye detector which is a fast counting detector based on Silicon strip technology. The samples are tested for scanning speed of 1 second with 0.05 increments at 2θ ranging between 10$^{o}$ and 60$^{o}$. The XRD pattern is analysed by Origin 2018 software.

#### *2.3.2 Fourier-transform infrared spectroscopy (FTIR)*

FTIR spectroscopy is adopted to identify functional groups in a material using KBr powder mix method. Samples are prepared in powder form and scanned across a frequency range of 4000 to 400 cm$^{-1}$ on Shimadzu's IR Prestige-21. The FTIR spectrum is analysed using Origin 2018 software.

#### *2.3.3 Field Emission scanning electron microscope (FESEM) and energy dispersive X-ray spectrometer (EDAX)*

Powdered samples are prepared and mounted on carbon tape for this test. The surface morphology is studied using Zeiss's ULTRA Plus, a high resolution field emission scanning electron microscope offering a theoretical resolution of 1.0 nm at 15 kV. Energy Dispersive X-ray spectrometer (EDAX) manufactured by Oxford Instruments Nano analysis is used to detect elements present.

#### *2.3.4 Normal consistency, setting time and soundness*

Normal Consistency can be defined as ability to flow for a freshly mixed GP paste. Normal Consistency and setting time are measured using Vicat's apparatus in accordance with IS 4031 (Part4): 1988 and IS 4031 (Part5): 1988, respectively. Soundness for excessive free lime and magnesia is tested using Le-Chatelier and autoclave apparatus respectively in accordance with IS 4031 (Part 3): 1988 for optimized proportion of GP Paste.

#### *2.3.5 Compressive strength test*

For preparation of GP paste specimens, geopolymeric precursor (GGBS+SMS) is taken and amount of SH solution to be added is worked out according to consistency of mix. After adding SH solution to precursor, a homogeneous mixture is prepared using gauging trowels for 3-4 minutes. The GP paste is poured in cube moulds of size 70.6 mm x 70.6 mm x 70.6 mm and compacted on vibrator for two minutes at the rate of 12000 +/- 400 vibrations per minute and its top surface is finished smoothly. 12 cubes are casted for each trial mix to determine compressive strength of GGBS based GP paste at the age of 1, 3, 7 and 28 days. The compressive strength test is performed on compression testing machine with capacity of measuring 1000 KN. For each observation, an average failure load of three cubes is recorded and divided by surface area of cubes for calculating compressive strength.

## 3 Results and Discussion

### 3.1 XRD analysis of GGBS and GP paste

The XRD spectrum of GGBS and GP paste are illustrated in Fig. 3. GGBS has vitreous structure with

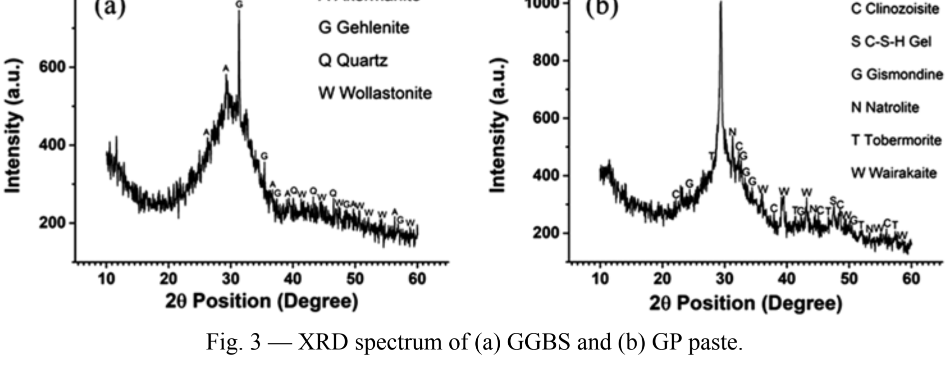


Fig. 3 — XRD spectrum of (a) GGBS and (b) GP paste.

amorphous nature as eminent from broad hump around 20° – 37° (2θ value) with peak postion at 31.31° while crystalline phases are indicated in Fig. 2(a) by sharp peaks primarily consisting of akermanite, gehlenite, quartz and wollastonite[30,31]. Amorphous nature is beneficial for geopolymerization reaction as, it aids in leaching of reactive Si and Al from their oxides and support in formation of $SiO_4$ and $AlO_4$ cross linkage[8]. The mineralogical phase is confirmed by the RRUFF project and corresponding 2 θ values along with their RRUFF ID. Peak positions of minerals along with their chemical formula are illustrated in Table 3. Akermanite, gehlenite, quartz and wollastonite has maximum intensity at 2θ value of 29.26°,31.31°,43.26° and 41.31°, respectively[32].

The XRD patterns of $_{2.0}GP^{2.0}$ paste is illustrated in Fig. 3(b). After the reaction of GP precursor (GGBS+SMS) and SH solution, a shift of broad hump is observed to around 25° – 39° (2θ value) in XRD pattern of GP paste with highest peak at 29.41° indicating the presence of alkaline alumino-silicate gel and C-S-H gel due to high calcium content in GGBS[33]. Similar observation of a broad and amorphous hump between 20° – 40° in XRD pattern due to geopolymerization which includes both geopolymeric gels and C-S-H gel suggesting that the geopolymeric and hydrate reaction occurred concurrently[34]. The new phases occurred in GP paste are clinozoisite, gismondine, natrolite to bermorite,wairakite and calcium silicate hydrate (C-S-H) in GP paste[32]. Peak positions corresponding to identified minerals and their chemical formula are tabulated in Table 4.

The highest peaks of clinozoisite, gismondine, C-S-H, natrolite, tobermorite and wairakite are observed at 2θ values of 32.24°, 29.41°, 32.63°, 31.26°, 28.14° and 35.94° respectively in Fig. 2(b). Transformation of GGBS to clinozoisite, gismondine, natrolite, tobermorite and wairakite phases indicates geopolymerization, consequently, formation of geopolymer[32]. Depending on proportions of Si, Al, Ca and Na contents in raw material, the geopolymer system comprises of C–S–H, (N, C)–A–S–H, C–(N)–A–S–H, N–(C)–A–S–H gels[35]. The reaction products are CASH or NASH gel in high calcium based geopolymers[36]. FA based GP paste containing GGBS forms C–S–H gel along with N–A–S–H gel or C–A–S–H gel on account high calcium oxide content in GGBS[15,37]. Thus, the presence of CASH gel may be confirmed by clinozoisite, gismondine, tobermorite and wairakite and NASH gel may be attributed to natrolite in GGBS based GP paste. Thus, co-existence of C-S-H gel and geopolymeric gel is evident from results obtained in XRD[9].

### 3.2 FTIR of GGBS and GP paste

FTIR characterization is adopted to investigate existence of various functional groups present in GGBS and GP paste. The FTIR spectrums of GGBS and GP paste are illustrated in Fig. 3. Significant peak positions are observed at 488 $cm^{-1}$, 692 $cm^{-1}$, 923 $cm^{-1}$, 1495 $cm^{-1}$ and 3517 $cm^{-1}$ in Fig. 4(a) for GGBS. The peak at 488 $cm^{-1}$ may be attributed to O-Si-O bending vibrations, which may confirm presence of orthosilicate unit[38]. Peaks at 692 $cm^{-1}$ correspond to Al-O symmetric stretching vibrations, might be ascribed to $AlO_4$ and 923 $cm^{-1}$ correspond to Si-O stretching vibrations of $SiO_4$[39]. C–O stretching vibration might be due to carbonate group, which is explicit from peak at 1495 $cm^{-1}$ and O-H group

Table 3 — Peak positions and chemical formula of mineral phase identified in GGBS by XRD.

| S. No. | Mineral phase | Chemical Formula | Peak Positions in degree (2θ value) for GP Paste | References |
|---|---|---|---|---|
| 1. | Akermanite | $Ca_2MgSi_2O_7$ | 26.29°, 29.26°, 36.72°, 39.36°, 49.74°, 56.91° | RRUFF ID: R061085 |
| 2. | Gehlenite | $Ca_2Al(SiAl)O_7$ | 31.31°, 35.41°, 37.41°, 48.62°,56.38° | RRUFF ID: R060841 |
| 3. | Quartz | $SiO_2$ | 40.04°, 43.26°, 46.53° | RRUFF ID: R040031 |
| 4. | Wollastonite | $CaSiO_3$ | 41.31°, 44.62°, 47.35°, 50.62°, 52.13°, 54.28°, 58.81° | RRUFF ID: R040131 |

Table 4 — Peak positions and chemical formula of mineral phase identified in GP Paste by XRD.

| S.No. | Mineralphase | Chemical Formula | Peak Positions in degree (2θ value) for GP Paste | References |
|---|---|---|---|---|
| 1. | Clinozoisite | $Ca_2Al_3[Si_2O_7][SiO_4]O(OH)$ | 22.14°, 32.24°, 37.94°, 45.06°, 48.52°, 55.99° | RRUFF ID: R040085 |
| 2. | Gismondine | $Ca_2(Si_4Al_4)O_{16}\cdot 8H_2O$ | 24.39°, 32.63°, 33.21°, 34.38°, 42.23°, 50.43° | RRUFF ID: R060809 |
| 3. | Natrolite | $Na_2(Si_3Al_2)O_{10}\cdot 2H_2O$ | 31.26°, 44.53°, 53.30° | RRUFF ID: R040102 |
| 4. | Tobermorite | $Ca_4Si_6O_{17}(H_2O)_2\cdot(Ca\cdot 3H_2O)$ | 28.14°, 41.36°, 46.67°, 51.94°, 57.40° | RRUFF ID: R060147 |
| 5. | Wairakite | $Ca(Si_4Al_2)O_{12}\cdot 2H_2O$ | 35.94°, 39.45°, 43.16°, 49.45°, 55.11°, 58.52° | RRUFF ID: R061075 |
| 6. | Calcium Silicate Hydrate | $Ca_2SiO_4\cdot 0.3H_2O$ | 29.41°, 47.55° | [32] |

stretching vibration peaks are spotted at 3517 $cm^{-1}$ probably indicating presence of silanol group[39, 40].

FTIR analysis of GP pastes is shown in Fig. 4(b). Significant peak is observed at 486 $cm^{-1}$, which may be attributed to O-Si-O bending vibrations confirming presence of polysialate-siloxo, 711 $cm^{-1}$ indicates presence of Si-O-Si symmetric stretching vibrations, which confirms the occurrence of polysialate-disiloxo. Peaks at 1641 $cm^{-1}$ might be attributed to bending vibration of H:OH and O-H group stretching vibration peaks are detected at 3467 $cm^{-1}$ indicating presence of absorbed water molecules[39]. The presence of geopolymeric gel may be confirmed by vibration bands of O-Si-O, Si-O-Si and absorbed water molecules confirming the formation of tobermorite.

Vibration peaks of 965 $cm^{-1}$ may be associated to Si-O stretching vibrations of $SiO_4$ tetrahedron confirming presence of C-S-H gel and vibration bands of 662 $cm^{-1}$ is probably due to symmetric stretching vibrations of Si-O-Al[39, 40]. Presence of Si-O-Al may be attributed to the formation of clinozoisite, gismondine, natrolite and wairakite. On solidification of alkali activated slag binders CSH and calcium and alumino-silicate hydrate are reaction products[25]. Thus, above observations imply that during geopolymerization of GGBS, both C-S-H and geopolymeric gel co-exist in GGBS based GP system. Peaks observed at 1444 $cm^{-1}$ possibly indicate presence of C–O stretching vibration due to carbonates formed in atmosphere[40].

### 3.3 FESEM and EDAX of GGBS and GP paste

The surface morphology of GGBS is examined using FESEM and its elemental classification using EDAX is shown in Fig. 5. It appears in FESEM images that GGBS particles are irregular shaped with particle size in the range of 1-19 μm. The energy-

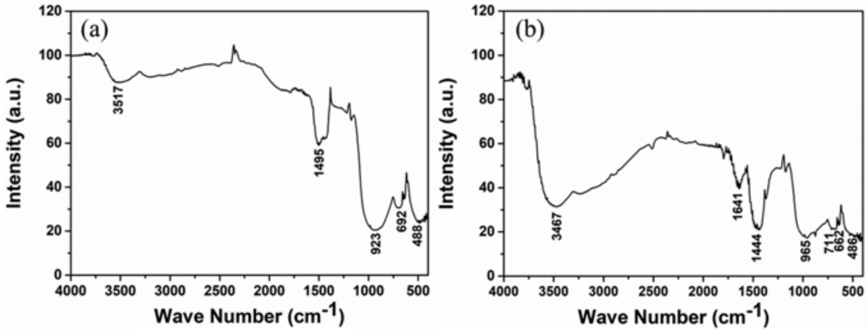
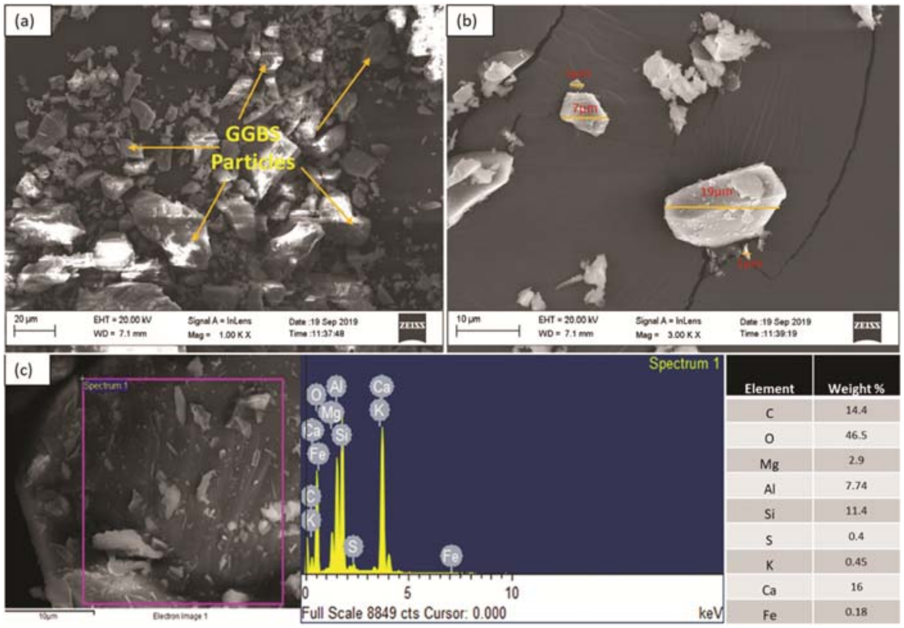


Fig. 4 — Different vibrational modes in FTIR region obtained for (a) GGBS and (b) GP paste.



| Element | Weight % |
|---|---|
| C | 14.4 |
| O | 46.5 |
| Mg | 2.9 |
| Al | 7.74 |
| Si | 11.4 |
| S | 0.4 |
| K | 0.45 |
| Ca | 16 |
| Fe | 0.18 |

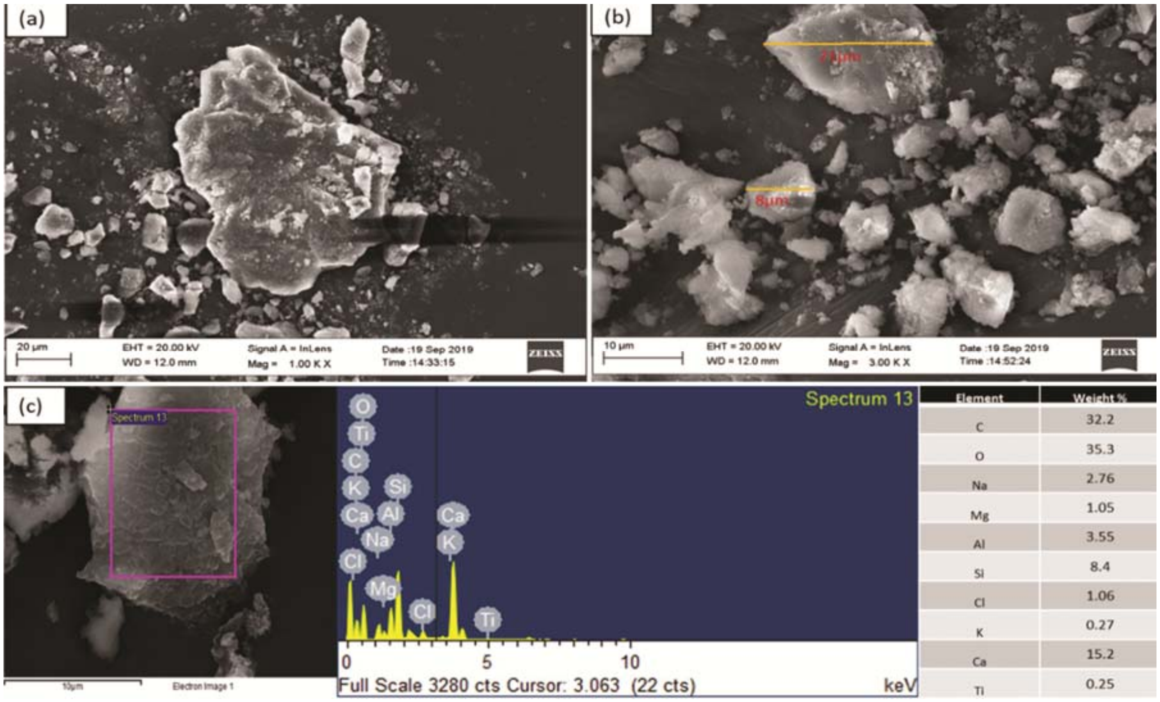
Fig. 5 — FESEM images of GGBS at magnification of (a) 1 KX, (b) 3 KX and (c) EDAX spectrum.

dispersive X-ray spectroscopy (EDAX) profile of GGBS is presented in Fig. 5(c), indicating presence of elements Calcium (Ca), Silicon (Si), Aluminium (Al), Oxygen (O), Carbon (C), Magnesium (Mg), Potassium (K), Sulphur (S) and Iron (Fe) in varying weight percentages. GGBS has reasonable amount of Al and Si, which are necessary component for geopolymerization. EDAX analysis further indicates that GGBS has high calcium content ensuring active participation information of hydration products. Basicity coefficient [$(CaO+MgO)/(SiO_2+Al_2O_3)$] is calculated as 0.80 for GGBS, which is neutral for an alkali-activated slag binder[41]. Hydration modulus [$(CaO+MgO+Al_2O_3)/(SiO_2)$] of GGBS is 1.88 (>1.4), which implies effective hydration property[42]. Chemical compositions of compounds present in GGBS are worked out from EDAX data and are compared with results of chemical analysis. The EDAX results are in good agreement with chemical composition obtained from chemical analysis.

GP paste images of FESEM at different magnifications and their respective EDAX spectrum are shown in Fig. 6. The EDAX profile of GP paste is tabulated in Fig. 6(c) indicating presence of elements Oxygen, Calcium (Ca), Carbon (C), Silicon (Si), Aluminium (Al), Sodium (Na), Magnesium (Mg), Potassium (K), Sulphur (S) and Iron (Fe) in varying weight percentage. The results obtained from EDAX show presence of sodium ions, may be attributed to formation of N-A-S-H gel during geopolymerization reaction[43]. The paste gains its strength due to formation of C-S-H/C-A-S-H gel or N-A-S-H gel, confirmed by EDAX.

### 3.3 Normal consistency, soundness and setting time

Measured Normal consistency, IST and FST are plotted in Fig. 7 indicating their variation with SH concentration for various R ratio. It is evident in Fig. 7(a) that normal consistency of GP paste varies in the range of 25% - 31.5%. This range of values is similar to range provided for OPC in relevant Indian Standard code of practices. The consistency lies in the range of 25% - 28.5% and 25% - 29% for SH solution with concentration of 1 M and 1.5 M, respectively. It varies in the range of 25% - 28% and 25% - 31.5% for molar concentration 2 M and 4 M respectively, for varying R ratios. Consistency of paste is dependent on concentration of SH solution and R ratio. As concentration of SH solution increases, its viscosity increases. Due to increase in viscosity of SH solution, leaching of Si and Al is likely to be disrupted and hence, the consistency increases with increase in SH concentration[44]. The decrease in consistency of GGBS paste with increasing R ratio is evident for all concentrations of SH solution. This may be explained by the fact that excess amount of SS hinders water evaporation and structure formation leading to decrease in consistency on increasing R ratios[45].



| Element | Weight % |
|---|---|
| C | 32.2 |
| O | 35.3 |
| Na | 2.76 |
| Mg | 1.05 |
| Al | 3.55 |
| Si | 8.4 |
| Cl | 1.06 |
| K | 0.27 |
| Ca | 15.2 |
| Ti | 0.25 |

Fig. 6 — FESEM images of GP paste at magnification of (a) 1 KX, (b) 3 KX and (c) EDAX spectrum.

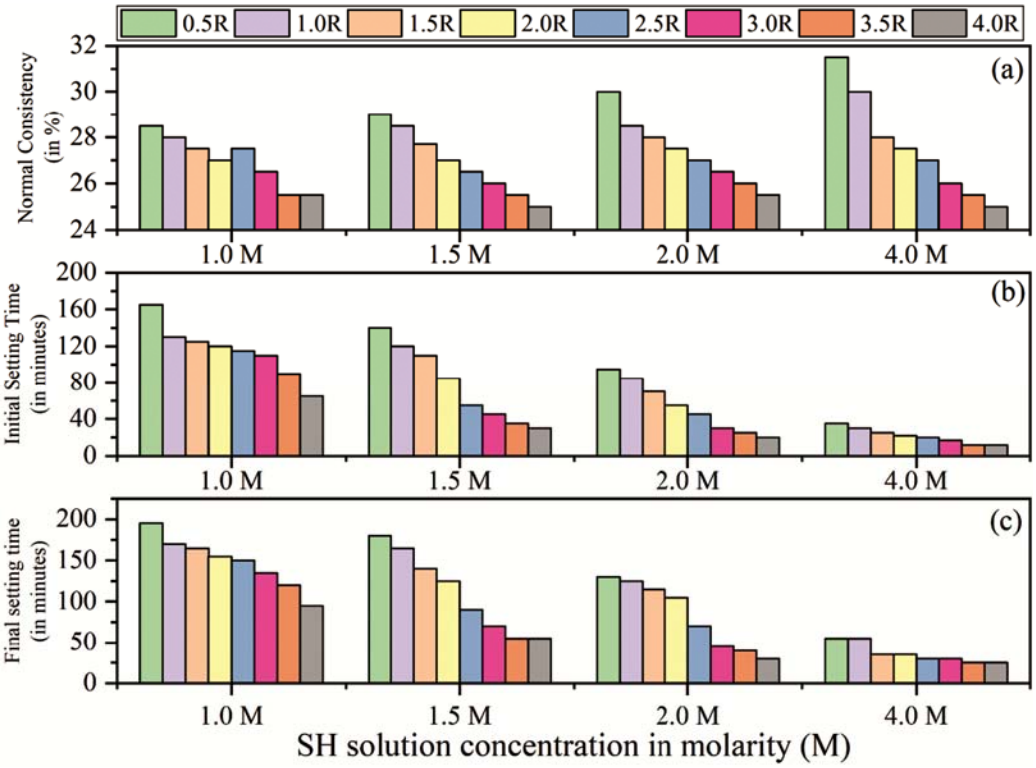


Fig. 7 — Measured variation in (a) Normal consistency, (b) Initial setting time and (c) Final setting time, with varying SH concentration and R ratio of pastes.

Volumetric changes occur in cement after setting due to the presence of excessive free lime and magnesia beyond permissible limit, resulting in disintegration of mortar/concrete and exposing the reinforcement to corrosion. In soundness test performed on GP paste, measured expansion of sample is 1.0 mm (lesser than permissible limit of 10 mm) in Le-Chatelier test and 0.028% (lesser than permissible limit of 0.80%) Autoclave test, which is well within allowable criteria. Hence, GGBS based GP binder satisfies the criteria for soundness and thus, it is appropriate for use as a substitute to conventional OPC.

The setting time of GP paste depends upon fineness of precursor, pH of activating solution, concentration of SH solution and R ratio[9,46,47]. Measured initial setting time of alkali-activated GGBS pastes with different concentrations of SH solution and R ratios are depicted in Fig. 7(b). It ranges from 10 - 165 minutes. The initial setting time lies in the range of 165 - 65 minutes, 140 - 30 minutes , 95 - 20 minutes and 35 - 10 minutes for 1 M, 1.5 M, 2 M and 4 M SH solution respectively with varying R ratios. Final setting time of alkali-activated GGBS paste ranges from 25 - 195 minutes in Fig. 7(c). The final setting time lies in the range of 195 - 95 minutes, 180 - 55 minutes, 130 - 30 minutes and 55 - 25 minutes for SH solution concentration of 1 M, 1.5 M, 2 M and 4 M respectively with different R ratios. The setting time decreases as concentration of SH solution increases from 1 M to 4 M in bar charts. This may be due to mechanochemical activation of GP precursor, which enhances reactivity of amorphous Si and Al, thereby increasing rate of geopolymerization due to higher specific area of source material. As concentration of SH increases, viscosity of SH solution also increases, which disrupts leaching of Si and Al, consequently, setting time shows a decreasing trend[44].

It is evident that, on increasing R ratio, setting time is observed to decrease. Excessive silicate in geopolymer system hinders evaporation of water and also interrupts formation of three dimensional matrix of alumino-silicate[9].

The research findings elucidate that setting and hardening process and its rate is prominently governed by $SiO_2$ and $Al_3O_3$ content in GP system. Varying trends are observed in research, for instance, with high $SiO_2$, more silicate species are available and the rate of condensation between silicate species is low, resulting in longer setting times. However, in a mix with high $Al_2O_3$ content, more $[Al(OH)_4]^-$ species are available for reaction, thus leading to higher rate of condensation between aluminate and silicate species resulting in shorter setting times[36].

Observed values of IST and FST are comparable to desirable setting times for OPC (minimum 30 minutes and maximum 600 minutes) in accordance with IS 12269. Observed setting time being comparable to desired range is noteworthy, as “quick setting” of GP paste prepared with sodium silicate solution is reported in previous research[26]. This correlation between measured and desired setting time is probably due to use of SMS powder, instead of SS solution, in this study.

### 3.4 Compressive strength

Compressive strength of GP paste for various R ratios at different ages at ascertained 1 M, 1.5 M and 2 M molar concentration of SH solution is shown in Fig. 8. The measured ranges of compressive strength for GP paste are elucidated in Table 5. Distinct increase in compressive strength with progressive ages of 1, 3, 7 and 28 days for various R- ratios is observed for all concentrations of SH solution. Similar increasing trend in compressive strength of GP paste with attempted trials of R ratios consistently for various concentrations of SH solution upto 2 M is evident and a maximum compressive strength of 87.76 MPa is achieved at 28 days at exceptionally low SH solution concentration of 2 M.

The paste specimens prepared with 4 M SH solution had shown brittle behavior while demoulding. During process of demoulding, cubes had broken and were not workable. Thus, compressive strength of cube could not be measured. Accordingly, compressive strength of cubes with 1 M, 1.5 M and 2 M concentration of SH are recorded. An increase in alkali concentration enhanced strength development of geopolymers upto certain limit, but beyond a limit excess hydroxide ion concentration offers hinderance to polycondensation and accelerates alumino-silicate gel precipitation at early stages, resulting in lower strength geopolymers[48, 49]. The

Table 5 — Compressive strength of GP paste at different ages.

| S.No. | SH Concentration | Compressive Strength (MPa) range for varying R Ratios | | | |
|---|---|---|---|---|---|
| | | 1 Day | 3 Days | 7 Days | 28 Days |
| 1. | 1.0 M | 9.10 - 19.39 | 21.43 - 39.80 | 26.53 - 43.88 | 30.61 - 59.18 |
| 2. | 1.5 M | 20.41 - 28.57 | 30.61 - 48.98 | 36.73 - 58.16 | 44.90 - 67.35 |
| 3. | 2.0 M | 21.43 - 42.86 | 37.76 - 59.18 | 43.88 - 66.33 | 59.18 - 87.76 |

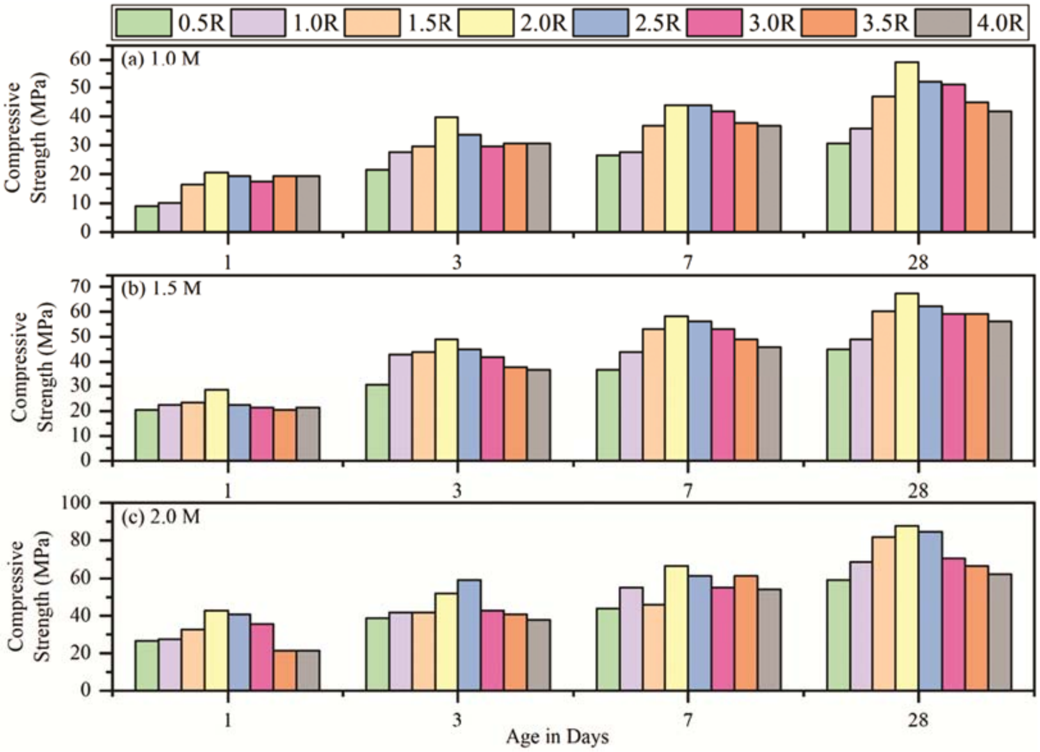


Fig. 8 — Variation of compressive strength of GGBS Paste at different ages for concentration of (a) 1 M, (b) 1.5 M and (c) 2 M.

probable reason for brittle nature of GP paste cubes prepared using 4 M SH solution is excessive SH that resulted in undesirable morphology and non-uniformity of hydration products in the pastes, thereby making it inelastic[50].

An early gain in compressive strength is also evident. Around 85 percent of 28 days compressive strength is achieved at 7 days[27]. It may be attributed to presence of higher content of calcium (due to 36.94% CaO) and amorphous nature of Si and Al in GGBS as observed from XRD analysis. Addition of calcium in GP system enhances overall strength properties of GP, as well as, its microstructure making it durable[51]. Dense geopolymer matrix (NASH/CASH) is formed along with co-existence of C-S-H gel due to high calcium content in GGBS based GP system which accelerates setting and hardening of pastes, resulting in early gain of strength[36]. C-S-H gel formation provided extra nucleation sites for precipitation of dissolved species which increased its solidification rate and caused rapid hardening[48]. The free calcium present in GGBS formed additional C–S–H and C–A–S–H, which co-existed with geopolymer products, lead to denser geopolymeric chain formation contributing to high strength of GP paste[52]. The C-S-H gel reduced the voids and pores within geopolymeric binder matrix which is perhaps the main reason of higher compressive strength at lower concentration of alkali.

Mechanochemical activation of GGBS with SMS in present experimentation may also be responsible for high compressive strength due to reduction in particle size, increase in surface area of precursor and change in morphology of GGBS particles permitting higher dissolution rate of Al and Si, when activated with alkaline solution, thereby increasing the rate of geopolymerization[27]. High degree of geopolymerization brings to dense matrix of geopolymer precluding the inter-connectivity of micropores, in turn, resulting in high compressive strength.

Measured compressive strengths are reorganized in Fig. 9 to depict variation in compressive strength of GP Paste with different trials of R-ratios and concentrations of SH solution at different progressive ages of 1, 3, 7 and 28 days separately. The compressive strength at various ages and SH concentrations increases, as R ratio increases upto 2.0, beyond that it decreases. Maximum compressive strength is visibly achieved for specimens with 2 M SH solution concentration and 2.0 R ratio for 1, 7 and 28 days age, except 3 days maximum strength achieved at 2 M SH solution concentration, but R ratio of 2.50, rather than 2. Thus, it is inferred that

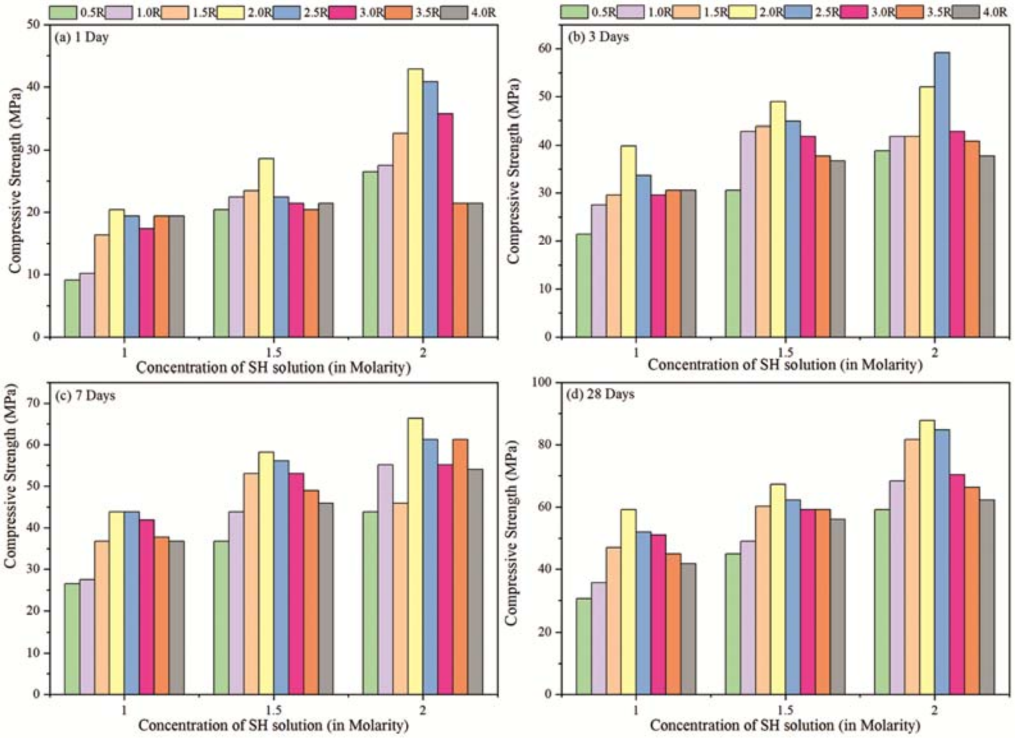


Fig. 9 — Variation of compressive strength of GP paste at different concentration for (a) 1 day, (b) 3 days, (c) 7 days and (d) 28 days

maximum compressive strength is achieved for SH solution of 2 M concentration and R ratio as 2.0, with few exceptions.

The early strength of paste is due to CSH/CASH gel and later age is due to NASH gel formation. This may be attributed to higher $Na^+$ ions which allows faster dissolution of alumino-silicate species resulting in formation of NASH gel responsible for high strength at later ages[13]. The compressive strength increases as R ratio increases upto 2.0, beyond that compressive strength decreases. It is due to the fact that for higher R ratio excessive SS is added to the system which hinders evaporation of water, disrupting formation of three dimensional networks of alumino-silicate, thereby retarding geopolymerization process[53].

Mechanochemical activation of SMS and GGBS increased the surface area of precursor thereby enhancing the dissolution rate of Si, Al and Ca on addition of SH solution to precursor and hence rate of geopolymerization increased. Increase in rate of geopolymerization produced denser matrix of GP gel. Also, co-existence of GP gel and C-S-H gel is evident from XRD and FTIR analysis which is probably responsible for higher compressive strength of 87.76 MPa at lower concentration of alkalis. Thus, it is evident that mild activation of GGBS by alkalis resulted in improved physical properties of geopolymer[54]. Desired compressive strength of FA GP was achieved by mechanical activation of FA under ambient temperature curing due to increase in its reactivity[27, 52].

For popularization of alkali activated GP mixes in construction practices, it is desired to optimize the cost of production. The quantity of alkali activators in binder (dependent upon normal consistency), concentration of SH solution and proportion of SMS to SH, apart from cost of other ingredients and construction cost, prominently governs economy of alkali-activated geopolymer. Optimum compressive strength has been achieved at low molar concentration of SH solution, which is contributing for economic viability and ease in on-site handling of geopolymer.

## 4 Conclusions

The present research has investigated engineering and microstructural properties of geopolymer paste prepared by alkali activation of GGBS solely with ambient temperature curing. A significant improvement in physical properties of GP is due to mechanochemical activation of GGBS and SMS. This has enhanced the reactivity of geopolymeric precursor and lead to generation of concurrent geopolymerization and hydration products by mild dosage of SH solution. The following inferences are drawn on the basis of present research:-

(i) Maximum compressive strength of 87.76 MPa is achieved at mild SH concentration of around 2 M and SMS/SH ratio of 2.0 with ambient temperature curing. This may be attributed to different route of blending GGBS and SMS by mechanochemical activation in addition to chemical activation by SH solution.

(ii) GGBS based GP paste exhibits mineralogical phases indicating formation of C-S-H gel and geopolymeric gel. These observations are further confirmed by post geopolymerization and hydration products identified in XRD and FTIR spectrum.

(iii) Co-existence of C-S-H gel and geopolymeric gel is responsible for achieving high strength at low concentration of alkali activators. These results are harmonious for making GP a robust and durable material for construction purpose.

(iv) Measured normal consistency, soundness and setting time of GP paste are in compliance with recommended values in accordance with Indian codal provisions, hence making it suitable for practical applications. It is further noteworthy on account of "quick setting" phenomenon of GP paste prepared with SS solution reported previously.

(v) Final Setting Time of 2-3 hours and realisation of 80-90 percent of ultimate compressive strength at 7-days observed during experimentation is significant for making GGBS based paste suitable for speedy construction and/or quick maintenance works. It may be attributed to the high calcium content in GGBS contributing to the formation of hydration products.

(vi) The experiments have confirmed suitability of alkali activated GGBS for substitution of conventional paste/concrete in construction practices. Optimal compressive strength achieved at mild concentration of SH solution and SMS indicates towards financial viability of GGBS based alkali activated paste/concrete, under ambient curing regime, for its upscaling and popularizationin construction.

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