



Ground granulated blast furnace slag based Geopolymer Mortar activated with low concentration alkali

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Infrastructural development requires large quantity of Ordinary Portland cement. Its manufacturing process emits enormous amount of carbon dioxide, thereby deteriorating the environment. Thus, an alternative binder known as geopolymer, developed using alumino-silicate rich materials like fly ash, silica fume, metakaoline etc., activated by suitable alkalis may serve as a substitute to conventional cement. The present research work focuses on preparation of ground granulated blast furnace slag based geopolymer mortar with ambient temperature curing at 27°C at low concentration of alkali activators. Compressive strength of geopolymer mortar at different concentration of activator and microstructure is studied. The concentration of alkali activators is optimized by trials in laboratory and maximum compressive strength of 64.29 MPa is achieved.

Keywords: Alkali activator, Fly ash, Ground granulated blast furnace slag, Geopolymer

1 Introduction

Ordinary Portland Cement (OPC) is most commonly used binding agent in concrete for infrastructural development. Large amount of carbon dioxide (CO₂) is released in environment during the process of calcination of limestone in rotary kiln at 1450°C temperature for production of OPC. This contributes approximately 5-7% of global anthropogenic CO₂ emissions¹. Excessive consumption of natural resources and energy in manufacturing of OPC leads to further enhancement in CO₂ emissions. Hence, suitable alternative binder with low carbon footprints is required for minimizing production of OPC².

Geopolymer (GP) binder is an eco-friendly solution to CO₂ emissions caused by cement production³. It is synthesized by reaction of alumino-silicate rich species dissolved under extremely high alkaline conditions releasing free [SiO₄]⁻ and [AlO₄]⁻ tetrahedral units during the process of geopolymerization. Tetrahedral units of Si-O-Al-O bonds are formed, alternatively, through linking of polymeric precursor by sharing oxygen atom⁴. A descriptive model of geopolymerization is illustrated in Fig. 1⁵.

Geopolymeric binder possesses excellent mechanical strength, low creep and shrinkage, higher resistance to elevated temperature and chemicals such as sulphates, acids etc.⁶. Researchers are, thus,

encouraged to focus on alternative geopolymeric binders to reduce use of cement⁷.

Geopolymerization of fly ash (FA) is possible when thermal curing is adopted, as it consists of Si and Al in crystalline state. Laboratory experiments have been performed on different pozzolanic materials with varying concentration of alkali activator, which is a combination of sodium hydroxide (SH) and sodium silicate (SS). Mostly, SH concentration is measured in molarity and the amount of SS added for activation is ascertained by weight which is denoted in terms of ratio of SS to SH, commonly denoted by SS/SH ratio. Summary of significant research made for preparation of GP mortar is enumerated in Table 1.

It is evident that FA based GP mortar achieved reasonable compressive strength when it was activated by 6 to 16M SH solution cured at 60 to 120°C for 20 to 48 hours. In-situ field applications of FA based GP become impracticable in view of prerequisite thermal curing at high temperature, thus, limiting its use to pre-cast concrete only. Such limitations have been overcome by partial replacement of FA by additives like OPC, ground granulated blast furnace slag (GGBS), etc. in FA based GP binder and compressive strength has been achieved at ambient curing temperature⁸.

Blast furnace slag (BFS) is an industrial by-product which is finely ground to produce GGBS⁹. It is suitable for production of GP as it is rich in oxides of Si, Al and Ca in amorphous phase. GGBS based

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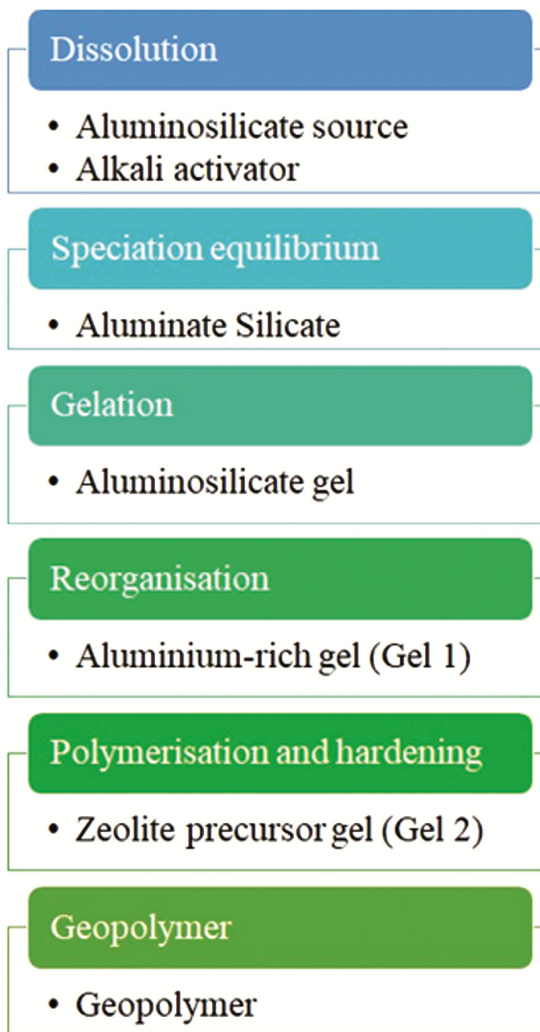


Fig. 1 — A descriptive model of geopolymerization⁵

GP achieved considerable compressive strength at ambient temperature curing, but, with higher concentration of 9M SH solution¹⁰. Use of higher concentration of alkali activator releases large amount of heat, posing handling trouble and high risk during its mixing with geopolymeric precursor, making GP synthesis uneconomical. Thus, further research is desired to focus on making safe (while field handling) and economically viable GP with appropriate engineering properties at lower concentration of alkali activator for making GP popular in field practices.

This research work focuses on utilization of GGBS exclusively for making GP mortar with low alkali concentration cured at 27°C ambient temperature. GGBS based GP paste and mortar are prepared using sodium metasilicate (SMS) and SH as alkali activators. Characterisation of GGBS and GGBS based GP paste is performed through microstructural

analysis and reported in authors' previous research¹¹. Engineering properties of GP mortar are determined. Laboratory trials are performed for achieving optimum compressive strength at low concentration of alkali activators for economy and feasibility in field applications.

2 Materials and Methods

2.1 Ground granulated blast furnace slag (GGBS)

Chemical composition of GGBS, procured from Jindal Steel and Power Limited, Raigarh, India is illustrated in Table 2. It is used as binding material for making GP. Its specific gravity and moisture content are 2.73 and 0.32% respectively. Specific surface area is 369 m²/kg with residue obtained on 45 μm IS sieve through wet sieve analysis as 1.93% measured in accordance with IS 4031.

2.2 Alkali activators and GP precursor

SMS powder and SH solution were used as alkali activators for synthesis of GGBS based GP. Alkali solution of SH was prepared with commercially available SH pellets of 97% purity. SH pellets were dissolved in water to obtain alkali solution of desired molar concentration. In this study, 1, 1.5, 2 and 4 molar (M) concentrations of SH solution were used for preparation of GP. The alkaline solution was prepared 24 hours prior to casting of GP.

GP precursor was prepared by mixing SMS powder with GGBS and it is ground in ball mill at the rate of 1000 rpm for 30 minutes for initiating mechanical activation. The fineness of precursor was maintained such that not more than 2% of precursor is retained on 45μm IS sieve. Quantity of SMS added to GGBS was ascertained in such a way that SMS/SH ratio (denoted by R ratio) varies from 0.5 to 4.0 with an interval of 0.5. The precursor obtained was activated using SH solution. The method of preparing precursor through mechanical activation was discussed in authors' previous research¹¹.

2.3 Experimental Methods

GP paste was prepared by thoroughly mixing GP precursor (GGBS+SMS) and alkali activator solution. Characterization of GGBS and GP paste was performed through microstructural investigations. The mineralogical phases were monitored by powder X-ray diffraction (XRD) measurements. Fourier-transform infrared spectroscopy (FTIR) was adopted to identify functional groups for analysis of GGBS and GP

Table 1 — Summary of significant research findings on GP mortars

| Pozzolanic Materials used for GP mortar | SH Concentration (M) | Ratio of SS/SH | Compressive Strength (MPa) | Age (Days) | Curing Regime | | Reference |
|--|----------------------|----------------|----------------------------|------------|-----------------------|--------------|-----------|
| | | | | | Temperature (°C) | Time (Hours) | |
| FA with sand:FA ratio of 2:1 | 10 | - | 35 | - | 65 | 48 | 33 |
| Coarse FA and Blast furnace slag | 14 | 2 | 27.30 | 7 | 60 | 48 | 34 |
| FA | 12 | 1 | 44 | 28 | 60 | 24 | 35 |
| FA | 14 | 2.5 | 60 | 7 | 70 | 24 | 36 |
| FA | 6 | - | 22 | 7 | 85 | 24 | 37 |
| Palm Oil Fuel ash/Fly-ash and GGBS (3:7) | 12 | - | 66 | 28 | 65 | 24 | 38 |
| FA | 10 | 2 | 33 | 28 | 120 | 20 | 39 |
| Low calcium FA with 5% OPC | 14 | - | 50 | 28 | Ambient Curing Regime | | 40 |
| FA and GGBS in 1:1 | 15 | - | 62 | 28 | Curing Regime | | 41 |
| FA (50%) and GGBS (50%) | 16 | 1 | 78.20 | 56 | | | 14 |
| FA and GGBS | 16 | 2.5 | 79 | 28 | | | 42 |
| GGBS | 9 | 1.5 | 65 | 28 | | | 10 |

Note: SH = Sodium hydroxide, SS = Sodium sulphate, FA = Fly ash, GGBS =Ground granulated blast furnace slag, OPC = Ordinary Portland cement, MPa = Megapascal

Table 2 — Chemical composition of GGBS

| Component | Percentage (%) |
|--|----------------|
| Silicon Dioxide (SiO ₂) | 33.89 |
| Aluminium Oxide (Al ₂ O ₃) | 20.43 |
| Calcium Oxide (CaO) | 36.94 |
| Magnesium Oxide (MgO) | 6.58 |
| Sulphur (S) | 0.55 |
| Sulphuric Anhydride (SO ₃) | 0.28 |
| Iron (III) Oxide (Fe ₂ O ₃) | 0.18 |
| Chloride (Cl) | 0.016 |
| Insoluble Residue (by mass) | 0.36 |
| Loss on Ignition (by mass) | 0.77 |

paste. Samples were scanned across a frequency range of 4000 to 400 cm⁻¹. The surface morphology was studied by Field Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-ray spectrometer (EDAX) was used to detect elements present in GP. Results of these microstructural investigations and engineering properties like normal consistency, IST, FST and soundness were reported in authors' previous research¹¹.

Different sets of GGBS based GP mortar were prepared with varying concentration of SH solution from 1M to 4M and R ratio in the range of 0.5 to 4.0 for making laboratory trials. GP mortar was prepared with precursor (GGBS + SMS) to standard sand ratio of 1:3, for determining 1, 3, 7 and 28 days compressive strength. This mixture of sand and

precursor was mixed thoroughly with SH solution of desired concentration to prepare 12 mortar cubes of size 70.6 mm x 70.6 mm x 70.6 mm for each trial mix. The cubes were prepared and tested as per recommendations in IS 4031:1988 and cured in ambient environment at 27°C±2 and relative humidity of 65±5% until the age of testing.

3 Results and Discussion

This study explored the feasibility of making solely GGBS based GP mortar cured under ambient environment. In order to achieve economy, GGBS was activated by different dosage of alkali activator in addition to mechanical activation of precursor. The results of compressive strength are elaborated in Table 3. It is evident that for 1M SH solution 1, 3, 7 and 28 day compressive strength ranges between 9.18 - 14.29 MPa, 17.35 - 28.57 MPa, 24.49 - 32.59 MPa and 26.53 - 39.76 MPa respectively, for 1.5 M SH solution 1, 3, 7 and 28 days compressive strength varies from 14.49 - 20.41 MPa, 21.43 - 33.67 MPa, 28.53 - 45.65 MPa and 32.65 - 55.02 MPa respectively, for 2M SH solution 1, 3, 7 and 28 days compressive strength lies in the range of 15.31 - 31.63 MPa, 27.55 - 42.86 MPa, 33.67 - 51.02 MPa and 35.71 - 64.29 MPa respectively and for 4M SH solution 1, 3, 7 and 28 days compressive strength lies in the range of 14.59 - 18.37 MPa, 24.67 - 30.43 MPa,

Table 3 — Compressive strength range of GP mortar for different molar concentrations and ages when cured at ambient temperature of 27°C

| SH concentration | Compressive strength (MPa) range for varying R ratio (0.5-4.0) | | | |
|------------------|--|---------------|---------------|---------------|
| | 1 Day | 3 Day | 7 Day | 28 Day |
| 1.0 M | 9.18 – 14.29 | 17.35 – 28.57 | 24.49 – 32.59 | 26.53 – 39.76 |
| 1.5 M | 14.29 – 20.41 | 21.43 – 33.67 | 28.53 – 45.65 | 32.65 – 55.02 |
| 2.0 M | 15.31 – 31.63 | 27.55 – 42.86 | 33.67 – 51.02 | 35.71 – 64.29 |
| 4.0 M | 14.59 – 18.37 | 24.67 – 30.43 | 26.55 – 35.71 | 40.82 – 53.10 |

Note: SH = Sodium hydroxide; MPa = Megapascal; R ratio = Sodium silicate to Sodium hydroxide ratio

26.55 - 35.71 MPa and 40.82 - 53.10 MPa respectively. These observations are discussed in detail in subsequent sections.

3.1 Microstructural analysis

Significant findings of micro-structural analysis are discussed here. X-Ray Diffraction analysis of GGBS and GP paste indicated that GGBS has vitreous structure with amorphous nature. 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 SiO₄ and AlO₄ cross linkage⁶. The mineralogical phases observed in GGBS are Akermanite, Gehlenite, Quartz and Wollastonite¹¹.

The XRD patterns of GP paste indicated enhanced crystallinity of GGBS after its reaction with alkali activators. XRD analysis identified presence of mineralogical phases of Clinozoisite, Gismondine, Natrolite, Tobermorite, Wairakite and C-S-H in GP paste^{11, 12}. Presence of Clinozoisite, Gismondine, Natrolite, Tobermorite and Wairakite indicates geopolymerization, consequently, formation of geopolymer¹². Thus, coexistence of C-S-H gel and geopolymeric gel is evident from XRD results⁷. 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 gels¹³. The reaction products are CASH or NASH gel in high calcium based geopolymers. 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 of high calcium oxide content in GGBS^{11, 14, 15}.

Existence of various functional groups present in GGBS and GP paste were investigated through FTIR

characterization. For GGBS, peaks corresponding to O-Si-O bending vibrations, Al-O symmetric stretching vibrations, Si-O stretching vibrations, C-O stretching vibration, O-H group stretching vibration were observed, which may confirm presence of ortho-silicate unit¹⁶, AlO₄, SiO₄¹⁷, carbonate group and silanol group^{11, 17, 18} respectively.

In FTIR spectrums for GP paste, significant peaks corresponding to O-Si-O bending vibrations, Si-O-Si symmetric stretching vibrations, bending vibration of H:OH and O-H group stretching vibration were observed, confirming presence of -Polysialate-Siloxo, Polysialate-disiloxo and absorbed water molecules¹⁷ respectively. The presence of geopolymeric gel may be confirmed by vibration bands of O-Si-O, Si-O-Si and absorbed water molecules¹¹.

Peaks associated to Si-O stretching vibrations of SiO₄ tetrahedron confirming presence of C-S-H gel and vibration bands probably due to symmetric stretching vibrations of Si-O-Si (Al) bridges were also observed^{17, 18}. On solidification of alkali activated slag binders, C-S-H and calcium alumino-silicate hydrate were reaction products¹⁹. 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^{11, 18}.

GGBS particles were found irregular in shape with particle size in the range of 1-19 μm in FESEM images. Al and Si are necessary components required for geopolymerization which, were witnessed in EDAX profile.

Also, high Ca content ensures active participation of GGBS in hydration process. Basicity coefficient $\left\{ \frac{\text{CaO} + \text{MgO}}{\text{SiO}_2 + \text{Al}_2\text{O}_3} \right\}$ was calculated as 0.80 for GGBS, which is neutral for an alkali-activated slag binder²⁰. Hydration modulus $\left\{ \frac{\text{CaO} + \text{MgO} + \text{Al}_2\text{O}_3}{\text{SiO}_2} \right\}$ of GGBS was 1.88 (>1.4), implying effective hydration property^{11, 21}.

The FESEM images and EDAX spectrum of GP paste were analysed. Presence of sodium ions is indicated, which may be attributed to formation of N-A-S-H gel during geopolymerization reaction²². The paste gains its strength due to formation of C-S-H/C-A-S-H gel or N-A-S-H gel. Particle size of GP paste increased due to geopolymerization of GGBS when it was activated by SH solution in addition to SMS. It may be attributed to enhancement of geopolymeric chain in GP matrix¹¹.

3.2 Compressive strength

Measured compressive strength of GP mortar for various R ratios and ascertained concentrations of SH solution at different ages is shown in Fig. 2. An increase in compressive strength with progressive age of 1, 3, 7 and 28 days for various R ratios was observed for all concentrations of SH solution. Maximum compressive strength of 64.29 MPa was achieved at 28 days at exceptionally low SH solution concentration of 2M and R ratio of 2.0. It may be due to the fact that presence of free calcium in GGBS formed additional C-S-H and C-A-S-H/N-A-S-H, which coexisted with geopolymer production. It resulted in denser geopolymeric chain formation contributing to high strength of GP mortar²³. The C-S-H gel reduced voids and pores within geopolymeric binder matrix. This is perhaps the main reason of higher compressive strength at lower molar concentration of alkali.

Mechanically activated GGBS with SMS in present research work 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^{23,24}. Higher degree of geopolymerization brings about denser matrix of geopolymer, in turn, resulting in higher compressive strength.

It is observed in Figs 2 and 3 that as the concentration of SH solution increases from 1M to 2M, compressive strength of mortar increases, but further increase in concentration of SH solution upto 4M, decreases compressive strength of specimens. It is probably due to excessive SH that resulted in undesirable morphology and non-uniformity of hydration products, thereby making it inelastic²⁵. 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^{26,27}. Similar compressive strength of 65MPa was reported for GGBS based GP activated by 9M SH solution¹⁰.

An early gain in compressive strength of mortar was also evident. Around 80 - 95% of 28 days compressive strength was achieved at 7 days^{10,24}. It may be attributed to the presence of high calcium

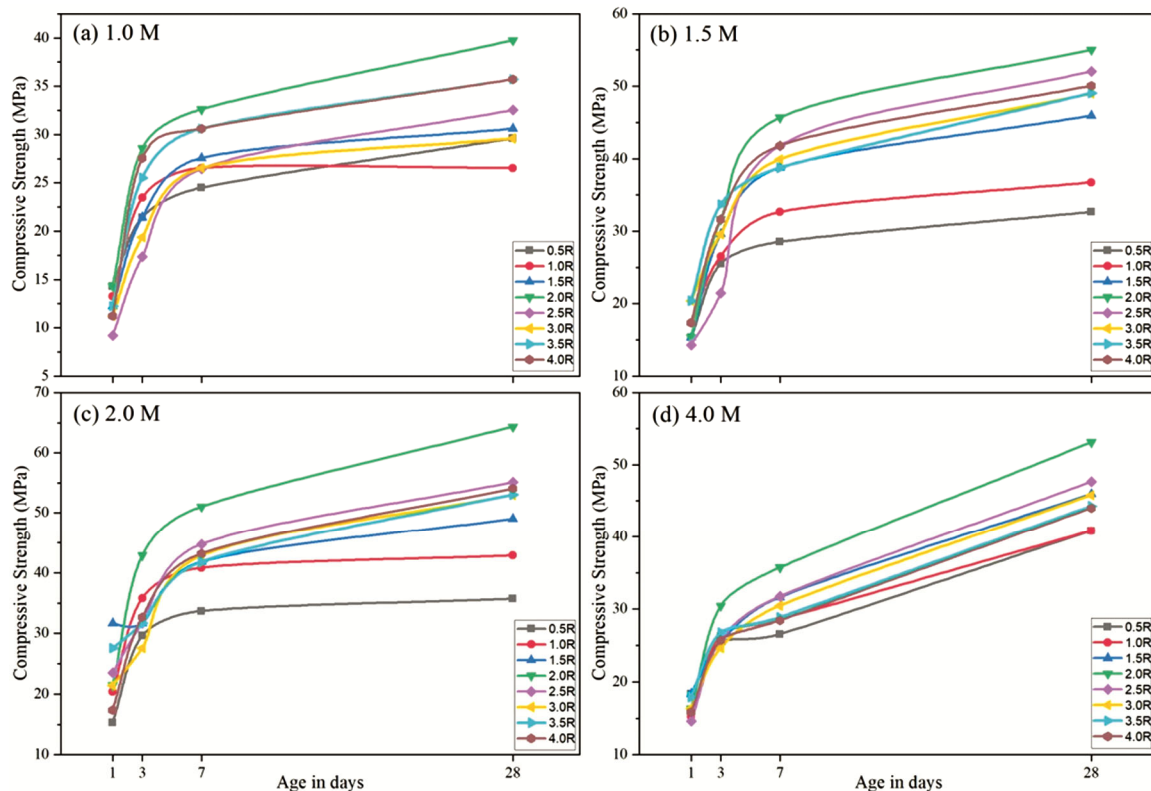


Fig. 2 — Variation of compressive strength of GP mortar at different ages for concentration of (a) 1 M, (b) 1.5 M, (c) 2 M, and (d) 4 M.

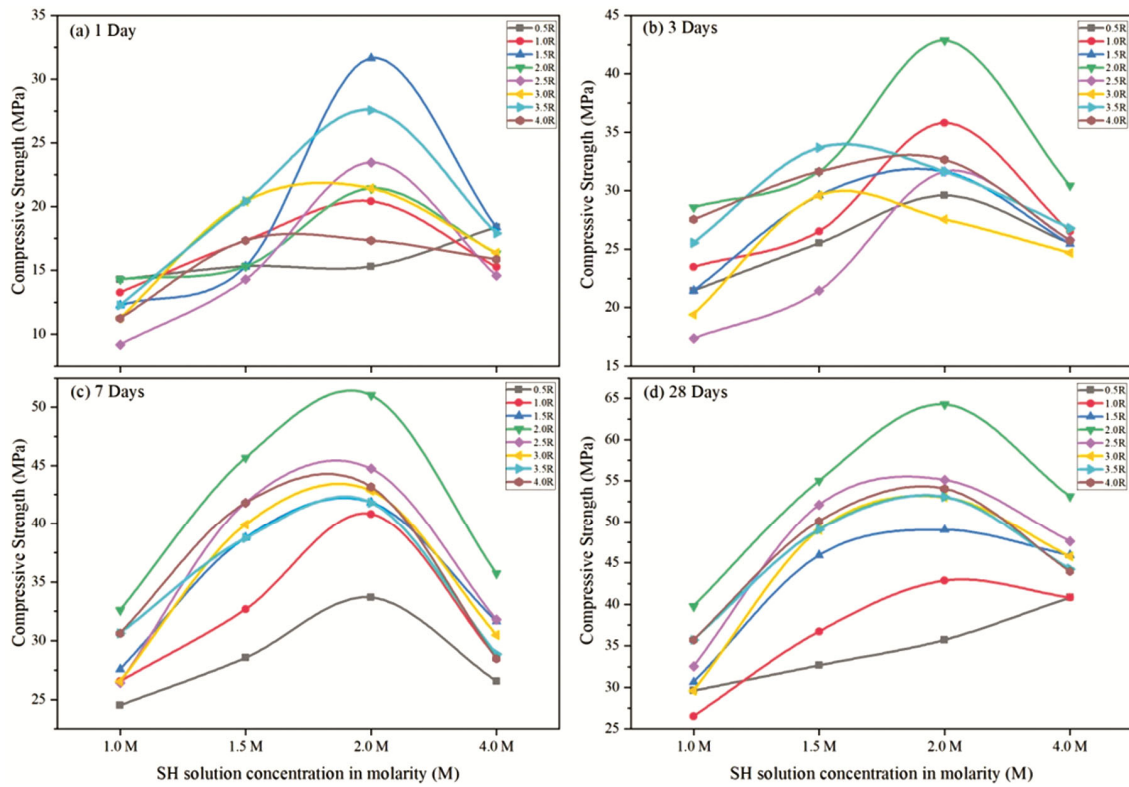


Fig. 3 — Variation of compressive strength of GP mortar at different molar concentration for (a) 1 Day, (b) 3 Day, (c) 7 Day, and (d) 28 Day.

content and amorphous nature of Si and Al in GGBS as observed from XRD analysis. Presence of calcium in GP system enhances overall strength properties of GP²⁸. Dense geopolymer matrix (NASH/CASH) was formed along with co-existence of C-S-H gels due to high calcium content in GGBS based GP system. It accelerates setting and hardening of mortar, resulting in early gain of strength²⁹. C-S-H gel formation provided extra nucleation sites for precipitation of dissolved species which increased its solidification rate and caused rapid hardening²⁶.

Compressive strength of GP mortar with different trials of R ratios and concentrations of SH at various progressive ages of 1 Day, 3 Days, 7 Days and 28 Days separately are depicted in Fig. 3. For all ages, compressive strength increases with increase in SH and SMS concentration upto 2M and 2R respectively, beyond that it decreases. Maximum compressive strength was visibly achieved for specimens with 2M SH solution concentration and 2 R ratio for 1,7 and 28 days age however, 3 days maximum strength is achieved at 2M SH solution concentration and R ratio of 2.50, rather than 2. Thus, it is inferred that maximum compressive strength is achieved for SH solution of 2M molar concentration and R ratio as 2.0,

with few exceptions. Similar findings were observed wherein maximum compressive strength was attained at 1.5 R ratio for varying concentration of SH solution¹⁰.

Hence, the early age strength of mortar is due to CSH/CASH gel and later age strength is due to NASH gel formation. This may be attributed to higher Na⁺ ions which allows faster dissolution of aluminosilicate species resulting in formation of NASH gel responsible for high strength at later ages³⁰. The compressive strength increases as R ratio increases upto 2.0, beyond that compressive strength decreases. It was due to the fact that for higher R ratio excessive sodium silicate is added to the system which hinders evaporation of water, disrupting formation of three dimensional networks of aluminosilicate, thereby retarding geopolymerization process³¹.

Addition of SS, along with alkali hydroxide enhances formation of geopolymer precursor³². The soluble silicate mixed with GGBS encourages gelation and precipitation of silicates¹⁹. In present experimentation, SMS was ground with GGBS for mechanical activation, which was chemically activated by SH solution. Use of SMS and SH solution with mechanical activation, in addition to chemical

activation, probably led to higher compressive strength of 64.29 MPa at lower SH solution.

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, concentration of SH solution and proportion of SMS/SH, prominently governs economy of GP. Optimum compressive strength was achieved at low concentration of SH solution, which is contributing for economy.

4 Conclusion

The present research has investigated compressive strength of geopolymer mortar prepared by activating GGBS 'exclusively' using alkalis cured at ambient temperature of 27°C. GP mortar achieved higher compressive strength of 64.29 MPa at lower SH concentration of 2 M and 2.0 R ratio. This may be attributed to mechanical activation of GGBS and SMS powder in addition to chemical activation. It may be due to the high calcium content in GGBS contributing to formation of hydration products. Simultaneous formation of C-S-H gel and geopolymeric gel is observed in mineralogical phase analysis. Functional groups in post geopolymerization and hydration products identified using FTIR spectroscopy further confirms it. Substantial component of compressive strength is achieved at early ages. High strength at low concentration of alkali activators is achieved on account of co-existence of hydration and geopolymerization products.

This experimentation has confirmed the feasibility of making GGBS based GP mortar for substituting conventional cement mortar in construction practices. High compressive strength achieved at lower concentration of SH solution indicates towards financial viability of GGBS based alkali activated mortar, under ambient curing regime, for its upscaling and popularization in construction. Thus, geopolymer mortar prepared by GGBS and alkali activators under ambient temperature curing can be used for quick repair and maintenance of concrete structures as it gains strength at faster pace along with quick setting.

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