RESEARCH

Influence of slag content and glass fibers on microstructure, durability and mechanical properties of geopolymer composites

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Abstract

Fibre reinforced geopolymer concrete holds the hybrid advantages of technical benefits and eco-friendliness. In this paper, an attempt was made to study the effect of glass fiber in an optimized geopolymer mix where the optimization was based on the dosage of slag replacement with fly ash as main precursor and different variations of the ratio of hydroxide to silicate since glass fibres proved to enhance the properties of cement-based composites, as per the literatures. The fresh and hardened properties of geopolymer mixtures, such as fluidity, setting time, water absorption, sorptivity, and mechanical properties (under ambient and heat curing temperatures) were considered for optimization process. The results revealed that there is a strong correlation noticed between physical and mechanical properties of the geopolymer samples and the chemical compositions of the binders and activator ratios. It was identified from the initial optimization process that 50% replacement of slag with fly ash along with 1.5 ratio of hydroxide to silicate oven cured composite (M11 mix) produced the maximum compressive strength of 33.59 MPa. M11 mix also produced enhanced tensile and flexural parameters with good resistance to free water and capillary water rise. The internal microstructure also showed denser nature and less micro cracks and flaws, thus considered as the optimized mix. Various dosages of glass fibres were then added to the optimized mix, say 0.5, 1, 1.5 and 2% which further revealed that 1% addition of glass fibre was efficient in all the mechanical, durability and microstructural aspects.

Introduction

Industrial activities generate a large number of industrial by-products, which have a negative impact on the environment. During the energy intensive traditional cement manufacturing processes, a considerable amount of carbon dioxide (CO_2) is released. Ordinary Portland cement (OPC) production contributes to environmental issues by releasing CO_2 into the atmosphere and also use of lime stone in the production of cement will also lead to considerable loss to environment. Geopolymers, as one of the possible future alternatives, have piqued the interest of many academics around the world due to their exceptional strength and endurance (Davidovits, 1989). An active silica–alumina source and alkaline solutions are used to start the synthesis of polymeric linkages (Si-O-Al-O), and then geopolymer resin is mixed with a filler to make a geopolymer binder (Deventer et al., 2007). Industrial waste materials such as fly ash, blast furnace slag, copper and zinc slag can be used as an aluminosilicate source in geopolymer synthesis because SiO_2 and Al_2O_3 are the major oxides in the process. Because fly ash is one of the most pozzolanic by-product material with low carbon footprint that can be used in the construction section due to its widespread availability and contribution to the production of binders with good properties, it has been studied by many geopolymer researchers in terms of mechanical characteristics, durability, and microstructural composition (Bellum, 2022; Bellum et al., 2022).

Researchers are interested in using fibres to build improved geopolymer materials because of the superior physical and mechanical qualities that can be obtained. Fibre

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reinforced geopolymer composites have been shown in a number of studies to increase the physical and mechanical properties of the composites significantly. However, understanding the behaviour of fibre, matrix, fiber-matrix interaction, adhesion as a function of temperature, and its contribution to the final strength of the composite is the subject of the fire proofing behaviour of geopolymer matrix and fibre sustainability. Fly ash (FA), ground granulated blast furnace slag (GGBFS), silica fume, and metakaolin, which are rich industrial by-products which have merely zero carbon footprint, can be used as raw materials for making geopolymers. Fly ash is an industrial by-product that is widely available in large quantities and has a strong pozzolanic character, making it suitable for use in building. Fly ash-based composites have shown to be a potential material for manufacturing binders with superior characteristics to OPC binders. As a result, the mechanical, durability, and microstructural features of FA-based geopolymers were examined by the majority of researchers (Naghizadeh & Ekolu, 2019). The effect of GGBFS on the strength parameters of FA-based geopolymers under ambient curing was discussed in several papers (Bellum et al., 2020). Carbon fibres have an effect on the tensile characteristics of geopolymer composites, according to Ranjbar et al. 2015 (Bellum et al., 2020). The addition of graphene-based nano-carbon fibres to geopolymer composites enhanced their flexural strength, according to the findings. Carbon fibres were also employed by Saafi et al. 2014 to create high-performance geopolymer binders. Polyvinyl alcohol fibres were used in geopolymer composites to improve the ductile nature and to enhance the strain hardening nature of the geopolymer composites (Alomayri et al., 2013; Rath et al., 2020). The inclusion of modest amounts of carbon nanofibers, on the other hand, enhanced the electrical and thermal conductivity of geopolymers. However, due to poor workability and strength qualities, the use of large-scale fibres in concrete is restricted. The influence of steel fibre on the mechanical characteristics and impact resistance of lightweight geopolymer concrete was investigated by Islam et al. 2017 (Islam et al., 2017).

Most of the studies recommended that the addition of calcium rich materials in the production of aluminosilicate binder-based geopolymers has better effect on strength characteristics. The majority of studies concluded that adding calcium-rich ingredients to the synthesis of aluminosilicate binder-based geopolymers improves strength properties (Bellum et al., 2020; Kaja et al., 2018). In sulphate settings (Madhuri & Srinivasa Rao, 2018) and acidic solutions (Bakharev, 2005), studies on the durability of alkali-activated materials (AAMs) revealed that calcium-free or low-calcium AAMs outlast OPC. The absence of sulpho-aluminate compounds during the polymerization process, such as ettringite and gypsum, as well as the creation of N-A-S-H gel, which has superior acid resistance, contribute to GP's improved durability.

Calcium-rich AAMs, on the other hand, form calcium-containing hydrates such C-S-H gel and C-(A)-S-H gel, which have lesser sulphate resistance and acid resistance than N-A-S-H gel (Elyamany et al., 2018). Aluminosilicate raw materials with low calcium fly ash (class F) have been used in the great majority of contemporary geopolymer experiments. While there is currently a dearth of knowledge about metakaolin-based geopolymers with calcium sources (OPC) in terms of mechanical performance and long-term durability concerns.

Tanyildizi and Yonar (Tanyildizi & Yonar, 2016) investigated the influence of high temperature on the mechanical properties of polyvinyl alcohol fibres fibre reinforced geopolymer concrete in the same field. According to the findings, raising the fibre ratio can improve the concrete's compressive and flexural strengths. Furthermore, as compared to the control sample, the samples exposed to high temperatures had smaller strength reductions. Researchers have focused in using fibres in the creation of innovative geopolymer materials because of the higher physical and mechanical qualities that can be attained. Fibre reinforced geopolymer composites have been proven in a number of studies to significantly improve the physical and mechanical properties of the composites (Alomayri et al., 2013; Rath et al., 2020).

This paper studies the efficiency of using glass fibers in fly ash and GGBFS blended optimized geopolymer mortar. The dosage of the glass fiber was considered as 0.5, 1, 1.5, 2% to the total weight of the binder in terms of rheological, mechanical and microstructural parameters. The process of optimization starts initially in terms of the effects of the content of slag in fly ash geopolymer and the effect of the ratio of hydroxide to silicate solutions in alkaline activators. With respect to aforesaid procedure, five different dosages of slag say 10, 20, 30, 40 and 50% replaced with fly ash content incorporated with two different ratios of hydroxide to silicate say 1:1 and 1:1.5 were incorporated in the mixture design. From the optimization process, in terms of fresh, mechanical, durability and microstructural properties, the best mix proportion was identified which was further tested with the various dosages of glass fiber. Even though literatures show good number of researches in this aspect, it demands the need for the development of two level optimization process between three materials in a geopolymer composite. The significance of this research exhibits the optimum content of three different materials in a composite which would help analyse the future researchers to adopt those optimized contents based on their required applications.

Experimental program

Materials

According to ASTM C 618–19 (2017), ASTM C 989–2018 (2018), locally available industrial by-products such as FA

(class-F) and GGBFS were used as binders in this investigation. GGBFS and fly ash were obtained from Ultratech Cement Limited, Rajahmundry. The constituents of the binder materials FA and GGBFS were obtained upon XRF analysis and tabulated in Table 1. It was identified that, FA and GGBFS have specific gravities of 2.30 and 2.81 respectively, with fineness modulus values based upon BET surface area was identified as 481 m^2/kg and 682 m^2/kg for FA and GGBFS respectively. Figures 1 and 2 illustrate the SEM with EDS analysis and XRD analysis of FA and GGBFS, respectively used in this study. The properties of glass fibres used in this paper are listed in Table 2.

In this paper, short length glass fibres were used to improve the characteristics of geopolymer composites as they are effective reinforcements in geopolymer materials as per the literatures. The influence of fibre length on

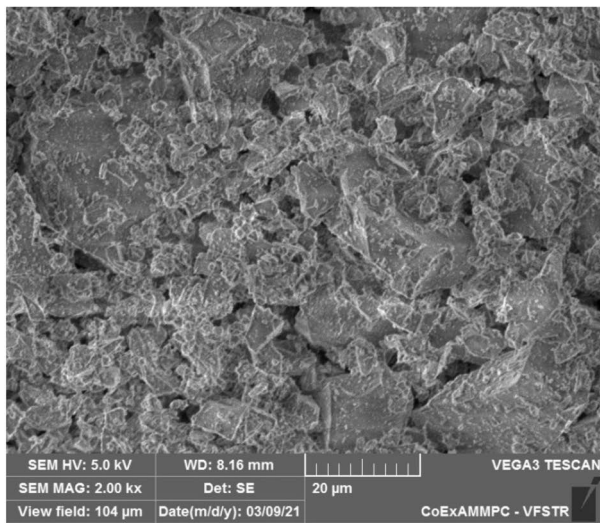**Table 1** Elemental composition of raw materials used in this study

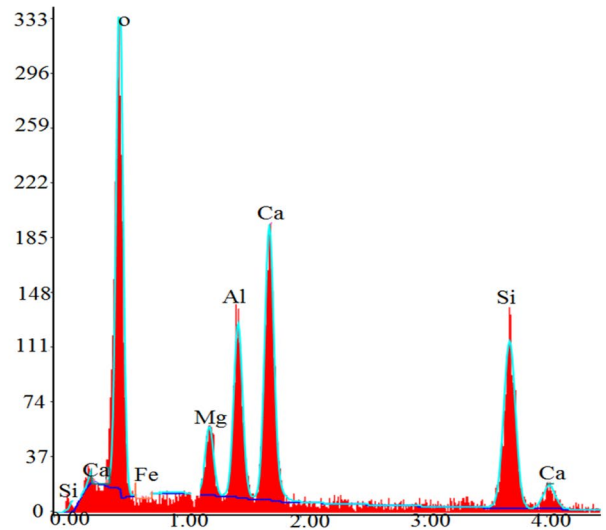| Material	Al_2O_3	Fe_2O_3	SiO_2	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	SO_3	TiO_2	LOI
Fly ash	24.11	5.06	59.13	2.55	1.18	0.38	0.91	2.76	0.31	1.25	0.72	1.64
GGBS	12.27	1.3	31.16	46.51	3.62	0.91	–	2.05	–	0.76	–	1.42
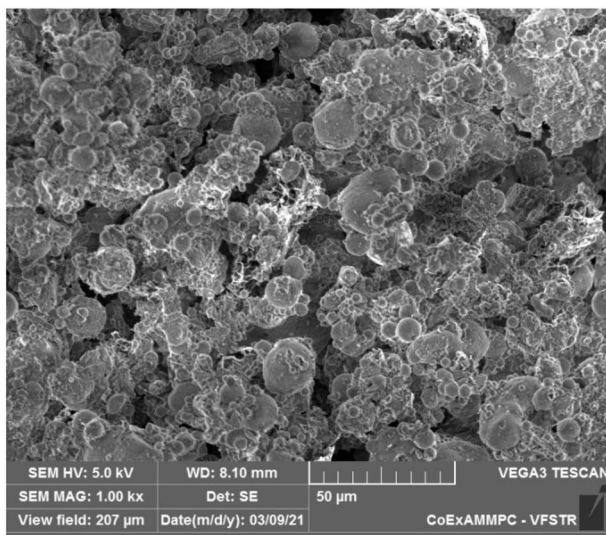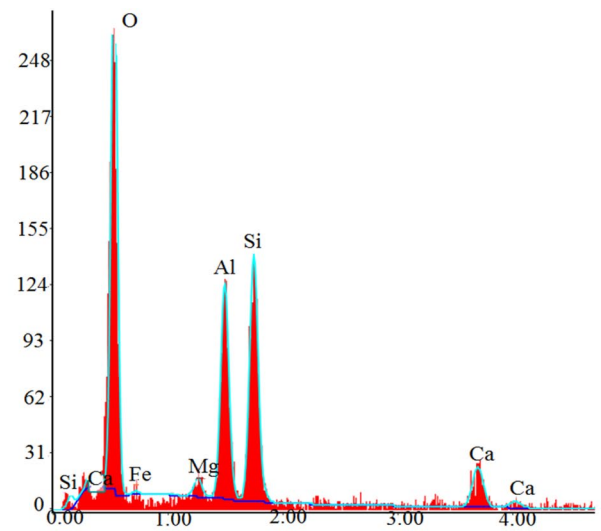
Fig. 1 SEM with EDS analysis, **a** Fly ash-SEM, **b** Fly ash-EDS, **c** GGBS-SEM, **d** GGBS-EDS
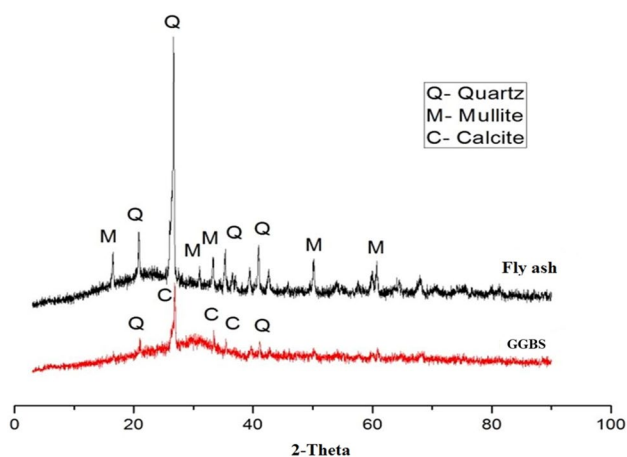
Fig. 2 XRD patterns of raw materials used in this study

Table 2 Properties of steel and glass fiber

	Length (mm)	Diameter (μm)	Density (g/cc)	Tensile strength MPa	Aspect ratio
Glass Fibre	6	9	2.54	1704	666.67

the mechanical properties of low-content short fibre reinforced composites is usually linked to the lengthening of the fibres. Alkaline solution is made with sodium-based compounds such as sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH). The Na_2SiO_3 solution had 27.8% SiO_2, 8% Na_2O, and 64.5 percent water, with NaOH pellets were 98% pure. The molarity of NaOH was fixed as 8 M for all the mixes. Locally available river sand was used as fine aggregate in the preparation of geopolymer mixes.

Mixture design and mix proportions

Initially for the optimization process, ten mixes were considered which incorporates the five variations of the dosage of slag over FA along with two variations of hydroxide to silicate ratio. The optimization process was followed by the studying the effect of the dosage of glass fibers, in four different dosages in the optimized mix. Both the optimization processes of materials and fibres, the optimization was carried out based on fresh, mechanical and microstructural parameters. The binder to fine aggregate ratio was kept constant as 0.3 for all the mixes. Even a small number of short-length fibres significantly boosted the strength of the geopolymer composite. For high fibre dose, however, dispersion and surface adhesion are poor, resulting in reduced stiffness and strength efficiency. The molarity of the sodium hydroxide solution was fixed as 8 M for all the mixes. Thus, from the aforesaid mixture design the following mix proportions, as summarized in Table 3, were considered for the further optimization process. The performance of the various geopolymer mixes were directly compared with the cement mortar of ratio 1:0.3 (v/v) as reference mortar.

Casting, curing and testing methods

Initially the activators were prepared by separate preparation of sodium hydroxide and sodium silicate solutions. Sodium hydroxide pellets in required weight were added in the water i.e., 320 g of sodium hydroxide pellets were added in water until the water reaches 1L mark. The hydroxide and silicate solutions were mixed based upon the proposed mix proportions, at least 24 h before preparing the concrete. The concrete was prepared with the help of a pan mixer as a proper mixing sequence. The proper mixing sequence was obtained

Table 3 Mix proportions of geopolymer composites (kg/m³)

Mix id	Proportion					Fine aggregate (sand)
	Binders			Alkaline solution		
	Cement	Fly ash	GGBFS	Na_2SiO_3	NaOH	
	(kg/m³)					
M1	360	–	–	–	–	1200
M2	0	342	38	57	57	1080
M3	0	304	76	57	57	1080
M4	0	266	114	57	57	1080
M5	0	228	152	57	57	1080
M6	0	190	190	57	57	1080
M7	0	342	38	45.6	68.4	1080
M8	0	304	76	45.6	68.4	1080
M9	0	266	114	45.6	68.4	1080
M10	0	228	152	45.6	68.4	1080
M11	0	190	190	45.6	68.4	1080

based on few trial mixes which gave maximum workability. The prepared geopolymer concrete specimens were subjected to temperature curing in which the specimens were kept under 100 °C in the oven for 24 h followed by ambient curing conditions. The average temperature for ambient curing was identified to be laboratory temperature range of about 25 ± 5 °C. Another set of specimens were subjected to only ambient curing i.e., heat temperature curing was not performed. The reference cement mortar specimen was subjected to different durations of saturated water curing.

The fluidity of the fresh composite was measured according to IS 1199–1991 (1991) i.e., the fresh mortar is placed in the slump cone (base diameter—60 mm, top diameter—36 mm, and the height—60 mm) and lifted such that the spread diameter was measured along perpendicular directions, say d_1 and d_2 and the fluidity was obtained as the average of d_1 and d_2. Similarly, the setting time of the fresh composites was determined as per IS: 4031 (Part 5)-1988 (1988).

To test the mechanical properties of the specimens, compressive strength (BS EN 12,390-3-09 (2009)), split tensile strength (ASTM C496/C496 M-17 (2017)), flexural strength (ASTM C78/C78M-18 (2023)) and elastic modulus tests as per the prescribed standards were adopted. Cubes of size 70.7 mm, prism of size $160 \times 40 \times 40$ mm and cylinders of size 75 mm diameter and 150 mm height were used for determining the compressive strength, flexural strength and split tensile strength respectively. The effect of the heat curing temperature was also analyzed in during the compressive strength test for which the specimens subjected with and without heat curing, for 7 and 28 days were considered. Curing duration of only 28 days were adopted to the specimens for testing split tensile and flexural strength, however the effect of curing duration does not affect the parameters largely. Similarly, 28 days Compression testing machine was used to measure the elastic modulus of 75×150 mm cylindrical samples that met ASTM C469/C469M-14 standards (2017).

Two significant durability parameters viz., sorptivity and water absorption tests were adopted in the geopolymer mixes. Disc specimens of size 150 mm diameter and 50 mm height were used in the sorptivity test in accordance with ASTM C1585-13 (1585) i.e., the sides of the specimens (heat + 28 days ambient cured) were sealed and the specimens were immersed in water for the depth of 5 mm to determine the capillary suction of water through difference in weight. Sorptivity was determined using the Eq. 1,

$$\text{Sorptivity} = \frac{i}{\sqrt{t}} \tag{1}$$

where, t = time in minutes and i = cumulative water absorption.

Cube specimens of size 70.7 mm were used to determine the water absorption, as per ASTM C642-13 (2001) i.e., heat cured followed by 28 days ambient cured specimens were saturated in the water for 28 days and the corresponding weights were noted before and after saturation. Increase in the percentage weight of specimens determines the rate of water absorption.

Microstructural categorization of specimens was carried out using an XRD test. The randomly selected samples were examined using the RIGAKU-6000 XRD with Cu-Ka radiation generated at room temperature under 15 mA and 40 kV. The XRD experiment was performed with a scanning angle of 2-Theta from 3 to 70 degrees. Analyses were performed using the "X' Pert High Score" software to detect auto-match peaks in the patterns.

An EDAX-EDS-SDD energy dispersive spectroscopy (EDS) analyzer with a high voltage of 5–35 kV was employed with SBH TESCAN Brno S.R.O-VEGA-3 equipment for morphological assessment of geopolymer samples using a low vacuum method. After the testing was completed, the SEM samples were collected from the broken pieces of geopolymer composites. The small pieces and powdered samples were used for Microstructural studies such as SEM, EDS and XRD.

Results and discussion

The experimental results were analyzed and discussed into two phases viz., optimization of material contents followed by the optimization of fibre dosage. Thus, the experimental results of two phases are discussed separately.

Optimization of materials

Fresh properties

Figures 3 and 4 shows the results of fluidity and setting time tests. A variable Initial Setting Time was observed for geopolymer concrete which varied non-Linearly based on the proportion of Fly ash and GGBFS. The Initial Setting Time of geopolymers was observed to be more than that of Cement when the proportion of Fly ash in the mix was higher. However, the Initial Setting Time of geopolymer concretes was found to be decreasing with increase in GGBFS content. Initially for mixes containing 90% Fly ash, the initial setting time was found to be 60 ± 5 min. For mixes having higher Fly ash content (90–70%), the Initial Setting Time decreased at a rate of 15 min for every 10% increase in GGBFS content. However, for mixes of relatively lower Fly ash content (70–50%), the rate of decrease
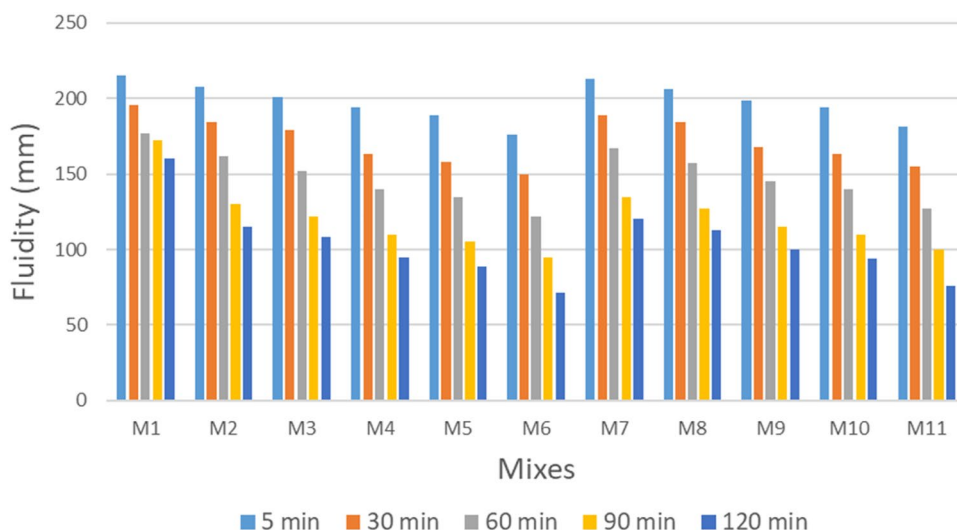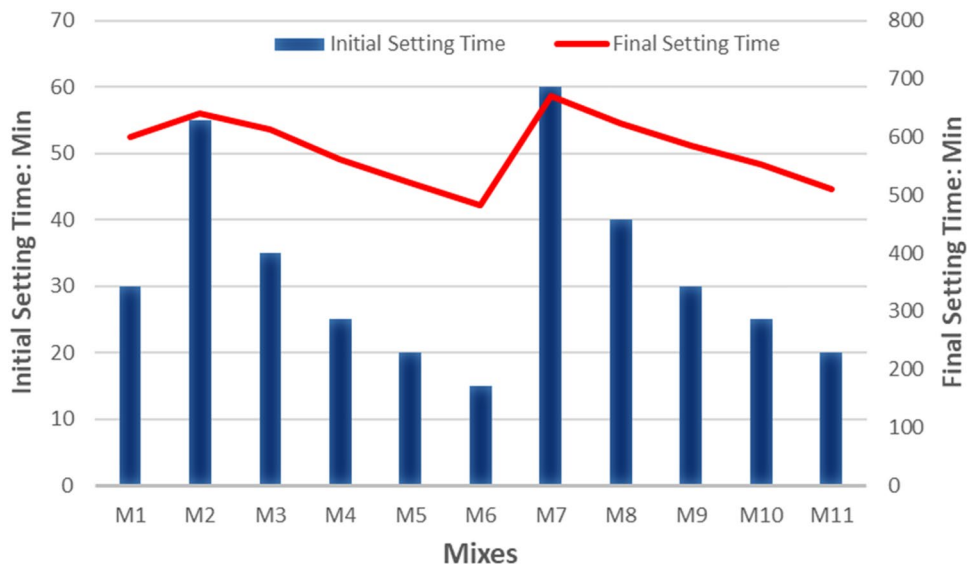
Fig. 3 Fluidity of geopolymer mixtures

Fig. 4 Initial and final setting times of mixes

was observed to be around 5 min for every 10% increase in GGBFS content.

Mechanical properties

Compressive strength Figure 5 shows the compressive strength of the geopolymer composites with different dosages of slag replacement and different ratio of hydroxide to silicate, under ambient and heat curing regimes, with the curing durations of 7 and 28 days. With the assistance from few literatures, it can be clearly inferred that the oven curing followed by ambient curing was very effective compared to simple ambient curing, irrespective of the curing durations. Most of the researchers have insisted that the geopolymerization reaction would be more effective only beyond 60 °C

exposure which was also revealed from the experiments. However, in some practical applications, high temperature curing cannot be performed for which the ambient curing results are to be adopted. It can be clearly inferred that; higher the dosages of slag content were efficient in improving the compressive strength of the specimens and it can also be observed that slight variation was observed with respect to the variation in the ratio of hydroxide to silicate, unlike 100% industrial by product precursors (Jounaghani et al., 2022). Though the magnitude of variation of test results was less among two ratios, the ratio of 1.5 was obtained to be relatively efficient. Thus, on comparison with the reference cement mortar, the mix M11 subjected to high temperature curing followed by 28 days ambient curing, provided significant result in both the aspects of optimization.
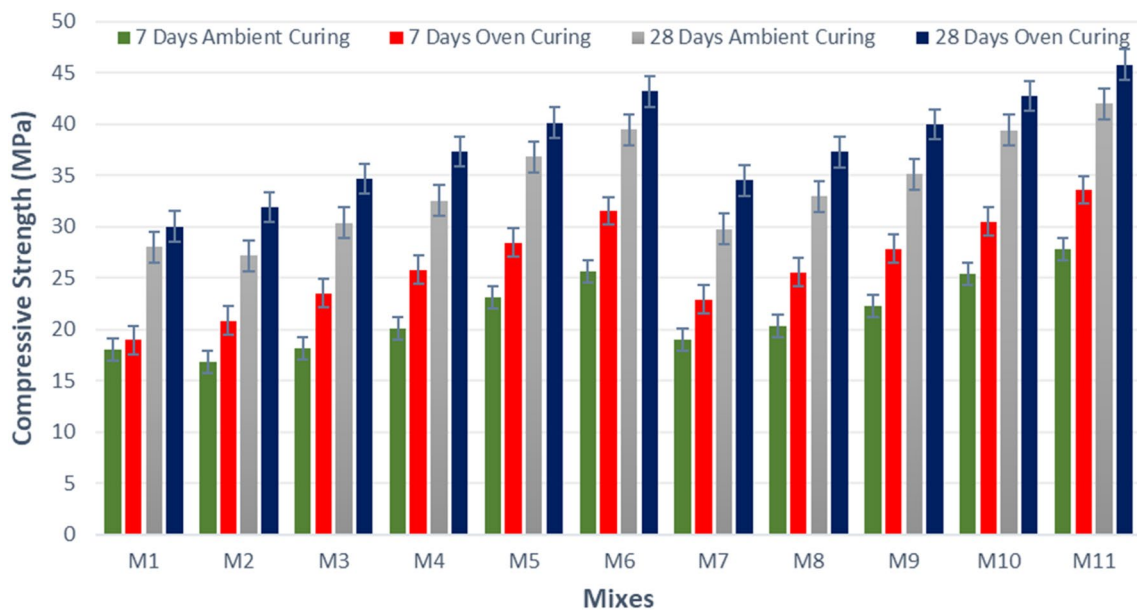
Fig. 5 Compressive strength of mixes at different curing regimes

Splitting tensile strength & flexural strength Figures 6 and 7 illustrate the splitting tensile strength and flexural strength respectively of the geopolymer specimens subjected to oven curing and ambient curing. The variation in the curing duration was not considered as it may not insist any significant variation with respect to tensile forces. The similar performance of the mixes was observed between compressive, splitting tensile and flexural strengths i.e., increase in the dosage of slag replacement dramatically increased the splitting tensile and flexural strengths. Replacement upto 50% of slag enhanced the splitting tensile strength upto 35% and flexural strength upto 40% respectively. It was quite obvi-

ous that, to optimize the content of slag, maximum replacement percentage of 50 can be considered and similarly, even though only a slight variation was observed between two ratios of hydroxide to silicate, ratio of 1.5 provided slightly greater results.

In contrary to compressive strength, splitting tensile and flexural strengths also rely largely on the matrix properties and the interface between the binder and fine aggregate. Thus, in this case also, the fineness of the GGBFS was observed to have good correlation with the particle sizes of flyash and thus upto 50% replacement of slag, the mechanical properties were enhanced greatly.

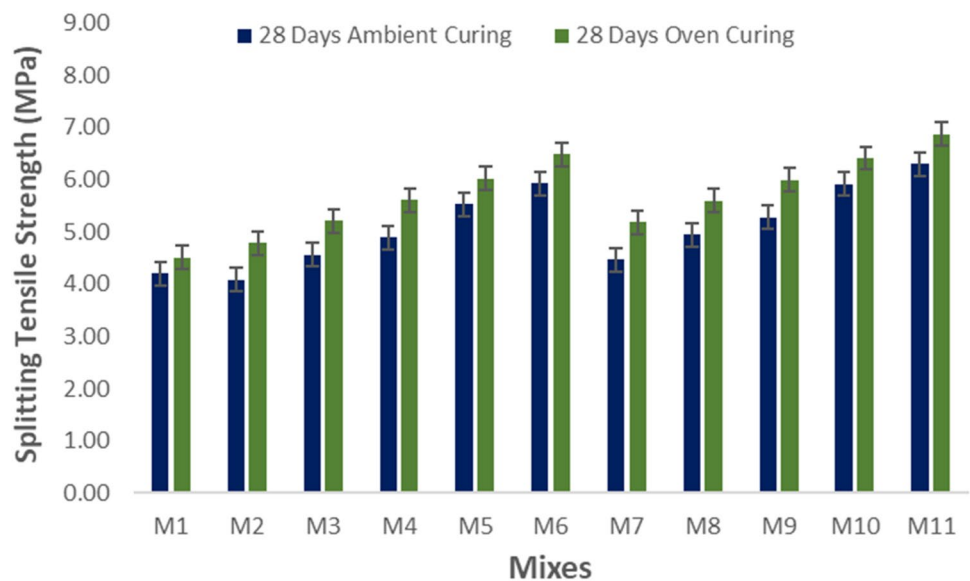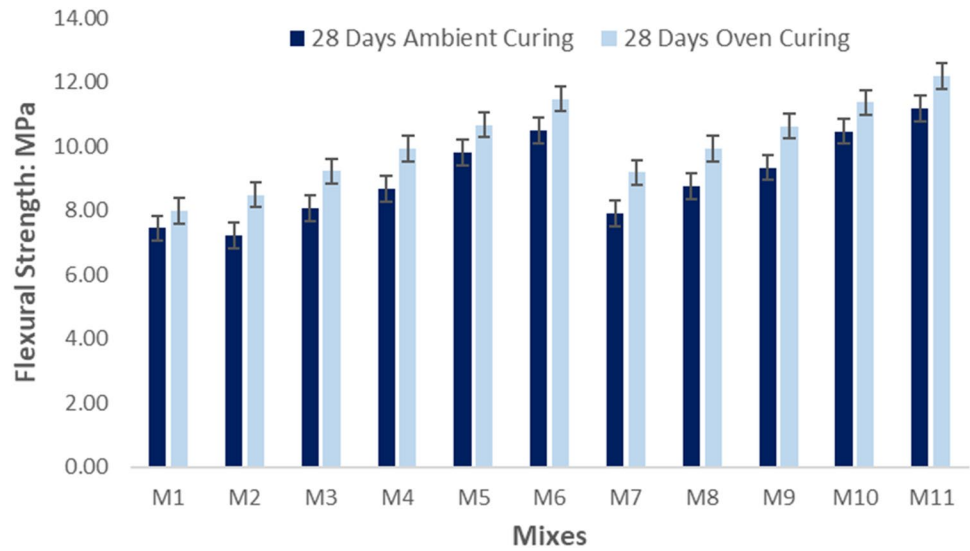**Fig. 6** Splitting tensile strength of mixes at different curing regimes

Fig. 7 Flexural strength of mixes at different curing regimes

Modulus of elasticity Table 4 shows the results of the geopolymer samples modulus of elasticity. The modulus of elasticity has been improved with the addition of GGBFS to FA-based geopolymer composites. There were elastic moduli of 28.48, 32.6, 25.42, 26.5, 27.49, 28.5, 29.57, 26.44, 27.48, 28.45, 29.42 and 30.45 GPa for the M2, M3, M4, M5, M6, M7, M8, M9, M10, and M11 mixes after 28 days of oven curing; the controlled mix (C) was 24.65 GPa. Geopolymer mortar samples with 50% GGBFS and 50% FA based samples had the best elastic modulus values, while samples with 10% GGBFS and 90% FA based geopolymer samples had the lower elastic modulus values. The modulus of elasticity increased as the GGBFS content in cement mortar samples was increased. There was a positive impact on mechanical properties and modulus of elasticity when GGBFS was replaced with FA in geopolymer composites under ambient curing conditions. It is clear from the experi-

mental results that the replacement of FA with GGBFS in geopolymer samples has improved the modulus of elasticity value compared to control mix.

The following are the equations reported by different codes and previous literatures to predict the modulus of elasticity of concrete.

(a) The young's modulus of OPC concrete (Density = 1442–2480 kg/m^3) can be calculated using Eq. (2) according to ACI 318–14 [60].

$$E_C = 0.043 \times \rho^{1.5} \times \sqrt{f'c} \tag{2}$$

Where, E_c = Modulus of elasticity (MPa)

Table 4 Modulus of elasticity from experiments and theoretical relations

Mix id	Compressive Strength (MPa)	Mean Density of GPC Kg/m^3	Modulus of Elasticity (E$_c$) GPa				
			Experimental (28 days), GPa	ACI 318 (2014)	AS 3600 (2001)	Diaz-Loya et al. 2011	Present paper
M1	30	2035	24.65	26.66	26.08	18.60	22.63
M2	31.91	2049	25.42	30.28	28.24	19.38	23.58
M3	34.68	2054	26.5	31.70	29.08	20.28	24.67
M4	37.34	2061	27.49	31.30	28.92	21.15	25.73
M5	40.12	2055	28.5	33.07	29.95	21.83	26.55
M6	43.18	2052	29.57	34.07	30.56	22.60	27.49
M7	34.541	2036	26.44	35.07	31.17	19.98	24.30
M8	37.311	2052	27.48	36.07	31.77	21.08	25.55
M9	39.971	2060	28.45	37.07	32.38	21.87	26.60
M10	42.751	2057	29.42	38.07	32.99	22.57	27.45
M11	45.811	2048	30.45	39.07	33.60	23.21	28.23

ρ = Density of concrete (kg/m^3)f'c = Characteristic compressive strength (MPa)fc = Average 28 days compressive strength (MPa)

(b) The elasticity modulus of OPC concrete can be calculated using Eq. (3) according to AS 3600 [61].

$$E_C = \rho^{1.5}\left(0.0243\sqrt{f'c} + 0.12\right) \qquad (3)$$

(c) According to Diaz-Loya et al. 2011 [62] the young's modulus of geopolymer presented through Eq. (4).

$$E_C = 37 \times \rho^{1.5} \times \sqrt{f'c} \qquad (4)$$

Based on the experimental compressive strength, Eq. (5) can be used to estimate the elastic modulus (in GPa) of FA\ and GGBFS-based geopolymer composites. At 28 days of oven curing, the young's modulus of geopolymer mortar samples can be estimated using the proposed model shown in Fig. 8.

$$E_C = 0.000045 \times \rho^{1.5} \times \sqrt{fc} \qquad (5)$$

Table 4 displays the geopolymer samples' modulus of elasticity as measured by standard codes, previous literature and this investigation. A higher modulus of elasticity was found in the 50% GGBFS and 50% FA-based geopolymer composite samples with 1.5:1 alkaline solution ratio at oven curing condition. The experimental data on modulus of elasticity was clearly indicating that the data obtained from experiment are almost equal to that of predicted values. This is positive sign that the predicted equation can be used to estimate the modulus of elasticity of FA-GGBFS-based geopolymer samples.

Durability properties

Figure 9 shows the water absorption results of the different mixes subjected to the optimization of geopolymer composites with respect to slag content and the ratio of hydroxide to silicate. It can be clearly observed from the figure that, the content of slag shows large influence towards the water absorption of the geopolymer mixes. It can also be figured out that the ratio of hydroxide to silicate has least effect on water absorption, as the set of specimens, say M2–M6 and M7–M11, do not show any major significant variation. Thus, the effect towards water absorption was completely based on the dosage of slag replacement with the flyash. The increase in the content of the slag greatly reduces the water absorption of geopolymer composites, as 50% replacement of slag with flyash has reduced upto 40% of the rate of water absorption. Irrespective of the ratio of the hydroxide to silicate, higher dosage of slag replacement is excellent in reducing the water absorption. It can also be inferred that 50% slag replacement with 1.5 ratio of hydroxide to silicate showed better results compared to that of the ratio 1.

In contrary to the water absorption results, similar effects can also be inferred in the sorptivity test as illustrated in Fig. 10. Both water absorption and sorptivity are the moisture related properties of the concrete specimens, however direct relations were not figured out in any of the past literatures. Thus, the effect of the dosage of slag replacement was crucial in sorptivity also, as the ratio of hydroxide to silicate did not show any significant variations. The presence of major pores might also contribute to the effect of both water absorption and capillary water rise. Because of its excellent fineness of GGBFS, the number of pores could be minimized which might be the scientific reason behind the reduction of water absorption and capillary water rise. Thus, it can be clearly inferred that M11 mix which was

Fig. 8 Young's modulus determination with experiment and theoretical equation compared with literatures
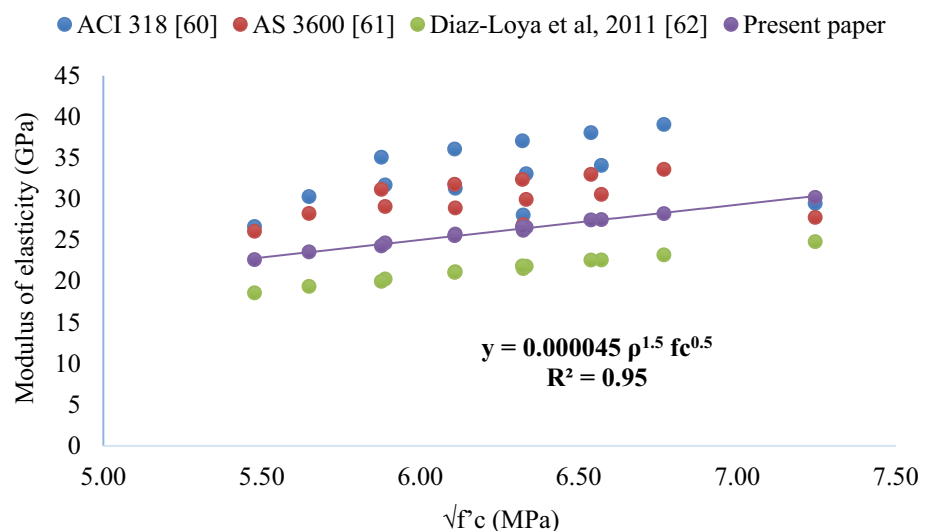
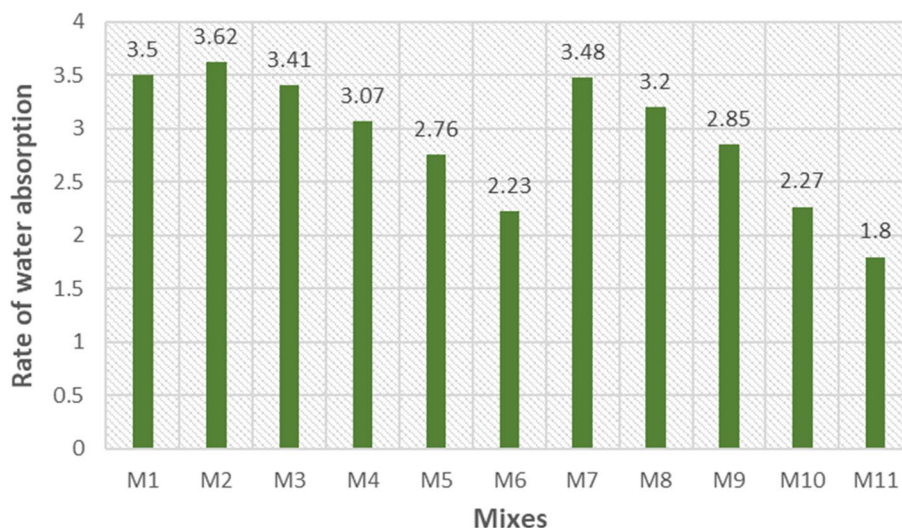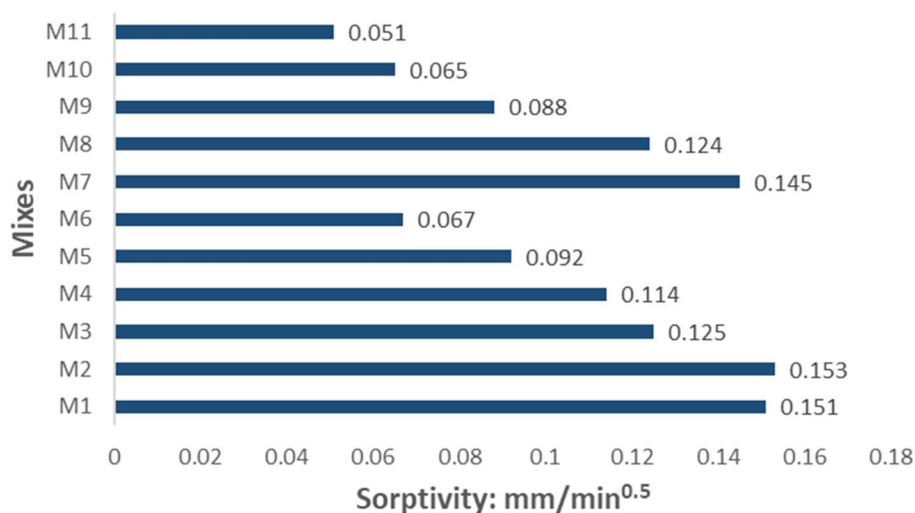**Fig. 9** Water absorption test results of different mixes



Fig. 10 Sorptivity test results of different mixes

excellent upon mechanical properties test, also showed good resistance to free waters and capillary water rise.

Microstructural properties

XRD Geopolymer samples were tested using an XRD analysis and the peaks are presented in Fig. 11. Out of 11 mixes from M1 to M13, few samples are selected for XRD analysis which are M1, M2, M6, M7 and M11 Scanning at a rate of one degree per minute from 3 to 87 degrees of 2-Theta yielded the XRD patterns. Figure 11 depicts the XRD patterns in amorphous and crystalline phases. It was found that calcite and quartz crystal peaks were prominent in the GGBFS and FA-based geopolymer concrete mixtures studied. Small peaks of mullite and alumina were also discovered. In FA-based geopolymer samples, a new peak of the C-A-S-H gel was observed in mixes M2, M6, M7, and M11, and it was found to form a steady polymeric reaction in

increasing amounts of GGBFS. GGBFS and FA hybrid gels are formed by alkali activation of GGBFS. GGBFS and FA samples were found to contain the C-A-S-H phase, which is one of the most common products of GGBFS and FA-based geopolymer concrete samples. The amalgamation of CO_2 from the environment during the analysis of the specimens led to the discovery of calcite peak. As the amount of GGBFS in geopolymer samples rises, so does the enrichment of the geopolymerization matrix. It is claimed that the GGBFS contains more soluble calcium oxide, which could improve geopolymerization products and increase gel production. The formation of geopolymeric gels and the improvement of geopolymer concrete strength qualities are thought to necessitate a highly alkaline media to increase the leaching of Si^{4+}, Al^{3+}, Ca^{2+}, and other minor ions. At 1.5:1 sodium silicate to sodium hydroxide, the strong alkaline medium was observed in geopolymer mixtures. Crystal phases like quartz and calcite are less prevalent in mixtures

Fig. 11 XRD charts for different significant mixes

1-Quartz, 2-Mullite, 3-Calcite and 4-C-A-S-H gel
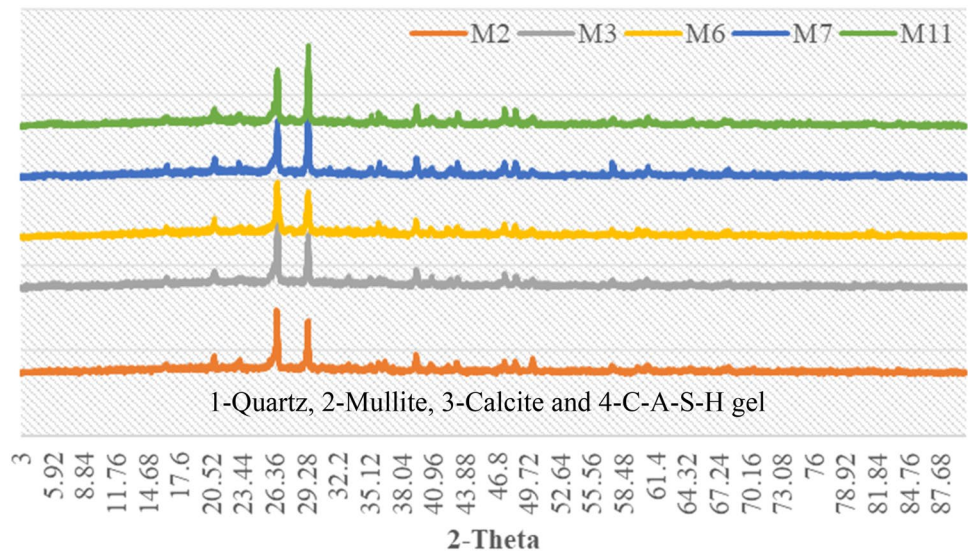
M2, M3, M7 and M8 because of the lower GGBFS content. In addition, the XRD peaks show that the amount of C-A-S-H gel decreases. The geopolymer samples has shown better results in case of alkaline ratio as 1.5:1 that is sodium silicate to sodium hydroxide as compared to 1:1 ratio sample. However, all the geopolymer samples shown improved properties with the binder combination of GGBFS and FA.

SEM with EDS Four geopolymer mixes (M2, M6, M7 and M11) based on FA-GGBFS are presented in SEM images in Fig. 12a–d. A standard medium, polymeric reaction, non-activated FA, and semi-activated FA particles were used to illustrate the microstructural images of geopolymer samples in Fig. 12. The non-activated particles are detected with the higher FA concentrations, such as M4 and M9 because of the decreased FA reactivity. Strength data support this conclusion, showing that specimens rich in GGBFS produce more geopolymerization matrix. A looser matrix is produced when the FA content of geopolymer mixtures is increased. In Fig. 13a, the geopolymer reaction is shown to have a low-density structure and few unreacted FA particles. Samples of Mix M13 that had more C-A-S-H gel formation, on the other hand, had a denser microstructure (50% GGBFS and 1.5:1 alkaline ratio). Sample M13 differed significantly from sample M8 when compared side by side. The lower sodium silicate content in the geopolymer mixes did not result in effective geopolymerization products, which explains why the strength attributes of the M8 samples are lower. Because the mix M4 had a lower salt concentration, the geopolymerization process was slow. The microstructure of the geopolymer system was altered as a result of the reaction of steel slag and FA with a 1.5:1 alkaline ratio. Images from scanning electron microscopy (SEM) show that the samples' surfaces had been subjected to the formation of inappropriate bonding. Because the geopolymer concrete mixtures con-

tained less GGBFS, it was discovered. Compared to other geopolymer mixes, these have a lower steel slag content, a less dense microstructure, and poorer mechanical properties. M4 and M9 mixtures were also found to have an interruption in the polymeric matrix. Figure 12a and c shows examples of inadequate gel formations.

The EDS peaks associated to the SEM images of geopolymer and control samples are presented in Table 5. Significant elements such as silica, calcium, sodium and alumina were discovered in selected areas of SEM images, as shown in Table 5. The EDS results were in good correlation with the XRD study. Si, Ca and Al are the most common elements found in geopolymer composites.

Optimization based on the effects of fibre

Different mixes including the effects of the content of slag, ratio of hydroxide to silicate were considered for optimization compared to cement mortar as reference upon fresh, mechanical, durability and micro-structural characteristics. Based on the optimization process, as shown in Fig. 13, it was identified that M11 mix has provided the maximum efficiency in all the parameters. i.e., 50% slag replaced with the content of fly ash with the hydroxide to silicate ratio of 1.5 provided the maximum efficiency in compressive, tensile and flexural strengths, as well as durability characteristics, which were cured under high temperature curing regime. Thus, M11 mix was considered as the optimized mix, which was further considered for optimization with respect to addition of glass fibres with various dosages. The mix proportion with respect to this optimization process is summarized in Table 6. The casting, curing and testing methods remains the same as in the case of previous optimization process.
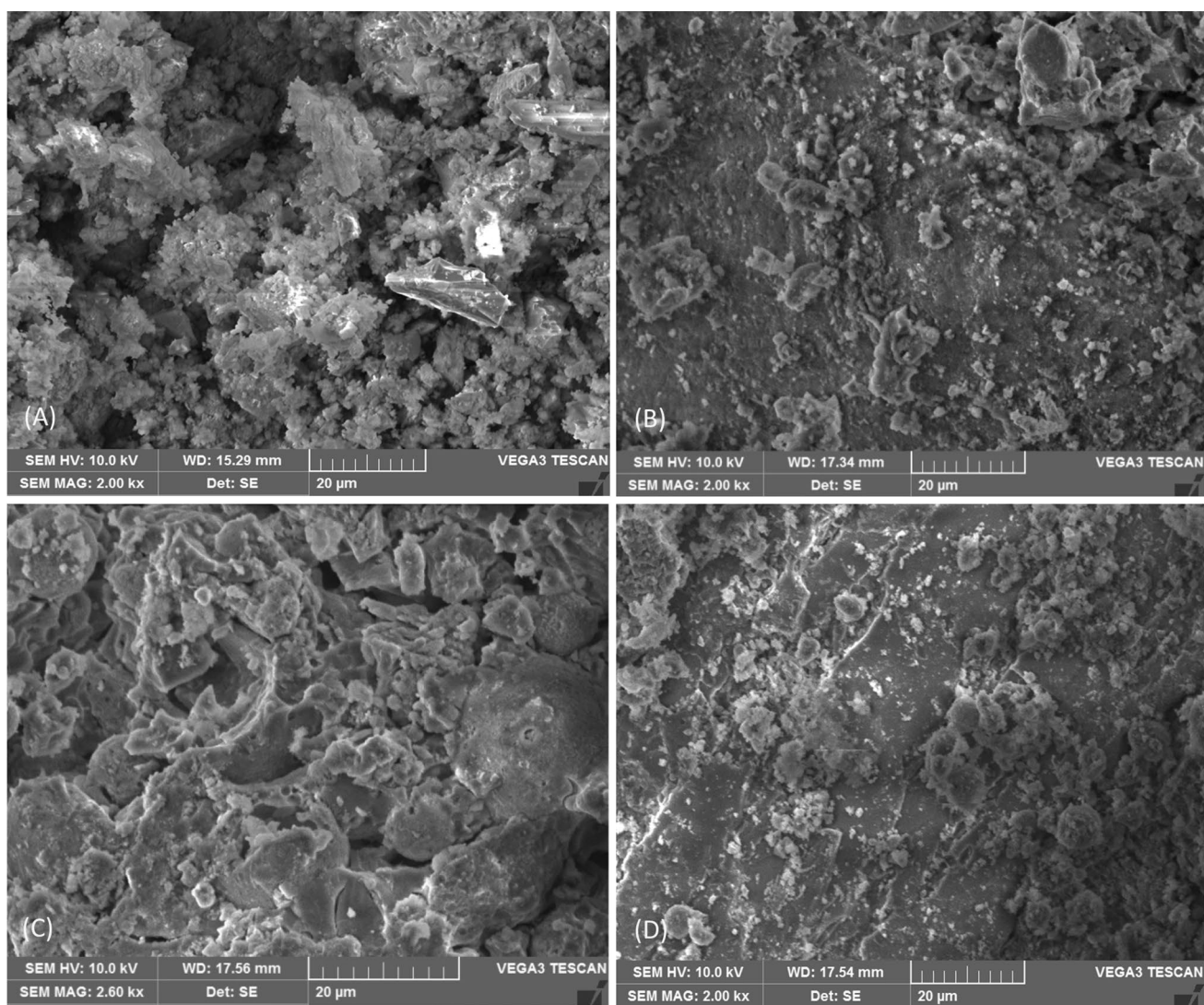
Fig. 12 SEM images for different significant mixes

Mechanical properties

Figure 14 shows the compressive strength test results of GFRGC specimens after 28 days of ambient curing and heat temperature curing. It can be clearly observed from the figure that the inclusion of glass fibre in flyash GGBFS blended geopolymer concrete is only optimum within a particular limit beyond which there is a sudden drop in the compressive strength. The optimum dosage of fibre was identified to be 1% which enhanced the compressive strength upto 8%. It can also be identified that though compressive strength of MF1.5 mix is slightly less than that of MF1 mix, the compressive strength was enhanced when compared to M11 mix, i.e., mix without fibres. Thus, in certain situations MF1.5 can also be adopted to the structure which may enhance other mechanical or durability parameters. Addition upto 2% of glass fibres shows decrement in strength when compared to M11 mix.

In eco-friendliness point of view, reduction in the content of chemicals in any fraction would certainly help the sustainability however if the technical requirements dominate, the MF2 mix can be avoided.

Figure 15 shows the flexural and splitting tensile performance of the GFRGC mixes cured at high temperature exposure followed by 28 days of ambient curing. In these tests, the simple ambient curing was not considered since it may not be effective as heat temperature curing, as observed from previous results. Similar to the compressive strength test results, the mix MF1 has provided the most optimized results as it has enhanced both the split tensile and flexural strength of the flyash and GGBFS blended geopolymer composite. But the critical observation made here is that anything beyond 1% addition of glass fibre results in deterioration in split tensile and flexural strengths. It is highly suggested that, in case of structural applications involving
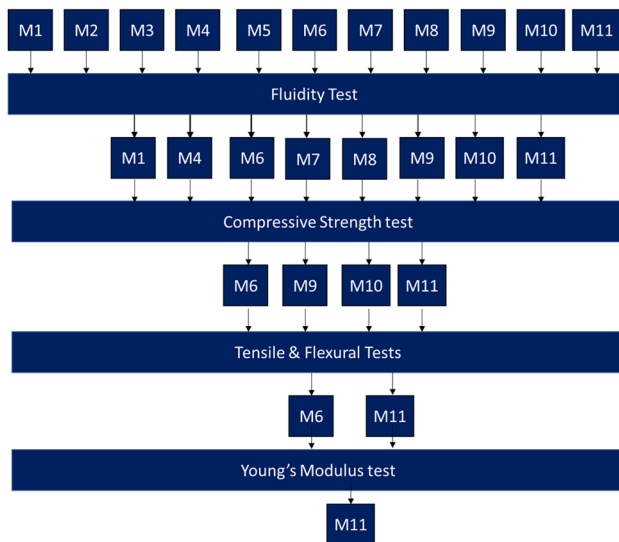
Fig. 13 First stage optimization process

Fig. 14 Compressive strength test results of GFRGC specimens

Table 5 Elements identified in EDS analysis

Element (Wt. %)	M2	M6	M7	M111
Si	43.36	28.44	42.05	32.75
Al	18.11	17.08	17.04	19.91
Na	8.20	10.25	12.89	16.10
Ca	8.95	21.00	11.95	26.21
Mg	0.41	0.29	0.67	–
C	1.34	2.40	2.09	1.48

Table 6 Mix proportions for glass fiber reinforced geopolymer composite

Mix id	Proportions				Glass fibers (%)
	Binders		Alkaline solution		
	Fly ash	GGBFS	Na_2SiO_3	NaOH	
	(kg/m³)				
MF 0	1008	432	259.2	172.8	–
MF 0.5	1008	432	259.2	172.8	0.5
MF 1	1008	432	259.2	172.8	1
MF 1.5	1008	432	259.2	172.8	1.5
MF 2	1008	432	259.2	172.8	2

any sort of tensile forces, the dosage of fibre can be limited to 1% for better results. 2% addition of glass fibres in geopolymer showed the results similar to that of M11 mix. Thus, the same mechanical performance can be obtained with the inclusion of 2% glass fibres, however, it may not be much economical compared to M11 mix.
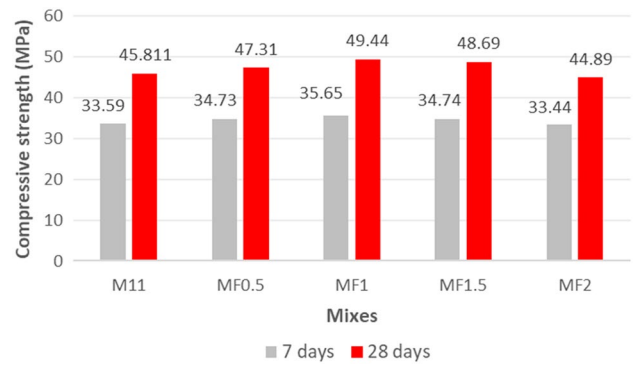
Durability properties

In this section, the influence of the dosage of the glass fibers in the geopolymer composite is significant towards the sensitiveness upon its durability properties. Figures 16 and 17 show the water absorption test and sorptivity test results respectively of M11 mix (without fibres) and mixes with several dosages of fibre. It was revealed previously that M11 mix (without fibres) showed 1.8% water absorption. The absorption of water by the composites may be primary due to absorption by the material or presence of free water in the pores. Whereas the water absorption of other mixes could be possibly influenced by the fibres. As it is clear from Fig. 15, that the water absorption range of GFRC is within 1.8–2.11% which showed incremental increase with increase in the dosage of fibers, however there is not much difference between GFRC mixes. This is well correlated with the literatures that glass fibres generally show very low water absorption compared to other natural and synthetic fibres. Similarly, the mixes also showed identical results on other water-based durability property i.e., sorptivity. 2% dosage of glass fibre in M11 mix was quite critical as it showed 25% more capillary water absorption compared to M11 mix. However, it is to be noted here that the magnitude of such durability results are quite very low and does not have any significant effects in the specimen.

Microstructural properties

To examine the effect of the addition of glass fibres in geopolymer concrete with respect to microstructural properties, only SEM images were taken, as shown in Fig. 18, since it is quite obvious that fibres do not create any influence over XRD or EDS analyses. The investigation upon microstructural properties through analysis with SEM images was to observe the nature of the matrix and fibre-matrix interface. Two best mixes viz., MF0.5 and MF1 were considered for SEM analysis, as these mixes provided better results over

Fig. 15 Splitting tensile strength and flexural strength test results of GFRGC specimens
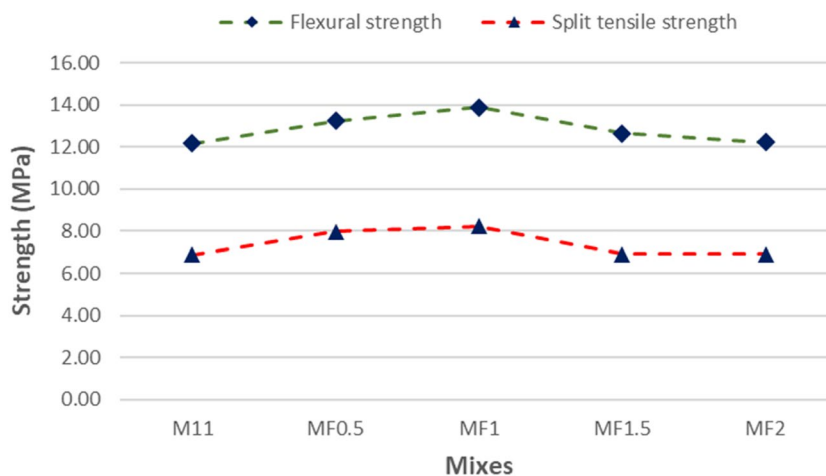

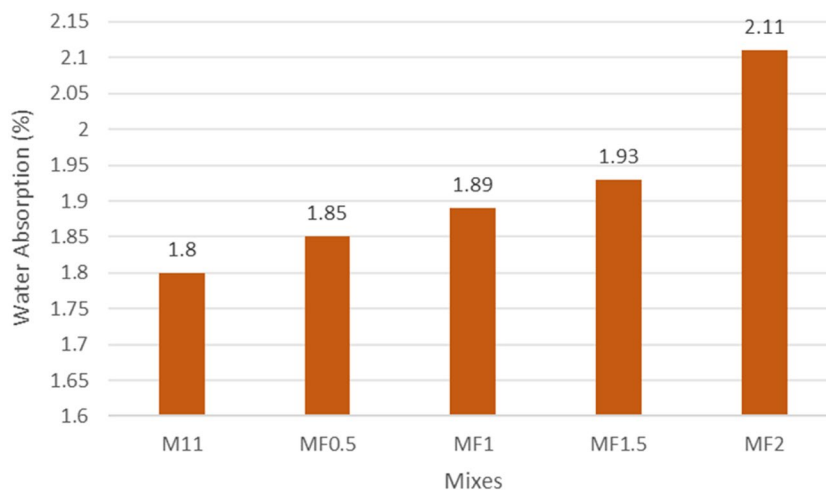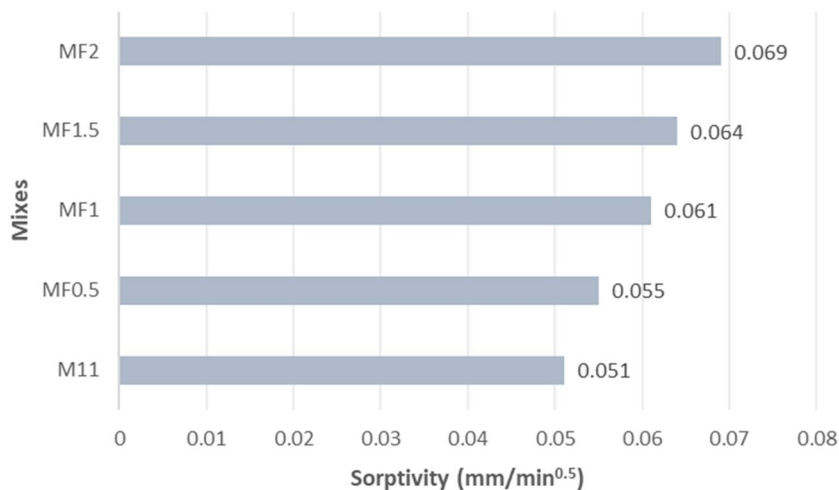**Fig. 16** Water absorption test results of GFRGC specimens

Fig. 17 Sorptivity test results of GFRGC specimens

mechanical and durability parameters. The presence of the glass fibers is particularly focused because of the analysis of the existence of transition zones around the fibers. These transition zones would lead to more micro cracks and pores around the fibres which may be the primary responsible for reduction in strength and poor durability properties. Slightly away from the fibres, good denser microstructure can be visible in both images MF0.5 and MF1 mixes, which shows
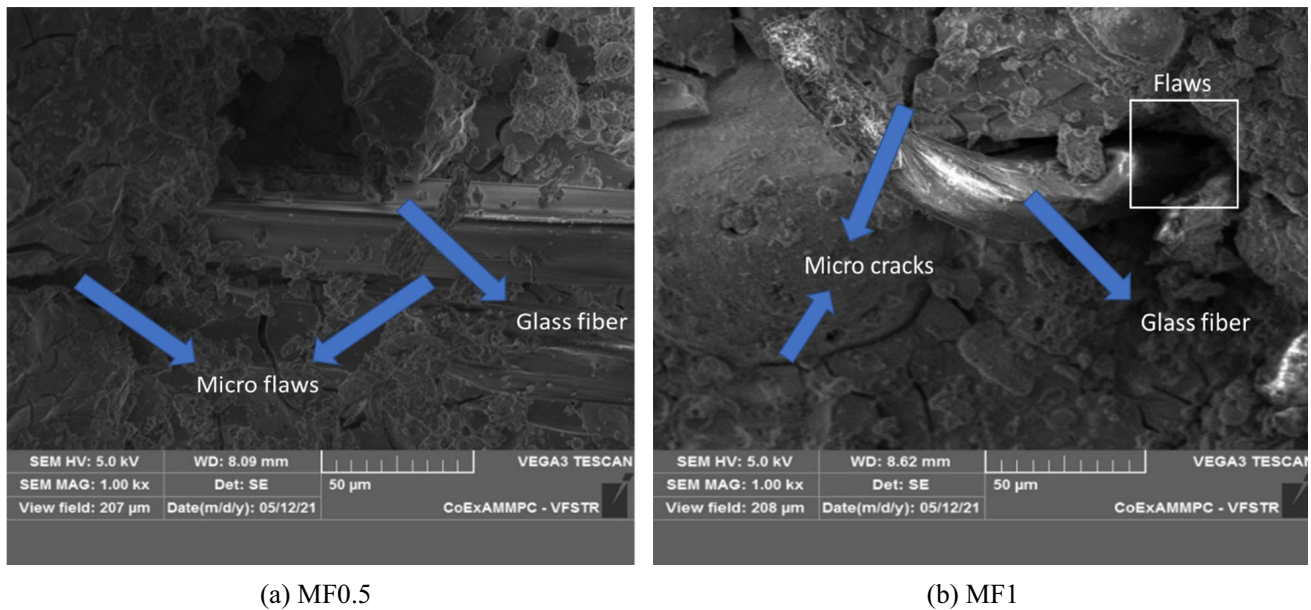
(a) MF0.5　　　　(b) MF1

Fig. 18 SEM images of GFRGC specimens

good agreement with the experiments respect to mechanical properties. Based upon durability tests, increase in dosage of fibre showed greater penetration of free water and capillary water which was primarily due to transition zones around the fibers. However, optimum dosage upto 1% dominates the denser microstructure, thus showed excellent mechanical properties.

Conclusions

This paper presents the influence of various factors such as alkaline ratio, variance in GGBFS dosage and incorporation of fibers on strength and microstructural characteristics of fly ash-based geopolymer samples. The optimization of aforesaid parameters was then followed by the optimization based on the involvement of glass fibers in various dosages. The experimental results about the effect of GGBFS and alkaline ratios of FA-based geopolymer samples were presented in this paper. However, the microstructural studies were conducted to understand the micro-level behavior of geopolymer samples under ambient and oven curing conditions. Moreover, the predicted modulus of elasticity equations will give an estimated value and it can be comparable to experimental value. From the experimental investigation on FA-GGBFS-based glass fibre reinforced geopolymer samples the following conclusions were drowned.

　　When geopolymerization times ranged from 5 to 120 min, FA-GGBFS mixtures revealed fluidity values ranging

between 76 and 195 mm. As the GGBFS content rises, the fluidity value was decreased. Flowability values of GGBFS-rich mixtures are more affected by alkaline ratios than those of fly ash-rich mixes.

Mixes rich in GGBFS set faster than mixes rich in fly ash. When compared to a 1:1 ratio sample, the alkaline ratio of 1.5:1 solution boosted the setting. The principal reaction products observed in FA-GGBFS geopolymer following setting and hardening of the paste/mortar include hybrid sodium–calcium alumino-silicate geopolymeric compounds, as well as calcium-based hydration compounds. Increased slag content improves geopolymer samples' mechanical characteristics. It was shown that geopolymer paste samples with 30% GGBFS and 70% FA had the highest compressive strength (52.5 MPa), while mortar samples with 50% GGBFS and 50% FA had the highest compressive strength (45.81), with a 1.5:1 alkaline ratio under oven curing. While geopolymer mortar samples have a higher compressive strength, those made from geopolymer paste have higher compressive strengths as well. For the GGBFS and geopolymers, the major reaction chemicals responsible for strength gain are calcium-based alumino-silicate hydration products and sodium-based alumino-silicon complexes, respectively.

In the optimization process with respect to materials, the mix M11, i.e., 50% replacement of slag with 1.5 ratio of hydroxide to silicate performed best which was further considered for further optimization with the dosage of glass fibre. The experiments were similar for both stages of optimization and it revealed that the mix MF1 performed best in all aspects i.e., addition of 1%

glass fibre has enhanced the mechanical, durability and microstructural properties of the optimized geopolymer composite and further increase in dosage deteriorated the performances.

Data availability Not Applicable.

Code availability Not applicable.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent Not applicable.

Consent for publication Not Applicable.

Replication of results No results are included.

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