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Influence of mix parameters on mechanical, microstructural, and durability properties of geopolymer mortar

Submitted

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CERTIFICATE

It is certified that the work contained in the project report entitled "**Influence of mix parameters on mechanical, microstructural, and durability properties of geopolymer mortar**", by **Nameet** (Roll No. 200104065) has been carried out under my supervision and that this work has not been submitted elsewhere for the award of a degree or diploma.

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ABSTRACT

The objective of this study is to investigate the effect of source materials, alkaline solution, and admixed chloride and sulphate salts on flowability, compressive strength, and microstructural properties of geopolymer mortar (GPM) mixes. The GPM mixes were made with different proportions of ground granulated blast furnace slag (BFS) and fly ash. The alkaline solution used was a mixture of sodium hydroxide solution (6 M and 10 M) and sodium silicate solution. The flow table test was conducted on fresh GPM to measure the flowability. The compressive strength test was carried out on control (without admixed salt) GPM cubes of size 50 mm at the age of 7 days. The XRD analysis was conducted to investigate the microstructure of GPM mixes. From the obtained results, the flow index (%) increased with fly ash content, and reduced with increase in NaOH solution molarity. The compressive strength of GPM mixes increased with BFS content and molarity of NaOH solution. The XRD analysis showed increase in the peak intensity of nepheline, anorthoclase, and C-S-H gel with increase in BFS content. Further, the increase in molarity of NaOH solution led to increase in peak intensity of nepheline and anorthoclase.

Keywords: Geopolymer mortar (GPM); Ground granulated blast furnace slag (BFS); Fly ash; Compressive strength; XRD analysis.

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Date: 27th November 2023

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Introduction

1.1. General

Concrete is the second most used material after water on the earth. Cement is one of the primary constituents, typically associated with the production of conventional concrete. It is well known that the production of one ton of cement releases one ton of carbo dioxide (CO_2) into the atmosphere. To overcome this problem, different alternate binding materials have been developed and one such emerging material that has gained interest in the scientific community is alkali activated material which is also termed as geopolymer binders [1,2]. Researchers have utilized different industrial by-products such as fly ash, ground granulated blast furnace slag, silica fume, etc., as a partial or full replacement of cement in the production of concrete through alkali activation [3]. Among different alkali activators, the combination of sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solution was commonly used in the production of geopolymer binders.

1.2 Geopolymerization mechanism

The polymerization process of geopolymer binders mainly includes four stages: (i) dissolution: the Si–O and Al–O bonds are broken in the aluminosilicate materials under the action of the alkali activator, and the Si–O and Al–O tetrahedral monomers are released; (ii) diffusion: dissolved Si–O and Al–O tetrahedral monomers diffuse into the reaction system. According to the principle of chemical equilibrium, the silicon and aluminium concentrations decrease on the particle surface owing to the diffusion, and the dissolution process continues, (iii) polycondensation: Si–O and Al–O tetrahedra form amorphous -Si-O-Al-O- structures through polymerization, and (iv) hardening: dehydration reaction occurs, forming a hardened geopolymer with high mechanical strength [4]. The geopolymer structure was given by Prof. J. Davidovits, which is shown in equation (1.1) [5,6].

$$M_n [(SiO_2)_Z - AlO_2]_n \, wH_2O \qquad (1.1)$$

where, M is an alkali metal cation; n represents the degree of polycondensation; w represents the number of chemically bound water molecules; z represents the silicon-to-aluminium ratio (Si/Al), which is 1, 2, or 3. The structures of the aluminosilicates are divided into three forms as per the Z value, which are illustrated in Fig. 1.1 [6].

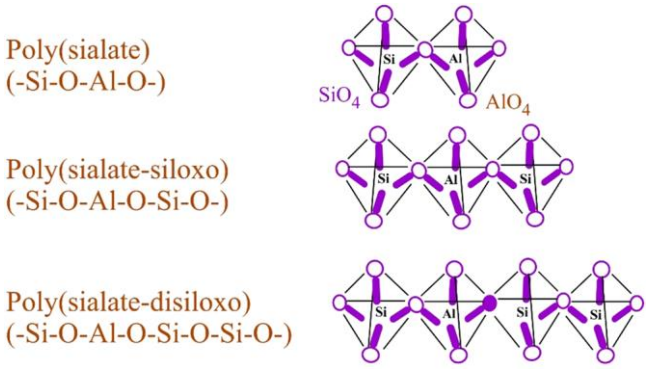

Fig. 1.1 Different types of aluminosilicate structures (1989) [6]

1.3 Properties of geopolymer composites

Geopolymer composites generally exhibit inferior fresh properties compared to OPC based composites. This is due to the higher viscous nature of silicate components present in the geopolymers [7]. Wong et al. investigated the fresh properties of fly ash based geopolymer mortar by varying the sodium silicate (Na_2SiO_3) solution to the sodium hydroxide (NaOH) solution, and concentration of NaOH solution. The authors found that the increase in sodium silicate solution to the sodium hydroxide solution, and concentration of NaOH solution reduced the flowability of fly ash based geopolymer mortars [8]. According to experimental results, utilizing geopolymer binders in place of ordinary Portland cement (OPC) in several applications significantly increased both compressive strength and resistance to sulfuric acid [9]. The performance of geopolymer binders depends on several factors which included type and size of precursor, concentration of alkaline activator solution, mix design, curing conditions etc. The increase in fineness of precursor material and molarity of alkaline solution improved the strength properties due to increase in specific surface area and increased polymerization rate, respectively [10]. The geopolymer composites cured at high temperatures showed better performance due to more stable crosslinked aluminosilicate polymer structure [11]. Generally, geopolymer composites made of 100% fly ash required curing at elevated temperatures due to slow polymerization reaction. However, this limitation can be overcome by replacing fly ash with calcium rich material such as ground granulated blast furnace slag [12]. When compared to OPC composites, geopolymer composites exhibits superior mechanical properties and offers greater resistance towards chemical attack. Additionally, water absorption, sorptivity, and porosity have a positive impact on the durability characteristics in ambient curing environments [13]. Verma et al [14]. stated that the fineness of fly ash, concentration of alkaline liquid, ratio of alkaline solutions, and ratio of fly ash to the

alkaline solution affects the properties of geopolymer composites. From the literature, it was found that the effect of variation in proportions of fly ash and GGBFS, and variation in concentration of sodium hydroxide solutions on mechanical properties of geopolymer mortar made with fly ash and GGBFS were studied [15–17]. These authors have reported that the compressive strength increased with slag content. However, the durability studies related to geopolymer mortar prepared with fly ash and GGBFS were limited.

Chapter-2

Literature Review

2.1 General

In this chapter, the review of previous research works carried out on different properties of geopolymer composites are presented.

Hager et al. [18] have investigated the microstructure and mechanical properties of geopolymer mortars made of fly ash and ground granulated blast furnace slag, and cured at room temperature and elevated temperatures (200, 400, and 800^{o}C). The fly ash was replaced with ground granulated blast furnace slag at 0, 10, 30, and 50% by weight. The authors have used sodium silicate solution as alkaline solution. The mortars were prepared with a sand to binder of 1.5, the ratio of alkaline solution to binder was 0.45, and water to binder weight ratio taken was 0.3. The authors have concluded that the addition of slag resulted in higher mechanical strength. In addition, the mortar specimens when subjected to 800^{o}C showed lower strength. From microstructural observation, the authors have reported that the fly ash geopolymer mortars showed better behavior when cured at high temperature.

H. Liu et al. [19] have investigated the impact of different alkali activators on the setting time of Class F fly ash paste, and also investigated the compressive strength and microstructure of paste by using FT-IR. The different alkaline activators that are used are sodium hydroxide (NaOH) solution, sodium silicate solution (Na_2SiO_3) solution, and a combination of NaOH and Na_2SiO_3 solutions. The authors have also investigated the influence of anhydrous borax as a retarder at varying dosages (5%, 10%, 15%, 20%, and 25% by fly ash mass). Cubes of 50 mm size were prepared and cured at 75°C in chamber with water bath for compressive strength test at 1 and 3 days. From the results, the authors have concluded that the setting time was extended with increased NaOH dosage when using combination of NaOH and Na_2SiO_3 solutions. Further, they reported that the addition of 10% and 15% borax to NaOH solution significantly extended setting time, but other dosages had less impact. The prolonged setting time with borax was due to the formation of $[BO_4^-]$ tetrahedron in Class F fly ash, which was observed by authors from FT-IR analysis. The authors have concluded that the borax's retarding effect was weakened with higher alkali concentration, and the compressive strength was decreased with addition of borax when compared with the mixtures without borax.

Ryu et al. [20] have investigated the mechanical properties of fly ash-based geopolymer concrete with different alkaline activators. The alkaline activators used in this study were sodium hydroxide (NaOH) solution at a concentration of 6 M, 9 M, and 12 M, and mixture of NaOH solution (9 M) along with sodium silicate (Na_2SiO_3) solution. Mortar cubes of 50 mm size were prepared for compressive strength test, and cylinders of 100 mm diameter and 200 mm height were prepared for split tensile strength test. The authors have also carried out microstructure studies i.e., X-ray diffraction (XRD) analysis, scanning electron microscope (SEM), and energy dispersive X-ray analysis (EDS) analyses. The authors have reported that the increase in molarity of NaOH solution led to increase in the compressive strength and split tensile strength. Further, it was found that the mixes made with mixture NaOH solution and Na_2SiO_3 solution had attained higher strength than mix made with NaOH solution. From the microstructural investigations, the authors concluded that the concrete mixes made with both NaOH solution and Na_2SiO_3 solution showed denser microstructure than the mixes made with NaOH solution.

Elyamany et al. [21] have investigated the setting time, compressive strength, and flexural strength at the age of 7 days of geopolymer mortars prepared with different binders. The geopolymer mortars were made with different proportions of fly ash, ground granulated blast furnace slag (GGBS), and silica fume i.e., 100% fly ash, 50% FA with 50 % GGBS, and 50% FA with 35 % GGBS and 15 % silica fume. The alkaline solution used was a combination of sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solution. The concentration of NaOH solution was varied at 10 M, 12 M, 14 M, and 16 M. The geopolymer mortars were made with different alkaline solution to binder ratios i.e., 0.35, 0.4, 0.45, and 0.50. The mortar specimens were cured at 30°C, 60°C, and 90°C. Cubes of 70 mm size were made for compressive strength test, and prisms of size 40 mm × 40 mm × 160 mm were made for flexural strength test. The authors have also conducted scanning electron microscope (SEM) as part of microstructure studies. From the results, the authors have reported that the increase in curing temperature led to increase in compressive strength and flexural strength of geopolymer mortars. In addition, the mortar mixes with fly ash, GGBS and silica fume had attained higher strength than other mixes. The increase in molarity of NaOH solution had enhanced the compressive strength and flexural strength of geopolymer mortar mixes. Further, the compressive strength and flexural strength decreased with increase in alkaline solution to binder ratio. SEM micrographs show that the mortar mix with fly ash, GGBS, and silica fume

had denser microstructure compared to that of mortar mix with fly ash, and mix with fly ash and GGBS.

Nazari et al. [22] have studied the compressive strength and microstructure of boroaluminosilicate geopolymer pastes made of fly ash. The authors have prepared anhydrous borax by heating borax decahydrate at 150°C for 30 min and further heated at 300°C for 15 hours, and mixed with NaOH solution. The authors have used this solution for the preparation of fly ash based boroaluminosilicate geopolymer. The mixes were made with different alkaline activator (NaOH plus anhydrous borax) to binder ratios i.e. 0.75, 0.80, 0.85, and 0.90. Cubes of 50 mm size were prepared and tested for compressive strength test at the age of 3, 7, 28 and 90 days. The microstructure studies i.e., scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FT-IR). The authors reported that the compressive strength was increased with increase in ratio of alkaline activator to binder ratio. Additionally, the authors did not observe any micro cracks in the pastes made of boroaluminosilicate geopolymers while conducting the SEM analyses. Further, from FT-IR analysis, the authors have identified additional bond i.e. B-O bond in boroaluminosilicate geopolymer paste, and it was not present in aluminosilicate geopolymer.

Somna et al. [23] have studied the compressive strength and microstructural properties of ground fly ash based geopolymer paste. The authors have used sodium hydroxide (NaOH) solution as an alkaline solution The molarity of NaOH solution was varied at 4.5 M, 7 M, 9.5 M, 12 M. 14 M, and 16.5 M. The ratio of NaOH solution to fly ash was taken as 0.3. The fly ash and NaOH solution were mixed for five minutes, and the samples of geopolymer paste were casted into cylindrical mould (size: 30 mm diameter and 60 mm height). The samples were maintained in a controlled environment at a temperature of 25 to 28°C till the day of testing. The compressive strength of geopolymer paste specimens was assessed at 7, 14, 28, 42, and 60 days. The authors have carried out X-ray diffraction (XRD) analysis, scanning electron microscope (SEM) analysis, and energy dispersive spectroscopy (EDS) analysis as part of microstructural studies. The authors have found that the highest compressive strength was attained in the pastes made of 14 M NaOH solution. Additionally, they reported that the compressive strength was increased with age. From microstructural investigations, the authors have found that the alkali activation of ground fly ash has occurred at room temperature. Further, they concluded that the ground fly ash can be used for preparation of geopolymers at room temperature.

2.2 Objectives of present study

The objective of the present research work are as follows:

- To study the flowability and compressive strength of geopolymer mortar (GPM) made with different proportions of ground granulated blast furnace slag (BFS) and fly ash, and different concentrations of sodium hydroxide solution.
- To investigate the durability and microstructural properties of BFS-fly ash based geopolymer mortar.

Chapter-3

Experimental Work

3.1 General

In this chapter, the materials used in the preparation of ground granulated blast furnace slag (BFS) and fly ash (FA) based geopolymer mortar (GPM) mixes, and the tests conducted on the GPM are presented.

3.2 Materials

3.2.1 Material details and mix proportions

For the preparation of geopolymer mortar (GPM), the precursor materials used are ground granulated blast furnace slag (BFS) and fly ash (FA). The alkali solution used was a mixture of sodium hydroxide (NaOH) solution, and sodium silicate (Na_2SiO_3) solution. The geopolymer mortar was designed for a wet density of 2125 kg/m^3, and the mass ratio of alkaline solution to binder content was taken as 0.5. The fine aggregate used in the preparation of GPM mixes was locally available river sand confirming to Zone II as per IS 383:2021 [24] that had a specific gravity of 2.65. The proportions and quantities of geopolymer mortar mixes are given in Table 3.1.

Table 3.1 Proportions and quantities of geopolymer mortar

BFS (%)	Fly ash (FA) (%)	Molarity of NaOH Solution	SS/SH ratio	Binder (kg/m^3)		Alkaline solution (kg/m^3)	Sand (kg/m^3)
				BFS	FA		
85	15	6 M	1.45	578	102	340	1105
		10 M					
65	35	6 M		442	238		
		10 M					

SS: Sodium silicate solution, SH: Sodium hydroxide solution

3.2.2 Source of chloride and sulphate ions

In the present study, sodium chloride (NaCl) was used as the source of chloride ions, and sodium sulphate (Na_2SO_4), and magnesium sulphate ($MgSO_4$) were used as the source of sulphate ions while preparing the salt admixed GPM mixes. In addition, to study the combined

effect of chloride and sulphate ions, the combination of NaCl with Na_2SO_4, and NaCl with $MgSO_4$ were also admixed during the preparation of salt admixed GPM mixes. The concentrations of admixed salts in the GPM mixes are given in Table 3.2.

Table 3.2 Details of admixed salts in GPM mixes

Admixed salt with concentration	Abbreviation
3.5% sodium chloride (NaCl)	3.5NC
7% sodium sulphate (Na_2SO_4)	7NS
7% magnesium sulphate ($MgSO_4$)	7MS
3.5% NaCl with 7% Na_2SO_4	3.5NC with 7NS
3.5% NaCl with 7% $MgSO_4$	3.5NC with 7MS

3.3 Preparation of GPM mixes

Initially, the alkaline solution was prepared for the preparation of GPM mixes. The preparation of alkaline solution involves dissolving the NaOH pellets in laboratory tap water as per the required molarity, i.e. 6 M and 10 M, 48 hours before the preparation of GPM mixes. After 24 hours, the Na_2SiO_3 solution was added to the NaOH solution. In case of salt admixed GPM mixes, the salts i.e., NaCl, Na_2SO_4, and $MgSO_4$ of different concentrations, as mentioned above, by mass of geopolymer solids were added to the alkaline solution prior to the preparation of mixes. During preparation of GPM mixes, the dry mixing of source materials and fine aggregate was carried out in a mortar mixer. Subsequently, the alkaline solution was added, and the mixing was continued to obtain a homogeneous mix. Then, flow table test was conducted on the fresh geopolymer mortar mix. Subsequently, the fresh mortar mix was placed in cube moulds of size 50 mm in 2 layers, and each layer was tamped 25 times with a tamping rod followed by vibration for 10–15 seconds on a vibrating table. After 24 hours of casting, the geopolymer mortar (GPM) cubes were demoulded and left to ambient laboratory condition till the day of testing.

3.4 Test methods

3.4.1 Flowability of GPM

The freshly prepared geopolymer mortar (GPM) flowability was measured by conducting the flow table test as per the procedure given in ASTMC 1437-20 [25]. The mould was lubricated with oil and then placed at centre of the flow table. The freshly prepared GPM mix was filled

in two layers, and each layer was tamped for 20 times with a tamping rod. The additional material was struck off, the top surface was levelled and the table was cleaned with a dry cloth. Then, the mould was lifted and flow table was dropped for 25 times within a duration of 15 seconds. To measure the flow value of the GPM mix, the diameter of the mortar spread was measured along four lines inscribed on top surface of the flow table. The average of these four measurements is the flow value of the GPM mix. The flow index was evaluated using the following equation.

$$FI\ (\%) = \frac{A}{B} \times 100 \qquad (3.1)$$

Where, *FI* (%) is the flow index, *A* is the average of four measurements of mortar spread minus the inner diameter of base of the mould, and *B* is the inner diameter of base of the mould.

3.4.2 Compressive strength test on GPM

The compressive strength test of GPM mixes was carried out on 50 mm cubes in a compression testing machine at the age of 7 days. Three replicate cubes from a given mortar mix were tested, and the average value was noted as the compressive strength value of that GPM mix.

3.4.3 XRD analysis of GPM

After strength test, the GPM specimens were further ground in a crusher, and passed through 75 μm sieve. The powder sample, thus obtained, was used for investigating the microstructure of GPM through X-ray diffraction (XRD) analysis. The analysis was carried out in X-ray diffractometer (Rigaku SmartLab 9 kW model) with CuKα radiation (λ = 1.5405 Å). The GPM powder sample was scanned from 5° 2θ to 65° 2θ at a step size of 0.03° 2θ.

Results and Discussion

4.1 General

In this chapter, the flowability of control and salt admixed GPM mixes, compressive strength, and XRD analysis of control GPM mixes made with different replacement levels of ground granulated blast furnace slag (BFS) and fly ash, and different molarity of NaOH solution are presented and discussed.

4.2 Flow index (%) of geopolymer mortar

The flow index (%) of control and salt admixed geopolymer mortar made with different proportions of ground granulated blast furnace slag (BFS) and fly ash (FA), and different concentrations of NaOH solution are depicted in Fig. 4.1. From this figure, it is inferred that the flow index increased with increase in fly ash content regardless of molarity of NaOH solution and type of admixed salt. This is due to the increase in the amount of spherical fly ash particles that resulted in higher flowability of GPM mixes [26]. Further, it was observed that the flow index mostly reduced with increase in concentration of NaOH solution regardless of fly ash content and type of admixed salt (Fig. 4.1). This is due to the greater amount of solids present in the GPM mixes made with 10 M NaOH solution than that made with 6 M NaOH solution.

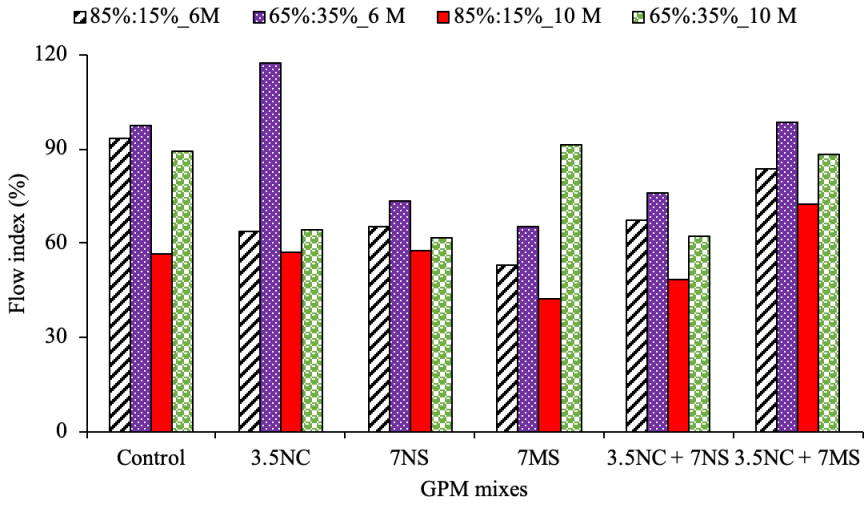

Fig. 4.1 Flow index (%) of control and salt admixed geopolymer mortar mixes

From Fig. 4.1, it was observed that the GPM mixes admixed with Na_2SO_4 mostly showed higher flow index as compared to the mixes admixed with $MgSO_4$. However, in case of GPM mixes admixed with combined chloride and sulphate salts, it was observed that the GPM mixes admixed with NaCl plus $MgSO_4$ had higher flow index than the mixes admixed with NaCl plus Na_2SO_4 regardless of fly ash content and molarity of NaOH solution (Fig. 4.1). Further, between chloride, and composite chloride plus sulphate salt admixed mixes, the flow index was mostly higher in case of NaCl admixed GPM mixes when compared with NaCl plus Na_2SO_4 admixed GPM mixes. However, mostly opposite variation in flow index was observed between NaCl, and NaCl plus $MgSO_4$ admixed GPM mixes. Further, the flowability as indicated by flow index was mostly higher in case of composite chloride plus sulphate salt admixed mixes GPM mixes as compared to GPM mixes admixed with only sulphate salt irrespective of cation type associated with sulphate ions (Fig. 4.1). The variation in flow index was mostly unsystematic between control and salt admixed GPM mixes as observed from Fig. 4.1. This could be due to the effect of presence of chloride and sulphate ions that altered the particle mobility in salt admixed GPM mixes.

4.3 Compressive strength of GPM

The compressive strength of control GPM mixes at the age of 7 days made with different proportions of BFS and fly ash, and different molarity of NaOH solution is shown in Fig. 4.2. From this figure, it was observed that the GPM mixes made with higher BFS content showed higher compressive strength than the mixes made with lower BFS content regardless of molarity of NaOH solution. This is attributed to the increase in calcium content present in BFS that led to formation of calcium based geopolymer gels [27], thereby resulting in formation of denser microstructure and higher compressive strength. From Fig. 4.2, the compressive strength of GPM mixes increased with increase in molarity of NaOH solution. This is due to the effect of greater leaching of silica and alumina from the precursor materials at higher molarity of NaOH solution that led to formation of denser geopolymer matrix, which increased the compressive strength of GPM mixes [28].

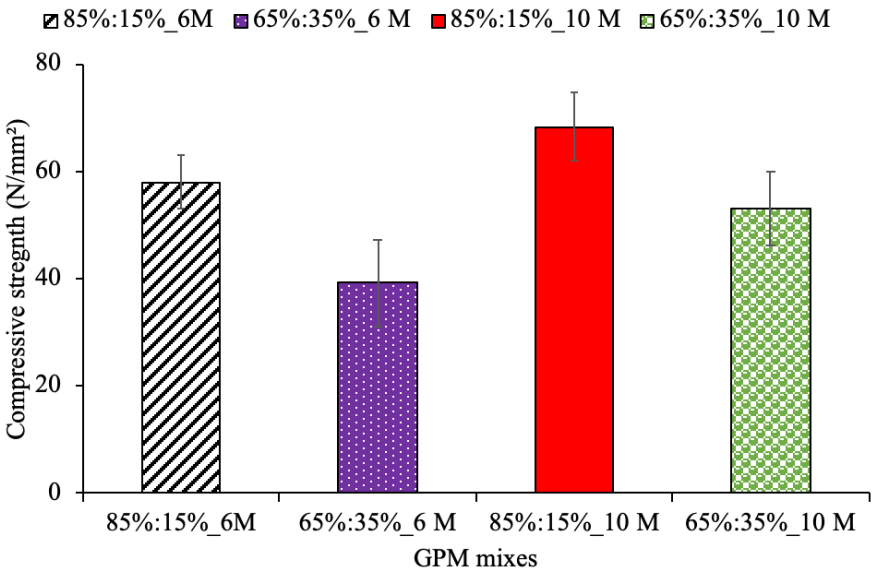

Fig. 4.2 Compressive strength of control GPM mixes at the age of 7 days

4.4 XRD analysis

The XRD spectra of GPM mixes made with different BFS and fly ash proportions, and concentrations of NaOH solution at the age of 7 days are shown in Fig. 4.3. From this figure, the crystalline phases of quartz and mullite are identified in all the XRD spectra, which indicates the presence of partially reacted fly ash particles [29]. In addition, the semi crystalline peaks corresponding to hydrotalcite at 10.5º 2θ, nepheline at 27.1º 2θ, anorthoclase at 27.5º 2θ, albite at 28.02º 2θ, calcite along with calcium silicate hydrate (C-S-H) gel at 29.5º 2θ, and aragonite at 45.8º 2θ were identified in all GPM mixes.

The peak intensity of albite varied unsystematically with BFS content. However, the peak intensity related to nepheline, anorthoclase, and C-S-H gel increased with increase in BFS content regardless of molarity of NaOH solution (Fig. 4.3). This is attributed to increase in calcium bearing compounds present in BFS that enhanced the geopolymerization reaction. As a result, the compressive strength of GPM mixes increased with increase in BFS content (Fig. 4.2). From Fig. 4.3, it was observed that there was unsystematic variation in the peak intensity of albite and C-S-H gel with molarity of NaOH solution. However, the peak intensity of nepheline and anorthoclase increased with increase in molarity of NaOH solution irrespective of BFS content. This is due to the effect of more leaching of silica and alumina species from the precursor materials in the presence of 10 M NaOH solution that enhanced the reaction between these species and alkaline solution, which resulted in greater formation of nepheline [30], and anorthoclase. Accordingly, the formation of higher amount of nepheline and

anorthoclase had dominant effect in the strength development of GPM mixes with increase in molarity of NaOH solution (Fig. 4.2).

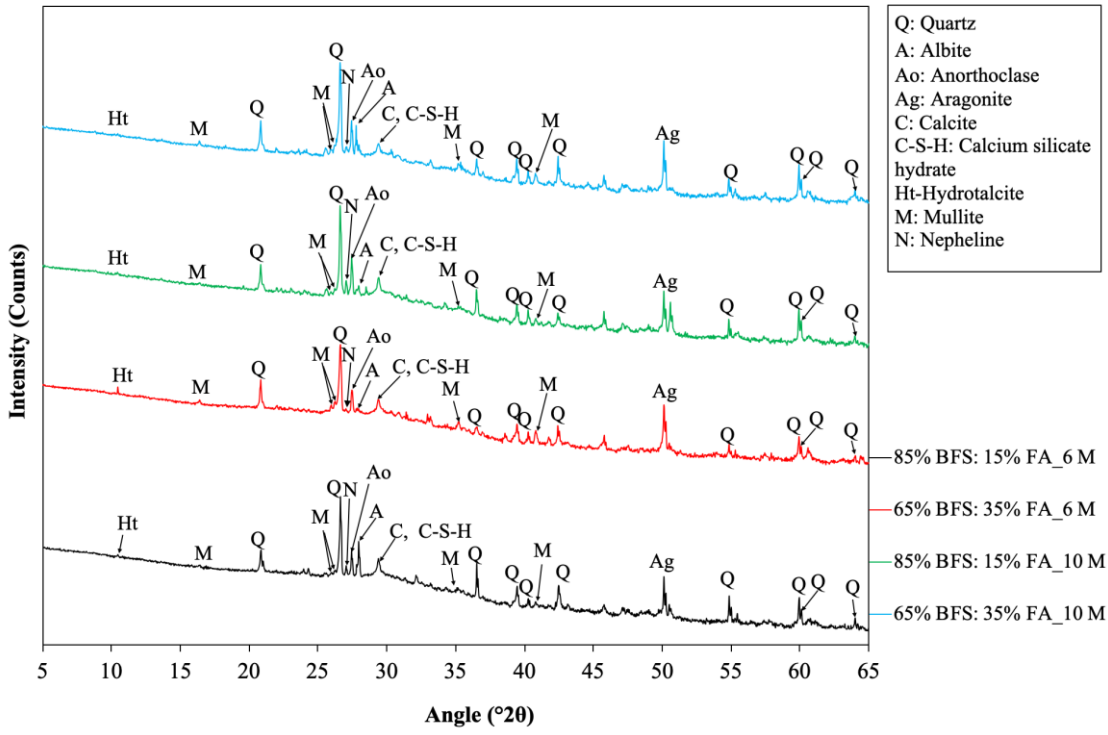

Fig. 4.3 XRD spectra of control GPM mixes at the age of 7 days

Chapter-5

Conclusions and Future work

5.1 General

In this chapter, the conclusions obtained from the present work, and the work to be done in the next phase are presented.

5.2 Conclusions

The conclusions obtained from the present work are as follows:

- The flowability of GPM increased with fly ash content and decreased with concentration of NaOH solution.
- The admixed salts altered the workability of GPM mixes, and the variation was found to be unsystematic with control GPM mix.
- The increase in BFS content and molarity of NaOH solution resulted in higher compressive strength of GPM mixes at the early age of 7 days.
- The XRD analysis showed that increase in BFS content led to increase in peak intensity of nepheline, anorthoclase, and C-S-H gel. Further, the peak intensity of nepheline and anorthoclase increased with molarity of NaOH solution.

5.3 Work to be done in the next phase

- The compressive strength test will be carried out at the age of 28 and 90 days on control and salt admixed GPM cube specimens.
- The mortar powder will be obtained from GPM specimens. This powder will be used for measuring the chloride ion, and sulphate ion concentrations of GPM mixes.
- The microstructural investigation will be conducted on control and salt admixed GPM powder samples at the age of 28 and 90 days.

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