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Effect of GGBS Addition on Reactivity and Microstructure Properties of Ambient Cured Fly Ash Based Geopolymer Concrete



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Abstract

Geopolymer concrete is an eco-friendly alternate to conventional concrete that considerably lower green house gases emitting into the atmosphere. Fly ash based geopolymer concrete is reported to become hardened during heat curing process which comes as a major constraint for cast in in-situ applications. In this study, the aluminosilicate materials such as Ground Granulated Blast Furnace Slag (GGBS) with varying percentages such as 0%, 10%, 20%, and 30% replaces the fly ash (FA) in geopolymer concrete was used. Manufactured sand (M-sand) is used as full replacement material for natural sand as fine aggregate owing to its increasing demand. This work aims at investigating the effect of alumino silicate materials on strength properties, characterization and micro structural analysis using Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray spectroscopy (EDX), Fourier Transform Infrared spectroscopy (FTIR) and X-ray diffraction (XRD) in geopolymer concrete under ambient curing condition. The SEM and EDX results reveals that, the micro structural properties of fly ash, GGBS materials, CaO, Si/Al ratio, and gel formation have a significant effect on compressive strength and setting time of geopolymer concrete. The FTIR analysis reveals that the stretching vibration of fly ash shifts to low wave number values due to changes in geopolymerization. The X-ray diffraction (XRD) reports show that the C-S-H gel formed around 27–30° 2theta value due to increase of GGBS in geopolymer concrete.

Keywords Fly ash · GGBS · Geopolymer · Compressive strength · Microstructure

1 Introduction

In construction industry, portland cement has become the foremost choice of binders in concrete. The huge production of portland cement annually exhausts 10–11 EJ which is $\sim 2-3\%$ of primary energy consumption globally [1]. Additionally, manufacturing of portland cement generates approximately one tonne of carbon dioxide for every tonne of cement produced [2] which contributes to 7% of CO₂ emissions in global warming [3]. Hence, most of the countries are consigning to reduce the green house gases to decrease their environmentally detrimental impact. In this context, exploring binder materials with low CO₂

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content or low energy materials, finding reuse possibilities for the byproduct materials from other industries gain attention. Already, a variety of byproduct materials such as fly-ash, slag and silica fume from coal, iron, and ferrosilicon production are used as an additional material for portland cement, usually on the order of 10–50% (and even in greater quantities sometimes). This attracts further development in obtaining binders made completely or predominantly from waste materials [2].

The new technology of alkali activated binders (clinker-free) including geopolymers which are formed by the reaction between aluminosilicate binder and alkali-activator solutions, help dissolution and polycondensation of raw materials to produce a hardened material [4, 5]. Geopolymer concrete is mainly produced by aluminosilicate materials like Fly ash, Metakaoline, Silica fume and GGBS reacts with alkaline activator solution such as sodium or potassium based [6]. Geopolymer cement emits six times lesser CO₂ than portland cement, which emits about 0.18 t of CO₂ in geopolymer cement as against 1 t of CO₂ in cement [7]. During geopolymerization process, the aluminosilicate materials are suspended into alkali activator solution to produce SiO₄ and AlO₄ tetrahedra (sialate network) linked

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Table 1	Fly ash and GGBS
Chemica	l compositions

Composition (%)	SiO ₂	CaO	MgO	Al ₂ O ₃	Na ₂ O	K ₂ O	Fe ₂ O ₃	SO_4	LOI ^a
Fly ash	63.32	2.49	0.29	26.76	0.0004	0.0002	5.55	0.36	0.97
GGBFS	35.05	34.64	6.34	12.5	0.9	0.6	0.3	0.38	0.26

alternately by all the oxygens [8]. The alkali metal cations provides the charge balancing cations like Na, K, Ca to these SiO_4 and AlO_4 tetrahedrons and provides polymeric precursors (– SiO_4 – AlO_4 – SiO_4 – SiO_4 – SiO_4 – AlO_4 –, or $-SiO_4$ – AlO_4 –, or $-SiO_4$ – AlO_4 –, SiO_4 – AlO_4 –) by sharing all oxygen atoms between two tetrahedral units and producing geopolymer [8, 9].

The byproduct material of fly ash is mainly produced from thermal power plants during combustion of pulverized coal which contains SiO₂ and Al₂O₃ along with the components of CaO, MgO, Fe₂O₃ etc. In geopolymer synthesis, fly ash has developed as a material of interest due to its availability, low water demand, high workability and alumino-silicate composition [10, 11]. The fly ash geopolymer concrete gains strength slowly when it is in ambient temperature around 25 °C [12]. To achieve reasonable strength in fly ash geopolymer concrete, the required curing temperature is 40–75 °C [13]. The fly ash geopolymer concrete shows good mechanical properties and enhanced durability [[10]; Fernandez and [14]]. However, the main limitation for using fly ash based geopolymers, are slow in setting and strength development due to its slow reactivity [15]. To resolve the issue of low reactivity of fly ash and to improve the strength development, two different ways are suggested. One is addition of GGBS and the other one is mechanical processing of fly ash [16]. The effect of mechanical processing on its geopolymerization and reactivity has been reported [16–19]. While some researchers have used additives such as GGBS, flue gas desulfurization gypsum, and portland cement [12, 20, 21].

The byproduct material of GGBS is mainly produced from iron making plant. GGBS is a granular material having CaO, MgO, SiO₂ and Al₂O₃. Calcium Silicate Hydrate (C-S-H) gel is the main reaction product during the activation of GGBS [22], which results in achievement of sufficient strength, also in ambient curing condition [23, 24]. The coupled materials of fly ash and GGBS are very efficient to give strength and stability since the alumina silicate materials undertake dissolution, polymerization with alkali, condensation, and solidification [25]. Now a days, M-sand is used for making concrete due to the lack of natural river sand and due to its quality controlled process. Also, full replacement of natural sand by M-sand didn't show any adverse effect on the compressive strength of concrete [26, 27]. Nevertheless, only few studies are available on using GGBS in fly ash based geopolymer concrete and the effect on strength properties and micro structural observations.

The mechanical properties by using the byproduct materials such as fly ash and GGBS as a replacement material for cement and M-sand as a replacement material for river sand in geopolymer concrete have been validated by developing a model in Levenberg–Marquardt algorithm using artificial neural network [28]. In this work, the mechanical property (compressive strength) of G30 grade geopolymer concrete have been determined using fly ash, GGBS, M-sand with 8 M concentration of sodium hydroxide and observe the microstructure of geopolymer concrete using Scanning Electron Microscopy (SEM) with Energy-Dispersive X-ray spectroscopy (EDX), Fourier Transform Infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analysis have been carried out under ambient curing condition.

2 Materials, Characterization and Techniques Used

2.1 Materials

For making the geopolymer concrete, main source of aluminosilicate material of class F fly ash was used which was acquired from North Chennai thermal power plant and GGBS, used as an additive [25] material was acquired from Astra chemicals, Chennai, Tamilnadu. The specific gravity of fly ash and GGBS are reported as 2.13 and 2.85, respectively. The fly ash and GGBS chemical composition obtained from X-Ray fluorescence spectroscopy are given in Table 1. The alkali activator solutions of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) solutions in the ratio of 2.5 were chosen for this work [29]. Ratio of SiO₂/ Na₂O by mass of 2.0 sodium silicate solution and 8 M concentration of sodium hydroxide solution was used. Combined crushed granite coarse aggregate were used with maximum sizes of 8, 12 and 20 mm. M-sand was collected from KMC blue metals, Theni to be used as fine aggregate. The constituent of M-sand is given in Table 2. The aggregates were used in saturated surface dry (SSD) condition [30]. To achieve the workability of geopolymer concrete, super plasticizer (naphthalene based) was used.

Та	ble	2	Constituents	of	M	-sand	
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Constituents (%)	CaO	SiO_2	MgO	SO_4	Cl	Al_2O_3	Fe ₂ O ₃	Na ₂ O	K ₂ O	РН
M-Sand	6	63.86	0.7	0.07	0.07	22.93	4.25	0.0001	Nil	8.74



Fig. 1 SEM analysis of i) Fly ash ii) GGBS iii) M-Sand



Fig. 2 (i). FT-IR spectra of Fly ash, (ii). FT-IR spectra of GGBS

800

Q 700 O- Quartz M - Mullite 600 500 Intensity (a. u.) 400 300 Q 200 100 0 20 30 **4**0 50 60 70 80 10 20 (Deg)

(i). XRD pattern of fly ash

Fig. 3 (i) XRD pattern of fly ash, (ii).XRD pattern of GGBS

2.2 Characterization of Materials

2.2.1 Scanning Electron Microscopy (SEM) with Energy-Dispersive X-Ray Spectroscopy

(EDX) Analysis Scanning Electron Microscopy (SEM) was used to find surface morphology which was conducted using EVO 18 research microscope, LaB_6 filaments electron source. The resolution used to take this image was 8 kV. The samples were evaluated in system vacuum technique. For observing the microstructure, SEM analysis was done on fly ash and GGBS as shown in Figs. 1(i) and 1(ii). The SEM analysis of M-sand as shown in Fig. 1(iii).

The Scanning Electron Microscope was equipped with Energy-Dispersive X-ray spectrometer used to characterize the micro structure of geopolymer concrete. The results showed that fly ash and GGBS having high amount of silica and alumina. Fly ash particles viewed as spherical, GGBS observed as Granular and M-sand as angular in shape.

2.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a method of chemical analysis to search Si-O & Al-O reaction zones and to find the degree of geopolymerization and



structure of reaction products in geopolymer concrete [31]. An Attenuated total reflection (ATR) accessory technique was used in FTIR analysis. Absorbance spectra collected from 4000 cm⁻¹ to 400 cm⁻¹ at a resolution of 2 cm⁻¹. The infrared spectroscopic results of fly ash and GGBS are shown in Fig. 2(i), (ii).

The IR spectrum of fly ash shows transmission bands at 3852, 3750, 3735, 3689, 3675, 3648, 1558, 1540, 1521, 1507, 1090 cm⁻¹. The main peaks are at 1540, 1507 and 1090 cm⁻¹. The IR spectrum of GGBS shows the main peak is at 920 cm⁻¹. Si-O-Si or Al-O-Si was observed at 1090 cm⁻¹ for FA and 920 cm⁻¹ for GGBS. The wave number of the band in the raw material of GGBS is decreased compared with FA. As calcium content increases in the raw materials, the band wave number match to low degrees of cross linking of the amorphous phase of raw materials is reduced [32].

2.2.3 X-Ray Diffraction (XRD) Analysis

A mineralogical analysis was performed by XRD used to explain the mechanical performances of materials. XRD data were obtained using a Bragg- Brentano geometry powder diffractometer with the parameters of 30 mA, 40KV and CuK α radiation. Scanning rate of XRD is one degree per minute from 10 to 90 degrees (2 θ) and steps of 0.05 degrees (2 θ).

Table 3 Mix Proportions (kg/m^3)

Mix Id	Fly ash	GGBS	OPC	M- Sand	Natural Sand	Coarse Aggregate	Alkali solution	Water
F100M100	380	0	-	660	0	1189	171	_
F90M100	342	38	_	660	0	1189	171	_
F80M100	304	76	_	660	0	1189	171	_
F70M100	266	114	_	660	0	1189	171	_
CR100	-	—	380	0	660	1189	_	171



Fig. 4 Geopolymer concrete in fresh mix condition

Wave length selected for XRD was 0.154 nm (Cu). X-ray diffraction (XRD) patterns of fly ash and GGBS are shown in Fig. 3(i) and 3(ii).

The XRD pattern of raw fly ash illustrates with single letter for easier representation. The crystalline phases namely Qquartz (SiO₂; JCPDS File card # 00–046-1045) and Mmullite (Al₆Si₂O₁₃; JCPDS File card # 00–015-0776) were determined in the diffractogram. Crystalline band of fly ash are attributed to the peaks at a 2 θ about 26°. The XRD pattern of GGBS is more amorphous and illustrates broad spectrum diffuse band in the range of 2 θ about 20–40° which has better reactivity when compared with crystalline phases of fly ash which contains crystalline phases of silica and alumina.

2.3 Techniques Used

Fly ash based geopolymer concrete samples with partial replacement of GGBS (0%, 10%, 20% & 30%) were cast with the ratio of alkali solution to total alkali binders as 0.45. The alkali solution prepared by mixing of NaOH and Na₂SiO₃

Fig. 5 Compressive strength Vs percentage variation of GGBS

emits heat in large quantities. Hence, the solution was mixed 24 h before making the concrete specimen [22]. Some studies showed that the solutions were directly mixed to the dry mixture of other materials [33]. NaOH solution is prepared 24 h before the casting of concrete, to avoid the extra heat in geopolymer. Before adding the alkalis, the Saturated Surface Dry (SSD) aggregates were mixed with the binders in the pan mixture for 5 min. To maintain the workability of concrete, 1% of super plasticizer was added to the total binder. The mix proportion has been adopted based on IS 10262 for attaining M30 grade of concrete. The same mix proportion has been taken for G30 geopolymer concrete since no mix design is available for geopolymer concrete [34]. The mix proportions are given in Table 3.

Geopolymer concrete was designated as FxMy, where F indicates fly ash, x indicates the percentage of fly ash used when replaced by GGBS, M indicates M-sand and y indicates replacing percentage of natural sand by M-sand. For example, F80M100 indicates that 20% GGBS replaced the fly ash and 100% river sand was fully replaced by M-sand. Geopolymer concrete was prepared and cast in moulds of size 150x150x150mm. To find the effect of GGBS and M-sand on compressive strength, the specimens were tested after 28 days under ambient curing conditions. For making ordinary portland cement concrete, cement was used as binder and river sand was used as fine aggregate for comparison purpose. CR100 indicates cement with natural sand. The fresh mix condition of geopolymer concrete is shown in Fig. 4.

After finding the compressive strength of geopolymer concrete mixtures, the micro structure of concrete specimens were examined by microscopic analysis.

3 Results and Discussions

3.1 Compressive Strength

Four geopolymer concrete and one conventional concrete cube specimens were tested to find the compressive strength of concretes and the test results are indicated in Fig. 5. The



Table 4Formulations ofgeopolymer concrete in terms ofSiO2/Al2O3 ratio and CaO

Mixture	Fly ash (wt%)	GGBS (wt%)	SiO ₂ /Al ₂ O ₃ ratio	CaO (wt%)
F100M100	100	0	2.36	2.49
F90M100	90	10	2.38	5.71
F80M100	80	20	2.41	8.92
F70M100	70	30	2.43	12.14

average of three specimens is considered for the compressive strength test result.

Mix numbers 1(F100M100), 2(F90M100), 3(F80M100), and 4(F70M100) were used to analyze the impact of increasing GGBS percentages with fly ash such as 0%, 10%,20%, and 30% in geopolymer concrete. It was noted from Fig. 5, that the compressive strength of geopolymer concrete increases about 1.18 times, 1.33 times, and 1.44 times while increasing the percentage of GGBS in the mixes of 2(F90M100) as 10%, 3(F80M100) as 20%, and 4(F70M100) respectively compared to the fly ash alone mix of 1(F100M100). The dissolution of fly ash is not completed in ambient curing. Also the setting time of mix 1(F100M100) increases when comparing with the other mixes of 2(F90M100), 3(F80M100), 4(F70M100) due to low reactivity of fly ash [35]. While comparing the Ordinary Portland Cement (OPC) concrete with geopolymer concrete, the G30 concrete could be achieved in the mixes of 3(F80M100) and 4(F70 M100) i.e. 20% and 30% replacement of fly ash with GGBS. Formulations in terms of SiO₂/Al₂O₃ ratio and CaO for geopolymer concrete are given in Table 4.

The compressive strength and initial setting time of geopolymer concrete increases with increase in replacement percentage of GGBS. This is due to an increase in the Si/Al ratios in source materials. As –Si-O-Si bonds are stronger than Al-O-Al and –Si-O-Al bond, increasing Si/Al ratio increases the number of –Si-O-Si bonds thereby achieving elevated compressive strength [36]. The fly ash/GGBS based geopolymer concrete speed up the initial setting time due to high calcium in GGBS [37].

The compressive strength of 20% replacement of GGBS and full replacement of natural sand with M-sand showed equal strength with the conventional concrete. Microstructural observations have been conducted to justify the better mix proportions.

3.2 Scanning Electron Microscopy (SEM) with Energy-Dispersive X-Ray Spectroscopy

(EDX) Analysis of Geopolymer Concrete The SEM with EDX of the geopolymer concrete samples is shown in Figs. 6, 7, 8 and 9. These are the micro structural images of the samples F100M100, F90M100, F80M100, and F70M100 of geopolymer concrete at age of 28 days. The geopolymer concrete having 30% replacement of GGBS (F70M100) is more compact, has less micro cracks and is less porous than other mixes of such as F100M100, F90M100 and F80M100 of geopolymer concrete. Higher portion of GGBS geopolymer concrete shows some non-reacted or partly reacted GGBS particles when compared with other mixes. The fly ash alone mix F100M100 geopolymer concrete produces geopolymer gel primarily sodium alumino silicate hydrate. Sodium alumino silicate hydrate (N-A-S-H) is the prime reaction product of the geopolymer gel which is produced by the low calcium fly ash [38]. While increasing GGBS, calcium alumino silicate hydrate (C-A-S-H) is reaction product when calcium compound rises in geopolymer concrete [39]. By adding the fine particles as an additive in geopolymer concrete, the density and homogeneity could be improved. The elemental



Fig. 6 SEM & EDX analysis of mix 1 F100M100



Fig. 7 SEM & EDX analysis of mix 2 F90M100



Fig. 8 SEM & EDX analysis of mix 3 F80M100

percentage by EDX analysis (Atomic %) is given in Table 5. The 3D networked polysialate- siloxo, and polysialatedisiloxo polymers are created by higher Si/Al i.e. Si/Al > 2.5 [40]. Silicon, oxygen and aluminium along with low concentrations of calcium and sodium are qualified to the formation of 3D network (Ca,K)-polysialate-siloxo [41].

X-Unreacted or partially reacted GGBS particles.

Y-Unreacted or partially reacted fly ash particles. Z-Geopolymer gel.



Fig. 9 SEM & EDX analysis of mix 4 F70M100

Table 5 Elements percentage by EDX analysis (Atomic %)										
Element	O K	Si K	Na K	Ca K	Al K	Si/ Al				
F100M100	65.80	20.46	5.75	0.63	5.81	3.52				
F90M100	65.49	16.91	5.65	1.57	4.62	3.66				
F80M100	63.06	14.33	4.69	2.87	3.85	3.72				
F70M100	55.86	14.13	3.54	4.78	3.68	3.83				

3.3 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Figure 10 shows the FTIR results of 28 days for replacing the fly ash with GGBS as 0%, 10%, 20% & 30%. The stretching vibration of fly ash occurred at 1062 cm^{-1} which is shift towards low wave number like 1011 cm^{-1} , 995 cm^{-1} , 986 cm^{-1} and 979 cm^{-1} for replacing percentage of 0%, 10%, 20% and 30% respectively in geopolymer concrete. The shift is approximately 51 cm^{-1} , 67 cm^{-1} , 76 cm^{-1} , 83 cm^{-1} . From the results, it reveals that due to increase amount of GGBS in the geopolymer concrete forming the C-S-H gel with N-A-S-H type gel with a reduction of Al. This leads to changes in geopolymerization and improve the strength properties.

Large bands around $3648-3852 \text{ cm}^{-1}$ are H-O-H stretching vibrations. Bands like $1507-1540 \text{ cm}^{-1}$ are -OH group of bending vibration of products of hydrated reaction of water. In geopolymer paste, these bands shows the reaction of alkaline activation products and water [42].

3.4 X-Ray Diffraction (XRD) Analysis

The XRD pattern of geopolymer concrete with percentages of GGBS is shown in Fig. 11. It reveals that there was a change from the raw materials chemistry since the reaction of alumino silicate materials with alkali activator solutions. The peaks around 27-30° corresponding to C-S-H gel are present found in GGBS increasing geopolymer concrete and also the intensity of crystalline phases decreases when increasing GGBS percentage. A-S-H and C-S-H gel are formed around 30° and 50° in all GGBS samples and crystalline phases decreases [43]. During first step of geopolymerization the amorphous compounds dissolve easier than crystalline compounds (i.e dissolution of species) which yield higher amounts of SiO2 and Al2O3 to combine geopolymerization reaction product. Due to this, the results give high degree of geopolymerization and higher mechanical strength when the replacement percentage of GGBS is higher in geopolymer concrete. Calcium aluminosilicate structure more reactive than siliceous structure.

4 Conclusion

The present study examined the compressive strength and micro structural studies of G30 grade geopolymer concrete at ambient curing conditions. The following conclusions are drawn from the results.

• G30 grade of geopolymer concrete is achieved by the combination of 80% fly ash and 20% GGBS alumino



Fig. 10 FT-IR spectra of geopolymer concrete



Fig. 11 XRD pattern of geopolymer concrete

silicate source material with the ratio of Na_2SiO_3 to NaOH as 2.5.

- When increasing GGBS percentage in fly ash based geopolymer concrete as 10%, 20%, and 30%, the compressive strength has been increased as 17%, 31%, and 41% respectively when compared with fly ash alone in geopolymer concrete.
- Geopolymer concrete is a replacement concrete for conventional concrete, since the compressive strength of GGBS-Fly ash based geopolymer concrete is mostly equal with the conventional concrete.
- SEM / EDX analysis showed a less dense structure with low content of GGBS or without GGBS while the compactness of geopolymer concrete increases when the content of GGBS increases.
- FTIR results showed the changes in geopolymerization and formation of C-S-H gel when increasing the percentage of GGBS, which in turn increases the strength properties.
- XRD analysis revealed that the intensity of crystalline phases decreased when increasing the percentage of GGBS and forming the C-S-H gel which has better reactivity when compared with fly ash.

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