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RESEARCH ARTICLE - CIVIL ENGINEERING

Final Setting Time and Compressive Strength of Fly Ash and GGBS-Based Geopolymer Paste and Mortar

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Abstract Geopolymer binders are attracting the attention of researchers as substitution to cement binder in conventional concrete. In manufacturing 1 ton of cement, 1 ton of CO_2 is released into the atmosphere. Thus, replacement of cement by geopolymer material in construction industry reduces pollution by two ways: reduction in carbon dioxide emission into atmosphere by reducing the consumption of cement and utilization of fly ash, which is another waste product piling in huge quantities in thermal power plants. To examine the use of geopolymer as a replacement to cement, it is essential to investigate normal consistency, final setting time and compressive strength of geopolymer which are routine tests generally conducted for cement. The procedure adopted for determining the normal consistency, final setting time and compressive strength of geopolymer is same as the procedure adopted for cement. In these tests, cement is replaced by geopolymer material and water is replaced by alkaline activator solution. The parameters considered in this investigation are geopolymer source material (fly ash and GGBS) and alkaline activator consisting of sodium meta silicate and sodium hydroxide of different molarities (8, 12, 16 M). The ratio of sodium meta silicate to sodium hydroxide considered in this study is 2.5. The test results indicated that combination of fly ash and GGBS results in decreased final setting time and increased compressive strength. It was also observed that increase in sodium hydroxide increases compressive strength of geopolymer mortar.

Keywords Geopolymer paste · Final setting time · Compressive strength · Normal consistency · Alkaline solution

1 Introduction

Concrete usage is second to water in this present-day world. Ordinary Portland cement (OPC) is conventionally used as the primary binder to produce concrete due to its availability of the raw materials over the world and ease of mold ability. The application of concrete in the realms of infrastructure, habitation and transportation has greatly promoted the development of civilization, economic progress, stability and quality of life. Nowadays with the advent of high-performance concrete (HPC), the durability and strength of concrete have been improved largely. The environmental issues associated with the production of OPC are well known. The amount of the carbon dioxide released during the manufacture of OPC due to the calcination of limestone and combustion of fossil fuel is in the order of 1 ton for every ton of OPC produced. In addition, the extent of energy required to produce OPC is only next to steel and aluminum. On the other hand, the abundant availability of fly ash worldwide creates opportunity to utilize this by-product of burning coal, as a partial substitute for OPC to manufacture cement concrete products.

Geopolymer has the potential to replace OPC in the construction sector. Geopolymer is used as the binder to completely replace OPC in producing geopolymer concrete. In order to produce geopolymer, low-calcium fly ash, a source material, needs to be activated by an alkaline solution to produce polymeric Si–O–Al bonds. The alkaline solution is the combination of sodium hydroxide and sodium meta silicate. Geopolymer concrete has the potential

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to reduce greenhouse emissions from the concrete industry by 80 % [1,2]. Geopolymer concrete is also known as alkali-activated concrete or inorganic polymer concrete. It is reported that fly ash-based geopolymer concrete possesses excellent physical properties such as high early strength, low shrinkage, high resistance to freezing and thawing, sulfate attack and corrosion [3]. A study which was carried out to investigate the effect of composition of source materials and curing process on the chemical and physical properties of geopolymer revealed that curing at high temperatures resulted in cracking and can impose negative effects on the physical properties of geopolymer [4]. Another study using alkali-activated ground-granulated blast-furnace slag concrete incorporated with sodium silicate as an activator gave compressive strength up to 50 MPa [5]. Fly ash-based geopolymer mortar cured at ambient temperature with addition of Cao and $Ca(OH)_2$ as calcium compounds had shown improved mechanical properties [6]. The term 'geopolymer' was coined in 1970s by the French scientist and engineer Prof. Joseph [7], and it was applied to a class of solid materials synthesized by the reaction of an aluminosilicate powder with an alkaline solution. A demonstration was given on the synthesis of construction materials by alkaline activation of solid, non-Portland cement precursors (usually high-calcium metallurgical slags) [8]. In particular, coal fly ash with low calcium content (<5 wt% CaO) is an abundant industrial by-product which is currently underutilized worldwide [9]. It is also well known that the reactions of slag are dominated by small particles. Particles above 20 microns in size react only slowly, while particles below 2 microns react completely within approximately 24 h in blended cements and in alkali-activated systems [10]. Clearly, when using slag in geopolymerization, careful control of particle size distribution can be utilized to control the strength development profile, as is done in OPC blends [11].

This paper presents the results of research that deal with study of normal consistency, final setting time and compressive strength of geopolymer mortar. The calcined source material class F fly ash (commonly used) is partially replaced with ground-granulated blast-furnace slag, and the mix is activated with alkaline solution of sodium hydroxide and sodium meta silicate. Molarity (concentration) of sodium hydroxide is varied as 8, 12 and 16. The ratio of sodium meta silicate to sodium hydroxide is maintained as 2.5.

2 Materials

Materials used in this research are GGBS obtained from Andhra Cements, Vishakhapatnam, India, and fly ash from Ramagundam Thermal Power Plant, India, with a specific gravity of 2.90 and 2.17, respectively. The chemical compositions of fly ash and GGBS are presented in Table 1. Fine aggregate used is clean dry river sand. It is sieved using 2.36 mm sieve to remove all the pebbles. Saturated surface dry specific gravity of sand is 2.67 which confirms to zone II as per IS: 383 [12].

Table 1 Chemical composition of fly ash and GGBS (% by mass)

Chemical composition	Fly ash	GGBS
SiO_2	60.11	34.06
Al_2O_3	26.53	20
Fe_2O_3	4.25	0.8
SO_3	0.35	0.9
CaO	4.00	32.6
MgO	1.25	7.89
Na_2O	0.22	NIL
LOI	0.88	NIL

2.1 Alkaline Solution

The alkaline activator is a combination of sodium hydroxide and sodium meta silicate solutions. Sodium hydroxide solution is used as alkaline activator because it is widely available and is less expensive than potassium hydroxide solution. Sodium hydroxide of 98 % purity is in flakes and pellets form. These pellets are dissolved in distilled water to obtain sodium hydroxide solution of required molarity. The mass ratio of SiO_2 to Na_2O of the sodium silicate solution is 2.61 ($SiO_2 = 30.0\,\%$, $Na_2O = 11.5\,\%$ and water $= 58.5\,\%$). Sodium hydroxide solution of required molarity and sodium meta silicate in liquid form are mixed and stored at room temperature of $25 \pm 2\,^{\circ}C$ and relative humidity of 65 % for 24 h before its use.

2.2 Paste

The source materials, i.e., fly ash and GGBS, with different proportions by weight are mixed in dry condition in a pan mixer. The mixture is activated by adding alkaline solution and mixed for 3 min to ensure homogeneity by uniform color. A series of geopolymer pastes are prepared by varying the proportions of calcined source material (fly ash and GGBS) as well as different concentrations of alkaline activator (molarities of sodium hydroxide).

2.3 Fly Ash and GGBS

Scanning electron microscopy is performed to ascertain the microstructure properties of the binder material fly ash and GGBS. The scanned images of fly ash and GGBS are presented in Figs. 1 and 3. Scanning electron microscopy image gives an approximate idea about the shape, angularity, size

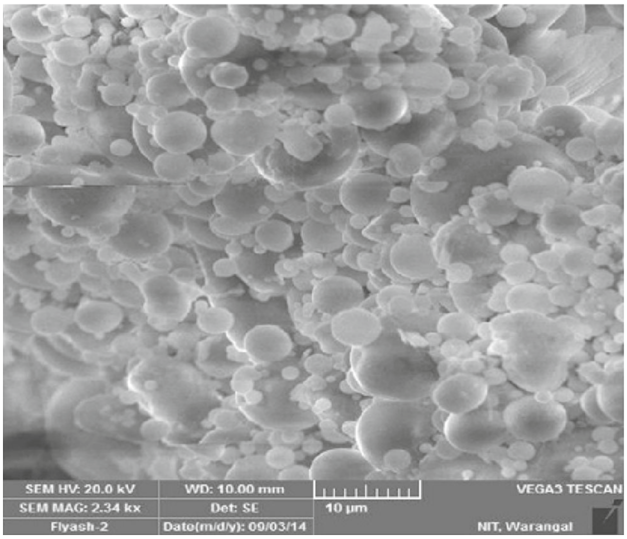

Fig. 1 SEM image of fly ash

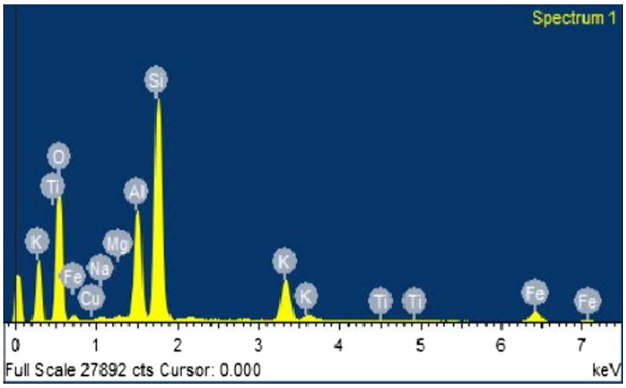

Fig. 2 EDXA of fly ash

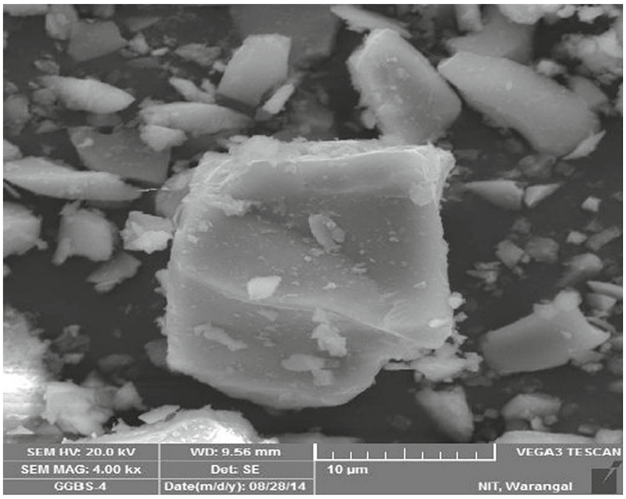

Fig. 3 SEM image of GGBS

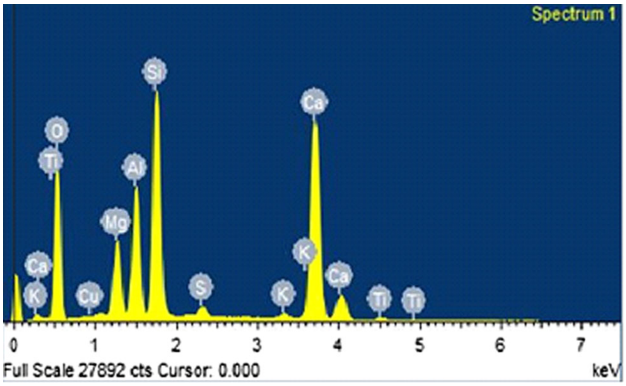

Fig. 4 EDXA of GGBS

and surface texture of the fly ash and GGBS particles used in the study. The GGBS particles appear to have straight, flaky-elongated shape with sharp-edged angularity, rough surface texture and large variation in size (1–10 microns). The fly ash particles appear to have spherical shape (i.e., curved, cubic shape with rounded angularity) with smooth surface in a broad range of sizes (with the lower limit being approximately 1/10 of the maximum fly ash particle size, i.e., 1–10 microns in size). The energy-dispersive X-ray spectroscopy of fly ash and GGBS is shown in Figs. 2 and 4. From the energy-dispersive X-ray spectroscopy, it is observed that GGBS is enriched with more silica percent than other elements. Fly ash particles contain more percent of silica and alumina.

2.4 Geopolymer Mortar

The ratio of calcined source material (binder) to fine aggregate is fixed as 1:1, and the alkaline liquid to binder ratios are fixed as 0.45. The required quantities of source material, fine aggregate and alkaline liquids are determined. The molarity of sodium hydroxide is varied as 8, 12 and 16 M. The proportions of calcined source material, fly ash and GGBS are given in Table 2. To prepare 8 molarity concentration of sodium hydroxide solution, 320 g (molarity × molecular weight) of sodium hydroxide flakes are dissolved in distilled water and makeup is done to 1 l. Sodium hydroxide solution thus prepared is mixed with sodium silicate solution 1 day before preparation of the mortar. Fine aggregate, fly ash and GGBS are dry mixed before adding to the alkaline solution for 2 min in an electrically operated mortar mixer. The calcined source materials, fine aggregate and alkaline solution are mixed for another 10 min in mortar mixer to ensure homogeneity. The fresh mixes prepared are cohesive and segregation resistant. Conventional table vibrator is used for compaction of the mortar. Steel molds of dimensions 100 mm × 100 mm × 100 mm are used for casting cube mortar specimens. The specimens are demolded after 24 h of casting and cured in outdoor and oven. For outdoor curing, specimens are left out in out-

Table 2 Mix proportions of geopolymer mortar

Mix ID/proportion of binders	Fly ash (kg/m^3)	GGBS (kg/m^3)	Fine aggregate (kg/m^3)	NaOH (kg/m^3)	Na_2SiO_3 (kg/m^3)	Alkaline liquid (kg/m^3)
$Na_2SiO_3/NaOH = 2.5$						
$M_1F_{100}G_0$	898.04	0	898.04	115.50	289.74	405.24
$M_2F_{90}G_{10}$	808.24	89.80	898.04	115.50	289.74	405.24
$M_3F_{80}G_{20}$	718.43	179.61	898.04	115.50	289.74	405.24
$M_4F_{70}G_{30}$	628.63	269.41	898.04	115.50	289.74	405.24
$M_5F_{60}G_{40}$	538.82	359.22	898.04	115.50	289.74	405.24
$M_6F_{50}G_{50}$	449.02	449.02	898.04	115.50	289.74	405.24
$M_7F_{40}G_{60}$	359.22	538.82	898.04	115.50	289.74	405.24
$M_8F_{30}G_{70}$	269.41	628.83	898.04	115.50	289.74	405.24
$M_9F_{20}G_{80}$	179.61	718.43	898.04	115.50	289.74	405.24
$M_{10}F_{10}G_{90}$	89.80	808.24	898.04	115.50	289.74	405.24
$M_{11}F_0G_{100}$	0	898.04	898.04	115.50	289.74	405.24

door (temperature—35 ± 2 °C and relative humidity—75 %) up to specified age of testing. Temperature and humidity control are not necessary for outdoor-cured specimens. The specimens, cured in hot air oven at 60 °C for 24 h duration, are considered as oven cured. After exposing to hot air in oven for 24 h, specimens are kept at outdoor until testing 28 days after casting. The variations considered in this study are: (1) variation in molarity of sodium hydroxide in the alkaline activator as 8, 12 and 16 M; (2) variation in the percentage of GGBS and fly ash; and (3) variation in the curing conditions, i.e., oven curing and outdoor curing.

3 Experimental Program

As per IS: 4031 (Part IV) [13], normal consistency or standard consistency of cement is determined using the Vicat's apparatus. Similar procedure is adopted for testing geopolymer material, and alkaline solution is used to produce geopolymer paste of standard consistency. The standard consistency or the normal consistency of the geopolymer paste is defined as the percentage of alkaline activator which allows the plunger of Vicat apparatus to penetrate to a depth of 33–35 mm from the top of Vicat's mold.

Final setting time of geopolymer paste was determined with the help of Vicat's apparatus taking 500 g of binder combinations (fly ash and GGBS) and 0.85 times of alkaline activator to produce geopolymer paste of normal consistency (0.85 P) [14].

Three cubes of each geopolymer mortar set with dimensions 100 mm × 100 mm × 100 mm are cast and tested in compression to determine 28-day compressive strength.

4 Test Results and Discussion

4.1 Normal Consistency

The values of normal consistency for different combinations of binder content are given in Table 3. It can be observed that geopolymer paste with 100 % fly ash requires less alkaline activator for normal consistency than geopolymer paste with 100 % GGBS. In case of intermediate mixes, increase in GGBS content resulted in increased normal consistency value. The reason for this behavior can be attributed to the fact that fly ash particles are spherical and exhibit less internal friction, allowing free movement of Vicat's plunger for lower alkaline activator content. On the other hand, GGBS particles are straight, flaky-elongated shape with sharp-edged angularity, rough surface texture possessing high internal friction compared to fly ash particles and hence need more alkaline activator to achieve normal consistency. The consistency is found to be 37 % with the combination of 70 % GGBS and 30 % fly ash. It can be observed that the concentration of sodium hydroxide in alkaline activator is not influencing the normal consistency of the geopolymer material of a given combination. From this discussion, it can be inferred that combination of fly ash and GGBS increases the normal consistency of the geopolymer.

4.2 Final Setting

Setting behavior of geopolymer is studied by varying sodium hydroxide concentration (8, 12, 16 M) and by varying the proportions of GGBS in fly ash. Final setting time reported in this paper is the final setting time of geopolymer paste. The procedure adopted is the same as the procedure followed for determining the final setting time of cement. The variation of

Table 3 Normal consistency of geopolymer paste for different molarities of NaOH

S.no.	Calcined source material combination		Percentage of alkaline activator required to produce geopolymer paste (P)		
	Fly ash (%)	GGBS (%)	Alkaline activator solution with sodium hydroxide 8 M	Alkaline activator solution with sodium hydroxide 12 M	Alkaline activator solution with sodium hydroxide 16 M
1	100	0	28	28	27
2	90	10	27	27	28
3	80	20	31	30	31
4	70	30	33	31	32
5	60	40	33	33	32
6	50	50	33	33	33
7	40	60	33	33	35
8	30	70	33	35	38
9	20	80	37	37	39
10	10	90	37	37	39
11	0	100	37	37	39

Table 4 Final setting time of geopolymer paste for various molarities of NaOH

S.no.	Calcined source material combination		Final setting time (minutes)		
	Fly ash (%)	GGBS (%)	Alkaline activator solution with sodium hydroxide 8 M	Alkaline activator solution with sodium hydroxide 12 M	Alkaline activator solution with sodium hydroxide 16 M
1	100	0	200	250	330
2	90	10	145	170	250
3	80	20	120	140	185
4	70	30	110	125	148
5	60	40	100	105	120
6	50	50	95	100	115
7	40	60	80	90	100
8	30	70	70	85	95
9	20	80	60	70	80
10	10	90	50	60	70
11	0	100	40	50	60

final setting time of geopolymer with the variation in concentration of sodium hydroxide in alkaline activator for different mixes of fly ash and GGBS is presented in Table 4. The final setting time of different mixes considered in this investigation varied from 40 to 330 min. It is found that increase in the concentration of sodium hydroxide solution resulted in increased final setting time. When fly ash content in the mix is 100 %, the final setting time increased from 200 min (with alkaline solution having sodium hydroxide of 8 M) to 330 min (with alkaline solution having sodium hydroxide of 16 M). When the fly ash content is totally replaced by GGBS, the final setting time increased from 40 min (with alkaline solution having sodium hydroxide of 8 M) to 60 min (with alkaline solution having sodium hydroxide of 16 M). This behavior clearly indicates that the GGBS readily reacts with the alkaline activator compared to fly ash. The setting aspects of GGBS with the alkaline activator are faster than fly ash. Thus for developing high early strength geopolymer material, GGBS is a better source material than fly ash. Partial replacement of fly ash by GGBS by 20 % decreased the final setting time from 200 to 120 min when the molarity of the sodium hydroxide is 8 M. From the test data, it is clear that final setting time has drastically reduced from 200 to 40 min when the total fly ash is replaced by GGBS for 8 M mix. Such fast setting behavior is not convenient for geopolymer mortar in conventional construction. Thus, it can be concluded that to have a desired value of final setting time suitable combination of GGBS and fly ash can be advocated.

Table 5 28-day compressive strengths of geopolymer mortars with alkaline activator sodium hydroxide 8 M

S.no.	Calcined source materials		Compressive strength (N/mm^2) at 28 days	
	Fly ash	GGBS	Alkaline activator solution with sodium hydroxide 8 M (outdoor curing)	Alkaline activator solution with sodium hydroxide 8 M (oven curing)
1	100	0	41	52
2	90	10	44	54
3	80	20	46	55
4	70	30	49	56
5	60	40	50	58
6	50	50	50	59
7	40	60	52	62
8	30	70	57	65
9	20	80	63	72
10	10	90	69	77
11	0	100	75	82

Table 6 28-day compressive strengths of geopolymer mortar with alkaline activator sodium hydroxide 12 M

S.no.	Calcined source materials		Compressive strength (N/mm^2) at 28 days	
	Fly ash	GGBS	Alkaline activator solution with sodium hydroxide 12 M (outdoor curing)	Alkaline activator solution with sodium hydroxide 12 M (oven curing)
1	100	0	44	53
2	90	10	45	55
3	80	20	47	57
4	70	30	50	59
5	60	40	52	61
6	50	50	53	65
7	40	60	55	68
8	30	70	62	74
9	20	80	65	77
10	10	90	72	82
11	0	100	78	85

The normal consistency of 100 % fly ash-based geopolymer paste is 28 %, and the alkaline activator used for estimating the final setting time is 0.85 P (0.85 × 28 = 23.8 %) [15]. Whereas for 100 % GGBS-based geopolymer paste, the normal consistency is 37 % and the alkaline activator used for estimating final setting time is 0.85 P (0.85 × 37 = 31.45 %). The chemical composition difference between fly ash and GGBS and difference in the quantity of alkaline activator perhaps decreased setting time for 100 % GGBS-based geopolymer paste.

4.3 Compressive Strength

The compressive strengths of geopolymer mortars having different proportions of fly ash and GGBS along with different concentrations of sodium hydroxide in alkaline activator are presented in Tables 5, 6 and 7. The compressive strength of geopolymer mortar ranges from 41 to 87 MPa. The outdoor-cured samples have shown variation in compressive strength with variation in molarity of sodium hydroxide in the alkaline activator from 41 to 69 MPa. Increase in molarity of sodium hydroxide in alkaline activator increased the compressive strength of the mortar, and similar trend is reported by Hardjito et al. [16]. In case of outdoor-cured samples, increase in percentage of replacement of fly ash by GGBS increased the compressive strength of geopolymer mortar. Total replacement of fly ash by GGBS has shown a compressive strength of 79 MPa. This indicates that geopolymer can attain strength even under outdoor curing if GGBS and fly ash together are used as source material. The reason for increase in compressive strength due to GGBS can be attributed to higher calcium content present in GGBS [17]. Oven curing

Table 7 28-day compressive strengths of geopolymer mortars with alkaline activator sodium hydroxide 16 M

S.no.	Calcined source materials		Compressive strength (N/mm^2) at 28 days	
	Fly ash	GGBS	Alkaline activator solution with sodium hydroxide 16 M (outdoor curing)	Alkaline activator solution with sodium hydroxide 16 M (oven curing)
1	100	0	45	54
2	90	10	47	58
3	80	20	52	62
4	70	30	56	63
5	60	40	59	65
6	50	50	63	66
7	40	60	65	69
8	30	70	67	73
9	20	80	69	78
10	10	90	75	83
11	0	100	79	87

results in an increased compressive strength in fly ash-based geopolymer mortar of 17 % when compared to that of outdoor sample. However, that increase is only 9.3 % for a similarly activated geopolymer mortar with 100 % GGBS as source material.

5 Conclusions

Based on the experimental work, the following conclusions are drawn:

1. Molarity of sodium hydroxide in the alkaline activator of geopolymer does not affect normal consistency significantly.
2. Final setting time increases with increase in molarity of sodium hydroxide in the alkaline activator.
3. Replacement of fly ash by GGBS decreases the final setting time of geopolymer paste.
4. High molarity of sodium hydroxide solution in the alkaline activator results in higher compressive strength of geopolymer mortar for all combinations of GGBS and fly ash as source material.
5. Compressive strength of geopolymer mortar increases with increase in percentage of replacement of fly ash with GGBS.
6. To develop geopolymer concrete under outdoor curing condition, combination of fly ash with GGBS can be a possible solution.
7. Method of curing plays an important role in geopolymerization process.

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