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Strength and Setting Times of F-Type Fly Ash-Based Geopolymer Mortar

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Abstract: Recently, great concern for many researchers has been the development of cement less concrete to reduce drastically the carbon dioxide emissions. A fair amount of research has been conducted on alkali-activated concrete using fly ash and blast furnace slag but no significant work reported so far in the area of geo-polymer mortars. It was assumed in the study that the factor governing the strength is the reaction between the fly ash and the alkaline activator. In this research , the influence of various parameters such as concentration of activator solution, the ratio of activator/binder ratio, the effect of curing temperature on the setting time, on the short term engineering properties of fresh and hardened F-Type fly ash based geo-polymer mortar were studied. The Test were carried out on 70.6x70.6x70.6 mm cube geo-polymer mortar specimens at 7 and 28 days. The test results revealed that the increase in fluid to binder ratio showed increase in compressive strength up to certain extent thereafter it showed decrease in compressive strength and geo-polymer paste did not set at room temperature which indicates that curing temperature plays an important role in the geo-polymerization. The geopolymer mortar setting time is much faster when it is cured at elevated temperature (Oven curing), where as in case of sunlight curing, setting time is comparatively better with respect to ambient curing.

Keywords: *Geo-polymer mortar; Fly Ash; setting time, strength;*

1. Introduction

Cement has been used since decades as a binding material in construction industry as concrete and mortar, But the manufacturing process of cement involves in emission of large amount of greenhouse gases like Carbon dioxide into atmosphere. As an alternative geo-polymers can be used as cementitious material that utilize high siliceous materials like fly ash and rice husk ash for binding which can produce an ecofriendly material. In geo-polymerization process no energy is utilized and no greenhouse gases are produced and also fly ash is by product obtained by combustion of pulverized coal of thermal power plant, it has been proved to be a harmful substance to environment. Therefore by incorporating fly ash as a source material in geo-polymers it can be turned out into a environment friendly material. Geo-polymers are a series of geochemical reactions of mineral dissolution, alumino silicate poly condensation and structural reorganization having potential application in manufacture of building materials. Industrial by products which are high in silica and alumina are generally used in producing geo-polymers because the reactions between silica and alumina bearing materials with alkaline solutions results into a gel like compounds which gains high strength eventually. In our study class-F fly-ash is used as binding material, because class-F fly-ash contains high amounts of silica(SiO_2 > 60%) and alumina (Al_2O_3 >30%). Geopolymer concrete produced without using elevated heat for curing will widen its application to the areas beyond precast members. Hence this study aimed to produce geopolymer concrete suitable for different curing conditions in addition to heat curing. Sunlight curing and ambient curing were suggested to explore the possibility of using low calcium fly ash to study the setting time, workability and the strength properties of geopolymer concrete

2. Literature Review

Geo-polymers consist of polymeric silicon-oxygen-aluminium framework [1] with silicon and aluminium tetrahedral alternately linked together in three directions by sharing all the oxygen atoms. The empirical formula of geo-polymers is like Mn[-(SiO2)z-AlO2]n· where – indicates the presence of bond between the atoms[2]. Geo-polymers are environmentally friendly materials, unlike cement they doesn't produce any harmful gases like CO, CO_2 , etc. and they need very little amount of energy to produce[3]. Moreover Geo-polymers have excellent physical and mechanical properties; they are intact to marine and acidic environment and they does not generate alkali aggregate reaction even they have high alkaline materials [4].

The suggested curing temperature for geo-polymers was 60^0C to 80^0C beyond that there was no significant strength gain was observed. Curing period was suggested for time periods of 24 hours to 48 hours, further increase in curing duration has no effect on geo-polymerization [5]. Increase in concentration of NaOH increases the compressive strength of geo-

polymer because higher alkaline imparts better dissolving nature to fly-ash which helps in forming stronger intermolecular bonding [6]. The concentration of NaOH solution directly affects the dissolution of the metakaolinite particulates, which affects the formation of the geo-polymer framework [7]. Addition of fibers in geo-polymer mortars helped in increasing compressive strength as well as flexural strength [8]. Geo-polymers have high thermal resistance i.e upto 800^0C, In fact, geo-polymer mortar does not release water in an explosive manner or dehydrate to a powder like OPC [9]. Geo-polymers are intact to marine, acidic environments which make them a better building material in environments which are prone to chemical changes like sea shores, industries, dumping sites etc [10]. Temuujin et al [11] attempted to study the effect of mechanical activation of fly ash on the properties of geopolymer cured at ambient temperature. The variables considered in their study are milled fly ash and raw fly ash. Mechanical activation of the flyash resulted in an 80% compressive strength compared to raw fly ash based geopolymer. Mustafa Al Bakri et al[12] investigated the effect of curing temperature on physical and chemical properties of geopolymers. geopolymer samples were prepared using different curing temperatures (Room temperature, 50^oC, 60^oC,70^oC & 80^oC), in which sodium hydroxide and sodium silicate were used as alkaline activators. The sample were cured for 24 hrs duration and tested on the seventh day. Based on the data from this study they concluded that the optimum curing temperature for fly ash based geopolymer is 60^oC which produced the maximum compressive strength 67.04 MPa, when the geopolymer cured at higher temperature the samples do not had enough moisture in order to develop better strength.

3. Significance and Scope of the Research

Several researchers [11,12] so far concluded that the increase in strength and decrease in setting time was observed with the increase of CaO content . Properties of calcium sources and the alkalinity of the activating solution also influences the geopolymerisation process . Recently, the suitability of fly ash based geopolymers mixed with silica fume, metakaolin and blast furnace slag has been studied by several investigators. However, most of the findings are reported for mortar and paste samples initially cured in temperature higher than ambient for variable lengths of time. The present study is aimed at evaluating the response of setting times of Fly ash based geopolymer mortars under different curing regimes. The study comprised determination of compressive strength of geopolymer mortar under suncured, ambient cured and heat cured conditions. The findings of the present study shall be useful in determining the applicability of geopolymer materials for use in cast-in-situ applications. In this research Geo-polymer mortar cubes of size 70.6x70.6x70.6 mm cube was prepared with fly ash: sand ratio of 1:2 which is constant for all the mixes. Alkali activator solution to binder (F/B) ratio was varied and taken 7 different proportions as 0.35, 0.375, 0.40, 0.425, 0.45, 0.475 & 0.50. The molar concentration of NaOH was taken as 4M and the ratio of NaOH: Na_2SiO_3 is taken as 2.5.Influence of various parameters such as the ratio of activator/binder ratio, the effect of curing temperature on strength and setting time were studied.

4. Materials Used

4.1 Fly Ash

Fly ash is a fine residual particle formed during combustion of powdered coal. Fly-ash mainly contains silica, alumina and minor amounts of oxides such as iron (Fe), sodium(Na), calcium(Ca), magnesium(Mg) and potassium(K). But major chemical constituents in class-F fly ash are alumina and silica, both together occupies more than 90% of fly ash. The class-F fly ash used in our work is obtained from Kakatiya Thermal Power Project (KTTP) jaggaipalli,, Kataram, Telangana. It confirms with grade I of IS: 3812 – 1981. It was tested in accordance with IS: 1727 –1967.The chemical composition of class-F fly ash is carried out by XRF analysis which shown in Table.1.

Table 1: *Chemical requirements of fly ash*

S. No.	Characteristics	Requirements (% by weight)	Fly Ash used (% by weight)
1	Silicon dioxide (SiO_2)plus aluminum oxide Al_2O_3 plus iron oxide Fe_2O_3	70 (minimum)	94.60
2	Silicon dioxide (SiO_2)	35 (minimum)	62.76
3	Magnesium Oxide (MgO)	5 (max.)	1.08
4	Total sulphur as sulphur trioxide (SO_3)	2.75 (max.)	0.23
5	Loss on ignition	12 (max.)	0.15

4.2 Fine Aggregate

Clean and dry river sand passing through IS 2.36 mm sieve was used for experimental study. Specific gravity is 2.46 and fineness modulus is 2.85. Natural river sand conforming to Zone II as per IS 383(1987) was used.

4.3 Sodium Hydroxide (NaOH)

Sodium hydroxide shown in fig.1(a) in flakes form were used. It is colour less substance with a purity of 98%.Sodium Hydroxide solid flakes of required concentration are dissolved water to make the solution. The mass of NaOH solid varies according to the Molarity required. The weight of NaOH solids is 160 grams per 1 lit of solution for 4M concentration.

Fig 1*(a): sodium hydroxide*

Fig 1*(b): sodium silicate*

4.4 Sodium Silicate(Na_2SiO_3)

Commercially available sodium silicate shown in fig.1(b) in liquid form was used. Colour of the liquid was white and the liquid was viscous in nature. The Sodium silicate used has a Na_2O to SiO_2 ratio of 2 was supplied by local manufacturer.

5. Experimental Work

5.1 Mix Proportions

The geo-polymer mortar was prepared by fly ash: sand ratio of 1:2, alkali activator solution to binder(F/B) ratio was varied and taken 7 different proportions 0.35,0.375,0.40,0.425,0.45,0.475&0.50 shown in Table.2 were adopted. The molar concentration of NaOH was taken as 4M and the ratio of NaOH:Na_2SiO_3 is taken as 2.5.

5.2 Preparation of Alkali-Activator Solution

Sodium Hydroxide in flakes form of 98% purity was used in the work, The mass of sodium hydroxide (NaOH) depends on the molarities of the solution. In the present work 4M solution was utilized where M is the molecular weight of sodium hydroxide (NaOH). Therefore 4M concentration solution consists of 4x40=160 grams of sodium hydroxide flakes in 1 lit1er of solution. The sodium hydroxide solution was prepared at least 1 day prior to the mixing. Sodium silicate was 2 available in liquid form. These two chemicals are mixed together at least 2 hours before the mix.

5.3 Preparation of Mortar Specimens

Fly ash and sand were mixed for 3 to 4 minutes manually then alkali activator solution is added to the mix and the mixing is continued for another 3 to 4 minutes till we get a homogenous mixture. The fresh mortar is now transferred into 70.6mm*70.6mm*70.6mm size moulds in 3 layers, tamping each layer 10 blows with mortar tamping rod and the moulds are kept on a vibrating for 120 seconds. A total of 95 cubes are casted and tested.

Table 2: *Mix Proportions*

S. No	Mix	Flyash: sand	Fluid/binder ratio
1	M1	1:2	0.35
2	M2	1:2	0.375
3	M3	1:2	0.40
4	M4	1:2	0.425
5	M5	1:2	0.45
6	M6	1:2	0.475
7	M7	1:2	0.50

Fig 2: *(a) Initial reading*

Fig.2 (b) *finale reading of initial setting time*

5.4 Curing

Three types of curing were adopted for the specimens Sunlight curing or atmospheric curing: specimens are cured in open air at 35^o centigrade.

Ambient curing: specimens are left to air in the Laboratory i.e curing was done at room temperature.

Oven curing: specimens are placed in oven along with the mould after 1 day of casting and cured at 60^0 degrees for 24 hours and then left in ambient conditions. Oven cured geo-polymer specimen attains its highest strength within 24 hours of curing.

Fig 3: (a) *Compaction*

Fig 3: (b) *Oven curing*

Fig 4(a): *Ambient curing*

Fig 4(b): *Sun (Atmospheric) curing*

The results show that fly ash based geo-polymer mortar could be handled up to 2 hours or 120 minutes without any sign of setting for curing temperature of 65^0 centigrade. For sun cured samples (temp $>35^0$ centigrade) it can be handled for more than 4 hours or 240 minutes. The procedure followed for this was similar to that of cement paste. Fluid to binder ratios of 0.40,0.45 were taken and samples were tested for atmospheric curing and oven curing. For every 15 minutes interval, the specimen was placed on the Vicat apparatus to measure the initial setting time. The results are given Table.3.

5. Results and Discussions

5.1 Initial Setting and Finale Setting Time

The activator-to-fly ash ratio was held constant at 0.35. Initial setting time is the time period between the alkali activator solution added to binder(Fly ash) and the time at which vicat's needle(1 mm square section) fails to pierce the test block by 33 to 35 mm from the top as shown in fig.2(a). Finale setting time is the time period between alkali activator solution is added to binder and the time at which vicat's annular ring (5mm section) doesn't make any impression on the surface of the paste as shown in fig.2(b).

Table 3: *Initial and Finale setting times*

	Oven cured		Atmospheric cured	
Fluid to binder ratios	**0.40**	**0.45**	**0.4**	**0.45**
Initial setting time	120 min	120 min	240 min	480 min
Finale setting time	150 min	150 min	280 min	520 min

5.2 Compressive Strength

Compressive strength is a mechanical test used to find the maximum amount of compressive load that can be taken by a material until failure. Compressive loading is applied on specimens by compressive testing machine of capacity of 2000kN (fig.5 (a)). Any two plane faces of the cube are inserted between platens of compressive testing machine and load is applied gradually until the specimen fails (fig.5 (b)). Test was conducted on 7 days, 28 days for the atmospheric cured specimens and for oven cured sample test was conducted on 3^{rd} day. The variation of compressive strength for atmospheric curing and heat curing is shown in the Table.4.

Fig. 5: *(a) compression test on specimens*

Fig 5(b) *failure pattern of specimen*

The initial and final setting time of Geopolymer establishes the time available for transport, placing and compaction of Geopolymer hence it is very important in practice. Mixture M3 & M5 minus the fine aggregate was used to investigate the setting time of geopolymer paste.

Table4*: Compressive strengths (in MPa)*

Mix	Ambient cured	Sunlight cured	Oven cured
M1	8.20	9.49	9.36
M2	11.18	12.40	12.43
M3	11.21	13.04	12.63
M4	12.42	17.38	14.17
M5	11.55	13.77	13.37
M6	11.38	12.87	11.90
M7	8.68	10.16	10.90

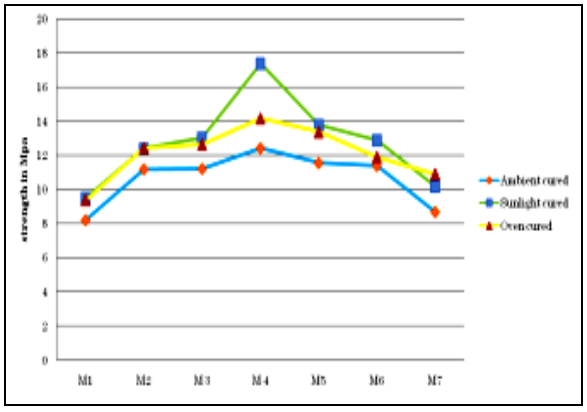

Fig.6 *Compressive strength for various mixes under different curing conditions*

Geo- polymerization is a slow process at room temperature; the initial strength gain is very low at ambient temperatures which can be seen from the fig.6 that compressive strength in the range of 8.2Mpa to 12.42MPa at 28 days. Where as in case of heat curing strengths are in the range of 9.36 to 14.17MPa which indicates that bulk strength attained by mortar at first 3 days itself. In case of sun light curing the strengths are in the range of 9.39MPa to 17.38MPa. Therefore, Temperature plays very important role in attaining the early strength. The setting time of geo-polymer paste at ambient temperature was more than 24hrs which indicates that geopolymer paste did not set even at 24hrs because of slow geopolymerization process.There was very little time interval was observed between initial setting and final setting time (30 to 40 min).The increase in fluid to binder ratio showed increase in compressive strength upto certain extent thereafter it showed decrease in compressive strength Fluid to binder ratio of 0.425 is optimum which shows highest strength of all mixes. Compressive strengths at 28 days for oven curing (for 24hrs at 60^0C) and sunlight curing for 28 days were almost similar. But the maximum strength was attained in oven curing within 48 hours but for atmospheric curing it took very long time(28 days). It was observed that fresh fly ash-based geopolymer paste did not harden at room temperature for at least one day.

6. Conclusions

1) Geo- polymerization is a slow process at room temperature; the initial strength gain is very low at ambient temperature.
2) Temperature plays very important role in setting time of geo-polymer paste and there was very little time interval was observed between initial setting and final setting time .
3) The increase in fluid to binder ratio showed increase in compressive strength upto certain extent thereafter it showed decrease in compressive strength.
4) Fluid to binder ratio of 0.425 shows highest strength of all mixes.
5) It is obvious that the geopolymer mortar setting time is much faster when it is cured at elevated temperature (Oven curing), where as in case of sunlight curing, setting time is comparatively better w.r.t ambient curing
6) Compressive strengths of oven curing and atmospheric curing for 28 days were almost similar but the maximum strength was attained in oven curing within 48 hours but for atmospheric curing it took very long time(28 days).
7) In tropical countries like India, sunlight curing seem to be advantageous compared to oven curing which requires a lot of energy

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