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Effect of Curing Conditions and Molarity on Compressive Strength of Fly ash based Geopolymer Mortar

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ABSTRACT: The geopolymers are eco-friendly materials as produces a very small quantity of CO_2 compared with the production of ordinary Portland cement. Geopolymers are prepared by fly ash which is a waste generated as by-product in thermal power plants by burning of coal. Fly ash is one of the major culprits responsible for causing air pollution. The utilization of fly ash in making geopolymers helps in the waste management of fly ash and also in reducing air pollution and carbon dioxide footprint. With the above objective the aim of present study is the optimization of effect of molarity and different thermal curing conditions for fly ash based geopolymers. Geopolymer results from the reaction of fly ash and alkaline solution. The alkaline solution comprises of sodium hydroxide and sodium silicate. Compressive strength test have been carried out on 70.6mm ×70.6mm × 70.6 mm cubes of geopolymer mortar samples. The ratio of alkaline liquid to fly ash is taken as 0.6. These samples were cured at 60°C for three different duration 24h, 48h, and 72 hours in hot air oven. It is found that compressive strength increases with the increases in molarity and thermal curing duration. This paper presents the results based on experimental study of molarity and effect of different thermal curing condition on compressive strength of geopolymer mortar.

KEYWORDS: Geopolymer mortar, Thermal curing, Molarity, Compressive strength, Class-F fly ash.

I. INTRODUCTION

An important ingredient in the conventional concrete is the Portland cement. The production of one ton of cement emits approximately one ton of carbon dioxide to the atmosphere. Moreover, cement production is highly energy intensive and also consumes significant amount of natural resources. Now a days, global warming is the major environmental problem in the world. It is causing environmental problems as requires large land area for disposal. CO_2 is the main green house gas responsible for this problem. It is produced from power plants, transportation and cement industries. India is the world's second largest producer of cement after China. Therefore, for a green environment, it is necessary to reduce CO_2 emissions. Apart from this, large quantity of fly ash is generated from power plants. More than 184 million tones of fly ash are generated every year in India. As the need for power increases, the volume of fly ash would increase. It is necessary and significant to use fly ash to produce cementitious binder without Portland cement. To overcome these problems, an alternative green cementatious material geopolymer is developed. In geopolymers, the CO_2 production is about $1/10^{\text{th}}$ of ordinary Portland cement. They are not only environment friendly but also most popular and widely used building materials.



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Vol. 5, Issue 10, October 2016

A. GEOPOLYMERS

Geopolymers were 1st developed by Joseph Davidovits in 1978 and named as "soil cement". They are produced at a temperature lower than required in manufacture of cement. Geopolymers are new group of building materials that has potential to transform the building products industries. Davidovits claims that the Egyptian Pyramids were built by casting geopolymer on site. He also reported that this material has excellent mechanical properties, does not dissolve in acidic solutions, and does not generate any deleterious alkali-aggregate reaction even in the presence of high alkalinity.

Geopolymers are a class of inorganic polymers formed by the reaction between an alkali and an aluminosilicate source. These materials have an amorphous 3- dimensional structure that gives geopolymers properties which make them an ideal substitute for Ordinary Portland Cement (OPC). Variations in the ratio of aluminium to silicon, and alkali to silicon, produce geopolymers with different physical and mechanical properties. It is an emerging class of cementitious material that utilizes 'fly ash', one of the most abundant industrial by-products on earth. Ever since the introduction of geo polymer binders, it has generated a lot of interest among engineers as well as in the field of chemistry. In the past few decades, it has emerged as one of the possible alternative to cement binders.

B. POLYMERIZATION OF THE GEOPOLYMERS

The polymerization process involves a substantially fast chemical reaction under alkaline conditions on silicon-aluminum minerals that results in a three-dimensional polymeric chain and ring structure. The reaction of Fly Ash with an aqueous solution containing Sodium Hydroxide and Sodium Silicate in their mass ratio, results in a material with three dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follows explained in Eq. 1.1

Mn [-(SiO₂) z–AlO₂] n. wH₂O.....1.1

Where: M = the alkaline element or cation such as potassium, sodium or calcium; the symbol –indicates the presence of a bond, n is the degree of polycondensation or polymerization; z is 1, 2, 3, or higher.

The schematic formation of geopolymer material can be shown as described by Equations 1.2 as (A)

and (B).

$$\begin{array}{c} n(Si_2O_5,Al_2O_2)+2nSiO_2+4nH_2O+NaOH \text{ or } KOH \blacktriangleright Na^+, K^+ + n(OH)_3-Si-O-Al^- -O-Si-(OH)_3 (A) \\ (Si-Al materials) \\ (Coopelumer propagation 1) \\ \end{array}$$

(Geopolymer precursor) (Equation 1)

$$n(OH)_{3}-Si-O-Al^{-}-O-Si-(OH)_{3}+ NaOH \text{ or } KOH \blacktriangleright (Na+,K+)-(-Si-O-Al^{-}-O-Si-O-) + 4H_{2}O (B)$$

$$(OH)_{2} O O O$$

(Geopolymer backbone)(Equation 2)

.....Eq as (A) and (B) (Panias.D and Giannopoulou.I 2004)

II. RAW MATERIALS USED

The main constituents of geopolymer mortar were fly ash, sodium hydroxide (NaOH) solution, sodium metasilicate (Na_2SiO_3) solution, sand and tap water. Source materials should be rich in silicon (Si) and aluminium (Al).

A. FLY ASH

In this study, low calcium fly ash (Class F) from Thermal Power Plant, Sarni (Madhya Pradesh) as shown in Fig 1(a) was used as the source material. The fly ash particles are spherical, and grey in colour. The fly ash was



(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 10, October 2016

characterized as Class F (conforming to IS 3812(Part-1):2013. The typical chemical composition and physical properties of fly ash are given in Table 1 and Table 2 respectively.

Compounds	Mass percentage (%)				
SiO ₂	64.2				
Al ₂ O ₃	28.2				
Fe ₂ O ₃	3.70				
Na ₂ O	0.17				
MgO	0.46				
CaO	0.06				
K ₂ O	1.34				
LOI*	1.79				

Table 1 Chemical Composition of Fly Ash

*LOI- Loss on ignition

About 75% of particles were finer than 45 micron having specific gravity of 2.1 and Blaine's specific surface area of fly ash was $360m^2/kg$. The pH of the fly ash used was 8.6.

Table 2 Physical Properties of Fly Ash

рН	8.6
Specific Gravity	2.1
Blaine fineness	360 m²/kg

B. FINE AGGREGATE

The local Narmada sand as fine aggregate having a Fineness Modulus 2.82 and specific gravity of 2.65 was used in dry clean form as shown in Fig 1(b).

C. ALKALINE ACTIVATOR

The alkaline liquid used for geopolymerization was prepared by dissolving sodium metasilicate (Na_2SiO_3) and sodium hydroxide (NaOH) in tap water as shown in Fig 1(c). Sodium metasilicate (SMS) in powder form and Sodium hydroxide in flakes form with 98% purity were used for the study. At the time of mixing of sodium metasilicate with sodium hydroxide solution it generates a huge amount of heat and the polymerization takes place by reacting with one another, which will act as a binder in the geopolymer mortar.



(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 10, October 2016



Fig. 1. Raw Material used with preparation of geopolymer mortar (a) Fly ash (b) Fine aggregates (c) Alkaline activator (d) Geopolymer mortar preparation

III. MIX PROPORTIONS

The alkaline activator is prepared at least one hour prior to cast as shown in Fig 1(c). Firstly, the Sodium hydroxide (NaOH) solution is prepared for the required molarity and then it is mixed with Sodium silicate (Na₂SiO₃) in different proportion. The samples were prepared for 6M, 8M, 10M, 12M and 14M. (where, M–molarity). For e.g. 6M means, concentration of NaOH in one litre of water is $6 \times 40 = 240$ gm, so for 0.7 litre of water 168gm of NaOH is used to get required molarity (where 40 is the molecular weight of NaOH). Mix proportions for various samples are illustrated in Table 3.

Sample	Fly ash (gm)	Sand (gm)	Molarity	NaOH	Water	SMS/NaOH (Ratio)	Sand to Fly ash ratio	Activator to fly ash ratio
Mix-1	2200	6600	6	168	700	0.5	3	0.6
Mix-2	2200	6600	8	224	700	1.0	3	0.6
Mix- 3	2200	6600	10	280	700	1.5	3	0.6
Mix-4	2200	6600	12	336	700	2.0	3	0.6
Mix- 5	2200	6600	14	392	700	2.5	3	0.6

Table 3 Mix proportions of materials used in experiment

Five mixes of geopolymer mortar were prepared. Activator to fly ash ratio was maintained at 0.6 and the ratio of sand to fly ash was taken as 3. The geopolymer mortar mix was prepared as shown in Fig 1(d) exhibited a thick sticky nature with good workability.



(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 10, October 2016

IV. CASTING & CURING

The fly ash and sand are first dry mixed for three minutes and then alkaline solution of different molarity i.e. 6M, 8M, 10M, 12M, and 14M is added in specified proportion for about 10 minutes so that the homogeneous mixture is obtained. The mortar samples have been casted in steel moulds of size 70.6mmX70.6mmX70.6mm. The fresh geopolymer mortar is then filled in mould immediately after mixing in two layers and hand compacted using cylindrical plunger to avoid the air bubbles and then vibrated for 15 s on a vibrating table. After casting, geopolymer mortar samples are left to room temperature for one hour under atmospheric pressure and uncontrolled humidity conditions and are cured in an oven at 60°C for three different durations 24hr, 48hr and 72hours. At the end of curing period the oven is turned off and the specimens is allowed to cool down inside the oven to room temperature. The samples are then removed from the mould and they are left to ambient curing (drying) at room temperature before being tested for compressive strength.

V. RESULTS

A. COMPRESSIVE STRENGTH TEST

The compressive strength values of fly ash based geopolymer mortar samples having different molar concentration of 6M, 8M, 10M, 12M and 14M are given in Table 4, 5 and 6.

Table 4 Compressive strength of geopolymer mortar after 7 days at 60°C aged for 24hr

Samples	6M	8M	10M	12M	14M
Compressive strength (N/mm ²)	12.43	14.52	19.82	24.12	25.60

Table 5 Compressive strength of geopolymer mortar after 7 days at 60°C aged for 48hr

Samples	6M	8M	10M	12M	14M
Compressive strength (N/mm ²)	15.10	18.25	21.38	28.72	30.10

Table 6 Compressive strength of geopolymer mortar after 7 days at 60°C aged for 72hr

Samples	6M	8M	10M	12M	14M
Compressive strength (N/mm ²)	15.65	19.20	22.86	29.32	33.18

The minimum compressive strength was found as 12.43, 15.10 and 15.65 N/mm² after 7 days for 6M geopolymer mortar samples oven cured at 24hr, 48hr and 72hr aged samples. The maximum compressive strength was found to be 25.6, 30.1 and 33.18 N/mm² after 7 days for 14M geopolymer mortar samples cured at 1 day, 2 days and 3 days respectively. However it was noted that there is minimal difference between the compressive strength of 12M and 14M geopolymer mortar samples. The compressive strengths of 6M, 8M, 10M, 12M and 14M geopolymer mortar samples increased with curing condition as can be seen graphically in Fig 2.

The variation in Compressive strength increment with molarity of NaOH (6M, 8M, 10M, 12M & 14M) for the geopolymer mortar specimens is shown in Figure 2. The compressive strength obtained was in the range of 10MPa to 30MPa. Higher concentration of sodium hydroxide solution yielded higher compressive strength. The Mix 1 (6M) gives the minimum compressive strength and the Mix 5 (14M) gives the maximum compressive strength among other geopolymer mortar samples.



(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 10, October 2016

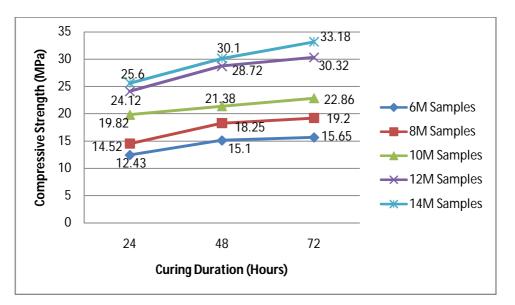


Fig. 2. Compressive strength values of geopolymer samples for different curing duration.

The compressive strengths of all the specimens increased with curing duration. For 24 hours curing duration, the thermal curing significantly affected the compressive strengths. However it was also noted that there is nominal difference between the compressive strength of 48hr and 72hr cured geopolymer mortar samples. The strength increased about 2 times at 7 days for 14M geopolymer samples as compared with 6M geopolymer mortar samples.

VI. DICUSSIONS

The results showed that the compressive strength of the geopolymer mortar samples increased with increase in curing condition and molarity. The effect of curing condition on the compressive strength was found to be more significant for curing times such as 48 hours. Longer curing time improved the polymerization process resulting in higher compressive strength. This might be due to relatively fast chemical reaction process in the geopolymer mortar.

A Compression testing Machine (CTM) model number AIM 308E-DG of AIMIL Ltd., India was used to test the compressive strength of mortar cubes. Compressive strength for the mortar cubes were tested by applying load of 2.9 KN/sec. The cubes were tested in compressive testing machine to determine their compressive strength at the age of 7 days for different curing conditions. Table 4, 5 and 6 shows the compressive strength of the cube specimens at 7 days age of mortar.

The variations in Compressive strength increment with molarity of NaOH (6M, 8M, 10M, 12M, 14M) for the geopolymer mortar specimens is shown in Figure 2. This is due to increase in release of aluminate and silicate species from amorphous silico aluminous phases of the fly ash. The compressive strength obtained was in the range of 10MPa to 30MPa. Higher concentration of sodium hydroxide solution yielded higher compressive strength. The Mix 5 (14M) gives the maximum compressive strength among other samples. It is comparable with 7 days compressive strength of Portland cement; 33 grade-specification.

VII. CONCLUSIONS

Geopolymer mortar which is produced by the inorganic polymeric reaction of fly ash and alkali activated solution is used for this experiment. Geopolymer are new materials which have various application fields. There are many parameters affecting the geopolymerisation. The study of influence of curing condition and molarity on compressive strength of geopolymer mortar have revealed as listed below:



(An ISO 3297: 2007 Certified Organization)

Vol. 5, Issue 10, October 2016

- It has been found that as the molarity increases, compressive strength increases. This is due to greater dissolution of source material.
- The compressive strength values indicated that the curing conditions influenced the physical properties of fly ash based geopolymer mortar samples. As the curing duration increased, the compressive strengths increased. This may attributed to higher degree of geopolymerization.
- The geopolymer mortar mixes were produced easily using conventional equipment similar to those used for production of conventional cement mortar.

The Geopolymer binder may be treated as future environment friendly alternative to Portland cement. Thus the geopolymer binders developed from Class F fly ash can be used as a substitute to Ordinary Portland cement.

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