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Effect Of Curing Temperature And Curing Hours On The Properties Of Geo-Polymer Concrete

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ABSTRACT:

Geopolymer, an inorganic alumina silicate polymer is synthesized predominantly from silicon and aluminum materials or from by-product materials like fly ash. In the present paper the effect of curing temperature, curing hours on Geo-polymer Concrete(GPC) specimens and also the effect of extra water on workability and compressive strength of GPC cubes were studied. Fly ash and GGBS were used as binder, combined with an alkaline solution to form geopolymer paste instead of cement paste to bind the aggregates. The experiments were conducted on GPC cubes for curing temperature of 80° C, 90° C and 100° C with curing period of 12 and 24 hours by adopting hot oven curing method. The constants used in the experiments were alkaline solution to binder ratio taken as 0.45, molarity of NaOH solution as 12M and ratio of sodium silicate to sodium hydroxide as 2.5. All the specimens were kept at one day rest period. The tests were conducted on each sample and results revealed that there is an increase in compressive strength for curing temperature 80°C to 90°C for both 12 and 24 hours of curing of GPC specimens. Maximum strength was obtained at temperature of 90°C for 12 hours of curing period. Beyond this, increase in curing temperature resulted in decrease in compressive strength of GPC specimen. Furthermore the study was continued by varying water to geopolymer solids ratio and addition of extra water to study the workability and compressive strength of GPC specimens at optimum temperature 90°C-12 hours. Results showed that increase in water to geopolymer solids ratio and extra water increased the workability of GPC and decreased the compressive strength of GPC.

KEYWORDS: *Curing hours, Curing strength, Curing temperature, Flyash, GGBS, GPC.*

I. INTRODUCTION

Demand for concrete as a construction material is on the increase so as the production of cement. The production of cement is increasing about 3% annually. The production of one ton of cement liberates about one ton of CO_2 into the atmosphere. Also, Portland cement is the most energy intensive construction material, after aluminum and steel [1]. In recent years, geopolymer has emerged as a novel engineering material in the construction industry [2, 3]. It is formed by the alkali activation reaction between alumina-containing and silica-containing solids and alkali activators. The raw material for geopolymer production normally comes from industrial by-products, for instance, fly ash and blast furnace slag. In India more than 100 million tons of fly ash is produced annually. Out of this, only 17 – 20% is utilized either in concrete or in stabilization of soil. Most of the fly ash is disposed off as a waste material that coves several hectares of valuable land [4]. There are environmental benefits in reducing the use of Portland cement in concrete, and using a by-product material, such as fly ash as a substitute. The industrial by-products can substitute cement clinker by 100% in the system of geopolymer. For this reason, geopolymer is generally considered as an environment-friendly construction material with great potential for sustainable development. Apart from the environmental advantages, pastes and concrete made of geopolymer can exhibit many excellent properties, for example, high early-age strength, low creep and shrinkage, high resistance to chemical attack and good fire resistance [5,6].

The process of geo-polymerization takes place by activating the alumino-silicate waste materials with high alkaline solution. The most crucial aspect which plays an important role in the polymerization process is the curing of freshly prepared geopolymer concrete. Proper curing of concrete has a positive effect on the final properties of the geopolymer concrete.

As the reaction of fly ash-based geopolymeric materials is very slow and usually show a slower setting and strength development, the curing of geopolymer concrete is mostly carried out at elevated temperatures [7]. Previous research has shown that both curing time and curing temperature significantly influence the compressive strength of geopolymer concrete. Several researchers have investigated the effect of curing time and curing temperature on the properties of geopolymer concrete. It is reported by Palomo et al. [8], in their study on fly ash-based geopolymers that the curing temperature and curing time significantly affected the mechanical strength of fly ash-based geopolymers. The results also revealed that higher curing temperature and longer curing time resulted in higher compressive strength. Hardjito et al. [9, 10] studied the influence of curing temperature, curing time and alkaline solution-to-fly ash ratio on the compressive strength. The authors confirmed that the temperature and curing time significantly improves the compressive strength, although the increase in strength may not be significant for curing at more than 60°C. The results also revealed that the compressive strength decreases when the water-to-geopolymer solids ratio by mass is increased. The drying shrinkage strains of fly ash based geopolymer concrete were found to be significant.

The present study was aimed at producing GPC cubes of strength 50MPa by using Fly ash and GGBS as binders for 12M molarity of NaOH solution. Samples were cured at 80ºC, 90ºC and 100°C with curing hours of 12 hours and 24 hours. From these results the optimum temperature and curing hours were obtained corresponding to high compressive strength of geopolymer concrete. Further study was extended to study the effect of extra water on workability and compressive strength of geopolymer concrete cured at optimum temperature and curing hours.

II. MATERIALS

Fly ash which was obtained from Raichur Thermal Power Station, India and GGBS obtained from JSW steel, Bellary, India were having specific gravity of 2.4 & 2.9 respectively. The chemical composition of Flyash & GGBS as obtained by X-ray fluorescence (XRF) is shown in Table-1 & Table-3 respectively. The IS code requirements & composition of Flyash is shown in Table-2. The class F fly ash used here confirms to requirement as per 3812-2003 IS code & shown in Table 2. Locally available Fine aggregate of specific gravity 2.8 & Coarse aggregate of specific gravity 2.7 were used in this experimental work. A combination of 12M sodium hydroxide and sodium silicate in the ratio of 2.5 was used as solution for activation. Sodium hydroxide solution NaOH (97% purity), in the form of pellets were used in this work. Sodium silicate also known as water glass is of industrial grade with SiO_2 as 34.8% by mass and Na_2O as 16.51% & water as 48.69%. Water used for the mix is of potable quality. The plasticizers are used to improve the workability of geopolymer concrete, the addition of super plasticizer, up to approximately 4% of fly ash by mass, improves the workability of the fresh fly ash-based geopolymer concrete; however, there is a slight degradation in the compressive strength of hardened concrete when the super plasticizer dosage is greater than 2% [11]. Hence in the present study dosage of plasticizer (conplast 430) was taken as 1% of Binder.

Table-1: Chemical composition of Fly ash as determined by XRF analysis in (mass %)

Binder	Fly Ash
S.Gr	2.4
*LOI	0.90
Al_2O_3	31.23
Fe_2O_3	1.50
SiO_2	61.12
MgO	0.75
SO_3	0.53
Na_2O	1.35
Chlorides	0.05
CaO	3.2

*LOI - Loss on Ignition

Table -2 : Constitution of Flyash and code requirements

Constituents	Composition in %	Requirements as per IS 3812- 2003
LOI	0.90	Max 5
$(Al_2O_3+Fe_2O_3+SiO_2)$	93.85	Min 70
SiO_2	61.12	Min 35
MgO	0.75	Max 5
SO_3	0.53	Max 3
Na_2O	1.35	Max 1.5
Chlorides	0.05	Max 0.05

Table -3: Chemical composition of GGBS as determined by XRF analysis in (mass %)

Binder	**GGBS**
S.Gr	2.9
LOI	0.19
Al_2O_3	13.24
Fe_2O_3	0.65
SiO_2	37.21
MgO	8.46
SO_3	2.23
Na_2O	- - -
Chlorides	0.003
CaO	37.2

III. EXPERIMENTAL WORK

3.1 Mix Design

Concrete mix design process is vast and generally based on performance criteria. Initially the density of Geopolymer concrete was assumed as 2400 kg/m^3. The coarse and fine aggregates were taken as 72% of entire mixture by mass as per Lloyd et al [12]. This value is similar to that used in OPC concrete in which it will be in the range of 75% to 80% of the entire mixture by mass. Fine aggregate were taken as 30% of the total aggregates. The remaining mass is the combination of Alkaline solution and Binder (Geopolymer paste). Assuming the Alkaline solution to Binder ratio as 0.45 the masses of Alkaline solution and Binder in kg/m^3 were obtained. Assuming Sodium silicate solution to Sodium Hydroxide Solution ratio as 2.5, mass of Sodium silicate solution and sodium hydroxide solution were obtained in kg/m^3. Assuming the molarity of sodium hydroxide solution as 12M, the geopolymer mix was designed. To study the effect of high temperature curing and to get optimum temperature and curing hours the mix proportions were prepared as listed in Table -4.

Water present in geopolymer concrete is of two types namely, water present in alkaline solution and extra water. Water present in alkaline solution is of very small quantity and hence geoplymer concrete mixes are usually very stiff. Therefore to improve the workability and to make geopolymer mix as a homogeneous mix an extra water is added to the mix, but this water is the main parameter which directly affects the strength of geopolymer concrete. Hence in the present work, the extra water was studied as varying parameter, to study the properties of geopolymer concrete. After obtaining the optimum curing temperature and curing hours the mix proportion details of geopolymer concrete to study the effect of extra water on workability and compressive strength of geopolymer concrete were prepared as shown in Table -5.

3.2 Preparation of fresh Geopolymer concrete

The manufacturing of geopolymer concrete was similar to cement concrete, the process involved preparation of alkaline solution, dry mixing, wet mixing, curing & testing of samples. To prepare sodium hydroxide solution of 12 molarity, 480 g (12 x 40) i.e (molarity x molecular weight) of sodium hydroxide pellets were dissolved in one litre of water. The mass of sodium hydroxide solids in the solution varies depending on the concentration of the solution expressed in terms of molar M. The prepared NaOH solution was added with sodium silicate solution proportionately according to the mix, 24 hours before casting.

Table -4: Mix design trials with different curing temperatures and curing hours

Materials	GPC1	GPC2	GPC3	GPC4	GPC5	GPC6
Coarse Aggregate (kg/m^3)	1123.2	1123.2	1123.2	1123.2	1123.2	1123.2
Fine Aggregate (kg/m^3)	604.8	604.8	604.8	604.8	604.8	604.8
Fly ash (kg/m^3)	231.72	231.72	231.72	231.72	231.72	231.72
GGBS (kg/m^3)	231.72	231.72	231.72	231.72	231.72	231.72
NaOH Solution (kg/m^3)	59.59	59.59	59.59	59.59	59.59	59.59
Molarity of NaOH	12M	12M	12M	12M	12M	12M
Na_2SiO_3 Solution (kg/m^3)	148.96	148.96	148.96	148.96	148.96	148.96
Temperature (ºC)	80	80	90	90	100	100
Curing Period (Hours)	12	24	12	24	12	24
Water to geopolymer solids ratio	0.201	0.201	0.201	0.201	0.201	0.201
Alkaline solution to Binder ratio	0.45	0.45	0.45	0.45	0.45	0.45
Rest period (days)	1	1	1	1	1	1
Extra Water (kg/m^3)	0	0	0	0	0	0
Slump (mm)	0	0	0	0	0	0
Super Plasticizers (Conplast 430) in % of binder	1%	1%	1%	1%	1%	1%

The coarse aggregate, fine aggregate, flyash and GGBS were taken in required amount in a mixing tray and dry mixed manually for about two minutes. The alkaline solution prepared 24 hours before was thoroughly stirred, then the required amount of superplastisizer was mixed with the alkaline solution and was added to the dry mix, addition of solution had to be done in small quantities so that there was no wastage of solution, usually the wet mixing time should be about 10 to 15 minutes or greater. The mixing of total mass was continued until the mixture became homogeneous and uniform in colour. After this the mix was left for 10 to 15 minutes then the extra water was added to the mix, again after mixing homogenously the slump test was carried out.
The fresh geopolymer concrete was casted in cubes of size 150 X 150 X 150 mm to three layers and was compacted by using the standard compaction rod so that each layer received 25 strokes followed by further compaction on the vibrating table. Then the cubes were kept at room temperature for one day rest period.

The casted specimens after one day rest period were demoulded and specimens were kept in oven for 80ºC, 90ºC and 100ºC for the curing period 12 hours and 24 hours as shown in Figure-1, after required curing period the specimens were removed from the oven and were kept open at room temperature until testing as shown in Figure-2.

The specimens were removed from the oven; after the cooling of the samples, the specimens for the required period (3rd day and 7th day) were tested as per IS 516 : 1979 in the Compressive Testing Machine of capacity 2000 kN for obtaining ultimate load of the specimens.

Fig-1: Specimens kept in Oven of 300°C capacity

Fig-2 : Specimens kept at room temperature after removing from oven

Table -5: Mix design trials with different water to geopolymer solids ratio and extra water

Materials	GPC7	GPC8	GPC9	GPC10
Coarse Aggregate (kg/m^3)	1123.2	1123.2	1123.2	1123.2
Fine Aggregate (kg/m^3)	604.8	604.8	604.8	604.8
Fly ash (kg/m^3)	231.72	231.72	231.72	231.72
GGBS (kg/m^3)	231.72	231.72	231.72	231.72
NaOH Solution (kg/m^3)	59.59	59.59	59.59	59.59
Molarity of NaOH	12M	12M	12M	12M
Na_2SiO_3 Solution (kg/m^3)	148.96	148.96	148.96	148.96
Temperature (°C)	90	90	90	90
Curing Period (Hours)	12	12	12	12
Water to geopolymer solids ratio	0.25	0.30	0.35	0.40
Alkaline solution to Binder ratio	0.45	0.45	0.45	0.45
Rest period (days)	1	1	1	1
Extra Water (kg/m^3)	27	55	83	111
Slump (mm)	10	68	126	170
Super Plasticizers (Conplast 430) in % of binder	1%	1%	1%	1%

IV. RESULTS AND DISCUSSIONS

4.1 Effect of Temperature and Curing Hours on Compressive Strength of Geopolymer Concrete

Compressive strength of geopolymer concrete mainly depends on the temperature of curing and corresponding curing hours. In the present investigation, 3rd day and 7th day compressive tests were conducted with concrete cubes of size 150mm X 150mm X 150mm.

Table -6 : Mean compressive strength of GPC specimens at curing temperature of 80°C

Sample Name	Mean Density (kg/m^3)	Mean Compressive Strength (N/mm^2)	Curing Temperature (°C)	Curing Period (hours)	Days Of Testing
GPC1	2468	38.71	80	12	3
GPC1	2387	51.15	80	12	7
GPC2	2450	50.22	80	24	3
GPC2	2505	57.53	80	24	7

Table -7 : Mean compressive strength of GPC specimens at curing temperature of 90°C

Sample Name	Mean Density (kg/m^3)	Mean Compressive Strength (N/mm^2)	Curing Temperature (°C)	Curing Period (hours)	Days Of Testing
GPC3	2430	67.91	90	12	3
GPC3	2434	76.53	90	12	7
GPC4	2398	66.20	90	24	3
GPC4	2462	60.91	90	24	7

Table -8 : Mean compressive strength of GPC specimens at curing temperature of 100°C

Sample Name	Mean Density (kg/m^3)	Mean Compressive Strength (N/mm^2)	Curing Temperature (°C)	Curing Period (hours)	Days Of Testing
GPC5	2357	57.24	100	12	3
GPC5	2499	54.18	100	12	7
GPC6	2505	47.24	100	24	3
GPC6	2481	46.12	100	24	7

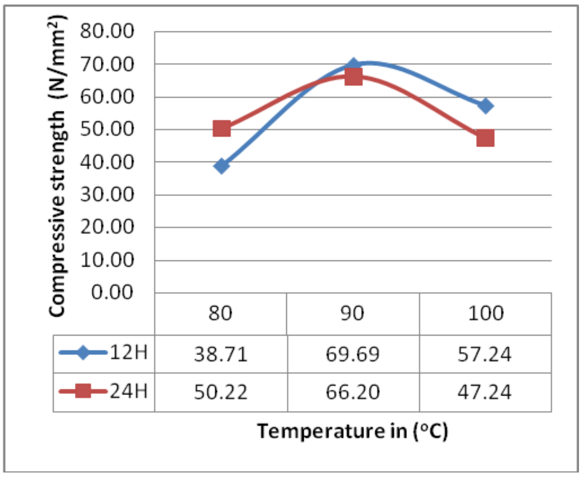

Fig-3 Variation of compressive strength on 3rd day represented graphically

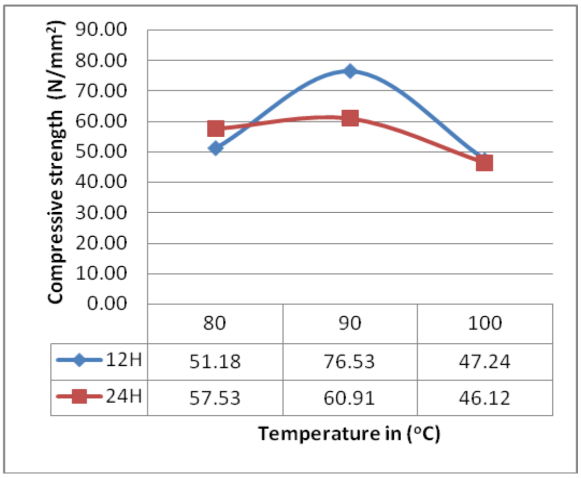

Fig-4 Variation of compressive strength on 7th day represented graphically

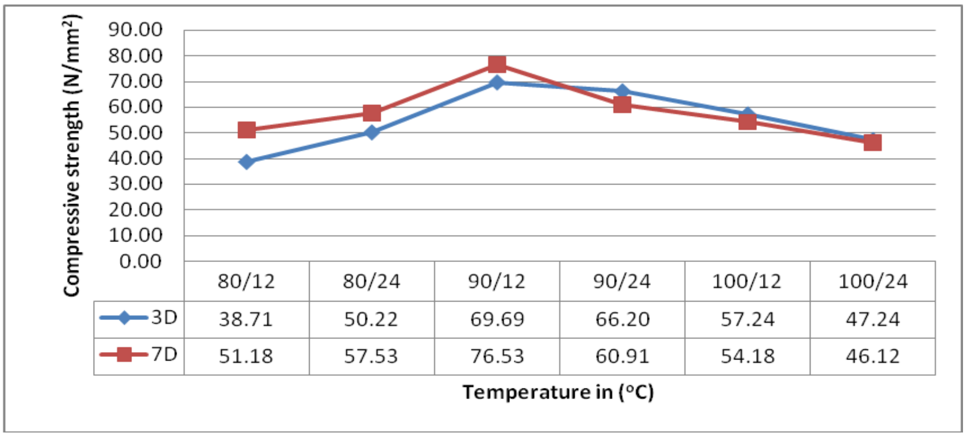

Fig-5 Spread compression strength for all combinations of curing temperatures and curing time

Fig-3 shows the compressive strength of geopolymer concrete specimen at different temperatures and different curing periods on 3rd day. The curing temperature at 90ºC showed better compressive strength than 80ºC and 100ºC. Furthermore 90ºC-12 hour curing produced maximum strength when compared with 90ºC-24 hours curing. This showed that curing hours play an important role in achieving the compressive strength of geopolymer concrete. This was because polymerization process increases with increase in the temperature and at a high temperature the 2D-polymer chains are converted into 3D-polymer chain with strong bond. At the same time the higher temperature results in increase in the rate of development of strength. This was same as observed in [13,14] and using high temperatures 90°C -12 hours produced compressive strength of 69.69MPa on 3rd day as compared to [15] in which 28th day compressive strength was 52MPa. Beyond this optimum temperature, increase in the curing temperature and curing hours reduced the compressive strength of geopolymer concrete specimens. The loss in compressive strength was due to continuous moisture loss from the specimens which produced voids and resulted in strength degradation. Fig-4 shows the compressive strength of geopolymer concrete specimen at different temperatures and different curing periods on 7th day. The curing temperature at 90ºC showed better compressive strength than 80ºC and 100ºC. Furthermore 90ºC-12 hours curing produced maximum strength when compared with 90ºC-24 hours curing. This showed that curing hours play an important role in achieving the compressive strength of geopolymer concrete. This was because polymerization process increases with increase in the temperature and at a high temperature the 2D-polymer chains are converted into 3D-polymer chain with strong bond. At the same time the higher temperature results in increase in the rate of development of strength. This was as same as observed in [13,14] and using high temperatures 90ºC-12 hours produces compressive strength 76.53MPa on 7th day as compared to [15] in which 28th day compressive strength was 52MPa.

Fig-5 shows that the compressive strength of GPC increased with increase in the temperature along with the curing hours from 80ºC to 90ºC for 12 hours curing on 3rd and 7th day of test. From the experiments it was observed that 7th day strength cured at 90ºC-12 hours produced maximum strength, which is the optimum temperature for the further study but when the curing period increased beyond 12 hours the compressive strength of geopolymer concrete decreased, this decrease in compressive strength would have been due to the continuous evaporation of moisture from the specimens. As the water content in geopolymer concrete was very less and when subjected to high temperature, there was loss of moisture on the surface which may have developed surface cracks, hence strength of geopolymer concrete decreased.

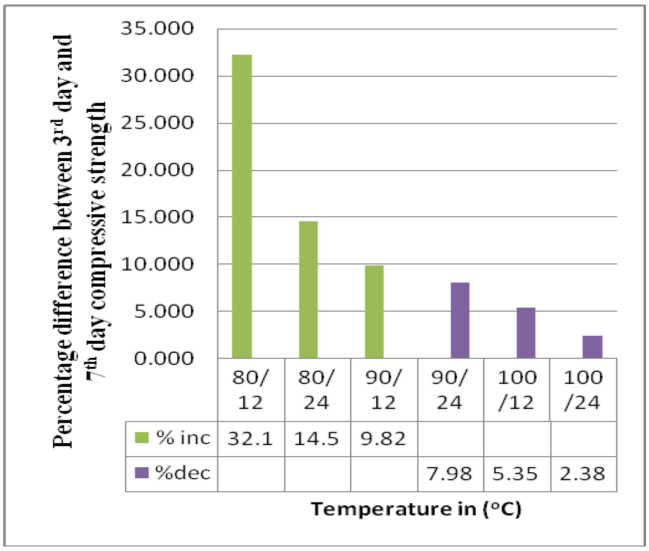

Fig-6 Variation in percentage change of compressive strength of GPC with varying temperatures and curing time

From fig-6 it is derived that, at 80º-12 hours the percentage increase in the strength from 3rd day to 7th day was 32.19%, at 80º-24 hours the percentage increase in the strength from 3rd day to 7th day was 14.55% and at 90º-12 hours the percentage increase in the strength from 3rd day to 7th day was 9.82%. This showed that increase in the temperature resulted in decrease in percentage difference from 3rd day to 7th day and also the increase in temperature up to an optimum temperature increased the rate of development of the strength. This was due to the degree of polymerization which directly depended on the temperature of curing, higher temperature resulted in the increase of degree of polymerization.

4.2 Effect of extra water & water to geopolymer solids ratio on the properties of Geopolymer concrete

In the present study, optimum temperature and curing hours which corresponded to maximum strength were 90^0-12 hours. When the water to geopolymer solids were kept as 0.201 the mix was not workable, but as per [12] when water to geopolymer solids was 0.20 the concrete showed moderate workability. The reason for this would have been the total aggregate to water ratio, coarse aggregate to water ratio and the size of coarse aggregate. As per the tests conducted by [12] the total aggregate to water ratio and the coarse aggregate to water ratio were taken as 21 and 14.7 respectively. But in the present study the total aggregate to water ratio and coarse aggregate to water ratio were taken as 15.31 and 9.96 and moreover half of the binder was replaced by GGBS which was much finer than the fly ash particles, hence these were the reasons for getting lower workability at water to geopolymer solids ratio of 0.201. Hence, further study was carried to make workable mix of geopolymer concrete by addition of extra water, this extra water was calculated by varying the water to geopolymer solids ratio and is tabulated in Table-9. The results of the slump and compressive strength of geopolymer concrete are tabulated in Table-9.

Table -9 : Mean Compressive Strength of GPC Specimens varying with addition of extra water and water to geopolymer solids ratio at an optimum temperature 90^{0}C-12 hours on 7th day

Sample Name	Density (kg/m^3)	Compressive Strength (N/mm^2)	Water to Geopolymer Solids ratio	Extra Water (kg/m^3)	Optimum Temperature (^{0}C) and optimum curing hours	Slump (mm)	% Decrease in Compressive Strength
GPC3	2434	46.53	0.2	0	90^0/12 hours	0	------
GPC7	2510	70.24	0.25	27	90^0/12 hours	10	8.22%
GPC8	2591	63.64	0.3	55	90^0/12 hours	68	16.84%
GPC9	2548	60.42	0.35	83	90^0/12 hours	126	21.05%
GPC10	2537	55.53	0.4	111	90^0/12 hours	170	27.44%

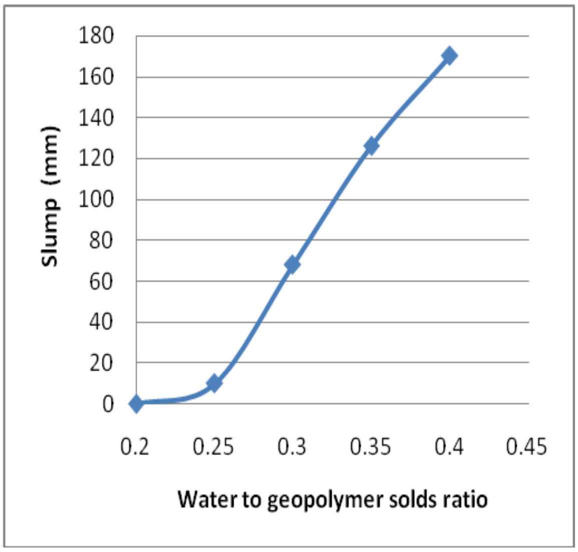

Fig-7 Water to geopolymer solids ratio Vs slump

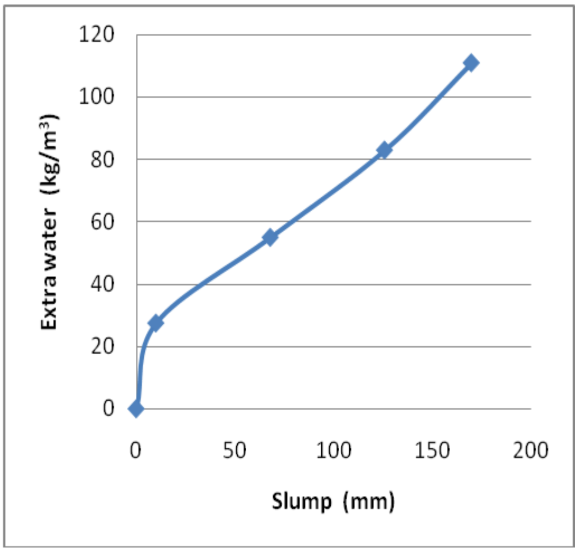

Fig-8 Variation of slump with extra water

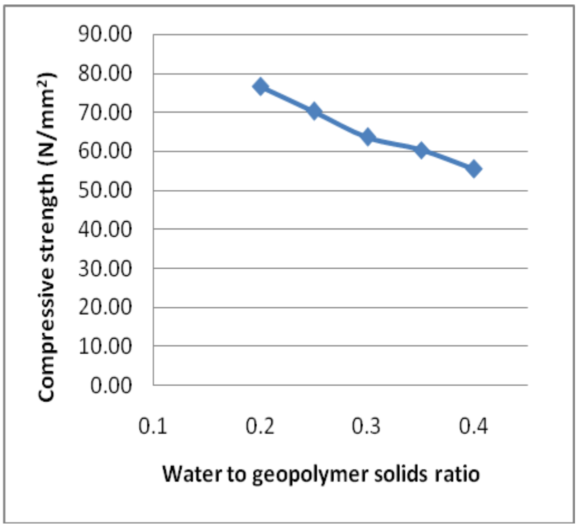

Fig-9 Variation of compressive strength with water to geopolymer solids ratio

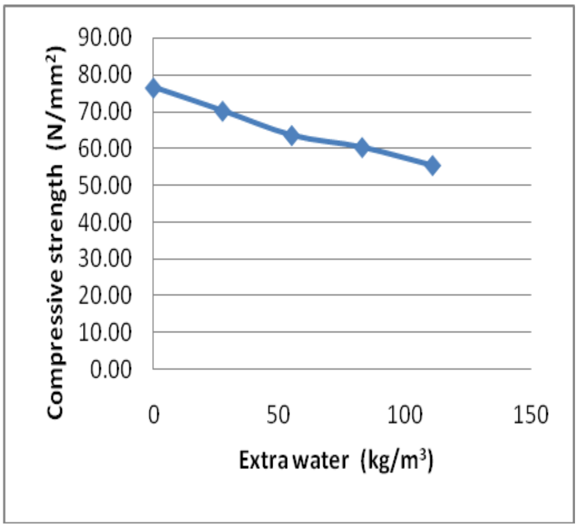

Fig-10 Variation of compressive strength with extra water

4.3 Effect of extra water and water to geopolymer solids ratio on the properties geopolymer concrete

From fig-7 and fig-8 it is clear that by increasing the water to geopolymer solids ratio and addition of extra water the workability of geopolymer concrete had increased as seen in Table-9. When the water to geopolymer solids ratio increased from 0.25 to 0.4 the corresponding slump was from 10 to 170 mm respectively. According to IS 456-2000 when slump is lesser than 25mm the concrete is said to be very low workable and when the slump is greater than 150mm it is said to be very high workable. In this study for water to geopolymer solids ratio of 0.3 slump obtained was 68 mm, hence this can be used for less reinforced concrete work and for water to geopolymer solids ratio of 0.35, the slump obtained was 126 mm, hence this can be used for congested reinforced concrete. Therefore by adjusting the water to geopolymer solids in the range 0.2 to 0.4, the desired slump for the desire work can be obtained.

From fig-9 and fig-10 it is clear that the compressive strength of geopolymer concrete depends on the water to geopolymer solids and extra water. Increasing the water to geopolymer solids ratio and extra water resulted in decrease of compressive strength of geopolymer concrete. This may be due to increase in the water content which resulted in the void formation after evaporation of water during curing process and also the increase in water resulted in the increase of H_2O to Na_2O ratio which further resulted in decrease of strength[13]. Based on the present study it is observed that high strength geopolymer concrete can be produced and strength can be achieved as early as in 7 days as compared to conventional concrete at 28 days. Hence it is much advantageous in fast track constructions.

V. CONCLUSIONS

Following conclusions were drawn from the experimental results of this study by varying curing temperature, curing hour, water to geopolymer solids ratio and extra water to achieve the compressive strength of 50MPa:

- The compressive strength of geopolymer concrete increases with increase in the curing temperature upto an optimum temperature of 90^0C & curing period of 12 hours to achieve the desired strength of 50MPa, beyond which the compressive strength of geopolymer concrete reduces.
- For the geopolymer solids to water ratio of 0.2-0.4, when extra water of 0-111kg/m^3 is added, the slump value increases from 0-170mm, while the compressive strength decreases from 0-27.44% respectively. Hence the design of GPC mix can be made for desired workability and compressive strength at the cost of extra water.

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