

Investigation on Mechanical Properties of Low Calcium Fly Ash and Slag based Geopolymer Concrete

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Abstract: Geopolymer is emerging as future cement as it not only meets the critical properties of cement but also falls under category of sustainability. An objective of this paper is aimed to investigate the compressive, tensile and flexural strength of low calcium fly ash (Class-F) and slag based geopolymer concrete for G50 which is equivalent to M50 grade of concrete. The alkaline solution used for the present study is combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH in the range of 12 to 18 molarity). The ratio of Na_2SiO_3 to NaOH is 2.5 and SiO_2 to Na_2O is 2.09 has been used since the compressive strength is maximum at these ratios. The test specimens were cast and after one day rest period, half of the specimens were cured in an oven at 60°C for 24 hours and the remaining period cured in sun light until the testing is done and remaining half of the specimens were ambient cured. The specimens were tested on 3rd, 7th and 28th day according to codal procedures. The comparative study is done among oven curing, ambient curing and control mix for compressive, tensile and flexural strength of concrete. From the test results it is observed that the compressive strength of G50 grade of geopolymer concrete was slightly increased compared to control mix of M50 grade and there is a slightly lower result in case of split tensile strength but there is no much variation in flexural strength.

Keywords: Geopolymer Concrete, Oven Curing, Compressive, Tensile and Flexural Strength

I. INTRODUCTION

After wood, concrete is the most often used material by the community. Concrete is conventionally produced by using the ordinary Portland cement (OPC) as the primary binder. 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 one ton for every ton of OPC produced. In addition, the amount of energy required to produce OPC is only next to steel and aluminium.

On the other hand, the abundance and availability of fly ash worldwide create opportunity to utilise this by-product of burning coal, as partial replacement or as performance enhancer for OPC. Fly ash in itself does not possess the binding properties, except for the high calcium or ASTM Class C fly ash. However, in the presence of water and in ambient temperature, fly ash reacts with the calcium hydroxide during the hydration process of OPC to form the calcium silicate hydrate (C-S-H) gel. This pozzolanic action happens when fly ash is added to OPC as a partial replacement or as an admixture. The development and application of high volume fly ash concrete, which enabled the replacement of OPC up to 60-65% by mass (Malhotra 2002; Malhotra and Mehta 2002), can be regarded as a landmark in this attempt.

In another scheme, pozzolanas such as blast furnace slag and fly ash may be activated using alkaline liquids to form a binder and hence totally replace the use of OPC in concrete. In this case the main constituents to be activated with high alkaline solution are mostly the silicon and the aluminium present in the by-product material such as low calcium fly ash (ASTM Class F) (Palomo, Grutzeck et al. 1999). The binder produced in this case is due to polymerisation. Several research publications were available regarding geopolymer pastes and geopolymer coating materials (Davidovits 1991; Davidovits 1994; Davidovits et al. 1994; Balaguru, Kurtz et al. 1997; van Jaarsveld, van Deventer et al. 1997; Balaguru 1998; van Jaarsveld, van Deventer et al. 1998; Davidovits 1999; Kurtz, Balaguru et al. 1999; Palomo, Grutzeck et al. 1999; Barbosa, MacKenzie et al. 2000). However, not a great deal was known regarding using the geopolymer technology to make fly ash based geopolymer concrete. The research reported in this paper is dedicated to investigate the process of making fly ash and slag based geopolymer concrete and the short-term engineering properties of the fresh and hardened concrete.

II. MATERIALS

2.1 Ordinary Portland Cement

In the experimental investigations, 53-grade of ordinary Portland cement of Ultra-tech Brand is used. The cement thus procured was tested for physical properties in accordance with the IS: 4031-1968 and found to be conforming various specifications of IS 12629-1987.

Table-1: Chemical Composition of Cement (Source: www.cement.org)

S.NO.	Constituent	Percentage
1	CaO	63.70
2	SiO ₂	22.00
3	Al ₂ O ₃	4.25
4	Fe ₂ O ₃	3.40
5	MgO	1.50
6	SO ₃	1.95

Table 2: Physical Properties of Ordinary Portland Cement of 53 Grade

S.No	Characteristics /Properties	Test Results	Requirements as per IS 12269-1987
1	Normal consistency	33%	----
2	Specific gravity	3.01	3.0 to 3.2
3	Setting time Initial setting time Final setting time	35 min 550 min	Not less than 30 minutes Not more than 600 minutes.
4	Soundness-Lechatlier method	1.55	Not more than 10 mm
5	Fineness of cement by sieving through sieve No.9(90 microns) for a period of 15 minutes	4%	<10%
6	Compressive strength at 28 days	55	----

2.2 Fine Aggregate

In the present investigation, fine aggregate used is obtained from local sources. The sand is made free from clay matter, silt, and organic impurities and sieved on 4.75mm IS sieve. The physical properties of fine aggregate like specific gravity, bulk density, gradation and fineness modulus are tested in accordance with IS: 2386 and the results are shown in table 3, 4 and 5. Grain size distribution of sand shows it is close to Zone II of IS 383-1970.

Table 3: Physical Properties of Fine Aggregate

S.No	Properties	Test Results	
1	Specific Gravity	2.63	
2	Bulk Density	Loose	1597 Kg/m ³
		Compacted	1725 Kg/m ³

Table 4 : Sieve Analysis of Fine Aggregate

Quantity of fine aggregate for sieve analysis = 1000gms

S.No	IS Sieve No	Weight Retained (gm)	Percentage Weight Retained	Cumulative Percentage Weight retained	Percentage Weight passed
1	40mm	0	0	0	100
2	20mm	0	0	0	100
3	10mm	0	0	0	100
4	4.75mm	3.5	0.35	0.35	99.65
5	2.36mm	15	1.5	1.85	98.15
6	1.18mm	96	9.6	11.45	88.55
7	600 μ	430	43	54.45	45.55
8	300 μ	420.5	42.05	96.5	3.5
9	150 μ	35	3.5	100	0
Total				264.6	

$$\begin{aligned} \text{Fineness modulus of fine aggregate} &= \text{Cumulative percentage retained}/100 \\ &= 264.6/100 = 2.65 \end{aligned}$$

Table 5 :IS Grading Requirements for Fine Aggregate

Sieve	Percentage by weight passing sieves IS:383-1970			
	Grading Zone I	Grading Zone II	Grading Zone III	Grading Zone IV
10mm	100	100	100	100
4.75mm	90-100	90-100	90-100	95-100
2.36mm	60-95	75-90	85-100	95-100
1.18mm	30-70	55-90	75-100	90-100
600 μ	15-34	35-59	60-79	80-100
300 μ	5-20	8-30	12-40	15-50
150 μ	0-10*	0-10*	0-10*	0-10*

* For crushed stone sand the permissible limit is increased to 20%

2.3 Coarse Aggregate

The crushed angular aggregate of 20mm maximum size obtained from the local crushing plants is used as coarse aggregate in the present study. The physical properties of coarse aggregate such as specific gravity, bulk density, flakiness and elongation index are tested in accordance with IS: 2386-1963. The results of coarse aggregate are shown in the table 6. The presence of elongated and flaky particles is 20% and 16.47% of the weight of the coarse aggregate. This shows that the coarse aggregate used in the concrete mixes is considered desirable as the indices are within 10-25%.

Table 6 : Physical Properties of Coarse Aggregate

S.No	Properties	Test Results	
1	Specific Gravity	2.71	
2	Bulk Density Kg/m ³	Loose	1597 Kg/m ³
		Compacted	1725 Kg/m ³
3	Elongation Index (%)	20	
4	Flakiness Index (%)	16.47	

2.4 Fly Ash

In the present study of work, the Class F-fly ash is used, which is obtained from Vijayawada thermal power station in Andhra Pradesh. The specific surface area of fly ash is found to be 4750 cm²/gm by Blain's Permeability Apparatus. The typical composition of fly ash and chemical requirements are shown in table 7 and 8 respectively.

Table 7: Typical Oxide Composition of Fly Ash

S.NO.	Constituent	Percentage
1	CaO(Lime)	0.7-3.6
2	SiO ₂ (Silica)	49-67
3	Al ₂ O ₃ (Alumina)	16-28
4	Fe ₂ O ₃ (iron oxide)	4-10
5	MgO(magnesia)	0.3-2.6
6	SO ₃ (Sulphur trioxide)	0.1-1.9
7	Surface area m ² /kg	230-600

Table 8: Chemical Requirement of Fly Ash (IS: 3812-part 1 2003)

S.N O.	Characteristics (Percent by mass)	Minimum Requirement in %	Composition of VTPS fly ash in %
1	SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃	70	86.75
2	SiO ₂	35	54
3	Reactive Silica	20	25
4	MgO	5	7
5	SO ₃ (Sulphur trioxide)	3	6
6	Available alkali as sodium oxide (Na ₂ O)	1.5	2.16
7	Loss of ignition	5	7.23

2.5 Ground Granulated Blast Furnace Slag

Ground Granulated Blast Furnace Slag (GGBS) shown in fig 2 is a byproduct of the steel industry. Blast furnace slag is defined as "the non-metallic product consisting essentially of calcium silicates and other bases that is developed in a molten condition simultaneously with iron in a blast furnace". About 15% by mass of binders was replaced with GGBS.

Table: 9 Chemical Compositions of GGBS

S.No	Constituent	Percentage
1	Silicon dioxide (SiO ₂)	33.2
2	Alumina tri-oxide (Al ₂ O ₃)	18.3
3	Ferric oxide (Fe ₂ O ₃)	0.6

4	Calcium oxide (CaO)	32.9
5	Magnesium Oxide (MgO)	11.6
6	Sulphur tri-oxide (SO ₃)	1.0
7	Potassium oxide (K ₂ O)	0.91
8	Sodium oxide (Na ₂ O)	0.21
9	Chlorides (Cl)	0.006

Table: 10 Physical Properties of GGBS

S No	Characteristics	Result
1.	Colour	Dull white
2.	Fineness(Blaine's) m ² /kg	450
3.	Specific Gravity	2.91
4.	Glass content percent	93
5.	Bulk Density kg/m ³	1100

2.6 Water

Water free from chemicals, oils and other forms of impurities is to be used for mixing of concrete as per IS: 456:2000.

2.7 Geopolymers

Geopolymers are member of the family of inorganic polymers, and are a chain structures formed on a backbone of Al and Si ions. The chemical composition of this geopolymer material is similar to natural zeolitic materials, but they have amorphous microstructure instead of crystalline (Palomo, Grutzeck et al. 1999; Xu and van (Deventer 2000).

2.7.1 Constituents of Geopolymer

2.7.1.1 Source Materials

Any material that contains mostly Silicon (Si) and Aluminium (Al) in amorphous form is a possible source material for the manufacture of geopolymer. Several minerals and industrial by-product materials have been investigated in the past. Low calcium fly ash (ASTM Class F) is preferred as a source material than high calcium (ASTM Class C) fly ash. The presence of calcium in high amount may interfere with the polymerisation process and alter the microstructure (Gourley 2003). On the nature of the source material, it was stated that the calcined source materials, such as fly ash, slag, calcined kaolin, demonstrated a higher final compressive strength when compared to those made using non-calcined materials, for instance kaolin clay, mine tailings, and naturally occurring minerals (Barbosa, MacKenzie et al. 2000). However, Xu and van Deventer (Xu and van Deventer 2002) found that using a combination of calcined (e.g. fly ash) and non-calcined material (e.g. kaolinite or kaolin clay and albite) resulted in significant improvement in compressive strength and reduction in reaction time. Natural Al-Si minerals have shown the potential to be the source materials for geopolymerisation, although quantitative prediction on the suitability of the specific mineral as the source material is still not available, due to the complexity of the reaction mechanisms involved (Xu and van Deventer 2000). Among the by-product materials, only fly ash and slag have been proved to be the potential source materials for making geopolymers. The other characteristics that influenced the suitability of fly ash to be a source material for geopolymers are the particle size, amorphous content, as well as morphology and the origin of fly ash.

2.7.1.2 Alkaline Activators

The most common alkaline activator used in geopolymerisation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na₂SiO₃) or potassium silicate (Davidovits 1999; Palomo,

Grutzeck et al. 1999; Barbosa, MacKenzie et al. 2000; Xu and van Deventer 2000; Swanepoel and Strydom 2002; Xu and van Deventer 2002). The use of a single alkaline activator has been reported (Palomo, Grutzeck et al. 1999; Teixeira-Pinto, Fernandes et al. 2002), Palomo et al (1999) concluded that the type of activator plays an important role in the polymerisation process. Reactions occur at a high rate when the alkaline activator contains soluble silicate, either sodium or potassium silicate, compared to the use of only alkaline hydroxides. Xu and van Deventer (2000) confirmed that the addition of sodium silicate solution to the sodium hydroxide solution as the alkaline activator enhanced the reaction between the source material and the solution. Furthermore, after a study of the geopolymerisation of sixteen natural Al-Si minerals, they found that generally the NaOH solution caused a higher extent of dissolution of minerals than the KOH solution.

2.7.1.3 Superplasticiser

High range water reducing (Master Glenium B233) super plasticizer was used in the mixtures at the rate of 1.5% of fly ash to improve the workability of the fresh geopolymer concrete.

III. EXPERIMENTAL INVESTIGATION

3.1 General

An objective of this paper is aimed to investigate the compressive, tensile and flexural strength of low calcium fly ash (Class-F) and slag based geopolymer concrete for G50 which is equivalent to M50 grade of concrete. The alkaline solution used for the present study is combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH in the range of 12 to 18 molarity). The ratio of Na_2SiO_3 to NaOH is 2.5 and SiO_2 to Na_2O is 2.09 has been used since the compressive strength is maximum at these ratios. The set of test specimens of 3 cubes of $150\text{mm} \times 150\text{mm} \times 150\text{mm}$, 3 cylinders of $150 \times 300\text{mm}$ and 3 prisms of $100 \times 100 \times 500\text{mm}$ for each composition were cast for testing compressive, tensile and flexural strength respectively then after one day rest period, half of the specimens were cured in an oven at 60°C for 24 hours and for the remaining period cured under sun light until the testing is done and remaining half of the specimens were ambient cured. The specimens were tested on 3rd, 7th and 28th day according to codal procedures. The results are tabulated and the required comparative study is done.

Table 11: Properties of Na_2SiO_3 Solution

Specific gravity	1.57
Molar mass	122.06 gm/mol
Na_2O (by mass)	14.35%
SiO_2 (by mass)	30.00%
Water (by mass)	55.00%
Weight ratio (SiO_2 to Na_2O)	2.09
Molarity ratio	0.97

Table 12: Properties of NaOH

Molar mass	40 gm/mol
Appearance	White solid
Density	2.1 gr/cc
Melting point	318°C
Boiling point	1390°C
Amount of heat liberated when dissolved in water	266 cal/gr

Table 13: Mix Proportions for G50 grade of Geopolymer Concrete

Grade of GPC	G50
Fly ash (Kg/m^3)	410
Fine Aggregate (Kg/m^3)	554.4
Coarse Aggregate (Kg/m^3)	1293.6

NaOH solids out of 46.86 Kg/m ³ (M) concentration in Kg/m ³	12	16.92
	14	18.93
	16	20.81
	18	22.49
Na ₂ SiO ₃ (Kg/m ³)		117.14
Extra water (Kg/m ³)		45
Super plasticizer (GLENIUM)@ 1.5% (Kg/m ³)		6.15
Ratio of mix proportions		1:1.35:3.16
Liquid/binder ratio		0.40
Workability (mm)		50

Table 14: Mix Proportions of OPC Controlled Concrete
Expressed as Equivalent Proportions of GPC

Grade of Concrete	M50
Cement (Kg/m ³)	410
Fine Aggregate (Kg/m ³)	554.4
Coarse Aggregate (Kg/m ³)	1293.6
Super plasticizer (GLENIUM)@1.5% Kg/m ³	6.15
Ratio of mix proportions	1:1.35:3.16
W/C ratio	0.40
Workability (mm)	50

3.2 Mixing and Casting of Geopolymer Concrete

Geopolymer concrete can be manufactured by adopting the conventional concrete techniques used in the manufacture of Portland cement concrete. In the laboratory, the fly ash and the aggregates were first mixed together dry in a pan mixer for about three minutes. The alkaline liquid was mixed with the super plasticizer and extra water if any. The liquid component of the mixture was then added to the dry material and the mixing continued usually for another four minutes. The fresh concrete was cast and compacted by the usual methods used in the case of Portland cement concrete. The workability of the fresh concrete was measured by means of the conventional slump test.



Fig. 1 Shows Cubes after Casting



Fig. 2 Shows Ground Granulated Blast Furnace Slag (GGBS)

IV. TEST RESULTS

4.1 Compressive Strength

After several trials of geopolymer mixes with different composition of fly ash and GGBS, an optimum compressive strength of the cubes is achieved at 85 % fly ash and 15% GGBS is given in table 15. From the table 15 and fig 3 it is observed that as the molarity of NaOH increases until 16M, the compressive strength is increased then it is decreased. As the compressive strength of concrete is reached to target mean strength at 16M on 28th day so an optimum compressive strength is considered at this molarity and further tests are also conducted based on this molarity.

Table 15: Compressive Strength (MPa) of G50 grade of Oven and Ambient cured Geopolymer Concrete

	Geopolymer Concrete (Ambient Curing) 85% FA +15% GGBS				Geopolymer Concrete (Oven Curing) 85% FA +15% GGBS			
	12M	14M	16M	18M	12M	14M	16M	18M
3 Days	10.17	10.39	11.15	9.83	44.11	47.69	54.19	41.05
7 Days	22.57	24.41	26.89	21.98	47.08	50.62	55.63	44.42
28 Days	49.61	51.94	58.36	47.27	50.36	53.78	59.75	48.29

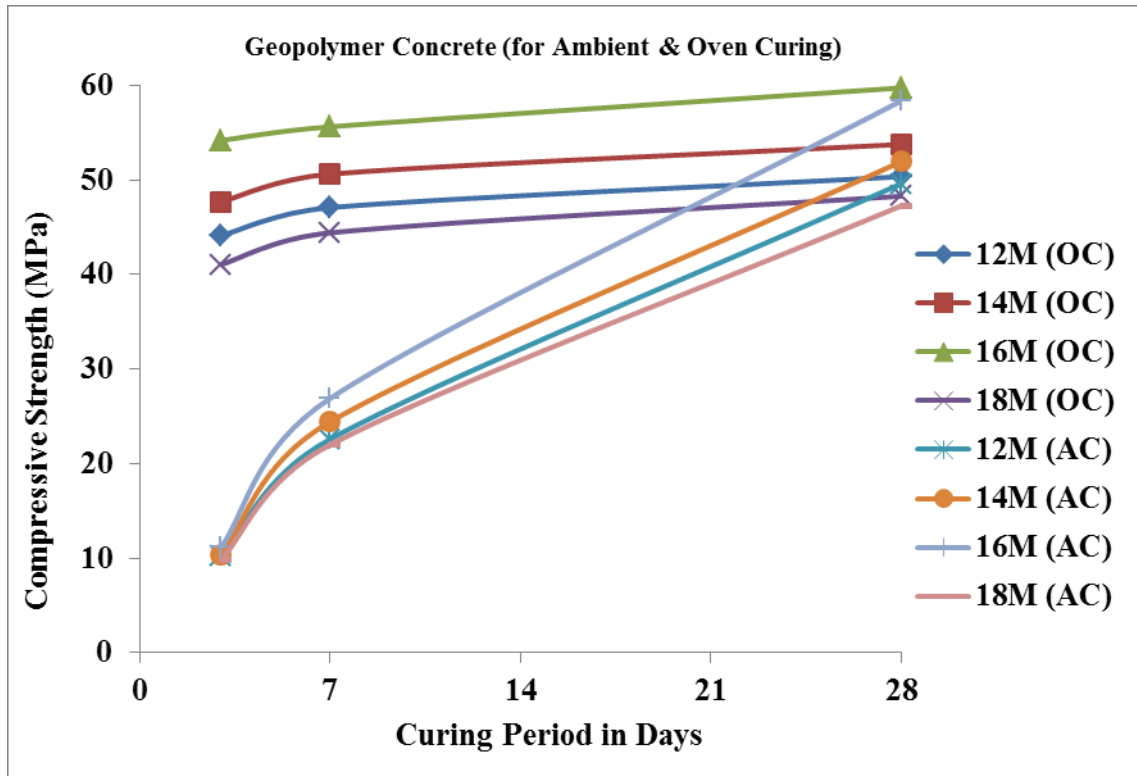


Fig. 3 shows Compressive Strength Vs Age of Concrete with various Molarity for Oven and Ambient Curing
 Table 16: Compressive Strength (MPa) of G50 equivalent to M50 grade of Concrete

	G50 (Ambient Curing)	G50 (Oven Curing)	M50 (Controlled Concrete)
3 Days	11.15	54.19	28.92
7 Days	26.89	55.63	41.07
28 Days	58.36	59.75	58.42

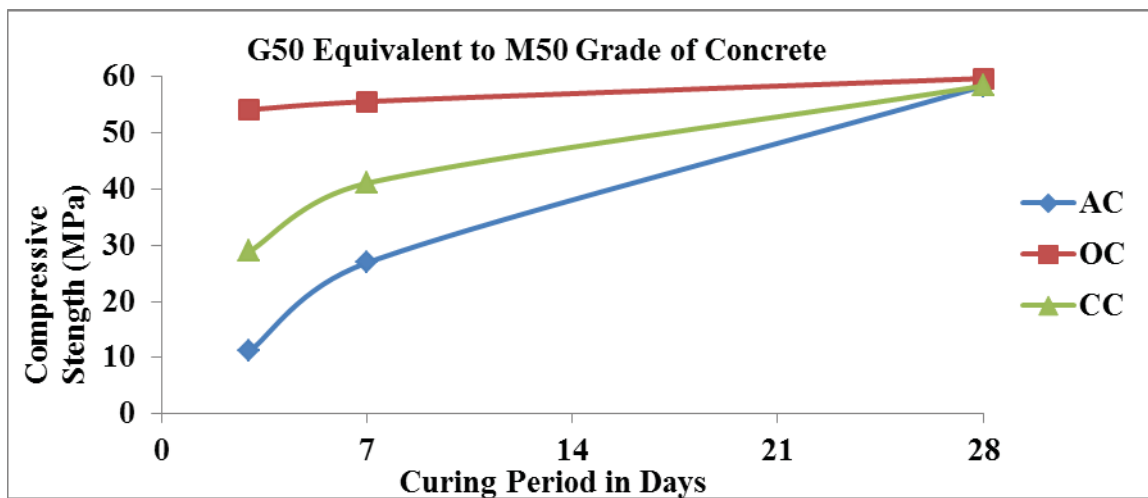


Fig. 4 shows Compressive Strength Vs Age of Concrete for various curing methods

4.2 Split Tensile Strength

After several trials of geopolymer mixes with different composition of fly ash and GGBS, an optimum compressive strength of the cubes is achieved at 85 % fly ash and 15% GGBS. Based on this composition the cylindrical specimens of 150x300mm were cast and tested on 3rd, 7th and 28th day. From the table 17 and fig 5 it is observed that split tensile strength of geopolymer concrete is slightly lower than the controlled concrete.

Table 17: Split Tensile Strength (MPa) of G50 of Geopolymer and M50 of Controlled Concrete

	G50 (Ambient Curing)	G50 (Oven Curing)	M50 (Controlled Concrete)
3 Days	0.68	3.13	2.12
7 Days	1.57	3.20	2.77
28 Days	3.41	3.52	3.85

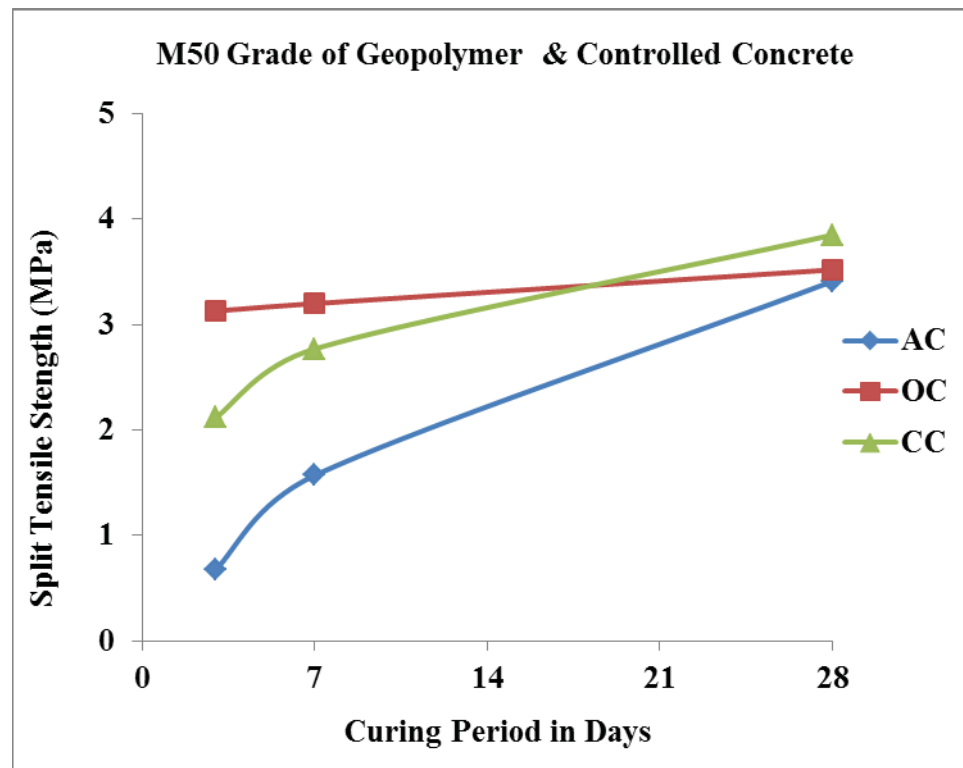


Fig. 5 shows Split Tensile Strength Vs Age of Concrete for various curing methods

4.3 Flexural Strength

After several trials of geopolymer mixes with different composition of fly ash and GGBS, an optimum compressive strength of the cubes is achieved at 85 % fly ash and 15% GGBS. Based on this composition the prism specimens of 100x100x500mm were cast and tested on 3rd, 7th and 28th day. From the table 18 and fig 6 it is observed that there is no much difference in flexural strength of geopolymer concrete compared to controlled concrete.

Table 18: Flexural Strength (MPa) of G50 of Geopolymer and M50 of Controlled Concrete

	G50 (Ambient Curing)	G50 (Oven Curing)	M50 (Controlled Concrete)
3 Days	1.17	4.85	2.99
7 Days	2.52	5.19	4.01
28 Days	5.59	5.71	5.65

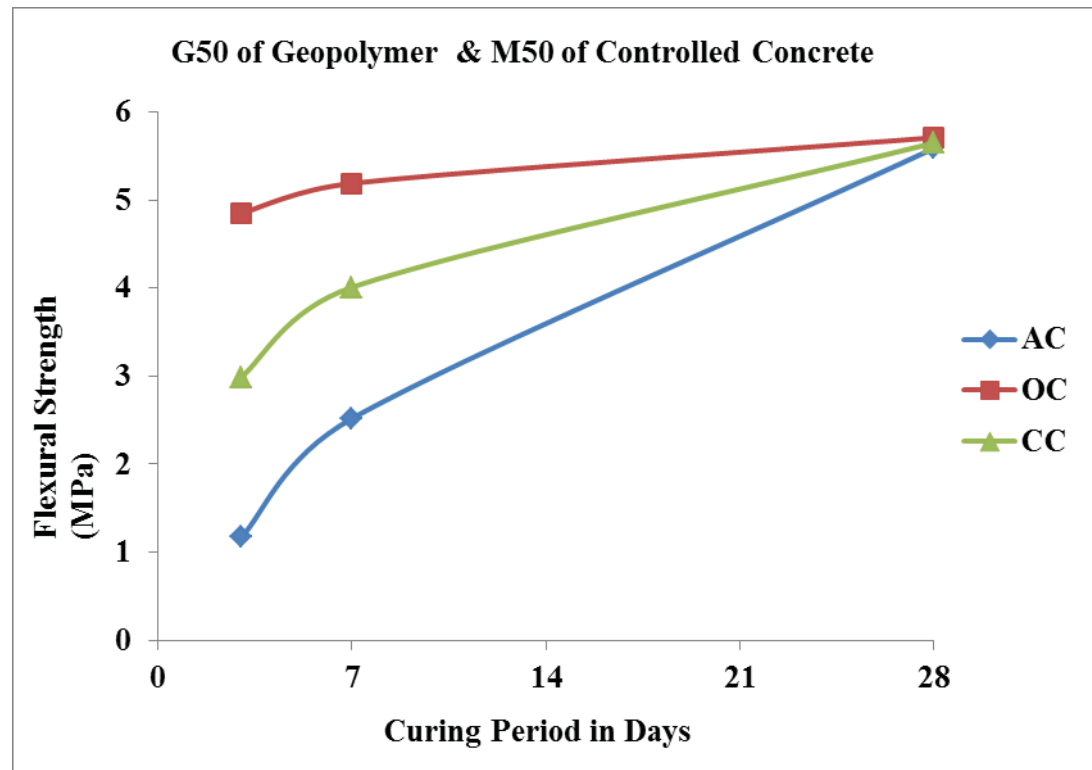


Fig. 6 shows Flexural Strength Vs Age of Concrete for various curing methods

V. CONCLUSIONS

The following specific conclusions can be drawn from the present experimental investigation

- i. It is clear that as the molarity of NaOH increases from 12 to 16M, the compressive strength is increased and from 16 to 18M it is decreased.
- ii. It is observed that an optimum compressive strength of the cubes is achieved at 85 % fly ash and 15% GGBS at 16 molarity since the compressive strength is reached to target mean strength on the 28th day.
- iii. The attainment of compressive strength is very early in the case of oven cured geopolymer concrete compared to controlled concrete by 87%, 35% on 3rd and 7th day respectively.
- iv. It is observed that split tensile strength of geopolymer concrete is slightly lower than the controlled concrete.
- v. It can be said that there is no much difference in flexural strength of geopolymer concrete compared to controlled concrete.

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