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Development and Optimization of Mix Design Of Low Calcium Fly Ash and Slag Based Geopolymer Concrete for Standard Grade

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Abstract: *Geopolymers are members of the family of inorganic polymers. The chemical composition of the geopolymer material is similar to natural zeolitic materials, but the microstructure is amorphous instead of crystalline. The polymerisation process involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals and that meets not only the critical properties of cement but also falls under category of sustainability. An objective of this paper is to develop and optimization of mix design for G30 and G50 grades of low calcium fly ash (Class-F) and slag based geopolymer concrete which is equilent to M30 and M50 respectively. The development of mix design is based on many factors such as alkaline liquid to fly ash ratio, Na_2SiO_3 to NaOH ratio, molarity of NaOH, type of curing methods, temperature and rest period etc. The concrete mix is designed by using all the above parameters and specimens were casted then tested on 3^{rd}, 7^{th} and 28^{th} day according to codal procedures. The comparative study is done on compressive strength for all the parameters and an optimum compressive strength has been selected for both G30 and G50 which is equivalent to M30 and M50 respectively.*

Keywords: *Geopolymer Concrete, Compressive Strength, Oven Curing, Alkaline Liquid and Molarity*

I. Introduction

Concrete usage around the world is second only to water. Ordinary Portland cement (OPC) is conventionally used as the primary binder to produce concrete. 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 extent of energy required to produce OPC is only next to steel and aluminium.

On the other hand, the abundant availability of fly ash worldwide creates opportunity to utilise this by-product of burning coal, as a substitute for OPC to manufacture concrete. When used as a partial replacement of OPC, 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. The development and application of high volume fly ash concrete, which enabled the replacement of OPC up to 60% by mass, is a significant development.

In 1978, Davidovits (1999) proposed that binders could be produced by a polymeric reaction of alkaline liquids with the silicon and the aluminium in source materials of geological origin or by-product materials such as fly ash and rice husk ash. He termed these binders as geopolymers. Palomo et al (1999) suggested that pozzolans such as blast furnace slag might be activated using alkaline liquids to form a binder and hence totally replace the use of OPC in concrete. In this scheme, the main contents to be activated are silicon and calcium in the blast furnace slag. The main binder produced is a C-S-H gel, as the result of the hydration process.

In 2001, when this research began, several publications were available describing geopolymer pastes and geopolymer coating materials. However, very little was available in the published literature regarding the use of geopolymer technology to make low calcium (ASTM Class F) fly ash-based geopolymer concrete.

This research is therefore dedicated to the development, the manufacture, and the engineering properties of the fresh and hardened concrete of low-calcium fly ash (ASTM Class F) and slag based geopolymer 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.

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 1, 2 and 3. Grain size distribution of sand shows it is close to Zone II of IS 383-1970.

Table 1: Physical Properties of Fine Aggregate

S.No	Properties		Test Results
1	Specific Gravity		2.63
2	Bulk Density	Loose	1597 Kg/m3
		Compacted	1725 Kg/m3

Table 2 : 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	600µ	430	43	54.45	45.55
8	300µ	420.5	42.05	96.5	3.5
9	150µ	35	3.5	100	0
Total				264.6	

Fineness modulus of fine aggregate = Cumulative percentage retained/100

= 264.6/100 = 2.65

Table 3:IS Grading Requirements for Fine Aggregate

Sieve	Percentage by weight passing sieves IS:383-1970			
IS	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
600µ	15-34	35-59	60-79	80-100
300µ	5-20	8-30	12-40	15-50
150µ	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 4. 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 4 : Physical Properties of Coarse Aggregate

S.No	Properties		Test Results
1	Specific Gravity		2.71
2	Bulk Density Kg/m3	Loose	1597 Kg/m3
		Compacted	1725 Kg/m3
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 cm^2/gm by Blain's

Permeability Apparatus. The typical composition of fly ash and chemical requirements are shown in table 5 and 6 respectively.

Table 5: 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_2O_3(Alumina)	16-28
4	Fe_2O_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 6: Chemical Requirement of Fly Ash (IS: 3812-part 1 2003)

S.NO.	Characteristics (Percent by mass)	Minimum Requirement in %	Composition of VTPS fly ash in %
1	$SiO_2 + Al_2O_3 + Fe_2O_3$	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_2O)	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: 7 Chemical Compositions of GGBS

S.No	Constituent	Percentage
1	Silicon dioxide (SiO_2)	33.2
2	Alumina tri-oxide (Al_2O_3)	18.3
3	Ferric oxide (Fe_2O_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_2O)	0.91
8	Sodium oxide (Na_2O)	0.21
9	Chlorides (Cl)	0.006

Table: 8 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 (Na_2SiO_3) 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 to develop and optimization of mix design for G30 and G50 grades of low calcium fly ash (Class-F) and slag based geopolymer concrete which is equilent to M30 and M50 respectively. The development of mix design is based on many factors such as alkaline liquid to fly ash ratio, Na_2SiO_3 to NaOH ratio, molarity of NaOH, type of curing methods, temperature and rest period etc. The concrete mix is designed by using all the above parameters and specimens were casted then tested on 3^{rd}, 7^{th} and 28^{th} day according to codal procedures. The alkaline solution used for the present study is combination of sodium silicate (Na_2Sio_3) and sodium hydroxide (NaOH in the range of 6 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 150mm*150mm*150mm for each composition were cast for testing compressive strength then after one day rest period, half of the specimens were cured in an oven at 60°C for 24hours 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 3^{rd}, 7^{th} and 28^{th} day according to codal procedures. The results are tabulated and the required comparative study is done.

Table 9: Properties of Na_2SiO_3 Solution

Specific gravity	1.57
Molar mass	122.06 gm/mol
Na_2O (by mass)	14.35%
SiO2 (by mass)	30.00%
Water (by mass)	55.00%
Weight ratio (SiO_2 to Na_2O)	2.09
Molarity ratio	0.97

Table 10: 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 11: Mix proportions for G30 & G50 grade of Geopolymer concrete

Grade of GPC		G30
Fly ash (Kg/m^3)		362
Fine Aggregate (Kg/m^3)		682.6
Coarse Aggregate (Kg/m^3)		1184.4
NaOH solids out of 46.54 Kg/m^3 (M) concentration in Kg/m^3	8	12.10
	10	14.61
	12	16.80
	14	18.80
Na_2SiO_3 (Kg/m^3)		116.36
Extra water (Kg/m^3)		20
Super plasticizer (GLENIUM B233)@ 1% (Kg/m^3)		3.62
Ratio of mix proportions		1:1.89:3.27
Liquid/binder ratio		0.45
Workability (mm)		50

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_2SiO_3 (Kg/m^3)		117.14
Extra water (Kg/m^3)		45
Super plasticizer (GLENIUM B233)@ 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 12: Mix Proportions of Controlled Concrete Expressed as Equivalent Proportions of GPC

Grade of Concrete	M30
Cement (Kg/m^3)	362
Fine Aggregate (Kg/m^3)	682.6
Coarse Aggregate (Kg/m^3)	1184.4
Super plasticizer (GLENIUM)@1% (Kg/m^3)	3.62
Ratio of mix proportions	1:1.89:3.27
W/C ratio	0.45
Workability (mm)	50

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 14. From the table 16 and fig 3 and 4 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 12M and 16M for G30 and G50 respectively on 28th day so an optimum compressive strength is considered at this molarity.

Table 13: Shows type of mix and composition of mix

Type of Mix	Composition of Mix
Mix1	100% fly ash
Mix2	95% FA+5%GGBS
Mix3	90% FA+10%GGBS
Mix4	85% FA+15%GGBS

Table 14: Compressive Strength (MPa) of G30 & G50 Grade of Oven Cured @ 60^{o}C Geopolymer Concrete

Grade of concrete	Type of Mix	3 days strength				7 days strength				28 days strength			
G30	Molarity	8M	10M	12M	14M	8M	10M	12M	14M	8M	10M	12M	14M
	Mix1	23.88	25.01	28.43	30.52	25.25	26.61	30.06	32.47	27.45	29.08	32.68	35.29
	Mix2	24.59	25.74	29.27	31.13	25.99	27.39	30.95	32.93	28.26	29.93	33.64	35.99
	Mix3	25.98	27.22	30.95	32.74	27.48	28.96	32.72	34.63	29.87	31.65	35.57	37.85
	Mix4	28.38	29.65	**33.45**	35.85	30.01	31.36	**35.29**	38.01	32.62	34.28	**38.45**	41.45
G50	Molarity	12M	14M	16M	18M	12M	14M	16M	18M	12M	14M	16M	18M
	Mix1	37.32	40.22	45.19	34.53	40.09	42.96	47.22	37.37	42.65	45.70	50.78	40.62
	Mix2	38.64	41.64	46.79	35.75	41.51	44.48	48.89	38.69	44.16	47.32	52.58	42.06
	Mix3	40.39	43.53	48.92	37.38	43.39	46.50	51.17	40.46	46.17	49.47	54.97	43.98
	Mix4	44.11	47.69	**54.19**	41.05	47.08	50.62	**55.63**	44.42	50.36	53.78	**59.75**	48.29

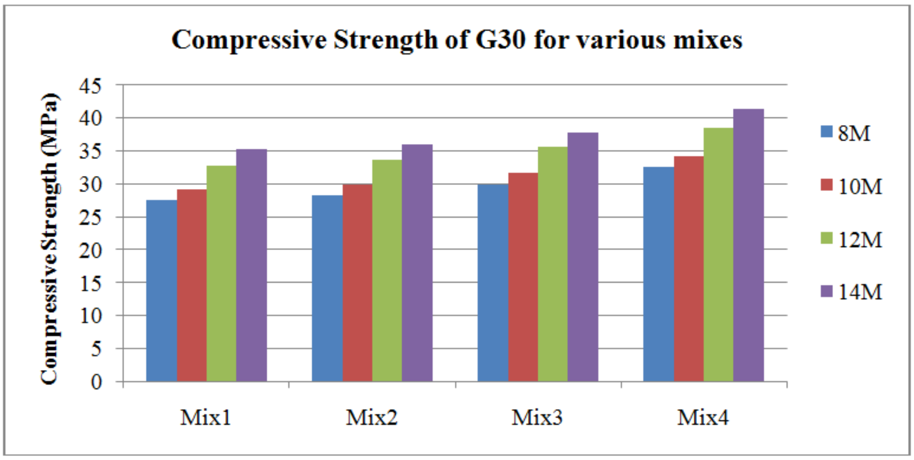

Fig. 3 shows Compressive Strength of G30 mixes for various molarities of NaOH

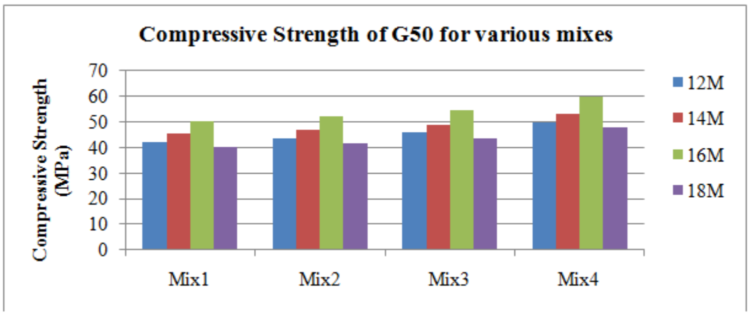

Fig. 4 shows Compressive Strength of G50 mixes for various molarities of NaOH

Table 15: Compressive Strength (MPa) of G30 grade of Oven and Ambient cured Geopolymer Concrete

	Ambient Curing				Oven Curing			
	85% FA +15% GGBS				85% FA +15% GGBS			
Molarity	8M	10M	**12M**	14M	8M	10M	**12M**	14M
3 Days	6.26	6.55	7.36	7.95	28.38	29.65	**33.45**	35.85
7 Days	14.69	15.39	17.29	18.67	30.01	31.36	**35.29**	38.01
28 Days	31.53	33.02	37.10	40.06	32.62	34.28	**38.45**	41.45

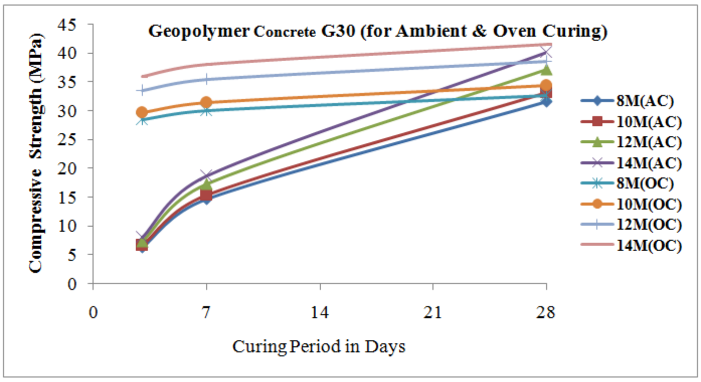

Fig. 5 shows Compressive Strength of G30 Vs Age of Concrete with various Molarity of NaOH for Oven and Ambient Curing

Table 16: 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			
Molarity	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

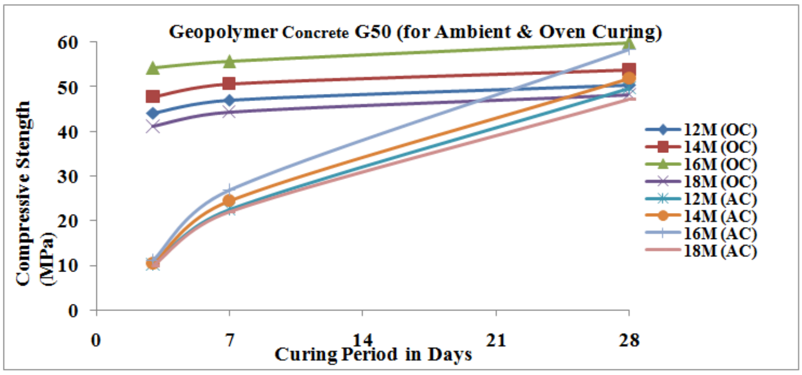

Fig. 6 shows Compressive Strength of G50 Vs Age of Concrete with various Molarity of NaOH for Oven and Ambient Curing

Table 17: Compressive Strength (MPa) of G30 equivalent to M30 grade of Concrete

Type of Curing	Ambient Curing	Oven Curing	Controlled Concrete
Grade of Concrete	G30	G30	M30
3 Days	7.36	**33.45**	19.45
7 Days	17.29	**35.29**	27.68
28 Days	37.10	**38.45**	38.62

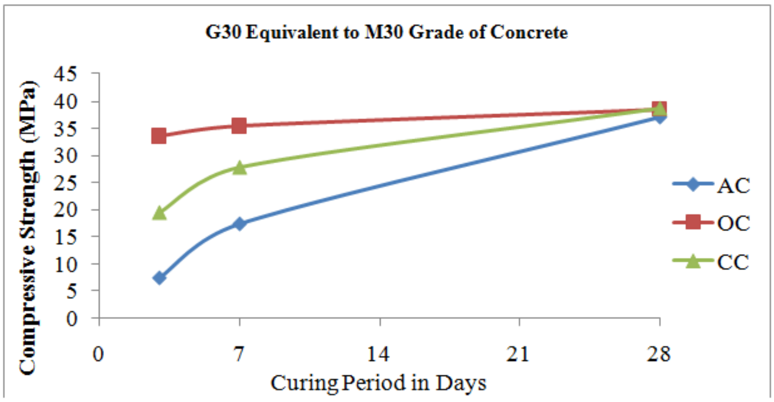

Fig. 7 shows Compressive Strength of G30 Vs Age of Concrete for various curing methods

Table 18: 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

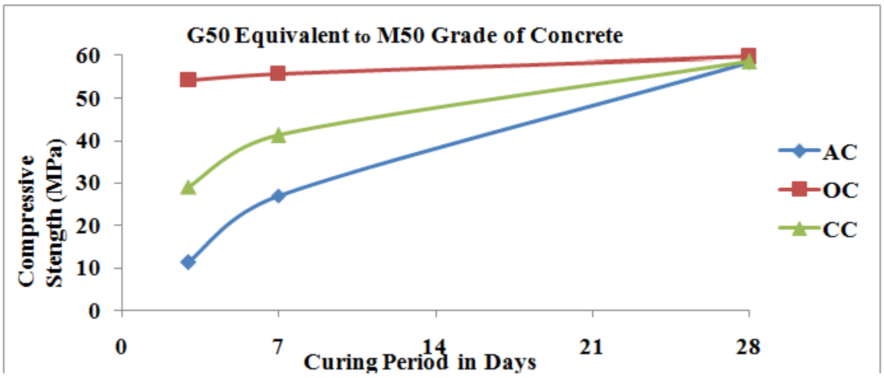

Fig. 8 shows Compressive Strength of G50 Vs Age of Concrete for various curing methods

Table 19: Compressive Strength (MPa) of G30 & G50 equivalent to M30 & M50 grade of Concrete

Type of Curing	Ambient Curing		Oven Curing		Controlled Concrete	
Grade of Concrete	G30	G50	G30	G50	M30	M50
3 Days	7.36	11.15	**33.45**	**54.19**	19.45	28.92
7 Days	17.29	26.89	**35.29**	**55.63**	27.68	41.07
28 Days	37.10	58.36	**38.45**	**59.75**	38.62	58.42

V. Conclusions

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

i. It is clear that alkaline liquid/fly ash ratio and molarity of NaOH are the governing factors in designing the geopolymer mix for various grades. The alkaline liquid/fly ash ratio of 0.45 & 0.4 and molarity of NaOH of 12M and 16M are suggested for G30 and G50 respectively.

ii. The ratio of Na_2SiO_3 to NaOH and SiO_2 to Na_2O is also very important factor in designing the mix. The ratio of Na_2SiO_3 to NaOH of 2.5 and SiO_2 to Na_2O of 2.09 are used for all grades since the maximum strengths are attained at this particular ratio.

iii. It is observed that an optimum compressive strength of the cubes is achieved at 85 % fly ash and 15% GGBS at 12 and 16 molaries for G30 and G50 respectively since the compressive strength is reached to target mean strength on the 28^{th} day.

iv. Low calcium fly ash and slag based geopolymer concrete has attained very early compressive strength under oven curing, it is about 86.6%, 91.3 % and 92.7%, 95.22 % of controlled concrete for G30 and G50 on 3rd day and 7th day respectively. So it can be used wherever early strength is required.

v. Is can be suggested that even though early strength is not attained under ambient curing, it can be used for structural applications as the 28th days strength is almost equal to oven curing of geopolymer and controlled concrete.

vi. 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.

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