

**EXPERIMENTAL INVESTIGATION ON STRENGTH PROPERTY OF LOW CALCIUM FLY ASH
BASED GEOPOLYMER CONCRETE**

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ABSTRACT

Considering the increase in demand for the developing of alternative construction materials due to growing environmental concerns. This paper is aimed to investigate mainly the compressive strength and workability of low calcium fly ash (Class-F) based Geopolymer concrete for M30 grade. Geopolymer G30 grade of concrete was investigated and it was compared with the control mix i.e.M30. The alkaline solution used for the present study is combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH in the range of 8 to 14 molarity). The ratio of Na_2SiO_3 to NaOH is 2.5 and SiO_2 to Na_2O is 2 has been used since the strength is maximum at these ratios. The test specimens were cast and cured in an oven at $60^\circ C$ for 24hours after a rest period of one, two and three days and then tested on 3rd, 7th and 28th day according to codal procedures. From the test results it is observed that the compressive strength of G30 grade of Geopolymer concrete was increased with the increase in molarity and rest period. The results are also compared to control mix of the respective grade of concrete and slight increase in strength is observed.

Keywords: Fly Ash, Geopolymer Concrete, Compressive strength, Oven curing, alkaline solution

1. Introduction

Soon after wood a new material with best strength came in to the world by the name called concrete. It is a usually used material by the group of people living together in a particular area and practicing common ownership. Concrete is conventionally produced by the binder called ordinary Portland cement (OPC). As we know the production of ordinary Portland cement (OPC) will arise several environmental issues. During the manufacture of 1tonne of Ordinary Portland Cement(OPC) will emit 0.8-1tonne of Carbon dioxide due to the calcination of limestone and combustion of fossil fuel.

On the other side, the plenty of availability of fly ash over the world create opportunity to utilize this product (by-product of burning coal), as performance enhancer or partial replacement in the place of OPC. The Fly ash which is directly from burning of coal will not possess any binding properties except for

high calcium or ASTM Class C fly ash. On the other hand, in the presence of water and surrounding temperature, fly ash reacts with $Ca(OH)_2$ (calcium hydroxide) during the hydration of OPC to form calcium silicate hydrate (C-S-H) gel. On the partial replacement of OPC with fly ash or an admixture will lead to pozzolanic action. So due to the application and development of high volume fly ash concrete, it has enabled the replacement of OPC up to (60%-65%) by mass (Malhotra 2002; Malhotra and Mehta 2002), and it can be taken 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



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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-based geopolymer concrete and the short-term engineering properties of the fresh and hardened concrete.

2. MATERIALS

2.1 Ordinary Portland Cement

In the experimental investigations, 53-grade of OPC (ordinary Portland cement) of Ultra-Tech Brand is used. The cement thus obtained was tested for the 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 research, fine aggregate which is used has obtained from local sources. This sand is made free from silt, organic impurities, and clay matter, then sieved on 4.75mm IS sieve. The physical properties of fine aggregate like gradation, fineness modulus, bulk density, and specific gravity, are tested in accordance with IS: 2386 and the results that are obtained were shown in table 3, 4 & 5. Grain size distribution of sand shows that the sand is close to Zone II of IS 383-1970.

2.3 Coarse Aggregate

The coarse aggregate which is crushed to a maximum size of 20mm and angularly shaped aggregate is obtained from the local crushing plants. The physical properties of coarse aggregate such as elongation index, flakiness, bulk density and specific gravity are tested in accordance with IS: 2386-1963. The results of coarse aggregate that are obtained in the tests are shown in the table 6.

The presence of flaky and elongated particles is 16.47% and 20% 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%.

2.4 Fly Ash

In the present study of work, the Class F-fly ash is used, which is obtained from NTPC (National Thermal Power Corporation) in Ramagundam, Telangana. By using Blain's Permeability Apparatus, it is found that the specific surface area of the fly ash is found to be 4750 cm²/gm. The typical composition of fly ash is shown in table 7 and chemical requirements are shown in table 8.

2.5 Water

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

2.6 Geopolymers

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

2.6.1 Constituents of Geopolymer



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2.6.1.1 Source Materials

For manufacturing of Geopolymer, we usually use the material which generally contains Silicon (Si) and Aluminium (Al) in amorphous form as a source material. In past, several minerals and industrial by-product materials have been considered and tested. ASTM Class F fly ash (Palomo, Grutzeck et al. 1999; Swanepoel and Strydom 2002), natural Al-Si minerals (Xu and van Deventer 2000), combination of calcined mineral and non-calcined materials (Xu and van Deventer 2002), Calcined kaolin or Metakaolin (Davidovits 1999; Barbosa, MacKenzie et al. 2000; Teixeira-Pinto, Fernandes et al. 2002), combination of fly ash and metakaolin (Swanepoel and Strydom 2002; van Jaarsveld, van Deventer et al. 2002), and combination of granulated blast furnace slag and metakaolin (Cheng and Chiu 2003) were investigated as source materials. Niche geopolymer product developers preferred metakaolin due to its high rate of dissolution in the reactant solution, and easier control on the (Si/Al) ratio and the white color (Gourley 2003). However, metakaolin is very expensive for making concrete in mass production state.

In fly ash there are different types of categories based on the percentage of calcium i.e. Low calcium fly ash (ASTM Class F) & High calcium fly ash (ASTM Class C), here low calcium fly ash (ASTM Class F) is preferred as source material. The presence of high amount of calcium may inhibit the process of polymerisation and change the microstructure (Gourley 2003). There are many source materials available in the nature and it was given that the calcined source materials i.e. fly ash, slag, calcined kaolin, are demonstrated with a higher final compressive strength when compared to those materials which are made with non-calcined source materials for example mine

tailings, naturally occurring minerals, kaolin clay (Barbosa, Mackenzie et al. 2000). However, Xu and van Deventer (Xu and van Deventer 2002) found it using a combination of calcined source material (e.g. fly ash) and non-calcined source material (e.g. kaolinite or kaolin clay and albite) were resulted in outstanding improvement in compressive strength and depletion in reaction time. Natural Alumina-silica minerals have shown the potential to be as a source material for the process of geopolymerisation, while the quantitative prediction on the suitability of the specific as the source material is still not available, because of the complexity of the mechanisms involved in the reactions (Xu and van Deventer 2000). In the available by-product materials, only slag and fly ash have been proved to be the potential source materials for the preparation of geopolymers. Due to its high reactivity which comes from the finer particle, fly ash is considered to be more advantageous than slag. Moreover, for the sake of geopolymer feedstock material fly ash is more desirable than slag. There were several studies that are done on the suitability of various types of fly ash, so as to use it as a geopolymer source material (i.e. Fernández-Jiménez and Palomo (2003). These researchers stated that, to produce optimal binding properties the low-calcium fly ash should have the amount of percentage of unburned material (LOI) less than 5%, low calcium oxide (CaO) content, Fe_2O_3 content must not exceed 10%, the content of relative silica should be in between 40-50%, and 80-90% of the particles should be smaller than $45\mu m$. On the contrary, van Jaarsveld et al (2003) found that fly ash with higher amount of calcium oxide (CaO) has produced geopolymer with higher compressive strength, due to the formation of calcium aluminate hydrate and other calcium compounds, especially in the early ages.



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2.6.1.2 Alkaline Activators

In geopolymerisation the most commonly used alkaline activator is prepared by the combination of potassium hydroxide (KOH) or sodium hydroxide (NaOH) and potassium silicate (K_2SiO_3) or sodium silicate (Na_2SiO_3) (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). By the use of single alkaline activator has been concluded that the type of activator plays major role in the process of polymerisation. When the alkaline activator contains soluble silicate (sodium or potassium silicate) then the reactions will occur at high rate when compared to use of only alkaline hydroxides (NaOH or KOH). It has been confirmed by Xu and van Deventer (2000) that, by the addition of sodium silicate (Na_2SiO_3) solution to the sodium hydroxide (NaOH) as the alkaline activator enhances the reaction between the solution and the source material. Moreover, after the study on geopolymerisation of sixteen natural Alumina-silica (Al-Si) minerals, they found that sodium hydroxide (NaOH) solution has caused higher extent of dissolution of minerals than potassium hydroxide (KOH) solution.

2.6.1.3 Superplasticizer

High range water reducing (Master Glenium B233) super plasticizer was used in the mixtures at the rate of 1% of fly ash to improve the workability of the fresh geopolymer concrete.

3. EXPERIMENTAL INVESTIGATION

3.1 General

This paper is aimed to investigate mainly the compressive strength and workability of low calcium fly ash (Class-F) based geopolymer concrete for M30 grade. Geopolymer G30 grade of concrete was investigated and it was

compared with the control mix i.e.M30. The alkaline solution used for the present study is combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH in the range of 8 to 14 molarity). The ratio of Na_2SiO_3 to NaOH is 2.5 and SiO_2 to Na_2O is 2 has been used since the strength is maximum at these ratios. The set of test specimens of 3 cubes of $150mm*150mm*150mm$ were cast for each composition and cured in an oven at $60^\circ C$ for 24 hours after a rest period of one, two and three days and then tested on 3rd, 7th and 28th day according to codal procedures. The results are tabulated in table 13 and the required comparative study is done.

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.

4. TEST RESULTS

4.1 Compressive Strength

Compressive strength of the cubes is given in table 13. From the table it is observed that as the molarity of NaOH and rest period increases the compressive strength is increased.

4.2 Workability



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The workability of concrete for G30 grade with different molarity of NaOH has been done and given in table 15. From this table it is seen that as the molarity of NaOH increases the workability is decreased.

5. CONCLUSIONS

- i. It is observed that as the molarity (8 to 14) of NaOH increases the compressive strength is increased.
- ii. It can be seen that as the rest period from one to three days increases the compressive strength is increased.
- iii. The workability of geopolymer concrete decreased as the molarity of NaOH increased.
- iv. The compressive strength of geopolymer concrete (G30) is slightly increased when it is compared with the conventional concrete i.e.M30 at 28 days, but the development of strength is very early in case of geopolymer concrete.
- v. The compressive strength of 14 Molarity is higher than the required target strength, so it is uneconomical because NaOH is costlier than Na_2SiO_3 . Hence the optimum compressive strength for G30 grade of concrete can be taken at 12 Molarity.

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List of Figures



Fig.1 Mixture of CA, FA, and Fly ash



Fig.2 Adding liquid component in Pan Mixer



Fig.3 Compacting using vibrator



Fig.4 Shows casting of cubes



Fig. 5 Shows curing of cubes at 60°C temperature in an oven



Fig.6 Compression Testing Machine

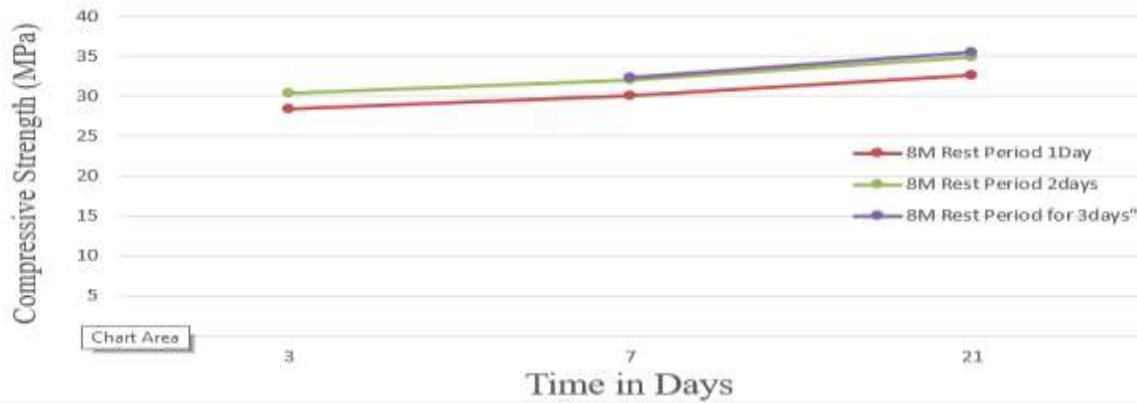


Fig.7 Shows graph of Compressive Strength Vs Time in Days for 8Molarity of NaOH

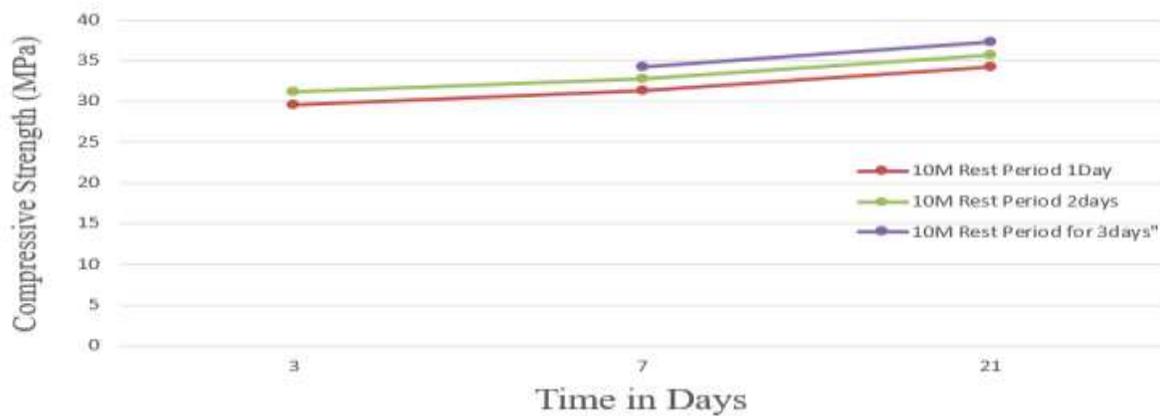


Fig.8 Shows graph of Compressive Strength Vs Time in Days for 10Molarity of NaOH

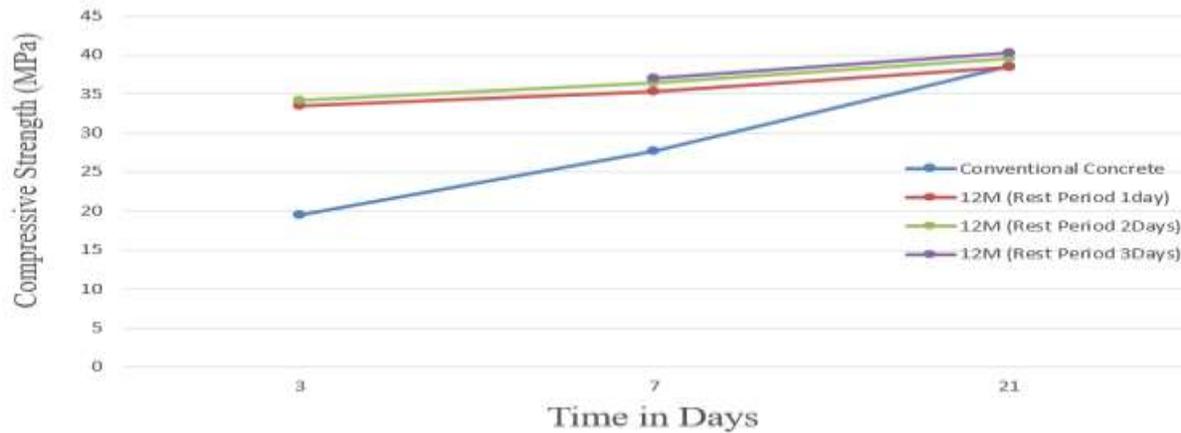


Fig.9 Shows graph of Compressive Strength Vs Time in Days for 12Molarity of NaOH

List of Tables

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	----

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 on 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%

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

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 %
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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

Table 9: Properties of sodium silicate solution

Specific gravity	1.6
Molar mass	122.06 gm/mol
Na ₂ O (by mass)	14.7%
SiO ₂ (by mass)	29.4%
Water (by mass)	55.90%
Weight ratio (SiO ₂ to Na ₂ O)	2
Molarity ratio	0.97

Table 10: Properties of sodium hydroxide solution

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 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	8	12.10
	10	14.61

(M) concentration in Kg/m ³	12	16.80
	14	18.80
Na ₂ SiO 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

Table 12: Mix proportions of OPC conventional 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

Table 13: Compressive strength of G30 grade of concrete when cured in an oven at 60°C

Molarity of NaOH	Rest period (in days)	3 rd day strength (MPa)	7 th day strength (MPa)	28 th day strength (MPa)
8	1	28.38	30.01	32.62
8	2	30.31	32.05	34.84
8	3	-	32.36	35.57
10	1	29.65	31.36	34.28
10	2	31.17	32.81	35.75
10	3	-	34.27	37.25



12	1	33.45	35.29	38.45
12	2	34.25	36.43	39.60
12	3	-	37.04	40.26
14	1	35.85	38.01	41.45
14	2	37.06	39.19	42.60
14	3	-	40.50	44.26

Table 14: Compressive strength of M30 grade of concrete when cured in water

3 rd day strength (MPa)	19.45
7 th day strength (MPa)	27.68
28 th day strength (MPa)	38.62

Table 15: Workability of G30 grade of concrete

Molarity of NaOH	Workability (slump in mm)
8	58
10	53
12	51
14	48