Durability of Geopolymer Concrete Based On Fly Ash Using Alkaline Solution ($Koh+K_2sio_3$)

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Abstract- Considering the increasing demand for developing alternative construction materials, due to the growing environmental concerns, this paper discusses the feasibility of alkali activated geo-polymer concrete, as a future construction material. The main objective of this study involves observation of structural behaviors of the fresh fly ash-based geo-polymer concrete, understanding the basic mixture proportioning of fly ash-based Geopolymer Concrete and evaluating various economic considerations.

Keywords- Alkali activated fly-ash based geo-polymer, construction material for green building, geo-polymer Concrete, eco-friendly construction material, low calcium based geo-polymer concrete.

1 Introduction

Concrete usage around the world is second only to water and Ordinary Portland Cement (OPC) is conventionally used as the primary binder to produce concrete. The environmental issues associated with the production of OPC are too many. The cement industry is held responsible for some of the CO_2 emissions. The amount of the carbon dioxide released during the manufacturing of OPC due to the calcinations 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 aluminum. The demand of Portland cement is increasing day by day and hence, efforts are being made in the construction industry to address this by utilizing supplementary materials and developing alternative binders in concrete; the application of geo-polymer technology is one such alternative.

Although the use of Portland cement is still unavoidable until the foreseeable future, many efforts are being made in order to reduce the use of Portland cement in concrete. These efforts include the utilization of supplementary cementing materials such as fly ash, silica fume, granulated blast furnace slag, rice-husk ash and metakaolin, and finding alternative binders to Portland cement.

Further, an environmentally compatible disposal of waste material by appropriate technologies is of increasing concern and imposes interesting technical challenges. Construction industry is the one where bulk utilization of waste materials can be effectively done without any compromise on quality performance. It has been established that fly ash can replace cement partially. It is essentially cement free concrete. The Geopolymer technology developed by Dr. Davidovits in the 1980s offers an attractive solution.

1.1 GEOPLOYMER

There are two main constituents of Geopolymer, namely the source materials and the alkaline liquids. The source materials for Geopolymer based on alumina-silicate should be rich in silicon (Si) and aluminum (Al). These could be natural minerals such as kaolinite, clays, etc. Alternatively, by-product materials such as fly ash, silica fume, slag, rice-husk ash, red mud, etc could be used as source materials. The choice of the source materials for making Geopolymer depends on factors such as availability, cost, type of application, and specific demand of the end users. The alkaline liquids are from soluble alkali metals that are usually sodium or potassium based. The most common alkaline liquid used in Geopolymerization is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate.

Geopolymer concrete utilizes an alternate material including fly ash as binding material in place of cement. It is essentially cement free concrete. This fly ash reacts with alkaline solution (e.g.KOH, NaOH) and Potassium Silicate (K_2SiO_3) to form a gel which binds the fine and coarse aggregates.

The schematic formation of Geopolymer material can be shown as described by Equation (1) and (2)

N (Si_2O_5,Al_2O_2) + $2nSiO_2$+$4nH_2O$+NaOH or KOH → Na^+,K^++ n$(OH)_3$-Si-O-Al—O-Si$(OH)_3$ (Si-Al materials)$(OH)_2$

(Geopolymer precursor) ------------- (1)

n $(OH)_3$-Si-O-Al—Si-$(OH)_3$ + NaOH or KOH (Na^+,K^+)-->(-Si-O-Al—O-Si-O-)+ $4nH_2O$ (OH)2 O-O-O

(Geopolymer backbone) ------------ (2)

Where; n = is the degree of poly-condensation or polymerization.

1.2 OBJECTIVE

- To determine the Optimum percentage of alkaline solution (KOH + K_2SiO_3) in Fly-ash for Geopolymer to be used.
- To determine the initial and final setting time of Geopolymer.
- To determine the Durability of Geopolymer concrete by following -
  a) Sulphate Resistance test
  b) Acid Resistance test
- To Determine the Correlation between OPC and GPC.

2 GEOPLOYMER CONCRETE

Geopolymer concrete is also known as Alkali-activated concrete or inorganic polymer concrete. Geopolymer is the most recently developed construction material for large scale utilization of fly ash without any cement. Compressive strength of fly ash based Geopolymer mortar depends on the strength of Geopolymer binder an excellent bonding between Geopolymer binder and aggregate.

Geopolymer concrete has excellent resistance to chemical attack and shows promise in the use of aggressive environments where the durability of Portland cement concrete may be of concern. This is particularly applicable in aggressive marine environments, environments with high carbon dioxide or sulphate rich soils. Similarly in highly acidic conditions, Geopolymer concrete has shown to have superior acid resistance and may be suitable for applications such as mining, some manufacturing industries and sewer systems. Commercial Geopolymer sewer pipes are in use today. Current research at Curtin University of Technology is examining the durability of precast box culverts manufactured from Geopolymer concrete which are exposed to a highly aggressive environment with wet-dry cycling in sulphate rich soils.

2.1 MATERIAL USED FOR GEOPOLYMER CONCRETE (GPC)

- Low Calcium Class F Type Fly Ash
- Potassium Hydroxide (98% Purity In Pure Form)
- Potassium Silicate Solutions (16M)

- Coarse Aggregate (10mm & 20mm)
- Fine Aggregate (Fineness Modulus 2.6 — 2.8)
- Distilled Water.

3 EXPERIMENTAL STUDIES

3.1MIX-PROPORTION OF GEOPOLYMER CONCRETE

The mixture proportion of concrete contains coarse aggregate, fine aggregate, fly ash, Potassium silicate solution and KOH solution. The mixtures with 16M were prepared and compressive strengths of these sample cubes were measured.

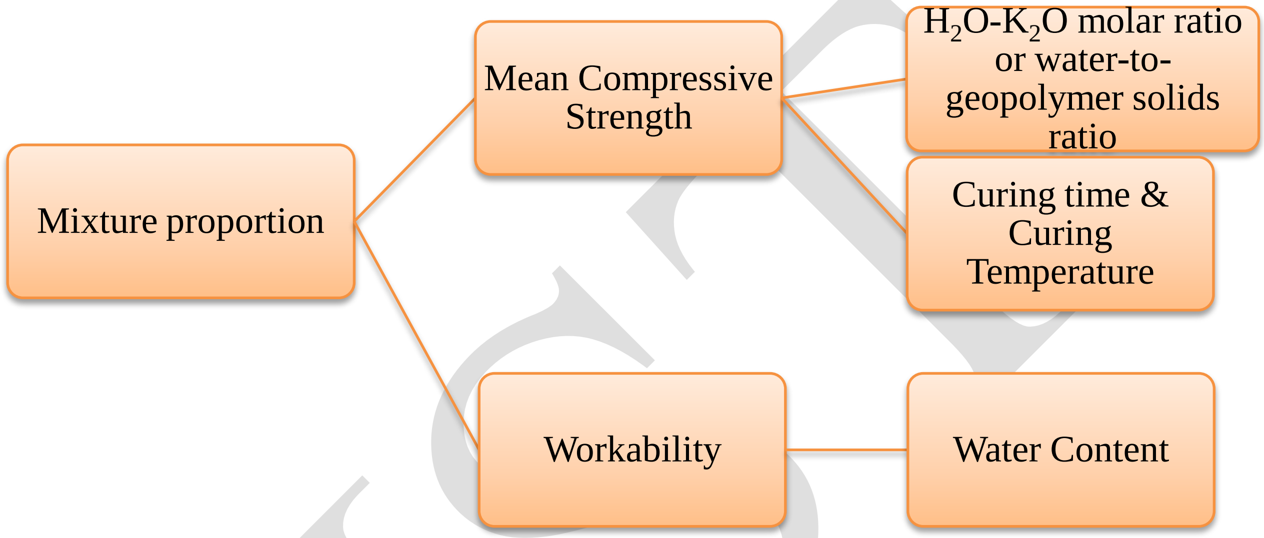

Figure 2 - Schematic Diagrams for Mixing Process

3.2 PREPARATION OF ALKALINE SOLUTION

The Potassium hydroxide (KOH) solids were dissolved in water to make the solution. The mass of KOH solids in a solution varied depending on the concentration of the solution expressed in terms of molar, M. For instance, KOH solution with a concentration of 16M consisted of 16x40 = 640 grams of KOH solids (in flake or pellet form) per litre of the solution, where 40 is the molecular weight of KOH.

1M = 1000gm of water + 40gm NaOH (40= molecular wt.)

Hence 14M Solution = 1000gm water + 40x14KOH
= 1000gm water + 560gm KOH
And 16M solution = 1000gm water + 16x40 KOH
= 1000gm water +640 KOH

3.3 MANUFACTURING OF GEOPOLYMER CONCRETE

Geopolymer concrete can be manufactured by adopting the conventional techniques used in the manufacture of Portland cement concrete. In the laboratory, the fly ash and the aggregates were first mixed together dry in 80-litre capacity pan mixer for about three minutes. The aggregates were prepared in saturated-surface-dry (SSD) condition. The alkaline liquid was mixed with the water. The liquid component of the mixture was then added to the dry materials and the mixing continued usually for another four minutes. The fresh concrete could be handled up to 120 minutes without any sign of setting and without any degradation in the compressive strength. The fresh concrete was cast and compacted by the usual methods used in the case of Portland cement concrete. Fresh fly ash-based geopolymer concrete was usually cohesive.

3.4 CURING OF SPECIMEN

Heat-curing of low-calcium fly ash-based geopolymer concrete is generally recommended. Heat curing substantially assists the chemical reaction that occurs in the geopolymer paste. Both curing time and curing temperature influence the compressive strength of geopolymer concrete. The test specimens were 150mmX150mmX150mm cubes heat-cured at 80^0C in an oven. The curing time varied from 4 hours to 96 hours (4 days). Longer curing time improved the polymerization process resulting in higher compressive strength. The rate of increase in strength was rapid up to 24 hours of curing time; beyond 24 hours, the gain in strength is only moderate. Therefore, heat curing time need not be more than 24 hours in practical applications.

3.5 CONSISTENCY, INITIAL AND FINAL SETTING TIME TEST

The initial and final setting time of geopolymer done according to IS: 4031 (Part 5) 1988.The geopolymer paste at curing temperature of 80^oC. The setting times of fly ash-based geopolymer mortar using Vicat needle. The needle used was 1.00 mm in diameter. In this case, fine aggregates were excluded from the mixture proportion. The fly ash and activator solution were mix in bowl. The Geopolymer paste was cast into the 40 mm height, 80 mm diameter conical mould in two layers.. The specimen was placed into the oven for curing at required elevated temperature, 60^oC. For every 5 minutes interval, the specimen was placed on the Vicat apparatus to measure the initial and final setting time.

3.6 COMPRESSIVE STRENGTH TEST

Compressive strength test was carried out in concrete cubes of size 150x150x150mm using 1:1.35:3 mix .Specimens with ordinary Portland cement concrete (control) were removed from the mould after 24h and subjected to water curing for 7 and 28 days. The geopolymer concrete specimens were prepared according to the method followed by Hardjito et. al. [2]. Geopolymer cubes of 16M were cast. During moulding, the cubes were mechanically vibrated.The specimens were wrapped by plastic sheet to prevent loss of moisture and placed in an oven. Since the process needs curing at high temperature, the specimens were cured at temperature of 80^0C for 24 h in the oven. They were then left at open air (room temperature 25^0C) in the laboratory until testing. Tests were carried out on triplicate specimens and average compressive strength values were recorded.

3.7 DURABILITY TEST

3.7.1 Acid Resistance

Acid resistance test was performed to determine the durability of samples. The 150x150x150 mm geopolymer concrete specimens were prepared and cured. After curing for 28 days, the specimens were taken out to measure the initial weights, and then transferred to 5% solution of Sulphuric acid (H_2So_4) and Phosphoric acid (H_3PO_4). The parameters investigated were the time and weight loss of fully immersed concrete specimens in the acid solution. The measurements of weight loss and compressive strength were performed at the age of 30 and 60 days.

3.7.2 Sulphate Resistance

Sulphate Attack test was performed to determine the durability of samples. The 150x150x150 mm geopolymer concrete specimens were prepared and cured. After curing for 28 days, the specimens were taken out to measure the initial weights, and then transferred to 5% solution of sodium sulphate (Na_2So_4) and magnesium sulphate ($MgSO_4$). The parameters investigated were the time and weight loss of fully immersed concrete specimens in the sulphate solution. The measurements of weight loss and compressive strength were performed at the age of 30 and 60 days.

4 TEST RESULTS

4.1 Specific Gravity

- Fly ash = 2.24

- Potassium silicate = 1.33
- Aggregates = 2.89
- sand = 2.68

4.2 CONSISTENCY, INITIAL AND FINAL SETTING TIME

Table 1- Consistency, Initial and final setting time of Geopolymer

Consistency (%)	65
Initial Setting Time (Min)	45
Final Setting Time (Min)	120

4.3 OPTIMUM PERCENTAGE OF ALKALINE SOLUTION (KOH + K_2SIO_3) IN FLY-ASH BASED GEOPOLYMER CONCRETE

Table 2 - The Compressive Strength of Geopolymer Concrete at 7 and 28 days

S. NO.	Ratio			A /FA	Alkaline ratio	Compressive Strength(Mpa)	
	Fly ash	Sand	Agg.		K2SiO3: KOH	7 days	28 days
1	1	1.40	3.5	0.8	2.51	22.3	30.4
2	1	1.40	3.5	0.7	2.51	20.5	28.4
3	1	1.35	3.5	0.8	2.51	26.5	34.2
4	1	1.35	3.0	0.8	2.51	30.2	41.05
5	1	1.35	3.0	0.7	2.51	22.2	32.4

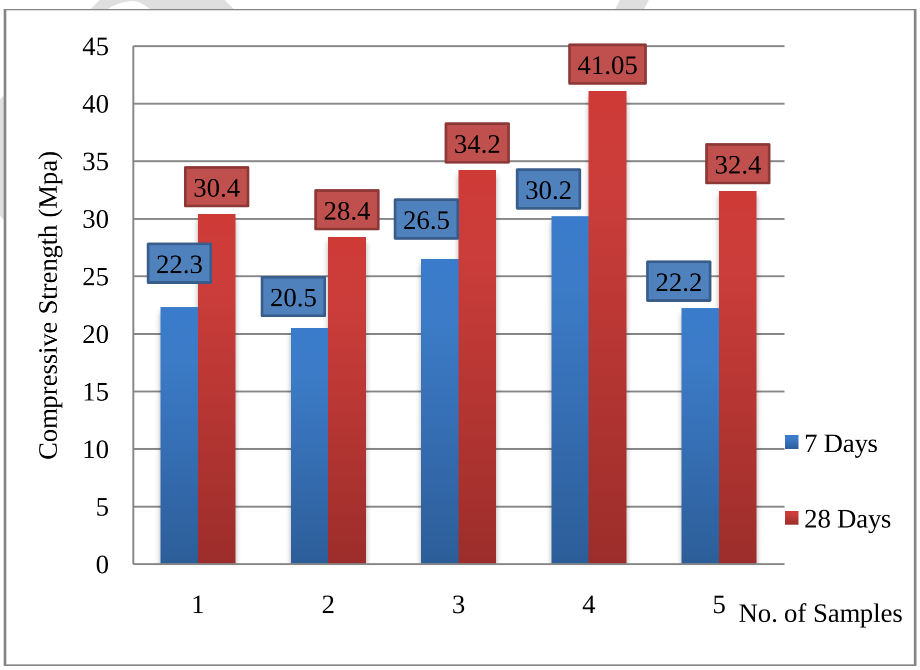

Figure 9 - Graph Showing the Compressive Strength of Geopolymer Concrete at 7 and 28 Days

4.4 DURABILITY OF GEOPOLYMER CONCRETE

4.4.1 Acid Resistance Test

Acid resistance test was performed to determine the durability of Geopolymer Concrete based on weight loss of samples after 0, 30 and 60 days curing in H_2SO_4 and H_3PO_4 acid solution.

a) Sulphuric Acid (H_2SO_4) Resistance Test

Table 3 - The Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after H_2SO_4 Curing

No. Of Days	Geopolymer Concrete Weight Loss (%)
0 Day	0
30 Days	2.64
60 Days	3.69

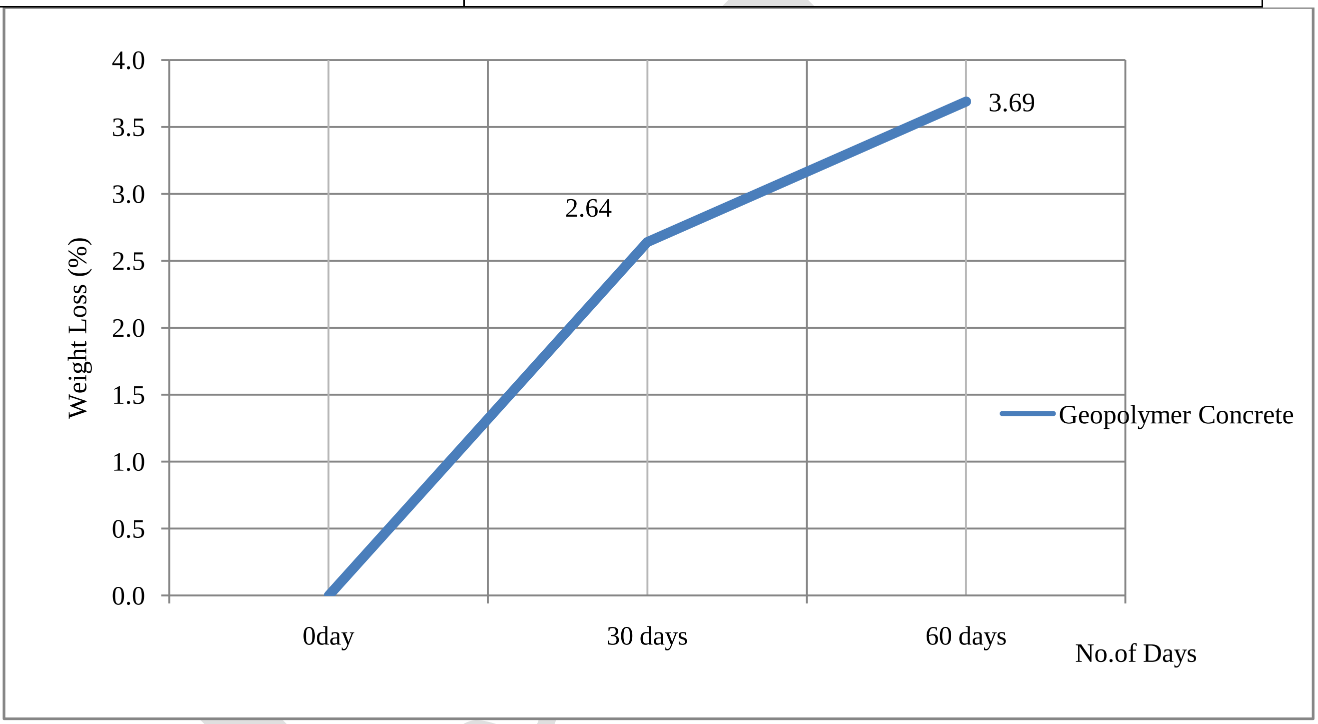

Figure 10 - Graph Showing the Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after H_2SO_4 Curing.

b) Phosphoric Acid (H_3PO_4) Resistance Test

Table 4 - The Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after H_3PO_4 Curing

No. Of Days	Geopolymer Concrete Weight Loss (%)
0 Day	0
30 Days	2.00
60 Days	2.37

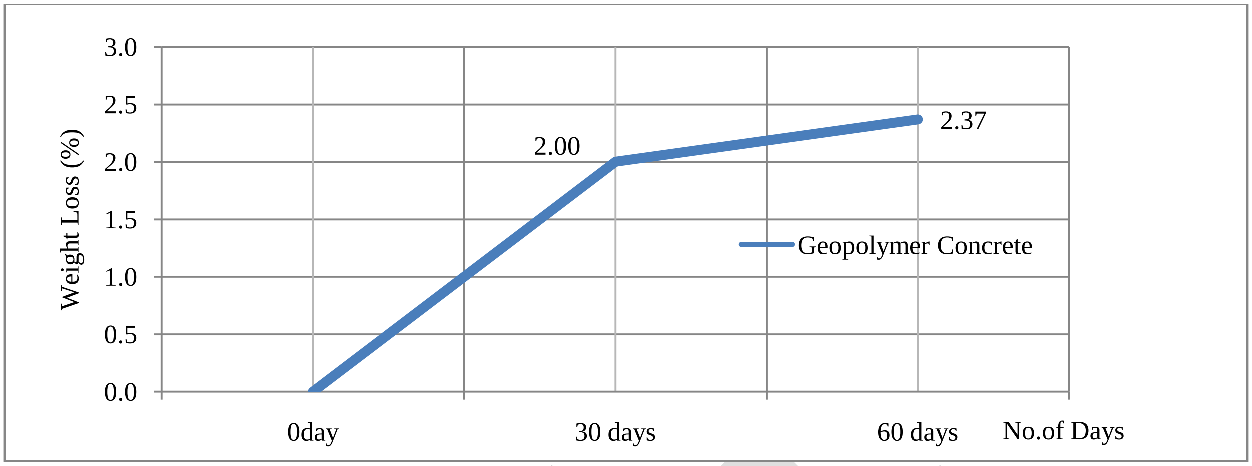

Figure 11 - Graph Showing the Weight Loss of Geopolymer Concrete at 0, 30 and 60 Day after H_3PO_4 Curing

4.4.2 Sulphate Resistance Test

Sulphate resistance test was performed to determine the durability of Geopolymer Concrete based on weight loss of samples after 30 and 60 days curing in Na_2SO_4 and $MgSO_4$ sulphate solution.

a) Sodium Sulphate (Na_2SO_4) Resistance Test

Table 5 - The Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after Na_2So_4 Curing

No. Of Days	Geopolymer Concrete Weight Loss (%)
0 Day	0
30 Days	0.68
60 Days	1.15

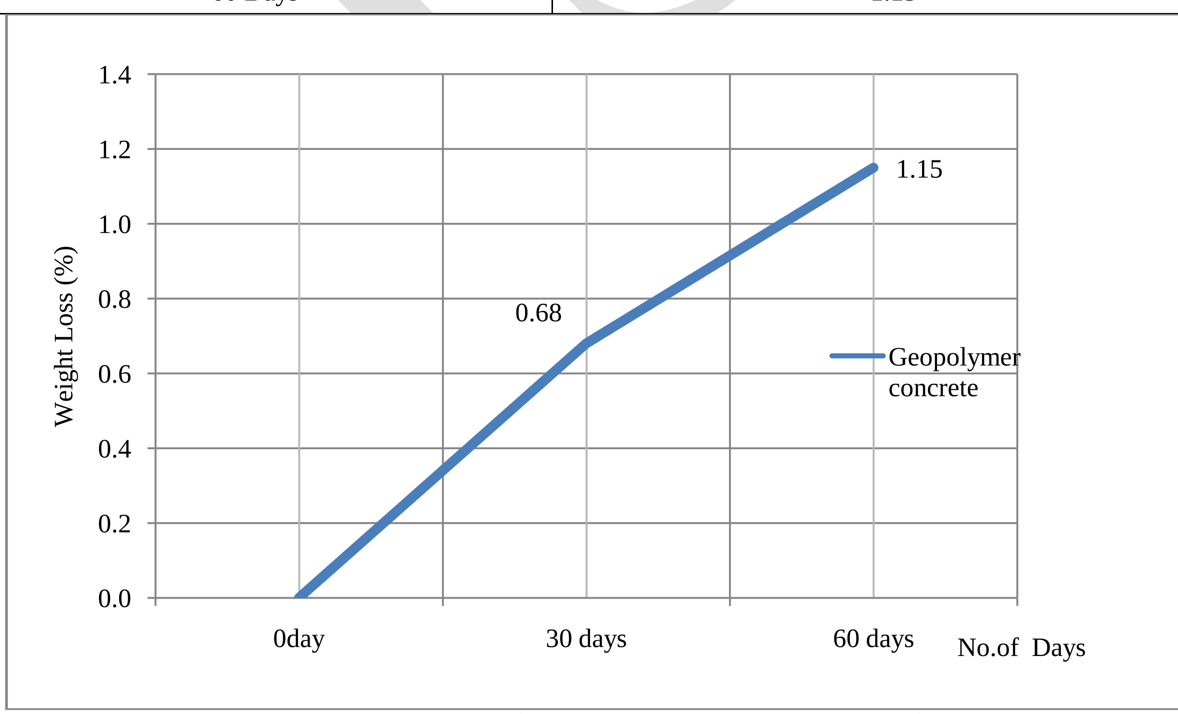

Figure 12 - Graph Showing the Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after Na_2So_4 Curing

b) Magnesium Sulphate ($MgSO_4$) Resistance Test

Table 6 - The Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after $MgSo_4$ Curing

No. Of Days	Geopolymer Concrete Weight Loss (%)
0 Day	0
30 Days	0.42
60 Days	0.78

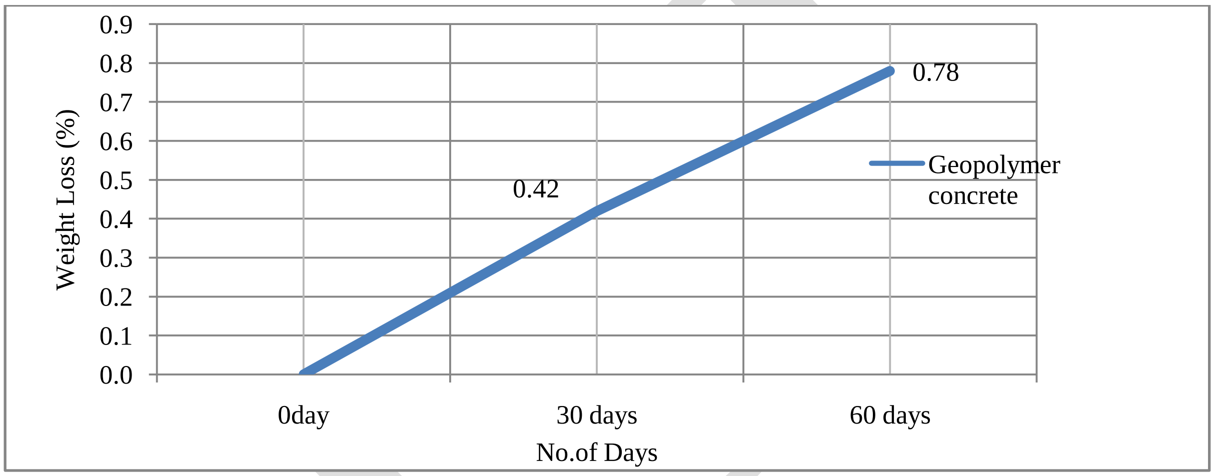

Figure 13 - Graph Showing the Weight Loss of Geopolymer Concrete at 0, 30 and 60 Days after $MgSo_4$ Curing

4.5 COMPARE THE PLAIN CONCRETE AND GEOPOLYMER CONCRETE

4.5.1 Compare the Compressive Strength of Plain Concrete and Geo-polymer Concrete

The Compressive Strength of Plain Concrete and Geopolymer Concrete after 0, 30 and 60 days curing in H_2SO_4, H_3PO_4, Na_2SO_4 and $MgSO_4$.

a) Sulphuric Acid (H_2SO_4) Test

Table 7 - The Compressive Strength of OPC and GPC at 0, 30 and 60 Days after H_2SO_4 Curing

No. Of Days	Plain Concrete (OPC) Compressive Strength (N/mm^2)	Geopolymer Concrete (GPC) Compressive Strength (N/mm^2)
0 Day	42.12	41.05
30 Days	28.45	30.95
60 Days	20.81	24.67

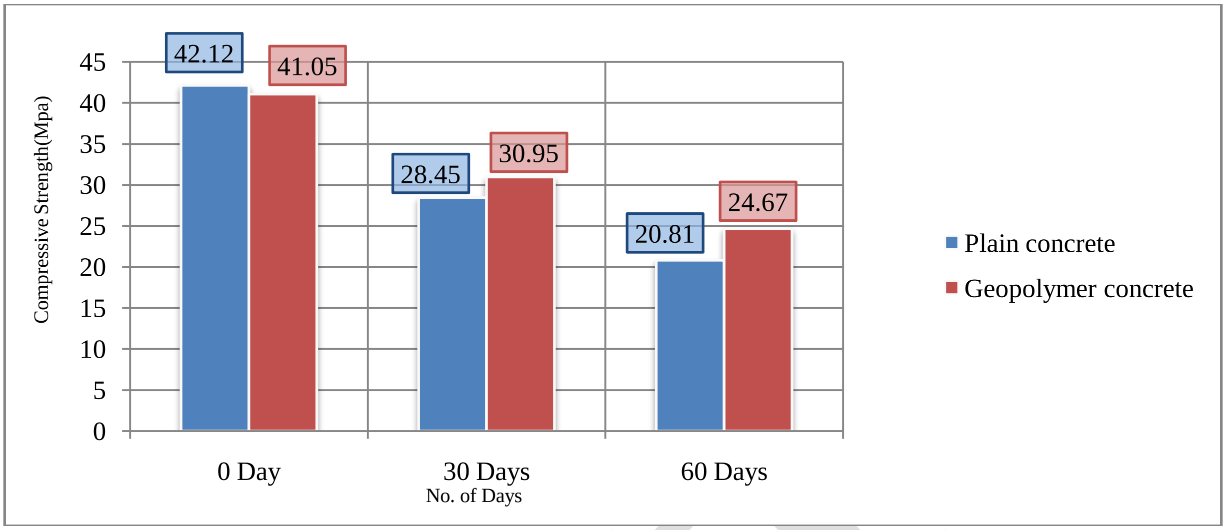

Figure 14 - Graph Showing the Compressive Strength of OPC and GPC at 0, 30 and 60 Days after H_2SO_4 Curing

b) Phosphoric Acid (H_3PO_4) Test

Table 8- The Compressive Strength of OPC and GPC at 0, 30 and 60 Days after H_3PO_4 Curing

No. Of Days	Plain Concrete (OPC) Compressive Strength (N/mm^2)	Geopolymer Concrete (GPC) Compressive Strength (N/mm^2)
0 Day	42.12	41.05
30 Days	33.56	35.74
60 Days	24.32	28.80

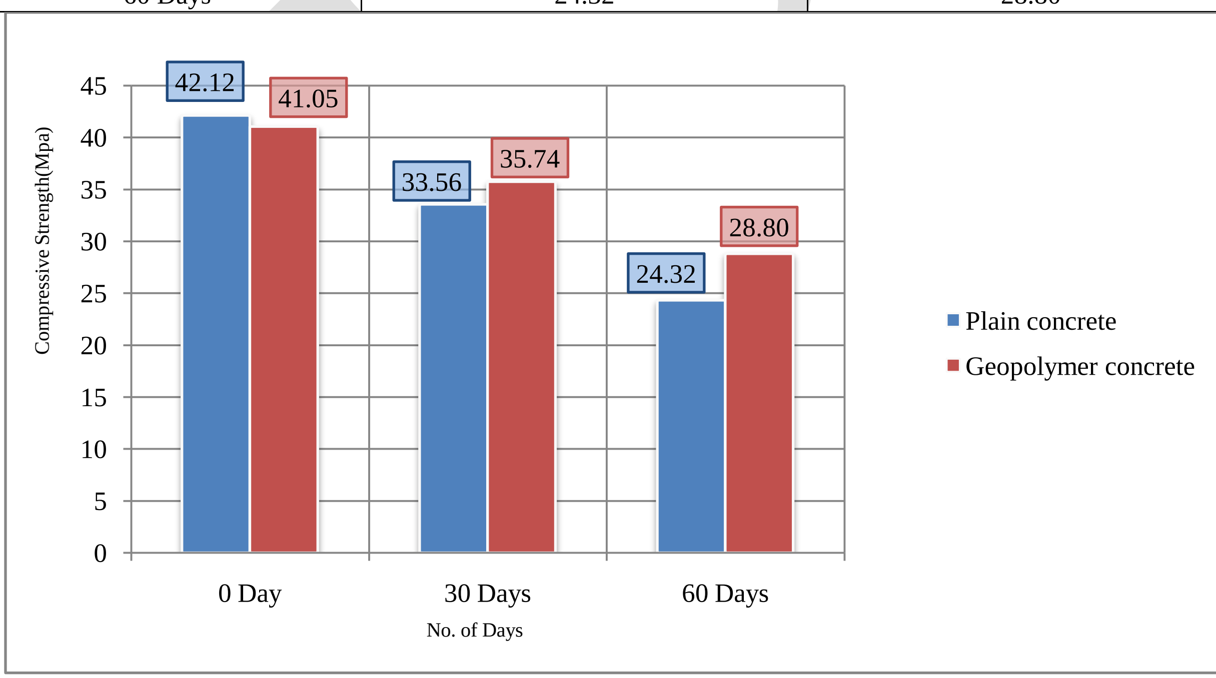

Figure 15 - Graph Showing the Compressive Strength of OPC and GPC at 0, 30 and 60 Days after H_3PO_4 Curing

c) Sodium Sulphate (Na_2SO_4) Test

Table 9 - The Compressive Strength of Geopolymer Concrete at 0, 30 and 60 Days after Na_2SO_4 Curing

No. Of Days	Plain Concrete (OPC) Compressive Strength (N/mm^2)	Geopolymer Concrete (GPC) Compressive Strength (N/mm^2)
0 Day	42.12	41.05
30 Days	34.15	37.65
60 Days	28.87	32.76

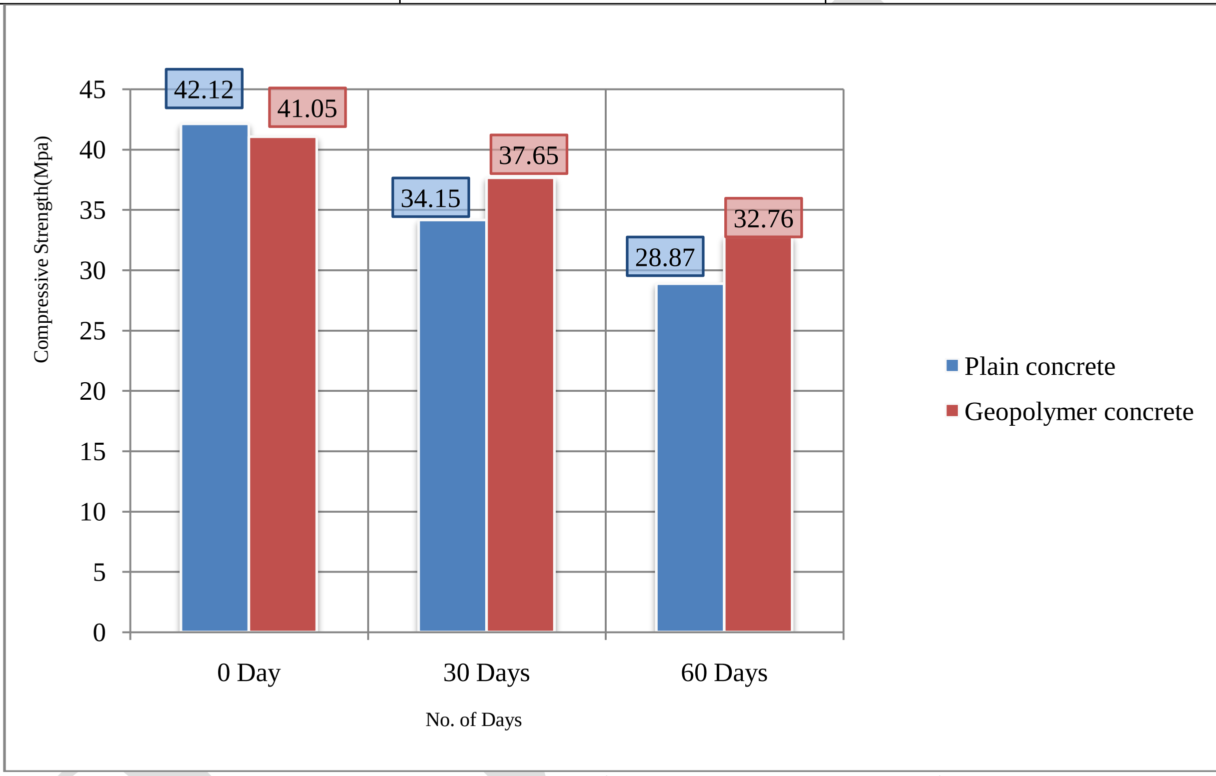

Figure 16 - Graph Showing the Compressive Strength of OPC and GPC at 0, 30 and 60 Days after **Na_2SO_4** Curing

d) Magnesium Sulphate ($MgSO_4$) Test

Table 10 - The Compressive Strength of OPC and GPC at 0, 30 and 60 Days after $MgSO_4$ Curing

No. Of Days	Plain Concrete (OPC) Compressive Strength (N/mm^2)	Geopolymer Concrete (GPC) Compressive Strength (N/mm^2)
0 Day	42.12	41.05
30 Days	35.77	38.18
60 Days	30.39	33.36

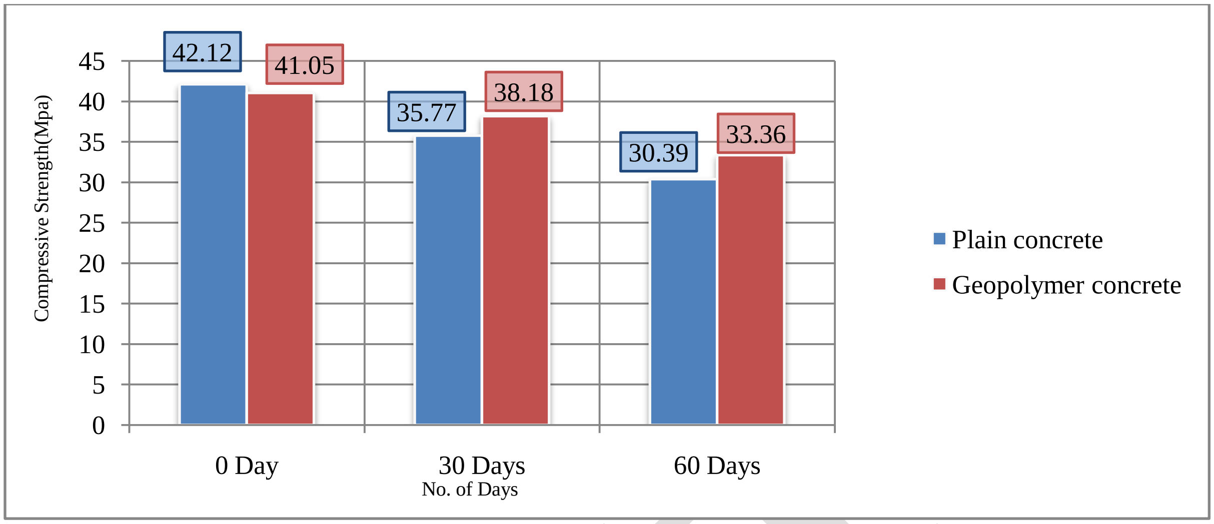

Figure 17 - Graph Showing the Compressive Strength of OPC and GPC at 0, 30 and 60 Days after $MgSO_4$ Curing

4.5.2 Compare the Weight Loss of Plain Concrete and Geopolymer Concrete

The Weight Loss of Plain Concrete and Geopolymer Concrete after 0, 30 and 60 days curing in H_2SO_4, H_3PO_4, Na_2So_4 and $MgSo_4$ are as following –

a) Sulphuric Acid (H_2SO_4) Test

Table 11 - The Weight Loss of OPC and GPC at 0, 30 and 60 Days after H_2SO_4 Curing

No. Of Days	Plain Concrete (OPC) Weight Loss (%)	Geopolymer concrete (GPC) Weight Loss (%)
0 Day	0	0
30 Days	3.25	2.64
60 Days	6.56	3.69

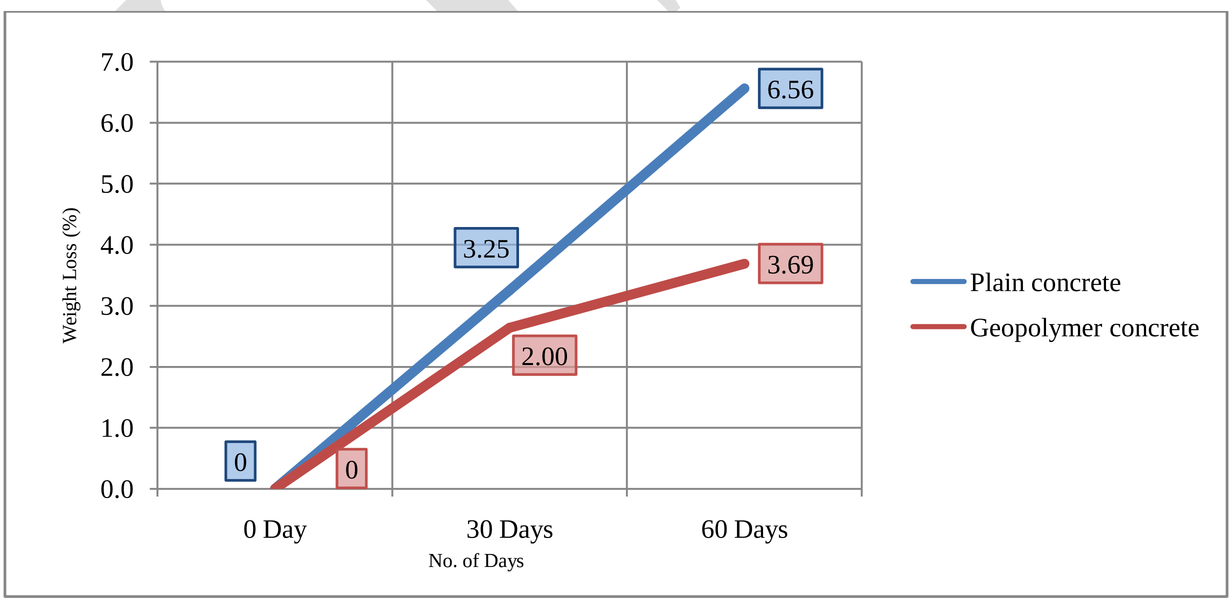

Figure 18 - Graph Showing the Weight Loss of OPC and GPC at 0, 30 and 60 Days after H_2SO_4 Curing

b) Phosphoric Acid (H_3PO_4) Test

Table 12 - The Weight Loss of OPC and GPC at 0, 30 and 60 Days after H_3PO_4 Curing

No. Of Days	Plain Concrete (OPC) Weight Loss (%)	Geopolymer concrete (GPC) Weight Loss (%)
0 Day	0	0
30 Days	2.45	2.00
60 Days	3.82	2.37

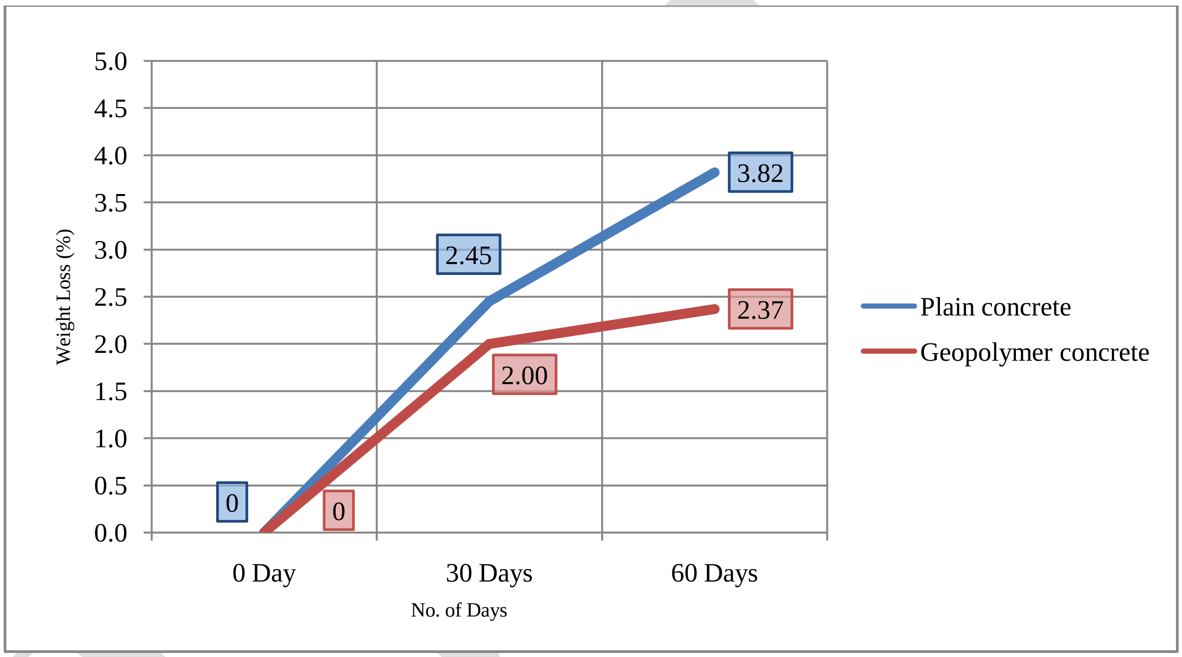

Figure 19 - Graph Showing the Weight Loss of OPC and GPC at 0, 30 and 60 Days after H_3PO_4 Curing

c) Sodium Sulphate (Na_2SO_4) Test

Table 13 - The Weight Loss of OPC and GPC at 0, 30 and 60 Days after Na_2SO_4 Curing

No. Of Days	Plain Concrete (OPC) Weight Loss (%)	Geopolymer concrete (GPC) Weight Loss (%)
0 Day	0	0
30 Days	1.78	0.68
60 Days	2.05	1.15

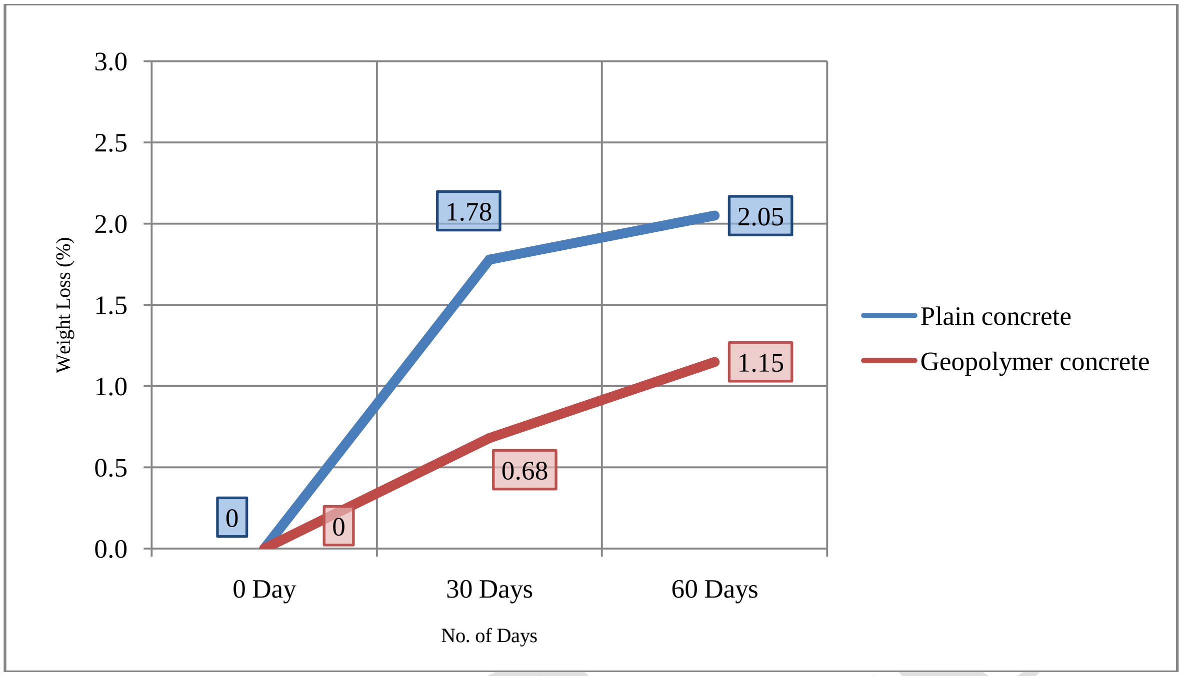

Figure 20 - Graph Showing the Wight Loss of OPC and GPC at 0, 30 and 60 Days after Na_2SO_4 Curing

d) Magnesium Sulphate ($MgSO_4$) Test

Table 14 - The Weight Loss of OPC and GPC at 0, 30 and 60 Days after $MgSO_4$ Curing

No. Of Days	Plain Concrete (OPC) Weight Loss (%)	Geopolymer concrete (GPC) Weight Loss (%)
0 Day	0	0
30 Days	3.15	0.42
60 Days	4.25	0.78

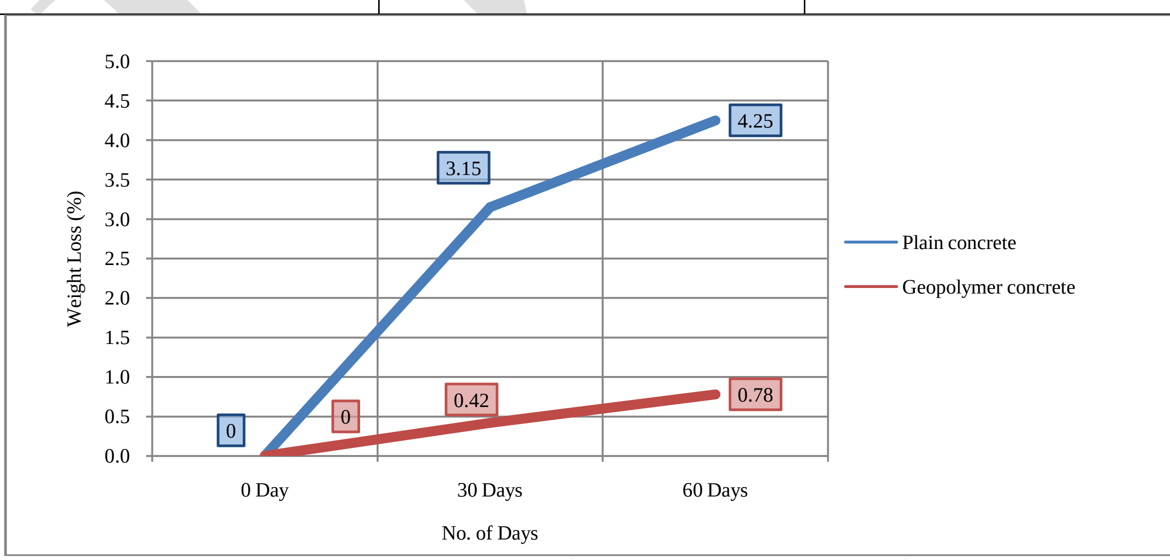

Figure 21 - Graph Showing the Weight Loss of OPC and GPC at 0, 30 and 60 Days after $MgSO_4$ Curing

Issue 4 volume 6, Nov. – Dec. 2014
ISSN 2249-9954

5 CONCLUSIONS

Based on the test results, the following conclusions are drawn:

- The initial setting time and final setting time ranged from 45 minutes to 120 minutes for Fly Ash Geopolymer.
- The highest Compressive strength (40Mpa) of the specimen produced by the 0.8 mass ratios (Activator / source material).
- The Compressive strength Loss of Plain Concrete and Geopolymer Concrete in the range after H_2SO_4, H_3PO_4, Na_2SO_4, and $MgSO_4$ curing are as following :-

Solutions	30 days	60 days
Acid solutions		
5% H_2SO_4 solution	8.43%	10.69%
5% H_3PO_4 solution	7.39%	12.42%
Sulphate solutions		
5% Na_2SO_4 solution	10.65%	11.26%
5% $MgSO_4$ solution	8.08%	9.11%

GPC – Geopolymer Concrete
OPC – Ordinary Plain Concrete

- Compressive strength loss (%) of OPC concrete in acid solution is varying from 7.39 to 12.42 % and in sulphate solution varying from 8.08 to 11.26 % more than the geopolymer concrete in 30 to 60 days.
- The Weight Loss of Plain Concrete and Geopolymer Concrete in the range after H_2SO_4, H_3PO_4, Na_2SO_4, and $MgSO_4$ curing are as following:-

Solutions	30 days	60 days
Acid solutions		
5% H_2SO_4 solution	0.61%	2.87%
5% H_3PO_4 solution	0.45%	1.45%
Sulphate solutions		
5% Na_2SO_4 solution	1.10%	0.90%
5% $MgSO_4$ solution	2.37%	3.47%

GPC – Geopolymer Concrete
OPC – Ordinary Plain Concrete

- Weight loss (%) of OPC concrete in acid solution is varying from 0.45 to 2.87 % and in sulphate solution varying from 0.9 to 3.47 % more than the geopolymer concrete in 30 to 60 days.
- OPC Concrete is more deteriorated in acidic as well as sulphate solution as compare to Geopolymer Concrete thus Geopolymer Concrete is more durable than OPC Concrete.

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