

# Experimental Study on Geopolymer Concrete subjected to Sulfuric Acid Solution

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**Abstract**— The most promising advantages of the geopolymer concrete are: lower harmful emissions, converting a variety of waste streams into useful by-products, higher resistance to corrosion and fire, higher compressive and tensile strengths and improved durability properties. This paper presents the experimental investigation done on performance of geopolymer concrete subjected to severe environmental conditions. Five geopolymer concrete mixes and one conventional OPC concrete mix were investigated. The source material for geopolymer concrete mixes were Fly ash (F), metakaolin (M) and ground granulated blast furnace slag (S). All mixes were labelled by the type and percentage of the source material like as S60% F40%, S80% F20%, S60% M40%, S80% M20%, S100%. The alkaline solution used for present study is the combination of sodium silicate and sodium hydroxide solution with the ratio of 3.50. The test specimens were 10x10x10 mm cubes. Durability of specimens were assessed by immersing the geopolymer concrete (GPC specimens in 25% sulfuric acid solutions, periodically monitoring surface deterioration, changes in weight and strength over a period of 42 days. The test results indicate that the fly ash- Slag- Metakaolin- based geopolymer concrete has an excellent resistance to acid and sulphate attack when compared to conventional concrete. Thus, it can be concluded that the production of geopolymers have a relative higher strength, excellent volume stability and better durability.

**Index Terms**— Minimum 7 keywords are mandatory; Keywords should closely reflect the topic and should optimally characterize the paper. Use about four key words or phrases in alphabetical order, separated by commas.

## 1 INTRODUCTION

In the context of increased awareness regarding the ill-effects of the over exploitation of natural resources, eco-friendly technologies are to be developed for effective management of these resources. Construction industry is one of the major users of the natural resources like cement, sand, rocks, clays and other soils. The ever-increasing unit cost of the usual ingredients of concrete have forced the construction engineer to think of ways and means of reducing the unit cost of its production. At the same time, increased industrial activity in the core sectors like energy, steel and transportation has been responsible for the production of large amounts like fly ash, blast furnace slag, silica fume and quarry dust with consequent disposal problem [9].

The geopolymer technology was first introduced by Davidovits in 1978 [1]. His work considerably shows that the adoption of the geopolymer technology could reduce the CO<sub>2</sub> emission caused due to cement industries. 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. Any material that contains mostly silicon (Si) and aluminium (Al) in amorphous form is a possible source material for the manufacture of geopolymer. Metakaolin or calcined Kaolin, low calcium ASTM Class F fly ash, natural Al-Si minerals, combination of calcined minerals and non-Calcined minerals, combination of fly ash

and metakaolin, combination of granulated blast furnace slag and metakaolin have been studied as source materials. The most common alkaline liquid used in geopolymerisation is a combination of sodium hydroxide or potassium hydroxide and sodium silicate or potassium silicate.

Ever since the introduction of geopolymer binders by Davidovits in 1978, it has generated a lot of interest among engineers as well as in the field of chemistry. In the past few decades, it has emerged as one of the possible alternatives to OPC binders due to their reported high early strength and resistance against acid and sulphate attack apart from its environmental friendliness. Though geopolymers can be manufactured from various source materials rich in silica and alumina such as fly ash, silica fume, ground granulated blast furnace slag and metakaolin etc, fly ash based geopolymers have attracted more attention. Geopolymer binders might be a promising alternative in the development of acid resistant concrete since it relies on alumina-silicate rather than calcium silicate hydrate bonds for structural integrity. Davidovits found that geopolymer bonding materials has very low mass loss of 5%-8% when samples were immersed in 5% sulfuric acid and hydrochloric acid solutions. In contrast, Portland cements were completely destroyed in the same environment [1].

Bakharev studied the resistance of geopolymer materials prepared from fly ash against 5% Sulfuric acid up to 5 months' exposure and concluded that geopolymer materi-

als have better resistance than ordinary cement counter- parts (Bakharev 2005 (a) and (b)). Wallah have shown that

geopolymer composites possesses excellent durability properties in a study conducted to evaluate the long-term

properties of fly ash based geopolymers [4]. The geopolymer has a very good resistance in acid media in terms of weight loss and residual compressive strength [5]. The performance on geopolymer concretes in aggressive environments was studied using tests on absorption and acid resistance (Manu Santhanam et al., 2008). Results indicated that the water absorption decreased with an increase in the strength of the concrete and the fly ash content. Based on summary of extensive studies conducted, a simple trial and error method was suggested to design the geopolymer concrete mixes [7]. The geopolymer concrete (GPC) was superior to plain Portland cement concrete (PPCC) when these mixes were subjected to sodium sulphate and magnesium sulphate solutions [8]

## 2. Objective

The main objective of this research is to study the effect of immersing in 25% sulfuric acid solution for different duration times on physical and mechanical properties of five different types of geopolymer concrete and conventional OPC concrete.

## 3. Experimental investigation

### 3.1 Materials

The cement used was CEMI 42.5 R that complies with the requirement of the Egyptian standard specifications (ESS 4756/2007). The coarse aggregate was crushed lime stone. Natural sand was used with fineness modulus of 2.40. The concrete mix was designed to achieve cube compressive strength after 28 days of 45 MPa.

### 3.2 Alkaline Solutions

To activate the used source materials, commercial grade sodium hydroxide (NaOH) and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) solutions were used as alkaline activator as shown in figure 1. Distilled water was used to dissolve sodium hydroxide pellets

to prevent any effect of unknown contaminants. The mass of NaOH solids in a solution varies depending on the concentration of the solution. In order to improve the workability, extra water was added to the mixture.



Sodium hydroxide solution

Sodium silicate solution

FIGURE 1: ACTIVATING SOLUTION COMPONENTS

### 3.3 Mix design of geopolymer concrete

In the design of geopolymer concrete mix, coarse and fine aggregates together were taken as 70% of entire mixture by mass. 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 was taken as 33% of the total aggregates. The density of geopolymer concrete is taken similar to that of OPC as 2400 kg/m<sup>3</sup> [7]. The details of mix design and its proportions for different grades of GPC are given in Table 1.

Table 1

Mix proportions of GPC mixes and OPCC mix with ratio of Na<sub>2</sub>SiO<sub>3</sub>/ NaOH as 3.5

Materials		Mass (kg/m <sup>3</sup> )					
		S60% F40%	S80% F20%	S60% M40%	S80% M20%	S100%	C100%
Coarse aggregates	10 mm	1093	1093	1093	1093	1093	1093
Fine sand		547	547	547	547	547	547
Cement		-----	-----	-----	-----	-----	450
Slag		270	360	270	360	450	-----
Fly ash		180	90	-----	-----	-----	-----
Metakaolin		-----	-----	180	90	-----	-----
Na <sub>2</sub> SiO <sub>3</sub> / NaOH		3.50	3.50	3.50	3.50	3.50	-----
SiO <sub>2</sub> /Na <sub>2</sub> O		2.00	2.00	2.00	2.00	2.00	-----
Sodium hydroxide solution		41	41	41	41	41	-----
Sodium silicate solution		131	131	131	131	131	-----
Extra water		112	112	112	112	112	200

### 3.4 Mixing, Casting, Compaction of Geopolymer Concrete

GPC can be manufactured by adopting the conventional techniques used in the manufacture of Portland cement concrete. In the laboratory, the GPC and the aggregates were first mixed together dry on pan for about three minutes. The liquid component of the mixture is then added to the dry materials and the mixing continued usually for another four minutes. (Figure 2 and 3) In preparation of NaOH solution, NaOH pellets were dissolved in one litre of water in a volumetric flask for one concentration of NaOH (8 M). Alkaline activator with the combination of NaOH and Na<sub>2</sub>SiO<sub>3</sub> was prepared just before the mixing with GPC. The addition of sodium silicate is to enhance the process of geopolymerization [10]. The ratio of GPC/ alkaline activator and Na<sub>2</sub>SiO<sub>3</sub> / NaOH used in the current study was 3.5 for all the mixes. The GPC and alkaline activator were mixed together in the mixer until homogeneous paste was obtained. This mixing process can be handled with

in 5 minutes for each mixture of NaOH. Fresh GPC based geopolymer concrete was usually cohesive.



Figure 2: Mixing of sodium silicate and sodium hydroxide solution.



Figure 3: Mixing of ingredients }

### 3.5 Test Procedure

The GPC and OPCC specimens were soaked in 25% Sulfuric acid solution after 28 days of casting. The specimens were kept fully immersed in these solutions for duration of 42 days. The effect of this solutions on the GPC and OPCC specimens were regularly monitored through visual inspection, measurement of weight change and compressive strength test. Samples for weight change test were primed in water for 3 days prior to immersion in these solutions and its saturated surface dry weight considered as initial weight. These samples were removed from the solution and weighed at various stages of exposure in similar condition as the final weight.

## 4. Results and discussions

Exposure to sulfuric acid solution led to the leaching of some component from the prepared specimens into the external solution, which resulted in the softening of the specimen structure and loss of specimen integrity. These changes were mostly visible on the outer surfaces of the exposed specimens, revealing the presence of aggregates on the surface, as can be seen in Fig. 4.

S80% M20%      C100%      S80% M20%      C100%

Figure 4: GPC and OPCC specimens exposed to sulfuric acid solution after (42) Day

### 4.1 Effect of the sulfuric acid solution on the weight loss

Fig. 5 & 6 shows the changes in the mass of GPC and OPCC specimens before and during sulfuric acid solution exposure. The initial values before immersion in sulfuric acid solution were taken as a reference value (i.e., 100%).

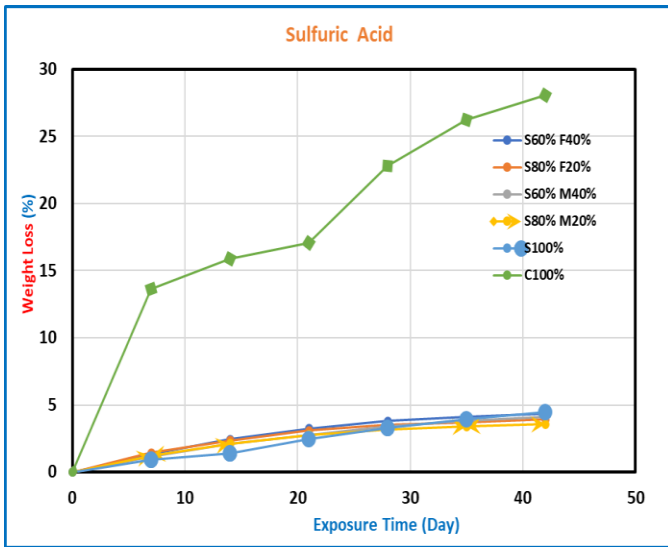


FIGURE 5: PERCENTAGE OF WEIGHT LOSS VERSUS EXPOSURE TIME

to mass of solution absorbed by concrete. The mass loss on exposure to Sulfuric acid in GPC was about 3 to 4%, where as in OPCC it was observed to be 28% after 42 days of exposure. In case of normal concrete, the hydration compounds were neutralized by sulfuric acid and gradually the binder disintegrated, thus exposing the aggregates.

#### 4.2 Effect of the sulfuric acid solution on the compressive strength

Fig. 7 & 8 shows the changes in the compressive strength of GPC and OPCC specimens before and after sulfuric acid solution exposure. The initial values before immersion in sulfuric acid solution were taken as a reference value (i.e., 100%).

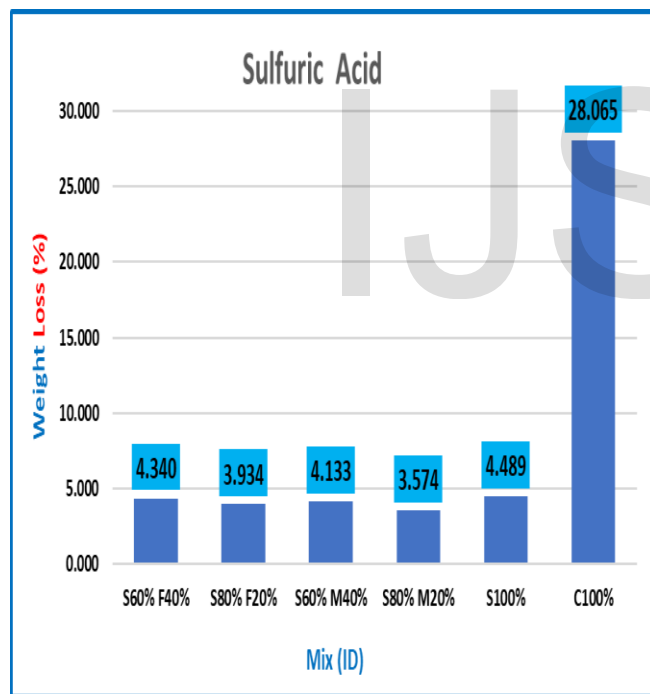


Figure 6: Percentage of Weight Loss for All Mixes after 42 days

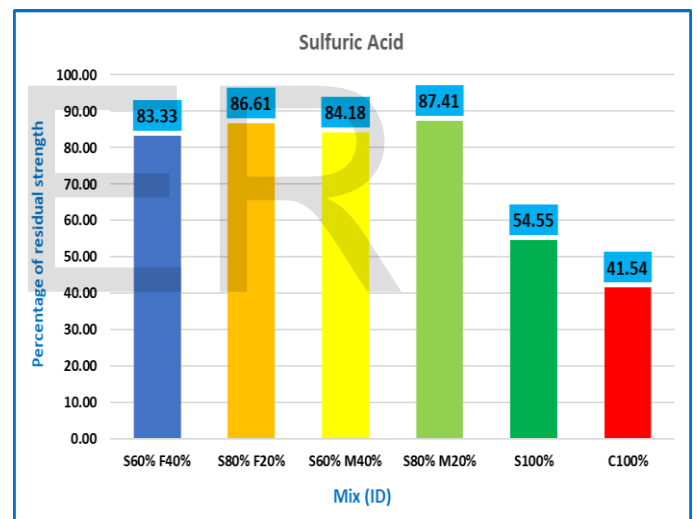


Figure 8: The Percentage of Residual Strength for All Mixes after 42 days

There is a slight mass loss during first week of exposure due

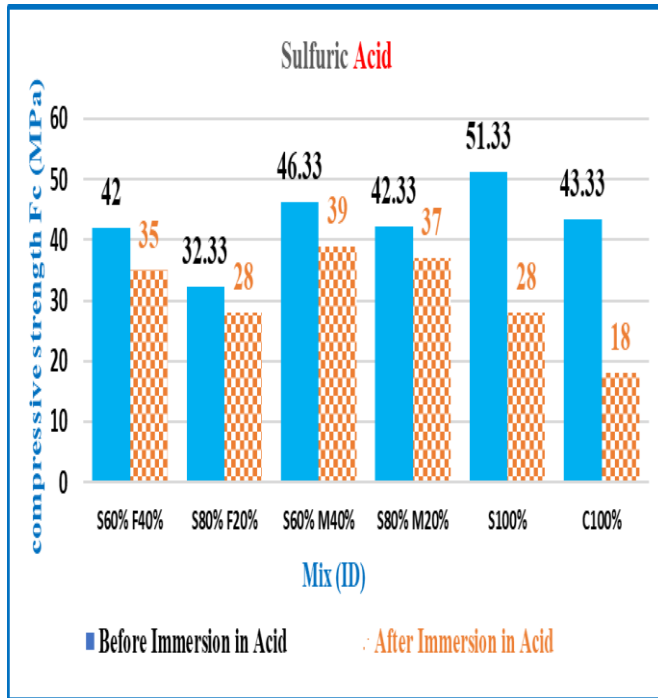


Figure 7: The Mean Compressive Strength for All Mixes before and after immersion in Sulfuric Acid Solution for 42 days

The details of compressive strength and for different types of concrete with different source materials exposed for a period of 42 days to sulfuric acid are shown in figure 7 & 8.

1. The strength of GPC and PPCC gradually decreases as the day of exposure increases.
2. The reduction in compressive strength observed for GPC and PPCC specimens were 17%, 14%, 16%, 13%, 45% and 59% respectively for 42 days of exposure.
3. The better performance of geopolymeric materials than that of Portland cement concrete in acidic environment might be attributed to the lower calcium content of the source material as a main possible factor since geopolymer concrete does not rely on lime like Portland cement concrete.

## 5. Conclusions

On the basis of results obtained during the experimental investigations, following conclusions were drawn:

1. Fly ash, slag, metakaolin was used in the present study to produce geopolymeric reactions with the help of sodium hydroxide-silicate based alkaline activator solutions. Conventional methods of mixing, compaction, molding and demolding can be adopted for GPCs mixes.
2. The degradation of geopolymer concrete and OPCC specimens under sulfuric acid attack were recorded in terms of decreasing in their strength and weight
3. Specimens received white deposits on the surfaces during exposure to sulfuric acid solution which gradually transformed from soft and flaky shape to hard and rounded shape.
4. The geopolymer concrete mixes indicated minor changes in weight and strength when the specimens were exposed to sulfuric acid.
5. The compressive strength loss for the specimens exposed in sulfuric acid was in the range of 59% in OPCC, where as it was about 13 to 17% in GPCs.

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