RESISTANCE OF GEOPOLYMER CONCRETE WITH FLY ASH AND SLAG TO SULPHATE AND ACID ATTACK

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ABSTRACT

This paper presents the study carried out on fly ash and slag based geopolymer concrete under sulphate and acid attack. Both types of geopolymer concretes and control concrete made from Ordinary Portland cement were subjected to 5% Sodium sulphate and 5% sulphuric acid solution and results were compared with control concrete. From the results, it can be concluded that geopolymer concrete undergoes very less change in compressive strength and hence durability under acid attack and sulphate attack of geopolymer concrete is much superior as compared to control concrete which suggests good applicability in field.

Key Words: fly ash geopolymer concrete, GGBFS geopolymer concrete, control concrete.

1. INTRODUCTION

Contribution of construction sector is US$ 3.3 trillion to the global economy in 2008[1] and the chief contribution is of cement and concrete. It is astonishing to note that worldwide production of cement in 2008 was around 2.9 billion tonnes [2], making it one of the highest volume commodities produced worldwide. It is estimated that usage of concrete is second only to water [3].Since the progress of any nations is measured by its infrastructure growth, this demand is going to increase exponentially. The popularity of concrete as construction material is due to its excellent mechanical properties, mould ability, adaptability, relatively good fire resistance capacity and availability. It is most intriguing material which can be reasonably engineered to satisfy given criteria within certain boundary limits [1]. Chief ingredients of concrete is cement. Being most popular and easily available material it is consumed in large amount, but it also has certain drawbacks which cannot be blinked upon. The production of cement is highly energy intensive process and also it releases significant amount of carbon dioxide in the atmosphere [3]. It is also well known fact, that for each ton
of Portland cement manufactured, it is estimated that one ton of carbon dioxide is released into the environment, thus contributing at least 5-8% of global carbon dioxide emission. Also the production process of cement involves very high temperature of around 1400°C – 1500°C, the destruction of natural resources like quarries for extraction of raw materials and emission of greenhouse gases like CO$_2$ and NO$_X$ [3]. There is ever growing concern and increasing emphasis on energy conservation and environmental protection. This is led to research and development of alternatives to customary building material which will help in reduction of greenhouse gas emissions and also minimize the energy required for material production. Again durability of the structures and cost of repair and rehabilitation of the structures constructed with Portland cement possess a serious issue and raise many questions.

On the other hand, industrialisation is increased by leaps and bounds, resulting into tremendous power consumption. Since majority of power is produced by use of coal, fly ash a waste product in power generation is produced in large amount. This fly ash is either used as land fill, resulting into turning fertile land barren and is also cause of lot of air pollution. It is estimated that the amount of fly ash produced by the year 2010 will be estimated to be about 780 million tons [4]. The recycled use of this waste material in construction will alleviate the cost of disposal elsewhere and reduce the cost of concrete manufacture overall. In Western Europe there is a trend to blend waste ashes and slag into OPC based concrete giving 100% utilisation [2, 4].

Alkali activation of material provide an excellent opportunity for utilisation of industrial waste like fly ash, blast furnace slag, metakaolin and converting these into useful product. The basic aim of Treaty of Rome is directed towards the prevention, reduction and elimination of environmental damage either at source or by careful management of natural resources [4]. Also there is anticipated increase in financial cost of carbon dioxide emission through national and international credits trading schemes or ‘carbon tax’ regimes, and simultaneously increase in cost of potable water which plays a pivotal role in construction industry. Geopolymer concrete is capable of meeting the performance criteria in construction industry.

2. GEOPOLYMER CONCRETE

Davidovits [6] proposed that an alkaline liquid could be used to react with the silicon (Si) and the aluminum (Al) in a source material of geological origin or in byproduct materials such as fly ash, GGBFS or rice husk ash to produce binders. Because the chemical reaction that takes place in this case is a polymerization process, Davidovits coined the term 'Geopolymer' to represent these binders. Geopolymer 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 polymerization process involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals, that results in a three- dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follows

$$Mn[(SiO_2)z AlO_2]n:wH_2O$$

Where: $M =$ the alkaline element or cation such as potassium, sodium or calcium; the symbol indicates the presence of a bond, $n$ is the degree of polycondensation or polymerisation; $z$ is 1,2,3, or higher, up to 32.

This paper presents the study of the durability fly ash based geopolymer concrete and slag based geopolymer concrete in sulphate and acid attack and comparison is made with concrete prepared from Ordinary Portland cement concrete.
3. EXPERIMENTAL

3.1 Materials: Chemical composition of two source materials i.e. low calcium processed fly ash and slag are summarised in Table 1 and Table 2. Low calcium Fly ash from Dirk India Private Limited under the name of the product POZZO CRETE 60. Ground granulated blast furnace slag was obtained for JSW cement, Pune.

Table 1: Fly Ash Properties

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Property</th>
<th>Fly Ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Colour</td>
<td>Light grey</td>
</tr>
<tr>
<td>2</td>
<td>Loss of ignition %</td>
<td>1.6</td>
</tr>
<tr>
<td>3</td>
<td>SiO$_2$ + Al$_2$O$_3$ + Fe$_2$O$_3$</td>
<td>92.5</td>
</tr>
<tr>
<td>4</td>
<td>SiO$_2$</td>
<td>57.3</td>
</tr>
<tr>
<td>5</td>
<td>MgO</td>
<td>2.13</td>
</tr>
<tr>
<td>6</td>
<td>SO$_3$</td>
<td>1.06</td>
</tr>
</tbody>
</table>

Table 2: Granulated blast furnace slag properties

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Loss on ignition %</td>
<td>0.07</td>
</tr>
<tr>
<td>2</td>
<td>Silica SiO$_2$ %</td>
<td>35.00</td>
</tr>
<tr>
<td>3</td>
<td>Iron oxide Fe$_2$O$_3$ %</td>
<td>0.5</td>
</tr>
<tr>
<td>4</td>
<td>Aluminum oxide Al$_2$O$_3$ %</td>
<td>10</td>
</tr>
<tr>
<td>5</td>
<td>Calcium oxide CaO %</td>
<td>37.00</td>
</tr>
<tr>
<td>6</td>
<td>Magnesium oxide MgO %</td>
<td>8</td>
</tr>
<tr>
<td>7</td>
<td>Manganese oxide MnO %</td>
<td>0.07</td>
</tr>
<tr>
<td>8</td>
<td>Sulfur S$_2$ %</td>
<td>0.54</td>
</tr>
</tbody>
</table>

Both fly ash based [7] and GGBFS based geopolymer [8] were prepared using sodium hydroxide and sodium silicate as alkaline activators. Sodium hydroxide (NaOH) in flakes form with 98% purity was purchased from local chemical supplier has been used. Sodium hydroxide solution was prepared by dissolving the flakes in tap water. The mass of NaOH solids in a solution varied depending on the concentration of the solution expressed in terms of molar M. Sodium Silicate used was in liquid form with Na$_2$O 16.84%, SiO$_2$ 35.01%, water : 46% and SiO$_2$/ Na$_2$O = 2.08, specific gravity : 1.48 g/cc. Alkaline solution was prepared one day before the casting of specimen. Naphthalene Sulphonate based superplasticiser, supplied by BASF, under the brand name Rheobuild 1125, has been used to improve the workability of the fresh geopolymer concrete. Coarse aggregate of 10 mm and 20 mm size were used confirming to Indian standards. Fine aggregates used was river sand.
Both geopolymer concrete and control concrete were designed for M25 (medium strength concrete) and comparison was made with OPC based concrete of same grade strength. For control concrete the proportions of various constituents arrived are shown in Table 3. Indian standard method IS 10262-2009, was used. Water/cement ratio was kept as 0.5 for control concrete.

Generally, in the design of geopolymer concrete mix, coarse and fine aggregates have been taken as 75% of entire mix by mass [9]. This value is similar to that used in OPC concrete in which they have been in the range of 75% to 80% of the concrete mix by mass. Fine aggregate has been taken as 30% of the total aggregate. The average density of geopolymer concrete has been considered similar to that of OPC concrete of 2400 kg/m$^3$ based on literature survey. Various trials were carried out for both fly ash and GGBFS as source materials with alkaline solutions, so that same strength can be achieved and proper comparison can be made. The mix proportions of geopolymer concrete is shown in Table 3.

### 3.2 Test Procedure for Acid Resistance

There is no standard test for acid attack on concrete and thus resistance to acid attack in present investigation was tested by immersion of concrete specimens in a solution of sulphuric acid and compared with control concrete [10,11]. Sulphuric acid ($H_2SO_4$) solution with 5% concentration was used as the standard exposure solution. The pH of the solution was checked at regular interval. The test specimen were 150 mm x 150 mm x 150 mm cubes. 3 specimens for each test were prepared compressive strength and change in mass to take average result of the specimen. The acid resistance of control concrete and geopolymer concrete were evaluated by measuring the residual compressive strength and change in mass after acid exposure. Cubes were immersed in solution after 28 days of curing period for a specific exposure period. The exposure period was 30, 60 and 90 days.

<table>
<thead>
<tr>
<th>Table 3: Mix design for Fly ash and Slag based Geopolymer concrete</th>
</tr>
</thead>
<tbody>
<tr>
<td>Properties</td>
</tr>
<tr>
<td>Density of concrete</td>
</tr>
<tr>
<td>Coarse aggregate</td>
</tr>
<tr>
<td>Fine aggregate</td>
</tr>
<tr>
<td>Source material</td>
</tr>
<tr>
<td>Alkaline liquid to source ratio</td>
</tr>
<tr>
<td>Ratio of $Na_2SiO_3$ to $NaOH$ hydroxide ratio</td>
</tr>
<tr>
<td>Molarity</td>
</tr>
<tr>
<td>Curing temperature</td>
</tr>
<tr>
<td>Curing time</td>
</tr>
<tr>
<td>Rest period</td>
</tr>
<tr>
<td>Mass of Sodium hydroxide</td>
</tr>
<tr>
<td>Mass of Sodium silicate</td>
</tr>
<tr>
<td>Extra water</td>
</tr>
<tr>
<td>Superplastizer</td>
</tr>
</tbody>
</table>
3.3 Test Procedure for Sulphate Resistance

For measuring sulphate resistance on concrete Sodium sulphate (Na₂SO₄) solution with 5% concentration was used as the standard exposure solution [9]. The test specimen were 150 mm x 150 mm x 150 mm cubes. 3 specimens for each test were prepared and the sulphate resistance of control concrete and geopolymer concrete were evaluated by measuring the residual compressive strength and change in mass after sulphate exposure. Cubes were immersed in solution after 28 days of curing period for a specific exposure period.

3.3 Change in mass

Change in mass of specimens was measured after various exposure period. The weight of each specimen was measured before immersion in to the solution. After the exposure period the specimen were taken out and left to air dry for a week in the laboratory condition. Then weights of the specimens were measured using the weighing scale available in laboratory and from that change in mass was calculated.

3.4 Change in Compressive strength

The change in compressive strength after acid exposure or sulphate exposure was determined by testing the compressive strength of the specimens after selected periods of exposure. The specimens were tested in saturated surface dry (SSD) condition. For the SSD condition, the specimens were removed from the solution, loose particles were removed using wire brush. Surface preparation was done using cement mortar (1:3) and then tested in compression testing machine available at laboratory. The reduction in compressive strength was found by % = (Average compressive strength of control specimen – average compressive strength of geopolymer specimen) * 100 / average compressive strength of control specimen, MPa

4. RESULTS

4.1 Sulphate Resistance of fly ash and slag based geopolymer concrete

4.1.1 Visual Appearance

The visual appearances of test specimens after different exposures are shown in Figure 1. It can be seen from the visual appearance of the test specimens after soaking in sodium sulfate solution for the exposure periods of 30, 60 and 90 days that there was no significant change in the appearance of the specimens compared to the condition before they were exposed. However, white patches were observed on the specimens both fly ash based and slag based geopolymer concrete. There was no sign of surface erosion, cracking or spalling on the specimens.

Figure 1: Geopolymer Specimen   Control Specimen after sulphate exposure
4.1.2 Change in Mass for Fly Ash and GGBFS Geopolymer concrete and control concrete

Figure 2 presents the test results on the change in mass of specimens soaked in sodium sulfate solution for 30, 60 and 90 days period as a percentage of the mass before exposure. That there was no reduction in the mass of the specimens, as confirmed by the visual appearance of the specimens. There was a slight increase in the mass of specimens of both type of geopolymer concrete and control concrete due to the absorption of the exposed liquid. The increase in mass of specimens soaked was 1.54%, 1.6% and 2.00% for fly ash based geopolymer concrete, GGBFS based geopolymer concrete had 1.47%, 1.8% and 2.24% increase and for control concrete increase was 0.30%, 0.39% and 0.42% after exposure of 30, 60 and 90 days respectively.

![Change in Mass after sulphate exposure](image)

Figure 2: Change in Mass of FA and GGBFS Geopolymer concrete and Control Concrete after sulphate exposure

4.1.3 Change in Compressive strength after sulphate exposure

The percentage reduction in compressive strength in geopolymer concrete with fly ash is 0.88%, 1.33 % and 2.66% after 30, 60 and 90 days interval. Geopolymer concrete with slag as source material had 0.12 %, 1.08% and 1.65% respectively after same time interval. The
reduction in control concrete was 1.89% for 30 days, 2.89% for 60 days and 3.77% for 90 days’ time interval. Figure 3 shows it in graphical form.

![Reduction in Compressive strength of Fly Ash GC, GGBFS GC, and Control Concrete](image)

**Figure 3:** Reduction in Compressive strength of Fly Ash GC, GGBFS GC & Control Concrete after Sulphate Exposure

### 4.2 Acid Resistance of Geopolymer and Control concrete

Acid resistance property of geopolymer concrete mixes has been studied by evaluating various parameters like visual appearance, change in mass and change in compressive strength after the 30 days, 60 days and 90 days of exposure period of both type of concrete.

#### 4.2.1 Visual Appearance

Figure 4 compares the visual appearance of the geopolymer and control concrete specimens after soaking in 5% concentrations of sulfuric acid solution for 30, 60 and 90 days.
It can be seen that the specimens exposed to sulfuric acid undergoes erosion of the concrete surface and control concrete of OPC shows higher erosion compared to Geopolymer concrete. After 4-5 days, coarse aggregates of control concrete were exposed as the surface undergoes erosion. Also initial surface erosion was significantly higher for control concrete.

**Figure 4:** Visual Appearance of Geopolymer (Left) and Control (Right) Concrete

4.4.2 Change in Mass of geopolymer and control concrete

Control concrete specimens have significant mass loss compared to geopolymer concrete having same exposure. Geopolymer concrete shows good resistance to acid and very less mass loss has been observed throughout the test. This is observed for both type of geopolymer concrete. Figure 5 shows graphical representation of mass loss of geopolymer and control concrete. The change in mass for Fly ash based GC was 0.31%, 0.43% and 0.51%. GGBFS based GC had reduction of 0.35%, 0.58% and 0.92% while in control concrete with OPC the reduction was 10.66%, 11.88% and 15.51% for 30, 60, 90 days of exposure period.

**Figure 5:** Percentage Change in mass for Fly ash, slag based Geopolymer concrete and Control concrete

4.2.3 Change in Compressive Strength for geopolymer and control concrete

Change in compressive strength for fly ash based geopolymer concrete was 2%, 5.3% and 7.53% after 30 days, 60 days and 90 days of exposure. For slag based geopolymer concrete it was 1.93%, 4.5% and 7.01% after 30 days, 60 days and 90 days of exposure periods. Control concrete had reduction in compressive strength of 10.66%, 23.75% and
36.61% after 30 days, 60 days and 90 days of exposure period. High reduction observed in control specimen up to 36.6% whereas in fly ash based geopolymer specimen 7.5% reduction has been observed and GGBFS geopolymer concrete had 7% reduction in strength. This suggests that the effect of acid exposure on geopolymer concrete with slag is very low. Figure 6 shows graphical representation of the test results.

![% Reduction in Compressive Strength](image)

Figure 6: Reduction in Compressive strength for FA GC, GGBFS GC and Control concrete in acid exposure

5. CONCLUSIONS

Fly ash and GGBFS based geopolymer concrete during the test showed excellent resistance to sulphate and acid attack. Following conclusions can be arrived.

- Initially increase in mass of GGBFS based geopolymer concrete was less compared to fly ash based geopolymer concrete, but after 60 days there was increase in mass, while control concrete had very little increase in mass due to sulphate exposure up to 90 days.
- GGBFS based geopolymer concrete showed less reduction in compressive strength which was 1.89% after 90 days as compared to fly ash which was 2.7%. More decrease was observed in control concrete strength which was found to be 3.8% after 90 days of exposure.
- When subjected to 5% sulphuric acid, fly ash based geopolymer concrete had less mass loss which was 0.5% after 90 days of exposure, while at slight higher mass loss of 0.9% was observed in GGBFS based geopolymer concrete while a huge mass loss of 15.5% was observed in OPC based control concrete after 90 days of exposure.
- GGBFS based geopolymer concrete had 7% reduction in compressive strength after 90 days of acid exposure compared to fly ash based geopolymer concrete which had 7.5% reduction in strength. Highest loss of compressive strength was observed in control concrete made from Ordinary Portland cement and which had 36.6% reduction in compressive strength.
Above observations suggest that Fly ash and slag based geopolymer concrete show excellent resistance against sulphate and acid resistance over control concrete. This indicates that geopolymer concrete is more suitable in sulphate infested and sewer pipe production, which has durability problems when Ordinary Portland Cement concrete is used.

6. ACKNOWLEDGEMENT

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