Study and Manufacture of Geopolymer Concrete using Class C type Fly Ash

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Abstract- Geopolymerization is a developing field of research for utilizing solid waste and by-products. It provides a mature and cost-effective solution to many problems where hazardous residue has to be treated and stored under critical environmental conditions. Geopolymer involves the sodium silicate and sodium hydroxide, by-products to undergo process of geopolymerization. Geopolymer is a material produced by inorganic poly-condensation. “Geopolymerization process comprises dissolution of aluminosilicates followed by condensation of free silicate and aluminates species to form a three-dimensional structure of silico-aluminate structures”. It is environmental-friendly and needs moderate energy for production. Literature demonstrates that certain mix compositions and reaction conditions such as Al2O3/SiO2, alkali concentration, curing temperature with curing time, water/solid ratio and pH significantly influences the formation and properties of a Geopolymer. It is utilized to manufacture precast structures and non-structural elements, concrete pavements, concrete products and immobilization of toxic metal bearing wastes that are resistant to heat and aggressive to environment. Geopolymer gain 70% of the final strength in the first 3-4 hours of curing. Geopolymer concrete based on fly ash is a new material that does not need the presence of Portland cement as a binder. This topic presents information on fly ash based geopolymer concrete using class C type fly ash, it covers the material and mixture proportions, the manufacturing process and the influence of various parameters on the properties of the concrete.

Keywords- Geopolymer concrete, Class C type Fly ash.

1. INTRODUCTION

1.1 Background of the Project:

Concrete which plays the most vital role in terms of construction material is usually associated with Portland cement (cement bricks). It is been estimated that the production of cement has increased from about 1.5 billion tons in 1995 to 2.2 billion tons in 2010 (Malhotra, 1999). Global Warming which is also another rising issue connected with production of cement and is becoming more significant day by day. This is mostly due to manmade activities which results in the emission of CO2. Among Green House Gases CO2 contributes about 65% of Global Warming. The Cement industry emits 6% of CO2 to atmosphere. The aggregates and cement industries together produce nearly 80% of CO2 emissions. The use of Portland cement is unavoidable until the foreseeable future, until a more viable alternative construction material is commercially found. Many efforts are being made in order to reduce the use of Portland cement in brick manufacture. These efforts lead to the utilization of supplementary cementing materials such as fly ash, silica fume, rice husk ash etc. and finding alternative binders to Portland cement. In this respect, the Geopolymer technology proposed by Davidovits (1988a; 1988b) exhibits considerable promise for application in brick industry as an alternative binder to the Portland cement. Inspired by the Geopolymer technology and the fact that fly ash is a waste material abundantly available, this project work on “Geopolymer Concrete Using Class C Type Fly Ash” is carried out to make use of this waste product profitably in an eco-friendly way.

1.2 Objectives of the Project:

The objective of the project is the attainment of maximum compressive strength in the fly ash concrete at the earlier stages using the process of geopolymerization. We are using Steam Curing and Dry Curing. Using the waste materials like fly ash, etc., as a raw material for manufacturing, we can also save our environment for our future generations by preserving the main resources like clay, red soil etc.

The test described in the project was performed to establish the physical and mechanical properties of the new geopolymer concrete. The mechanical properties for different ratios of concrete were determined and the results were also compared. The result shows that the concrete possess good physical and mechanical properties that qualify them for producing high quality geopolymer fly ash concrete.

1.3 Scope of the Project:

This experimental work involved includes conducting short-term compressive strength
tests on class C fly ash based geopolymer concrete.

The scope of the work is that

- Geopolymer concrete manufacturing is a more beneficial technology in utilization of fly ash which might otherwise pose serious disposal issues.
- It reduces the requirement of water and curing period.
- The cement product reduces the CO2 emission by a greater degree and supports an eco-friendly “Industrial Culture”.

2. RAW MATERIALS

2.1 Fly ash:

Fly ash consists of finely divided ashes produced by pulverized coal in power stations. The chemical composition depends on the mineral composition of the coal gangue (the Inorganic part of the coal). Silica content usually varies from 40 to 75% and alumina from 20 to 30%. The iron content varies quite widely. Alkalis are present in an appreciable amount and potassium prevails over sodium. During the hydration process, fly ash chemically reacts with the calcium hydroxide forming calcium silicate hydrate and calcium aluminate, which reduces the risk of leaching calcium hydroxide and concrete's permeability. Fly ash also improves the permeability of concrete by lowering the water content, which reduces the volume of capillary pores remaining in the mass. The spherical shape of fly ash improves the consolidation, which also reduces permeability. The color of fly ash can be tan to dark grey, depending upon the chemical and mineral constituents. Aside from the chemical composition, the other characteristics of fly ash that are generally considered are Loss On Ignition (LOI), Fineness and Uniformity. LOI is a measurement of un-burnt carbon remaining in the fly ash. Fineness of fly ash mostly depends on the operating conditions of coal crushers and the grinding process of the coal itself. Finer gradation generally results in a more reactive ash and contains less carbon.

2.1.1 Types:

The chemical composition of various fly ash shows a wide range, indicating that there is a wide variations in the coal used in power plants all over the world (Malhotra and Ramezanianpour 1994). There are two types of fly ash: Class C and Class F type fly ash.

2.1.1.1 Class C Fly Ash:

Fly ash that results from burning sub-bituminous coals is referred as ASTM Class C fly ash or high-calcium fly ash, as it typically contains more than 20 percent of CaO.

- Class C fly ash is typically not as effective as Class F fly ash in mitigating ASR; one may need to add Class C fly ash at higher amounts in order to achieve the same level of mitigation.
- Class C type will generate more heat of hydration than Class F type.
- Class C type is generally not as resistant to sulphate attack.
- Class C fly ash will generate more strength at early ages than class F type.

2.1.1.2 Class F Fly Ash:

Fly ash from the bituminous and anthracite coals is referred as ASTM class F fly ash or low calcium fly ash. It consists of mainly an aluminosilicate glass and has less than 10 percent of CaO.

2.2 Fine Aggregate:

The Fine aggregate to be used in the SCC has the following properties.

- Natural river sand
- Specific Gravity 2.78
- Fineness Modulus 2.65
- Water absorption 1.05%
- Density 2.3 gm/cm³
- Dry rodded Bulk Density 1610 kg/m³
- Loose Bulk Density 1430 kg/m³

2.3 Coarse Aggregate:

The Coarse aggregate to be used in the SCC has the following properties.

- Crushed blue granite
- 12.5 mm size
- Angular shape
- Specific Gravity 2.61
- Fineness modulus 5.9
- Dry rodded bulk density 1480 kg/m³
- Loose bulk density 1290 kg/m³

2.4 Water:


2.5 Sodium Hydroxide Solution:
The most common alkaline activator used in geopolymerization is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate. The type and concentration of alkali solution affect the dissolution of fly ash. Leaching of Al\(^{3+}\) and Si\(^{4+}\) ions are generally high with sodium hydroxide solution compared to potassium hydroxide solution. Therefore, alkali concentration is a significant factor controlling the leaching of alumina and silica from fly ash particles, subsequent to geopolymerization and mechanical properties of hardened geopolymer. Duchesne et al (2010) confirmed that in presence of NaOH in the activating solution, the reaction proceeds more rapidly and the gel is less smooth. The gel composition analyzed in the sample activated with the mixture of sodium silicate and NaOH is enriched in Na and Al.

Fig2.1 Sodium Hydroxide Pellets

2.6 Sodiun Silicate Solution:

Palomo et al (1999) concluded that the type of activator plays an important role in the polymerization 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. Tempest et al (2009) state that the sodium silicate activator dissolves rapidly and begins to bond fly ash particles. Open porosity can be observed and is rapidly filled with gel as soon as the liquid phase is able to reach the ash particles. The liquid phase is important as a fluid transport medium permitting the activator to reach and react with the fly ash particles.

Fig2.2 Sodium Silicate

3. GEOPOLYMERIZATION

3.1 Polymerization:

Test methods used to study the characteristics of fresh concrete include slump test, U – tube, V – funnel and L – Box. These tests had been conducted to determine the filling ability, passing ability and resistance to segregation of the SCC mix. The process in which many simple monomers (small molecules) join together to form a large complex macromolecule called polymer is termed as the “polymerization”.

e.g. \[ A + B + C + D \rightarrow A \cdot B \cdot C \cdot D \]

Monomers polymer

3.1.1 Types:

Polymerization reaction is of two types:

- **Addition Polymerization**: In which monomers simply join together to form a polymer.
- **Condensation Polymerization**: In which the loss of some small molecule (like H\(_2\)O) also takes place during the addition of monomers to form a polymer.

3.2 Geopolymer:

Geopolymer is a class of inorganic polymer formed by the reaction between an alkaline solution and an aluminosilicate source or feedstock. The hardened material has an amorphous 3-dimensional structure similar to that of an aluminosilicate glass.

3.3 Alkali Activation:

Figure 3.1 show the dissolution process of the Si and Al occurring when the fly ashes are submitted to the alkaline solution. Then the higher molecules condense in a gel form and the alkali attack on the surface of particle, and then expand to larger hole, exposing smaller particles whether hollow or partially filled with other yet smaller ashes to bidirectional alkaline attack from the outside in and from the inside out. Consequently, reaction product is generated both inside and outside the shell of the sphere, until the ash particle is completely consumed.
Fig 3.1 Descriptive model of the alkali activation of fly ash.

Alkali activation of alumino-silicate materials represents a complex process that has not been described to the full yet. The reaction of alumino-silicate materials in a strong alkaline environment results, first of all, in a breakdown of Si-O-Si bonds; later, new phases arise and the mechanism of their formation seems to be a process that includes a solution (“synthesis via solution”). The penetration of Al atoms into the original Si-O-Si structure represents a substantial feature of this reaction. Alumino-silicate gels (geopolymer precursors) are mostly formed. Their composition can be characterized by the formula Mn [-(Si-O) z -Al–O] n. wH2O. The C-S-H and C-A-H phases may also be originated in dependence on the composition of the starting materials and the conditions of the reaction. Even secondary H2O may be formed during these (poly-condensation) reactions. Amorphous (gel-like) or partially amorphous or crystalline substances may be originated in dependence on the character of starting raw materials and on the conditions of the reaction. The concentration of the solid matter plays a substantial role in the process of alkali activation.

3.4 Microstructure of the Geopolymer:

Unlike ordinary Portland cement, geopolymer do not form calcium silicate-hydrates (CSHs) for matrix formation and strength, but utilize the polycondensation of silica and alumina precursors and a high alkali content to attain structural strength. Composition of the geopolymer is similar to natural zeolitic materials, but the microstructure is amorphous instead of crystalline.

Fly ash particles: SEM was used to investigate the surface of fly ash, before and after reacting with NaOH. NaOH reacted with fly ash.

Particles resulted in the roughness of surface as shown in Fig 3.2 and 3.3.
4. EXPERIMENTAL WORK

4.1 Introduction:

This chapter describes the experimental work. First, the materials, mixture proportions, manufacturing and curing of test specimens are explained. This is then followed by descriptions of types of specimens used, test parameters and test procedures.

4.2 Manufacture of Test Specimens:

4.2.1 Preparation of Liquids:

The sodium hydroxide (NaOH) solids were dissolved in water to make the solution. The mass of NaOH solids in a solution varied depending on the concentration of the solution. Similarly the required amount of sodium silicate solution was made. The sodium silicate solution and the sodium hydroxide solution were mixed together with the extra water (if any) to prepare the liquid component of the mixture.

4.2.2 Manufacture of Fresh Concrete and Casting:

The fly ash and the fine sand were first mixed together for about 3 minutes. The liquid component of the mixture was then added to the dry materials and the mixing continued for further about 4 minutes to manufacture the fresh concrete. The fresh concrete was cast into the mould immediately after mixing and well compacted and the top surface is finished.

4.2.3 Curing of Test Specimens:

After casting, the test specimen was placed in the oven for dry curing at optimum temperature of 60°C - 75°C.

After the curing period, the test specimens were left in the mould for at least six hours in order to avoid a drastic change in the environmental conditions. After remolding, the specimens were left to air-dry in the laboratory until the day of test.

4.2.4 Curing Time:

The test specimens were cured for 24 hrs in oven and were found that prolonged curing time was not found to alter the compressive strength of concrete.

4.2.5 Workability of Fresh Geopolymer:

Water plays an important role in geopolymer concrete as much as normal concrete. The used water in the geopolymer is to improve the workability, but it will increase the porosity in concrete due to the evaporation of water during curing process at elevated temperature discovered an increase in sodium hydroxide and sodium silicate concentration will reduce the flow of concrete. To improve the workability of concrete, extra water can be added.

4.3 Manufacture of Concrete:

Geopolymer fly ash brick is manufactured by mixing sand and fly ash in the ratio of 1:2. The concentration of sodium hydroxide solution and sodium silicate solution in the ratio of 10M: 1M and 12M: 1M.

<table>
<thead>
<tr>
<th>Concentrations</th>
<th>Samples</th>
<th>Sodium silicate (ml)</th>
<th>Sodium hydroxide (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>Sample 1</td>
<td>300 (60%)</td>
<td>200 (40%)</td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td>350 (70%)</td>
<td>150 (30%)</td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td>400 (80%)</td>
<td>100 (20%)</td>
</tr>
<tr>
<td>750</td>
<td>Sample 1</td>
<td>360 (60%)</td>
<td>240 (40%)</td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td>420 (70%)</td>
<td>180 (30%)</td>
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<tr>
<td></td>
<td>Sample 3</td>
<td>480 (80%)</td>
<td>120 (20%)</td>
</tr>
<tr>
<td>700</td>
<td>Sample 1</td>
<td>420 (60%)</td>
<td>280 (40%)</td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td>490 (70%)</td>
<td>210 (30%)</td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td>560 (80%)</td>
<td>140 (20%)</td>
</tr>
</tbody>
</table>

Table 4.3 Mix Proportions of Activator Liquids

<table>
<thead>
<tr>
<th>MIXTURE</th>
<th>SODIUM HYDROXIDE SOLUTION</th>
<th>SODIUM SILICATE SOLUTION</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8M</td>
<td>1M</td>
</tr>
<tr>
<td>2</td>
<td>10M</td>
<td>1M</td>
</tr>
<tr>
<td>3</td>
<td>12M</td>
<td>1M</td>
</tr>
<tr>
<td>4</td>
<td>14M</td>
<td>1M</td>
</tr>
</tbody>
</table>

Table 4.1 Molar Concentration of Solution

<table>
<thead>
<tr>
<th>MIXTURE</th>
<th>SODIUM HYDROXIDE SOLUTION (%)</th>
<th>SODIUM SILICATE SOLUTION (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>2</td>
<td>30</td>
<td>70</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>80</td>
</tr>
</tbody>
</table>

Table 4.2 Proportions of Activator Liquids
5. RESULTS AND DISCUSSION

5.1 Introduction:
The experimental results are presented and discussed. The parameters are considered as follows:

- Concentration of sodium hydroxide solution in molarity
- Curing temperature
- Curing time
- In this case high calcium (ASTM Class C) fly ash was used.

5.2 Effects of Salient Parameters:

- Higher the concentration of sodium silicate solution, higher is the compressive strength.
- Higher compressive strength was obtained when the ratio of sodium silicate to sodium hydroxide was 2.33 by mass.
- An optimum curing temperature of nearly 70°C gave higher compressive strength.
- Dry- hot oven curing for 24 hrs gave better results. Curing time can be reduced with an increase in curing temperature.
- Water may be added to the required workability.
- Increased wet mixing time was found to increase the compressive strength to some extent.

Table 4.4 Concrete specimens

<table>
<thead>
<tr>
<th>Molarity</th>
<th>Concentration</th>
<th>Specimen 1</th>
<th>Specimen 2</th>
<th>Specimen 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>10M</td>
<td>500</td>
<td>19.40</td>
<td>20.53</td>
<td>21.09</td>
</tr>
<tr>
<td>600</td>
<td>Sample 1</td>
<td>21.00</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td></td>
<td>21.30</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td></td>
<td></td>
<td>21.50</td>
</tr>
<tr>
<td>700</td>
<td>Sample 1</td>
<td>20.50</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td></td>
<td>21.80</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td></td>
<td></td>
<td>22.10</td>
</tr>
</tbody>
</table>

Table 5.1 Compressive Strength at 7 days

<table>
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<th>Concentration</th>
<th>Specimen 1</th>
<th>Specimen 2</th>
<th>Specimen 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>12M</td>
<td>500</td>
<td>19.90</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 1</td>
<td></td>
<td>21.07</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td></td>
<td>21.50</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td></td>
<td></td>
<td>21.89</td>
</tr>
<tr>
<td>600</td>
<td>Sample 1</td>
<td>20.18</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td></td>
<td>22.60</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
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<td>22.90</td>
</tr>
<tr>
<td>700</td>
<td>Sample 1</td>
<td>22.40</td>
<td></td>
<td></td>
</tr>
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<td></td>
<td>Sample 2</td>
<td></td>
<td>22.10</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td></td>
<td></td>
<td>23.10</td>
</tr>
</tbody>
</table>

6. CONCLUSIONS

From the Exhaustive experimental study presented in the project, the following conclusions are made:

- Higher concentration of sodium silicate results in higher compressive strength of fly ash-based geopolymer concrete.
- The compressive strength of heat cured fly ash-based geopolymer concrete does not depend upon the age.
- Water Absorption is less.
- Since wet curing is not needed, substitution of water is conserved.
- Geopolymer concrete is ecofriendly.
7. REFERENCES