Study of Geopolymer Paste Considering Equivalent Silicate Modulus

Krishnendu Banerjee¹, Ankush Banik¹, Surosrita Pal¹, D. Dutta²,
Dr. T. G. Biswas¹

¹(Department of Civil Engineering, Supreme Knowledge Foundation Group of Institutions, Mankunda, Hooghly-712139, WB, India)
²(Department of Civil Engineering, Camellia School of Engineering & Technology, Barasat, Kolkata-700124, WB, India)

ABSTRACT: This study has been carried out to find out the details of development of various physical properties of geopolymer. Compressive strength, water absorption and sorptivity are some of the most important physical properties which are broadly discussed here. Grey Fly Ash (Class F) is the main constituent material of this geopolymer. Potassium hydroxide and sodium silicate are used here as activator. Percentages of alkali changes in grey fly ash have made an obvious impact on it. This below investigation is broadly depends upon the variation in silicate modulus and also percentage of K₂O but with fixed sample of fly ash. The details and discussion of the experimented properties are discussed below broadly.

KEYWORD: Geopolymer, Fly ash, Potassium hydroxide, Sodium silicate, Alkali activator.

I. INTRODUCTION

Geopolimerisation is nothing but a process of geo-synthesis that includes occurring of silica-aluminates [1]. Geopolymers are generally of aluminium silicate with cross-linked networks. It has charge balancing alkaline cat-ions with water, which retained in the internal pores [2]. The reports suggests the contribution of Ordinary Portland Cement (OPC) production which leads to emission of greenhouse gas, estimated to be approximately 1.35 billion tons annually or approximately 7% of the total greenhouse gas emissions [3]. Geopolymers are activated by alumina-silicate source material such as fly ash, with a highly alkaline solution and also with moderate thermal curing [4]. These tend to exhibit high compressive strength with better durability [5]. Readily dissolved pozzolanic compound or source of silica and alumina in the alkaline solution is a well-known source of geopolymer, contributes itself in geopolymerization [6]. The polycondensation reaction of geopolymeric precursors involves yielding of polymeric Si-O-Al-O bonds. This is also one of the most important mechanisms of geopolymerization [7, 8, 9, 10]. Researches largely on slag, a supplementary material in fly ash based geopolymer, suggests having a favourable effect on physical properties of geopolymer [11]. Addition of calcium compound has been proved to bring quick setting behaviour and more over it enhances strength of geopolymer[12]. These materials are as a result likely to be cement in coming future [13].

The objective of this paper is to observe the development of physical properties of geopolymer considering same type of fly ash but with varying silicate modulus. The silicate modulus (SiO₂/K₂O) are 1 and 1.43 respectively.

II. EXPERIMENTS

A. Materials Used

The type of fly ash has been used here is Grey Flyash and it has been taken from Kolaghat thermal power plant, Kolaghat, West Bengal. Available potassium hydroxide in palettes form and sodium silicate solution is supplied from Loba Chemie Pvt. Ltd, India. Locally available fly ash is sieved by 75 micron. Fly ash of specific gravity 2.04 is used. The provided sodium silicate is with silicate modulus 3.31, silicate solution (SiO₂=26.5, Na₂O =8%, H₂O=65.5%). The supplied KOH palettes are with 84% purity.

Table-1: Chemical analysis report of Fly ash

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>Class F fly ash (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>46.5</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>24.1</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>5.4</td>
</tr>
<tr>
<td>CaO</td>
<td>7.9</td>
</tr>
</tbody>
</table>
Study of Geopolymer Paste Considering Equivalent Silicate Modulus

<table>
<thead>
<tr>
<th>MgO</th>
<th>2.1</th>
</tr>
</thead>
<tbody>
<tr>
<td>K2O</td>
<td>0.9</td>
</tr>
<tr>
<td>Na2O</td>
<td>2.5</td>
</tr>
<tr>
<td>SO3</td>
<td>0.9</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>0.36</td>
</tr>
</tbody>
</table>

Figure-1: Chemical Composition of Class F (Gray) Fly Ash

B. Preparation of specimen and testing

At first the sample fly ash is sieved by 75 microns. At the very out set water is added to KOH and left for over-night [14]. Sodium silicate was added here to maintain silicate modulus in activator i.e. SiO2/K2O ratio is 1 and 1.43 for 8% and 5.58% K2O respectively. Water is incorporated to create a sticky mix in a percentage of 32% of weight of fly ash (Class F). A recent term named equivalent silicate modulus is also incorporated in a manner to find out the combinational effect of potassium and sodium as cations in the process of polymerization[14].

Cubes sizes of 50mmx50mmx50mm were taken and the mix was transferred into it. For 2 minutes vibration was provided in order to remove any entrapped air. At 85°C the cubes were cured in an oven for 2 hours and then allowed to cool inside the oven. [15]. Then the specimen are removed and kept in a dry place. Specimens were tested for compressive strength and micro structural after 3 days rest period.

Table-2: Combination of Geopolymer Specimens

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>K2O content in Activator (%) of fly ash</th>
<th>Equivalent Silicate modulus (SiO2/K2O)</th>
<th>Equivalent Silicate modulus (SiO2/X2O)</th>
<th>Type of specimen</th>
<th>Water / fly ash ratio</th>
<th>Curing temp. and duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>GPG1</td>
<td>8</td>
<td>1</td>
<td>0.77</td>
<td>Paste</td>
<td>0.32</td>
<td>85°C and 48 hrs</td>
</tr>
<tr>
<td>GPG2</td>
<td>5.58</td>
<td>1.43</td>
<td>1</td>
<td>Paste</td>
<td>0.32</td>
<td>85°C and 48 hrs</td>
</tr>
</tbody>
</table>

*Here X indicates the summation of Potassium and Sodium ions.

C. Test Setup:

Sorptivity test:

Sorptivity test of specimen was conducted on specimens, which are previously painted with waterproof enamel paint on all four sides. It is thus set up by which water can only be sucked from bottom. A curve of cumulative mass gained per exposed surface area was drawn against square root of time and the slope of the linear portion was considered for determination of sorptivity values.

a. Test specimens:

50x50x50mm³ cubes are used for this test. There are 4 samples of geopolymer paste cubes from each series.

b. Test Procedure:

The samples were kept in the hot air oven about 85 degree celsius for 48 hours. four sides of the specimen were sealed for achieving unidirectional flow. After sealing, the samples are weighed and was noted
as initial weight. The initial mass was taken when it was immersed into the water at a depth of 5 mm. The reading was taken at predetermined interval (around 2\textsuperscript{nd}, 5\textsuperscript{th}, 10\textsuperscript{th}, 15\textsuperscript{th}, 30\textsuperscript{th}, 60\textsuperscript{th}, 120\textsuperscript{th} minutes). The wet samples were wiped off with a cloth and weighed again. The gain in mass per unit area over the density of water versus the square root of the elapsed time is plotted. The slope of the linear portion of was considered for determination of sorptivity.

**Water Absorption & Apparent Porosity Test:**

For the concrete, water absorption and apparent porosity test plays an important role to indicate its durability and apparent porosity test method indicate the determinations of density, percent absorption and percent voids in concrete paste. This test was carried out to measure the absorption of water by the geopolymer paste.

\textit{a. Test Specimens:}

50×50×50 mm\textsuperscript{3} cubes are used for this test also. There are 4 samples of paste which contain different amount of materials.

\textit{b. Test Procedure:}

The samples were kept in the hot air oven about 85-90 degree Celsius for 48 hours until the mass became constant. After that, the samples were weighed and it is called as dry weight of the cubes. The samples were then immersed in water for 24 hours and the final weight was recorded as its saturated surface dry weight. At the same time water absorption of specimens is recorded as the percentage weight. The saturated specimen is then boiled for 5 hours and allowed to cool for not less than 14hrs to a final temperature of 20 to 25\degree C. The specimen is then suspended by a wire and its apparent mass in water is taken.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{sample_absorption.png}
\caption{Sample subjected to water absorption}
\end{figure}

**Compressive strength test**

A compressive strength machine was used to carry out the compressive strength to evaluate the strength development for the samples the compressive strength was carried out. The curing was done for 48 hours at 85 degree Celsius and then it was kept in room temperature. Two samples were tested for compressive strength in compression testing machine is reported. In the figure below Compressive strength of geopolymer sample is represented. By the result we can see with lower concentration of sodium hydroxide with higher concentration of potassium hydroxide and higher concentration of sodium hydroxide with lower concentration of potassium hydroxide remains same.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{compressive_strength.png}
\caption{Compressive Strength Test}
\end{figure}
III. RESULTS AND DISCUSSIONS

Sorptivity test result:
In this below study the curves for cumulative absorption of water schemed beside square root of time as shown in Fig.2. Though the alkalinity of activator differs in the series of casting but the pore distribution does not affected much. The rate of sorption or the sorptivity is almost same for every. It can be demonstrated in this manner that higher ratio of Si to Ca in grey fly ash emphasize most amorphous geopolymeric structure even at lower alkali content.

| Table3. Values of Water Sorptivity Test |
| Sample Id | Dry Weight (gm) | Weight of saturated Sample at different time interval (gm) |
|  |  | 2min | 5min | 10min | 15min | 30min | 60min | 120min |
| GBG1 | 185.5 | 187 | 187 | 187.5 | 187.5 | 188 | 189 |
| GBG2 | 181.5 | 183 | 183 | 183.5 | 183.5 | 183.5 | 185 |

![Figure 2. Sorptivity Graph](image)

![Figure 3. Results of water sorptivity](image)

Result of water absorption & apparent porosity
Table-4 depicts the results of water absorption and apparent porosity of two respective samples. Water absorption and apparent porosity both are seemed to be almost same for both specimens. As the percentage of void volume occupied by moisture is reflected by apparent porosity so it can be concluded that both the specimens are here with significant permeable pores which is latter occupied by moisture during test. The sample GPG1 has water absorption and apparent porosity of 7.06 and 10.5 respectively. The sample GPG2 has water absorption and apparent porosity of 6.88 and 10.09 respectively. So it is interesting to find the matter that variation of alkali content in activator does not have significant effect on microstructure or pore size distribution.

| Table-4: Results of water absorption, apparent porosity |
| Specimen ID | Water absorption (%) | Apparent porosity (%) |
| GPG1 | 7.06 | 10.5 |
| GPG2 | 6.88 | 10.09 |
Result of compressive strength:
This test suggests that the sample GPG1 is having highest strength of 22MPa and sample GPG2 is having least strength of 15MPa among the two specimens. It is interesting to see that GPG1 has achieved 46.66% higher strength than GPG2. Despite having same pattern of pore morphology GPG1 possess better strength because of higher alkalinity. Actually it can elaborated in the way that in case of GPG1 $K^+$ acts as primary charge compensator of aluminium due to higher presence of $K_2O$. Whereas for GPG2 $Ca^{++}$ acts as primary charge compensator. So probability of formation of pure amorphous structure is feasible for GPG1. But GPG2 brings the possibility of crystalline structure formation.

<table>
<thead>
<tr>
<th>SAMPLE ID</th>
<th>LPAD</th>
<th>SF.AREA</th>
<th>STRENGTH (MPA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GPG1</td>
<td>30</td>
<td>2500</td>
<td>22</td>
</tr>
<tr>
<td>GPG2</td>
<td>30</td>
<td>2500</td>
<td>15</td>
</tr>
</tbody>
</table>

Figure 6. Apparent Porosity Chart

Figure 8. Compressive Strength Chart
IV. CONCLUSION

From the following experiment we conclude the following:
1. The compressive strength of GPG1 is higher. It is having the compressive strength of 22MPa. The sample GPG2 is having the compressive strength of 15MPa. Formation of pure amorphous structure is feasible for GPG1.
2. Variation of alkali content in activator does not have significant effect on microstructure or pore size distribution.
3. GPG1 and GPG2 has almost same result in connection with sorptivity, water absorption and apparent porosity.
4. Design mix considering actual silicate modulus and equivalent silicate modulus effects on strength remarkably.

REFERENCE