Effect of synthesizing parameters on workability and compressive strength of Fly ash based Geopolymer mortar
Anupam Bhowmick¹, Somnath Ghosh²
1- Post Graduate Student, Civil Engg. Deptt. Jadavpur University, Kolkata, India
2- Professor, Civil Engg. Deptt. Jadavpur University, Kolkata, India
anupam.bhowmick@rediffmail.com
doi:10.6088/ijcsen.201203013016

ABSTRACT

Geopolymer is known for its durability and environmental sustainability. The findings of this research paper are based on the experiments conducted on geopolymer mortar synthesized from locally available fly ash. Various engineering properties are highlighted. Influence of processing parameters such as Fly ash/ Sand ratio, SiO$_2$/Na$_2$O ratio, and Water/ Fly ash ratio on workability and compressive strength are studied which may be useful to develop mix design methodology. Pore size distribution and total pores volume are appreciated by Mercury Intrusion Porosimetry (MIP) and their influence on compressive strength. Scanning electron microscopy (SEM) along with Energy dispersive X-ray spectroscopy (EDX) was done for micro-structural and elemental analysis.

Keywords: Geopolymer, fly ash, workability, compressive strength, microstructure.

1. Introduction

Sustainable practice of industrial ecology demands the use of byproduct of one industry as source materials to other industrial application. This will help in complying environmental parameters for both the industry. Environmental concerns compelled us to reduce the material consumption by enhancing the durability and resource efficiency using advancement in technology. (Mehta P.K. 1990). Fly ash based geopolymer may be a solution in this regard. Fly ash is a residue from combustion of coal, improper retention of which cause environmental concern. Fly ash is activated with alkaline solutions and soluble silicates. Sodium / Potassium hydroxide are generally used as alkaline solution successfully supplemented by Sodium / Potassium Silicate.

A lot of research was carried out on the geopolymer paste to understand the basics of geopolymer chemistry. Parameters such as Si/Al ratio, SiO$_2$/Al$_2$O$_3$, Na$_2$O/ Al$_2$O$_3$, Na$_2$O/ SiO$_2$ in the activator solution, water/fly ash ratio were varied in the synthesis of geopolymer and found to affect the geopolymer properties. Choice of source material is important parameters which affect the processing of geopolymer. Thermal activation is an essential requisite of geopolymer formation. However level and duration of thermal activation differs for various source materials. Heat overcomes activation barrier & enhances the dissolution rate of solid alluminosilicate material from byproduct materials such as ASTM class F fly ash. (Thakur R.N., Ghosh S., 2009) Curing at ambient temperature is also possible.

However, exact properties of geopolymer mortar cannot be predicted from the properties of geopolymer paste. Plastic shrinkage induced cracking especially due to thermal curing affect. Addition of filler materials like sand up to a certain extent plays an important role in reducing
Effect of synthesizing parameters on workability and compressive strength of Fly ash based Geopolymer mortar

Anupam Bhowmick, Somnath Ghosh

plastic shrinkage cracking. However, thermal incompatibility between geopolymer paste and sand particles will affect strength. Addition of sand significantly alters the pore structure which affects the processing and properties of geopolymer mortar. Hence it is felt that there is need for research on geopolymer mortar for wider application using locally available fly ash.

2 Experimental programs

2.1 Materials

Geopolymer Mortar was made from low calcium fly ash activated with a combination of sodium hydroxide (NaOH) and sodium silicate solution (Na$_2$SiO$_3$) and mixed with local sand. Fly ashes from Kolaghat thermal power plants were used for investigation. 75% of particles are smaller than 45 micron and specific surface area was 380 m$^2$/kg (Blaine). Sand was having Fineness modulus of 3.2. Table 1 shows the chemical composition of fly ash.

<table>
<thead>
<tr>
<th>Elements</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
<th>TiO$_2$</th>
<th>CaO</th>
<th>MgO</th>
<th>K$_2$O</th>
<th>Na$_2$O</th>
<th>SO$_3$</th>
<th>P$_2$O$_5$</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>% by mass</td>
<td>56.01</td>
<td>29.8</td>
<td>3.58</td>
<td>1.75</td>
<td>2.36</td>
<td>0.30</td>
<td>0.73</td>
<td>0.61</td>
<td>0.44</td>
<td>0.40</td>
<td></td>
</tr>
</tbody>
</table>

Sodium hydroxide in pellets form (Na$_2$O=77.5%, H$_2$O=22.5%) were supplied by Merck Chemical with 97% purity. Sodium Silicate solution (Na$_2$O=8%, SiO$_2$=26.5%, H$_2$O=65.5%) were supplied by Loba Chemie. The SiO$_2$-to-Na$_2$O ratio (Silicate modulus) in the alkaline activating agent was adjusted by adding NaOH to soluble glass in order to obtain silicate modulus values ranging from 0.5 to 1.8. The overall concentration of the alkaline activating agent is 8 % Na$_2$O (expressed as percentage of the fly ash weight).

2.2 Preparation of specimen

Mix Sodium hydroxide pellets and Sodium silicate solution added with extra water (if required) so that desired parameters of geopolymer mortar are achieved. In a Hobart mixer, fly ash was first mixed with the activator solution for 5 minutes before sand was gradually introduced and further mixed for another 5 minutes. The geopolymer mortar mix was then transferred into 50 mm cube moulds and vibrated on a vibrating table for 2 minutes. Specimens were cured along with the moulds in an oven for a period of 48 hours and 72 hours at 80°C and allowed to cool inside the oven before being removed to room temperature until tested. The manufacturing procedure followed was after Thakur and Ghosh. (7)

2.3 Brief description of experimental methods

The workability of fresh geopolymer mortar was determined using Flow Table. Flow test was carried out as per ASTM C1437. The direct compressive strength was obtained at the age of seven days, using 2000kN capacity digital compressive testing machine. Three identical specimens were weighed, tested and the mean values of compressive strength are reported.

3. Results and discussions

3.1 Workability

3.1.1 Effect of Fly ash/Sand ratio
As the proportion of sand is increased in respect to fly ash, the mix becomes non-cohesive leading to decrease in flow value. Percentage of flow value decreases with decrease in Fly ash/Sand ratio. (Ref. to figure no.1)

3.1.2 Effect of SiO$_2$/Na$_2$O ratio in activator

Increase in SiO$_2$/Na$_2$O ratio in activator increases the cohesiveness and flow ability of the mortar mix. At SiO$_2$ / Na$_2$O= 0.5 mortar mix becomes dry, stiff when water/Fly ash ratio is 0.37 and Fly ash/Sand ratio=2:1. It was difficult to fill and compact the mould. But when SiO$_2$/Na$_2$O=1 the fluidity of the mix increases substantially. Increasing SiO$_2$/Na$_2$O at 1.5 flows slightly increases but after that increase in SiO$_2$/Na$_2$O results decrease in % of flow. (Ref. to Figure no.2)

3.1.3 Effect of Water/Fly ash ratio

Particle size, grading and also mix proportion influences the consistency and workability of the mortar. But ultimately workability is the result of the roller bearing effect of the aggregate particles lubricated by geopolymer paste. Increase in water content provides mix flow ability remarkably. (Ref. to Figure no. 3)
Effect of synthesizing parameters on workability and compressive strength of Fly ash based Geopolymer mortar

Anupam Bhowmick, Somnath Ghosh

Figure 3: Flow value vs. Water/Fly ash ratio
(Fly ash/Sand=2:1, SiO$_2$/Na$_2$O=1.5, % of Na$_2$O=8)

3.2 Compressive strength

3.2.1 Effect of Fly ash/Sand ratio

Compressive strength at the end of 48 hours and 72 hours thermal curing is shown in graphical mode (refer Figure no. 4) which clearly show that strength increases with increase in fly ash/sand ratio. At higher sand content, the quantity of the geopolymer gel formed during the process of geopolymerisation may not be adequate to bind all loose aggregates and reduction in compressive strength was observed.

Figure 4: Compressive strength vs. Fly ash/Sand ratio
(Water/Fly ash=0.37, SiO$_2$/Na$_2$O =1.5, Curing temperature=80$^\circ$C, % of Na$_2$O=8)

3.2.2 Effect of SiO$_2$/Na$_2$O ratio in activator

It is observed that the compressive strength increases when SiO$_2$/Na$_2$O ratio increases from 1 to 1.5. But the compressive strength decreases when SiO$_2$/Na$_2$O ratio increases from 1.5 to 1.8. Alkalis promote the dissolution of solid fly ash particles. Its presence is important in
Effect of synthesizing parameters on workability and compressive strength of Fly ash based Geopolymer mortar

Anupam Bhowmick, Somnath Ghosh

controlling the quantity of geopolymeric reaction product without affecting the nature of the product. However soluble silicates affects the degree of polymerization of the dissolved gel. Hence its presence modifies the nature of reaction product. Excess of silicates retard the geopolymeric reaction.

Figure 5: Compressive strength vs. SiO\textsubscript{2}/Na\textsubscript{2}O ratio in activator
(Water/Fly ash=0.37, Fly ash/sand=2:1, Curing temperature=80\textdegree{}C, \% of Na\textsubscript{2}O=8)

3.2.3 Effect of Water/Fly ash ratio

Less water means greater concentration of alkaline solution which accelerates the dissolution rates of aluminosilicate oxides. But water content cannot be lowered beyond a certain limit. It affects the workability of the Geopolymer mix substantially. At lower water content, proper mixing and compaction of the mixes could not be achieved which results in porous material lead to lesser strength. Again, the quantity of water present in the geopolymer mix depends on the quantity of fly ash with respect to sand. If percentage of fly ash in mortar is more, sufficient wetting of fly ash particles causes better geopolymerisation which lead to higher strength. From the study water/fly ash ratio can be taken as 0.40 as for producing a satisfactory mix for locally available fly ash. (Ref. to Figure no. 6, 7, 8)

Figure 6: Compressive strength vs. Water/Fly ash ratio
(Fly ash/Sand=2:1, SiO\textsubscript{2}/ Na\textsubscript{2}O =1.5, \% of Na\textsubscript{2}O=8, Curing temperature=80\textdegree{}C)

International Journal of Civil and Structural Engineering
Volume 3 Issue 1 2012
Effect of synthesizing parameters on workability and compressive strength of Fly ash based Geopolymer mortar
Anupam Bhowmick, Somnath Ghosh

Figure 7: Compressive strength vs. Water/Fly ash ratio
(Fly ash/Sand=1:1, SiO₂/ Na₂O =1.5, % of Na₂O=8, Curing temperature=80⁰C)

Figure 8: Compressive strength vs. Water/Fly ash ratio
(Fly ash/Sand=1:1, SiO₂/ Na₂O =1, % of Na₂O=8, Curing temperature=80⁰C)
3.3 Microstructural studies

3.3.1 Mercury Intrusion Porosimetry (MIP)

Two test specimens were selected for MIP characterization having different SiO$_2$/Al$_2$O$_3$ ratio. Sample 1 marked as SiAl-1 have SiO$_2$/Al$_2$O$_3$ ratio=3.652 (Molar Si/Al=1.826) and the other specimen marked as SiAl-2 have SiO$_2$/Al$_2$O$_3$ ratio=3.88 (Molar Si/Al=1.940). The MIP results are presented graphically for better understanding of the geopolymer mortar.

![Figure 9: MIP graph for specimen SiAl-1](image1.png)

![Figure 10: MIP graph for specimen SiAl-2](image2.png)

Porosity of specimen SiAl-1 is 34 % higher compared to porosity of SiAl-2. Compressive Strength of SiAl-1 is found 19.5Mpa whereas compressive strength of SiAl-2 is found 22.9, MPa. This establishes the fact that lesser porosity results higher compressive strength.
**Table 2:** Different properties of Geopolymer mortar test specimens

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Molar $\text{SiO}_2/\text{Al}_2\text{O}_3$</th>
<th>Molar $\text{Si}/\text{Al}$</th>
<th>$\text{SiO}_2/\text{Na}_2\text{O}$ in activator</th>
<th>Compressive strength (MPa)</th>
<th>Total Pore volume (cc/g)</th>
<th>Minimum Pore diameter ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiAl-1</td>
<td>3.652</td>
<td>1.826</td>
<td>1</td>
<td>19.5</td>
<td>0.1747</td>
<td>$3.909 \times 10^{-3}$</td>
</tr>
<tr>
<td>SiAl-2</td>
<td>3.88</td>
<td>1.940</td>
<td>1.5</td>
<td>22.9</td>
<td>0.1300</td>
<td>$4.039 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

### 3.3.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy along with EDX was done with an EDAX Genesis 2000 X-ray microanalysis system. From the micrographs below it is observed that geopolymer matrix comprises of gel phase and unreacted/partially reacted fly ash particles. Some crystal cracks are appeared on the specimens.

![Figure 11: SEM images of geopolymer specimen](image-url)

(Fly ash/Sand ratio=1:1, $\text{SiO}_2/\text{Na}_2\text{O}$=1.5, Water/Fly ash=0.42, % of $\text{Na}_2\text{O}$=8, curing temperature=$80^\circ\text{C}$, Curing duration=48 hours)
3.3.3 Energy dispersive X-ray Spectroscopy (EDX)

EDX was done to obtain the elemental analysis of the geopolymeric gel produced. All the basic elements in the fly ash, activators are found in the analysis. Si, Al, Na, O, Mg, Fe all elements are shown in the spectrum taken from Geopolymer specimen during X-ray micro analysis. Relative % of weight and atomic weight percentage of all the elements are also obtained from the EDX results.

![Figure 14: EDX Spectrum of geopolymer specimen](c:edw032geenesidgymspec.spc)
(Fly ash/Sand ratio=1:1, SiO$_2$/Na$_2$O=1.5, Water/Fly ash=0.42, % of Na$_2$O=8, curing temperature=80°C, Curing duration=48 hours)

4. Conclusions

Geopolymer mortar of reasonable workability and strength can be produced by activating locally available fly ash with sodium hydroxide and sodium silicate solution. Flow value of geopolymer mortar depends on Fly ash/Sand ratio, SiO$_2$/Na$_2$O ratio, and Water/Fly ash ratio. Workable mortar of moderate flow values ranges from 50% to 80% may be produced by suitably adjusting the above mentioned ratios. Geopolymer mortar of compressive strength of 15 to 28 MPa could be produced. Fly ash / Sand ratio as 2:1, SiO$_2$/Na$_2$O in the activator as 1.5, and Water/Fly ash ratio in the range of 0.37 to 0.40, may be considered for producing good quality mortar. MIP analysis establishes the dependence of compressive strength on the volume of pores. SEM along with EDX confirms the successful geopolymerisation of the fly ash activated by alkaline solution.

5. References


