Mechanical Characteristics of Geopolymer Mortar in Ambient Air and Immersion Curing

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Abstract: Geopolymer mortar is an environmentally sound alternative to the conventional concrete, which could assist in reducing the carbon footprint that augments the process of global warming. The current work presents a comparative study on the compressive strength and ultrasonic pulse velocity of geopolymer mortar mixes at activator modulus (SiO₂/Na₂O) of 1.25, 1.50 and 1.75 subjected to ambient air curing and immersion curing. The dynamic modulus of elasticity determined from the UPV results are analogized with the compressive strength. The study also aims at developing economic geopolymer mortar with acceptable mechanical properties. The three mixes considered for the study include geopolymer mortar with 100% slag, 50% slag and 50% fly ash and 50% red mud and 50% slag. The molarity of alkaline solution and sand-binder ratio is fixed as 8M and 1.50 respectively. The results indicate that the immersion cured specimens of the mix with 50% slag and 50% fly ash corresponding to the activator modulus 1.50 has satisfactory strength performance. The dynamic modulus of elasticity was found to be higher for the immersion cured specimens.

Keywords: Geopolymer, mortar, Activator modulus, Fly ash, Slag, Red mud, Strength, Ultrasonic Pulse Velocity

1. Introduction

The geopolymers are inorganic polymers that are capable of efficaciously reducing the emission of carbondioxide, significantly contributed from the construction industry. Geopolymers are produced by reacting the alkaline solution with the silicates and aluminates to produce binders. The waste products and industrial by products such as ricehusk ash, flyash, slag, metakaolin, redmud and volcanic ash are the source materials for the silicates and aluminates [1]. Geopolymer concrete is a cementless concrete that helps in achieving sustainability in the construction field. The effective utilization of the waste materials produced is possible with the systematic usage of geopolymer concrete.

The prime focus of the study is on the compressive strength development of the geopolymer mortar which is highly influenced by the parameters namely activator modulus (SiO₂/Na₂O), water to geopolymer solids (w/s) ratio, workability, curing conditions, silica content, alkali content, alkali to binder ratio (a/b), sand to binder ratio, temperature and duration of curing. Murali et al. [2] suggested that the compressive strength of fly ash based geopolymer mortar increases linearly with an increase in the alkali content. There is a remarkable decrease in the strength development with increase in the w/s ratio. It is well established in the study concluded by Islam et al. [3] that the early strength development in geopolymer mortar is significantly higher than the ordinary cement mortar. About 90
percent of compressive strength is achieved during the first 7 days. Razak et al. [4] reported that the strength attainment was greater when the specimens were subjected to curing in 1M NaOH compared to ambient curing. Ma et al. [5] reported that curing temperature considerably affected the strength enhancement of the flyash - slag blends. Temperature is responsible for the activation of aluminosilicates. The slag activation occurs at a lower temperature whereas higher temperature promote both the flyash and slag activation. Nuruddin et al. [6] observed that when the molarity of the solution exceeded 12M, the strength attainment was negatively affected.

The need of the hour is to optimise the activation condition so as to obtain environmental as well as economic sustainability by producing geopolymer with minimum activator modulus. This could be satisfied if researches are done by focussing on the activator modulus. Activator modulus (AM) is a vital parameter which governs the activation condition. It can be defined as the ratio of SiO₂ to Na₂O of the alkaline solution [7]. This paper presents a comparative study on the compressive strength of different geopolymer mortar mixes in ambient air and immersion curing condition by varying the activator modulus.

2. Experimental Programme

The objective of the paper is to conduct a comparative study of the mechanical property of the geopolymer mortar. Three different mixes were prepared using slag (GGBS), fly ash (FA) and red mud (RM). Mix 1 (M1-100% Slag), Mix 2 (M2-50% Slag and 50% FA) and Mix 3 (M3-50% Slag and 50% RM) were prepared with activator modulus of 1.25, 1.50 and 1.75. The specimens cast for each mix were subjected to both ambient and immersion curing. The work is also meant to assess the possibility of producing economic geopolymer mortar with minimum alkali content by varying the activator modulus. The mix design is done as per the standard codal provisions [7]. AM and w/s ratio was chosen based on the codal provisions which specified that AM is to be between 1 and 2 and w/s to be less than 0.50 respectively. The w/s ratio was fixed as 0.30 and the sand to binder ratio was fixed as 1.50 respectively. The ratio of Na₂SiO₃/NaOH was calculated corresponding to the fixed activator modulus of 1.25, 1.50 and 1.75. From the ratio obtained, the quantity of alkaline solution required for the each mix was calculated.

Materials procured for producing geopolymer mortar are as given below:

- Ground Granulated Blast Furnace Slag (GGBS)
- Fly ash (FA)
- Red Mud (RM)
- Sodium Hydroxide (NaOH) pellets
- Sodium Silicate (Na₂SiO₃) solution
- Graded M-Sand (passing 2 mm sieve)

Fly ash (Class F) and slag was obtained in powdered form. Red mud from an aluminium industry was sieved through IS 150 micron sieve and used. Measured quantity of NaOH pellets was dissolved in appropriate amount of water and then mixed with Na₂SiO₃ solution to form the 8M alkaline solution required to produce the geopolymer mortar mix. The 8M solution was prepared atleast 24 hours prior to mixing. Na₂SiO₃ was obtained in solution form from Minar Chemicals, Ernakulam, Kerala which contained 50.3 percent solids with an AM of 2.20. Graded M-Sand was used for the casting purpose. The sand passing 2 mm and retained in 1 mm, 500 micron and 90 micron sieve was taken in equal quantity for obtaining the standard gradation of sand. The mixes used are shown in the Table 1 given below:
Table 1. Details of mixes used in the study

| Mix      | Binder       | AM | (Na₂SiO₃)/(NaOH) | Alkali/Binder |
|----------|--------------|----|-----------------|---------------|
| Control  | 100% OPC     |    | -               | -             |
| Mix 1 (M1) | 100% Slag | 1.25 | 1.70   | 0.60          |
|          |              | 1.50 | 2.77   | 0.67          |
|          |              | 1.75 | 5.02   | 0.73          |
| Mix 2 (M2) | 50% Slag + 50% FA | 1.25 | 1.70   | 0.60          |
|          |              | 1.50 | 2.77   | 0.67          |
|          |              | 1.75 | 5.02   | 0.73          |
| Mix 3 (M3) | 50% Slag + 50% RM | 1.25 | 1.70   | 0.60          |
|          |              | 1.50 | 2.77   | 0.67          |
|          |              | 1.75 | 5.02   | 0.73          |

The specific gravity of the binders to be used in the mortar mix was obtained as shown in Table 2 as given below:

Table 2. Material Property of Binders

| Binder     | Specific Gravity |
|------------|------------------|
| Cement     | 3.14             |
| Slag       | 2.92             |
| Fly ash    | 2.21             |
| Red Mud    | 2.63             |

2.1 Casting of Specimens

The specimens were cast in plastic cylindrical moulds, including the control specimens. The control specimens were cast with a w/c ratio of 0.30 by the addition of 1% superplasticizer (Conplast SP430). Specimens of 50mm height and 25mm diameter were cast. The compaction was provided in form of tamping of the filled cylinders. After 24 hours, the specimens were demoulded and kept for ambient as well as immersion curing. Control mix specimens are cured by immersing in water only. Potable well water was used for the immersion curing. The cast specimens are shown in the Figure 1. Figure 2 shows the demoulded specimens.

Figure 1. Cast Specimens in plastic moulds
2.2 Compressive Strength Test
The compressive strength test is done in digital compression testing machine on cylindrical mortar specimens of 25 mm diameter and 50 mm height with the aid of a fabricated test rig. The loading rate is kept at 0.5 kN/sec. The test is done after 7 and 28 days for the specimens cured in ambient condition and specimens cured by water immersion. The strength of different mixes with varying activator modulus is then analysed. Figure 3 shows the compression testing of a cylindrical specimen in progress.

2.3 Ultrasonic Pulse Velocity (UPV) Test
UPV test is a non destructive technique intended to establish the homogeneity of the mortar and also to assess the quality of mortar. The UPV values obtained from the test indicate indirectly about the strength of mortar. The test apparatus comprise of an electrical pulse generator, a pair of transducer, amplifier and an electronic timing device. The transducer used for the current experiment is 150kHz. The test is done in accordance with [8]. The machine is calibrated before testing. When the pulse is induced by transducer at one end, it undergoes multiple reflection and is received at the opposite end. The velocity and time taken to pass through the specimen is digitally captured. The quality of specimen is denoted by the ultrasonic pulse velocity. A couplant gel is employed to ensure smooth transmission of the pulse. Table 3 below shows the quality grading of the concrete specimens corresponding to different velocity ranges.
Table 3. Velocity Criterion for Concrete Quality Grading (Source: IS 13311-1992)

| Pulse velocity by cross probing (km/sec) | Concrete Quality Grading |
|------------------------------------------|--------------------------|
| Above 4.5                                 | Excellent                |
| 3.5 - 4.5                                 | Good                     |
| 3 - 3.5                                   | Medium                   |
| Below 3                                   | Doubtful                 |

Figure 4 demonstrates the ultrasonic pulse velocity test done on the cylindrical specimens. The dynamic Young’s modulus of elasticity ($E_d$ in MPa) of concrete can be obtained from the pulse velocity ($V$ in m/s) and dynamic poisson's ratio ($\mu$) as per the formula given below (Eq. 1).

$$E_d = \frac{\rho(1+\mu)(1-2\mu)}{(1-\mu)} V^2$$  \hspace{1cm} \text{Eq. (1)}

Where,

- $E_d$ - Dynamic Young’s modulus of Elasticity in GPa
- $V$ - Pulse velocity in m/sec
- $\mu$ - Dynamic Poisson's ratio
- $\rho$ - density in g/cm$^3$

The value of dynamic poisson's ratio varies between 0.20 to 0.35, with 0.24 as average. The poisson's ratio of mortar assumed for the study is 0.20. The bulk density was computed from the weighed mass and volume of each specimens.

3. Results and Discussions
The results obtained are discussed in the following sections.

3.1 Compressive Strength Test Results
The compressive strength test results of 7th and 28th day under two different conditions of ambient and immersion curing are presented in Tables 4 and 5.

From the results obtained, it could be inferred that there is an inverse relation of compressive strength with the activator modulus (AM). There is no significant change in the compressive strength up to an AM of 1.50. As the AM exceeds 1.50, a noticeable decrease in the strength is observed. This could be due to the existence of poorly geopolymerised products at higher AM. Activator modulus in geopolymer concrete design could be closely associated to the water-cement ratio in the conventional mix design. Both the parameters affect the development of compressive strength in a similar manner. It could be recognized from the results that the immersion cured specimens exhibit greater strength.
compared to the air cured specimens, except for the mix M1 with 100% slag. For M1, there is marginal decrease in the compressive strength at 28th day when compared to 7th day.

Table 4. Compressive Strength Test Results of 7th day

| Mix         | Activator modulus | 7th day Compressive Strength (MPa) | Ambiant curing | Immersion curing |
|-------------|-------------------|-----------------------------------|----------------|------------------|
| M1 (100% slag) | 1.25              | 55.62                             | 56.03          |
|             | 1.50              | 47.63                             | 55.30          |
|             | 1.75              | 29.88                             | 49.00          |
| M2 (50% slag & 50% FA) | 1.25              | 21.65                             | 40.703         |
|             | 1.50              | 20.36                             | 36.05          |
|             | 1.75              | 12.68                             | 23.36          |
| M3 (50% slag & 50% RM) | 1.25              | 22.70                             | 33.19          |
|             | 1.50              | 20.30                             | 32.44          |
|             | 1.75              | 13.30                             | 27.45          |
| Control (100% OPC) | -                 | 47.78 (immersion curing)          |                |

Table 5. Compressive Strength Test Results of 28th day

| Mix         | Activator modulus | 28th day Compressive Strength (MPa) | Ambiant curing | Immersion curing |
|-------------|-------------------|-----------------------------------|----------------|------------------|
| M1 (100% slag) | 1.25              | 54.26                             | 46.21          |
|             | 1.50              | 40.21                             | 46.79          |
|             | 1.75              | 28.47                             | 44.97          |
| M2 (50% slag & 50% FA) | 1.25              | 20.15                             | 46.55          |
|             | 1.50              | 18.47                             | 46.39          |
|             | 1.75              | 16.04                             | 31.27          |
| M3 (50% slag & 50% RM) | 1.25              | 22.60                             | 37.69          |
|             | 1.50              | 19.44                             | 36.89          |
|             | 1.75              | 13.19                             | 28.41          |
| Control (100% OPC) | -                 | 62.52 (immersion curing)          |                |

Figure 5. Variation of 7th day Compressive Strength with Activator Modulus
Figures 5 and 6 show the variation in compressive strength with activator modulus (AM) for 7 day and 28 day curing respectively. The 7 day and 28 day strength was found to decrease with an increase in the activator modulus irrespective of curing conditions. Among the three mixes considered for the study, the mix M1 showed the highest strength whereas the mix M3 showed the least strength. The inferior strength performance exhibited by M3 may be ascribed to the low reactive Al₂O₃ content [9]. Immersion cured M2 specimens showed similar strength performance level of M1 at the 28th day.

The obtained strength results indicate that M1 is better for constructional purposes. But the setting time of M1 observed is too early that it is practically difficult to be used for construction and this concern needs further study. It would be more productive if a percentage of slag in M1 is replaced by other binders like fly ash so that the setting time could be delayed further. Hence M2 under immersion curing yields better results and is practically possible to be used for construction. Compared to ambient curing, immersion curing was found to give better strength results for the geopolymer mortar.

### 3.2 Ultrasonic Pulse Velocity Test Results

Table 6 and Table 7 provide the UPV results of three different mixes in ambient and immersion curing for 7th and 28th day. It is found that UPV decreases with increase in activator modulus. The quality of mortar is better if the UPV is higher. Thus from the obtained results, it could be concluded that the mortar quality is better for immersion cured specimens. The quality of mortar increases with duration of immersion curing. Excellent (>4500m/s) quality of mortar is visible for all immersion cured specimens at 28th day. There is a variation in the results of Mix 2 and Mix 3 subjected to immersion curing. This may be due to variation in compaction, surface irregularities and height variation occurred during the casting of small sized specimens.

Table 8 elucidates the mass density values of 7 day and 28 day cured specimens subjected to ambient and immersion curing. These values are employed for obtaining the dynamic modulus of elasticity (E_d). The dynamic modulus of elasticity of 7 day and 28 day cured specimens, computed from the UPV values are illustrated in Figure 7 and Figure 8. The E_d values of immersion cured specimens is progressively greater when compared to the ambient air cured mortar specimens. Additionally, it could be apparently deduced that the E_d values declines with the increase in activator modulus. Immersion cured Mix 3 specimens show higher dynamic modulus of elasticity than the immersion cured Mix 2 and Mix 1 specimens (for AM values 1.5 and 1.75). This behaviour is contrary to the strength characteristics exhibited by the immersion cured Mix 3 specimens.
### Table 6. Ultrasonic Pulse Velocity Results of 7th day

| Mix                  | Activator modulus | 7th day UPV(m/s) |  
|----------------------|-------------------|-----------------|  
|                      |                   | Ambient curing  | Immersion curing |  
| M1 (100% slag)       | 1.25              | 4439.50         | 4555.00          |  
|                      | 1.50              | 3838.50         | 4309.50          |  
|                      | 1.75              | 3106.50         | 4122.50          |  
| M2 (50% slag & 50% FA) | 1.25            | 3000.00         | 4015.62          |  
|                      | 1.50              | 2599.50         | 3762.50          |  
|                      | 1.75              | 2460.50         | 4488.00          |  
| M3 (50% slag & 50% RM) | 1.25            | 3213.50         | 4554.00          |  
|                      | 1.50              | 2369.00         | 4619.50          |  
|                      | 1.75              | 2249.50         | 4276.50          |  
| Control (100% OPC)   | -                 |                 | 4975.45          |  

### Table 7. Ultrasonic Pulse Velocity Results of 28th day

| Mix                  | Activator modulus | 28th day UPV(m/s) |  
|----------------------|-------------------|-----------------|  
|                      |                   | Ambient curing  | Immersion curing |  
| M1 (100% slag)       | 1.25              | 5186.00         | 5593.00          |  
|                      | 1.50              | 4019.00         | 4420.00          |  
|                      | 1.75              | 3681.00         | 4024.00          |  
| M2 (50% slag & 50% FA) | 1.25            | 3335.00         | 4388.00          |  
|                      | 1.50              | 2913.00         | 4120.50          |  
|                      | 1.75              | 2908.00         | 4595.50          |  
| M3 (50% slag & 50% RM) | 1.25            | 3472.50         | 5057.00          |  
|                      | 1.50              | 3027.00         | 5011.00          |  
|                      | 1.75              | 2816.50         | 4342.50          |  
| Control (100% OPC)   | -                 |                 | 6049.38          |  

### Table 8. Density values of 7 day and 28 day cured specimens

| Mix                  | AM | Density(kg/m³) 7th day | Density(kg/m³) 28th day |  
|----------------------|----|------------------------|--------------------------|  
|                      |    | Air Curing | Immersion Curing | Air Curing | Immersion Curing |  
| M1 (100% slag)       | 1.25 | 2307.64        | 2337.28 | 2399.16        | 2396.8         |  
|                      | 1.50 | 2301.48        | 2311.68 | 2304.84        | 2310.04        |  
|                      | 1.75 | 2155.20        | 2180.72 | 2287.04        | 2315.92        |  
| M2 (50% slag & 50% FA) | 1.25 | 2148.92        | 2188.28 | 2186.16        | 2217.44        |  
|                      | 1.50 | 2155.20        | 2180.96 | 2158.88        | 2191.52        |  
|                      | 1.75 | 2124.52        | 2149.48 | 2089.96        | 2177.44        |  
| M3 (50% slag & 50% RM) | 1.25 | 2327.72        | 2320.04 | 2308.64        | 2288.56        |  
|                      | 1.50 | 2291.24        | 2319.00 | 2278.52        | 2287.64        |  
|                      | 1.75 | 2277.80        | 2297.48 | 2237.96        | 2263.64        |  
| Control (100% OPC)   | -   | 2336.38        | 2413.85 | 2413.85        |  

Figure 7. Dynamic Modulus of Elasticity values for different mixes at 7th day

Figure 8. Dynamic Modulus of Elasticity values for different mixes at 28th day

Figure 9. Variation of 7th day compressive strength with 7th day dynamic modulus of elasticity
Figure 10. Variation of 28th day compressive strength with 28th day dynamic modulus of elasticity

Figure 9 and Figure 10 represents the variation of the compressive strength with the dynamic modulus of elasticity for the 7 day and 28 day cured specimens respectively. The graphs depict that the compressive strength is directly related to the dynamic modulus of elasticity. Mix 1 gives higher $E_d$ values when compared to the other mixes investigated. The compressive strength and dynamic modulus of elasticity could be reasonably positively correlated for all the geopolymer mortar mixes considered in the study.

4. Conclusion
The geopolymer concrete is a promising alternative to conventional concrete as it can contribute emphatically in reducing the carbon footprint produced largely by the cement manufacturing industries. The present study focussed on optimising the activation condition so that economical achievement of environmental sustainability in geopolymer system could be possible by minimising activator modulus.

The specific conclusions drawn from the study are as follows:
1. Immersion cured geopolymer mortar specimens have higher strength performance compared to the ambient cured specimens for all the mixes investigated. The mix containing slag alone (M1) exhibits enhanced strength performance in both the ambient and immersion curing conditions for the activator modulus studied.
2. The compressive strength has an inverse relation with the activator modulus. A significant reduction in compressive strength is observed when activator modulus value exceeds 1.50.
3. Based on the strength requirements, Mix 1(100% slag) is superior to both Mix 2 (50% slag and 50% fly ash) and Mix 3 (50% slag and 50% red mud). But Mix 2(50% slag and 50% fly ash) is adjudged to be a better option for practical constructions, considering the delayed setting time and the enhanced workability observed at the time of casting. Mix 3(50% slag and 50% red mud) shows the least strength performance among the mixes investigated but more research may be done on processing red mud to utilise it in a better way.
4. Ultrasonic pulse velocity and dynamic modulus of elasticity has an inverse relation with the activator modulus.
5. Overall, the mortar compressive strength is observed to have good positive correlation with dynamic modulus of elasticity.
6. The Mix 2 (blend of 50% slag and 50% fly ash) prepared with an activator modulus of 1.50 subjected to immersion curing could be adopted for construction purpose to achieve environmental sustainability when compared to Ordinary Portland Cement based concrete.
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