The Effect of Sodium Hydroxide Molarity and Other Parameters on Water Absorption of Geopolymer Mortars

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Abstract

Objective: Durability is one of the critical role effects in geopolymer serviceability. The capability of water to penetrate the mortar microstructure is mainly used to measure the durability, which is also called permeability.

Methods/Statistical Analysis: The influence of sodium hydroxide (NaOH) molarity, alkali Solution to Binder ratio (S:B), binder to aggregate ratio (B:A) and sodium silicate to sodium hydroxide ratio (NS:NH) on water absorption of Geopolymer Mortar (GPMs) are investigated in this article. Geopolymer mortar specimens of different NaOH molarity, S:B, B:A, and NS:NH were prepared and cured at different temperatures, 27°C and 60°C. Granulated blast furnace slag, fly ash, waste ceramic and waste glass bottle were used as a binder and mixed with river sand as fine aggregate.

Findings: The results of experimental indicated that water absorption significantly affected by curing temperatures. The lower water absorption results were observed of the samples cured at ambient temperature (27°C) compared to samples cured at oven temperature (60°C).

Applications/Improvements: The geopolymer specimens’ water absorption observed positively enhanced with increase concentration of alkaline solution.

Keywords: Geopolymer, Sodium Hydroxide Molarity, Water Absorption.

1. Introduction

Undoubtedly, the exposure of concrete to external environmental conditioning including extreme heat, cold and stress during service life is inevitable. Concrete is known to shrink and expand with the variations of moisture contents, pressure and temperature. Many researchers reported in aggressive environments the conventional concrete showed short service life and start deteriorate even designed for more than 50 years. Also the saturated concrete is liable to freeze-thaw damage. Scherer and valenza have reported the high water absorption can be stimulated disruptive alkali-aggregate expansion, freeze-thaw damage and chloride ingress and reduce the concrete durability. Many researchers have been published. In absorption tests the volume of voids in the concrete that have been filled or partially filled with water is measured for specific conditions of immersion and time intervals. The water absorption obtained by total immersion of specimens in water gives an indication of the open pore volume. However, if water is absorbed into the concrete by capillary action, a measure of the rate of absorption provides a useful allusion of the pore structure of concrete. The high rate of water absorption is indicative of a relatively poor structure; the higher rate of water absorbed, the poorer the quality of concrete. The tests do not measure true permeability, but they are simple and quick, and can provide a convenient means of obtaining a permeability index and are relevant to the durability of concrete.

In reported the durability of GPMs significantly

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influenced by water absorption and porosity rate. The lowest compressive strength observed with high rate of water absorption, porosity and water sorptivity. The geopolymerization mechanism plays the critical role in microstructure composition of hardened GPMs. Many factors effect on geopolymerization mechanism as NaOH molarity, silicate content, curing temperature.

The aim of this study to assess the influence NaOH morality and other factor such as solution type, sodium silicate and binder content on GPMs water absorption, also study the effect factors on microstructure and compressive strength development.

2. Methodology

This section describes materials, mixture proportioning, specimen preparation, curing conditions and the procedures of the testing in detail. Eight various sodium hydroxide concentrations have depended to design the GPMs mixtures to evaluate the effect of alkaline solution concentration on GPMs water absorption under various curing temperatures. After 28 days from casting date, the compressive strength and water absorption have been evaluated.

2.1 Materials

Granulated Blast Furnace Slag (GBFS), low calcium Fly Ash (FA), Ceramic Powder (CP) and Glass Powder (GP) used as binder to prepare the mixtures. The results of X-Ray Fluorescence Spectroscopy (XRF) test presented in Table 1 shows the chemical composition of materials. Their XRD and SEM are shown in Figures 1 and 2.

| Material | OPC | GP | FA | GBFS | CP |
|----------|-----|----|----|------|----|
| SiO$_2$  | 20.8| 69.14| 57.20| 30.8| 72.8|
| Al$_2$O$_3$ | 4.7 | 13.86| 28.8| 10.9| 12.2|
| Fe$_2$O$_3$ | 3.4 | 0.24| 3.67| 0.64| 0.56|
| CaO      | 65.3| 3.16| 5.16| 51.8| 0.01|
| MgO      | 1.5 | 0.68| 1.48| 4.57| 1.0|
| K$_2$O   | 0.4 | 0.01| 0.94| 0.36| -|
| Na$_2$O  | 0.12| 0.01| 0.08| 0.45| 13.5|
| SO$_3$   | 2.7 | 4.08| 0.10| 0.06| -|
| LOI      | 0.90| -   | 0.12| 0.22| -|

Figure 1 illustrates the XRD patterns of GP, FA, GBFS and CP. The XRD pattern of GP and GBFS verifies their highly amorphous nature because of the absence of any sharp peak. The reactive content of SiO$_2$ and Al$_2$O$_3$ significantly effect on GPM formation. However, incorporation of FA is required to overcome the low Al$_2$O$_3$ content (2.48 wt.%). The XRD pattern of FA and CP revealed pronounced diffraction peaks around 2q = 16-30°, which are attributed to the crystalline silica and alumina compounds. Nonetheless, the occurrence of other crystalline peaks is ascribed to the presence of crystalline quartz and mullite phases.

Figure 2 shows the SEM images of FA, GBFS, WC and, WBG, respectively, which are blended in the GPMs to achieve enhanced comprehensive strength and improved micro structural properties. It is evident that FA consists of spherical particles with smooth surface, while the GBFS comprises irregular and angular particles.
Fine aggregate, available river sand had a specific gravity of 2.74 passing from sieve 2.36 used. Sodium hydroxide of 98% purity used to prepare various NaOH molarities after added water. Sodium silicate had modulus elasticity 2.0 was added to sodium hydroxide to prepare final alkaline solutions 15. In this study sodium hydroxide prepare before 24 hours with different concentration and mixed with sodium silicate and used as an alkali activator solution.

2.2 Mixture Proportions

Various sodium hydroxide molarities, binder to aggregate (B:A), sodium silicate to sodium hydroxide (NS:NH) and solution to binder (S:B) used to prepare GPMs mixtures as presented in Table 2. Two mixtures prepared by using different two solution types, the first one prepared with normal solution NHNS and a second mixture prepared with sodium silicate solution (NS) without any addition of NaOH, the binder was fixed for all types of mixtures. Multi blend content four types of waste materials furnace slag (GBFS), fly ash (FA), waste ceramic (CP) and waste glass bottle (GP) were mixed together till be homogenous and used as GPMs binder Table 3.

Ordinary Portland cement mortar prepared with (1:3) cement to sand ration and 0.48 water: cement (W:C), cured for 7 days in water and testing after 28 days was used a control sample in this laboratory experiment.

Table 2. GPMs binder content

| No. | GBFS | FA | CP | GP |
|-----|------|----|----|----|
| 1   | 55   | 15 | 15 | 15 |

2.3 Specimen Preparation

Mixing was performed in accordance with ASTM C109. The multi blend content GBFS, FA, CP and GP was mixed two minutes, then followed by add the sand and mixed for three minutes till it became homogenous. Followed by alkaline solution addition and mixed for another three minutes.

50 mm cube specimens casted in steel moulds. In two layers and shaken for 15 second for each layer using vibration table, mortar was placed in the moulds with two layers. At laboratory atmosphere (27°C and 75% RH), specimens was left for hardened then the moulds was opened after 24 hours.

Table 3. GPMs mix proportion

| Information | Alkali solution |
|-------------|-----------------|
| Factor      | M   | S:B | NS:NH | B:A |
| NaOH Molarity |     |     |      |     |
| 1           | 2   | 0.35| 3.0   | 1.5 |
| 2           | 4   |     |      |     |
| 3           | 6   |     |      |     |
| 4           | 8   |     |      |     |
| 5           | 10  |     |      |     |
| 6           | 12  |     |      |     |
| 7           | 14  |     |      |     |
| 8           | 16  |     |      |     |

| S:B          | 1   |     |      |     |
|              | 2   | 0.40|      |     |
|              | 3   | 0.45|      |     |
|              | 4   | 0.50|      |     |
|              | 5   | 0.55|      |     |

| NS:NH        | 1   |     |      |     |
|              | 2   |     | 1.5  |     |
|              | 3   |     | 2.0  |     |
|              | 4   |     | 2.5  |     |
|              | 5   |     | 3.0  |     |
|              | 6   |     | 3.5  |     |
|              | 7   |     | 4.0  |     |

| B:A          | 1   |     |      |     |
|              | 2   |     | 0.30 |     |
|              | 4   |     | 1.0  |     |
|              | 5   |     | 1.5  |     |

| Type of solution | 1   |     |      |     |
|                 | 2   | 0.35| 100% | 1.5 |

2.4 Curing Conditions

Two different curing methods were employed for the study of absorption characteristics; after de-moulding the samples, a set of cubes were cured at ambient temperature while the other set were initially kept in the oven at 60°C for 24 hours before been taken out and left in ambient temperature till testing date.

2.5 Water Absorption Test

Most publications dealing with water absorption in concrete are concerned with samples that have been oven-dried to constant weight. A procedure according to ASTM C140-07 was used; however, after the specimens matured, they were immersed in water at a temperature of 26°C for 24 hours. The specimens taken outside the water and measure the Saturated Weight (WS). After that, all specimens were dried in a ventilated oven at 105°C for not less than 24 hours as shown in Figure 3. For the determination of water absorption by total immersion, the
The relationship between Water Absorption (WA) and Compressive Strength (CS) was also reported. Figure 5 presented the results of average of three specimens tested after 28 days and cured at different conditions. Increase sodium hydroxide molarity significantly effect on development of compressive strength and enhance the geopolymerization process.

Also the results showed the compressive strength increased with decrease water absorption. Highest compressive strength observed of specimens prepared with 12 M of alkaline solution molarity and for two types of curing. Samples cured at ambient temperature showed higher compressive strength (54 MPa) than samples cured at elevate temperature (52.6 MPa).

The effect of sodium hydroxide molarity and other parameters on water absorption of geopolymer mortars

Dry mass (Wd) for each sample was recorded and then WA was calculated using the formula shown in equation (1):

$$WA(\%) = \frac{W_s - W_d}{W_d} \times 100$$  \hspace{1cm} (1)

Figure 3. Water absorption test process.

### 3. Experimental Results

#### 3.1 Effect of NaOH Molarity

Figure 4 display the results of WA tests after 28 days. Increase sodium hydroxide molarity from 2 M to 16 M lead to enhance the density of specimens and reduce the voids content, increase molarity contributed to reduce the water content and improve the microstructure of samples. Most samples cured at ambient temperature showed lower water absorption compared to samples cured in oven condition, increased curing temperature effect on CSH mechanism which led to the microstructure of the GPM is much coarser and porous, which explains the reason of increased water absorption and reduced strengths at elevated temperatures curing. Samples prepared with 6, 8, 10, 12, 14 and 16 M showed lower water absorption compared to control sample (OPC mortar). The increased molarity of sodium hydroxide enhance the geopolymerization mechanism and microstructure composition and presented low water absorption as depicted clearly in Figure 5, 6a, 6b and 6c.

Figure 4. Effect of NaOH molarity on WA of GPMs.

Figure 5. Effect of NaOH concentration on geopolymer water absorption and strength.

Figure 6. Effect of NaOH molarity on microstructure of GPMs (a) 2M (b) 8M (c) 14 M.
3.2 Effect of Solution to Binder Ratio

Five mixtures were prepared with different solution for binder content (S:B) as shown in Figure 7. NaOH molarity, NS:NH and B:A were fixed at 6 M, 3 and 1.5, respectively for all mixtures. The results indicated that the water absorption, increase alkaline solution content to binder lead to increase the water absorption of mortar, increase solution content contributed to increase water content and effect in the negative side of microstructure building. Most samples cured at ambient temperature showed lower water absorption than samples cured at elevated temperatures for, effect of high temperature on calcium silicate hydration CSH and final effect on microstructure of GPMs explain clearly the reason of increase WA with the increase amount of solution in the mixture.

Figure 7. Effect of (S:B) on geopolymer water absorption.

3.3 Effect of Na2SiO3 Content

Various ratios of NS:NH used to prepare seven geopolymer mixtures. Figure 8 presented the Results of influence sodium silicate content on GMs absorption. The results revealed that water absorption reduced with increase in sodium silicate content.microstructure and strength of GPMs improved with increase sodium silicate (NS) content, Whereas the use of NS solution resulted in mainly the amorphous products with only a small amount of crystalline CSH in GPMs and show the lower water absorption for samples prepared with high content sodium silicate 3, 3.5 and 4 was presented 10.1, 9.8 and 9.2 respectively and lower than control sample was present 11.75.

Figure 8. Effect of (NS:NH) on water Absorption of GPMs.

3.4 Effect of (B:A) Ratio

The impact of binder content to fine aggregate (B: A) ratio on water absorption of GPMs presented in Figure 9. Five mixtures were prepared with different binder content. NaOH concentration, S:B and NS:NH were fixed with 6 M, 0.35 and 3.0 respectively. The results indicated that an increase in binder ratio contributed to increase water absorption of samples. GPMs prepared with low binder content (0.30) display the lower WA compared to other ratios 1, 1.5, 2 and 2.5.

Figure 9. Effect of B:A on water Absorption of GPMs.

3.5 Effect of Solution Types

To evaluate the effect of solution type in water absorption two types of solution were used in this laboratory, experimental, Normal solution (NHNS) was prepared by mixed sodium hydroxide with sodium silicate; the
second solution prepared with sodium silicate only as alkali activator. Figure 10 shows the results of WA, specimens prepared with NS alkaline solution presented lower absorption results compared to results of specimens prepared with alkaline solution containing sodium hydroxide and sodium silicate. The using of sodium silicate (NS) with 100% amount as alkali solution lead to increase the dissolution of silicate and enhance structure and strength of GPMs. Whereas the use of NS solution resulted in mainly the amorphous products with only a small amount of crystalline CSH in GPMs compared to normal solution which produced the highest amount of crystalline and low amount of amorphous and clearly depicted the reason of reduced water absorption of GPMs prepared with the NS solution as shown in Figure 11.

Figure 10. Effect of solution types on WA of GPMs.

Figure 11. Microstructure of GPMs prepared with various solution types (a) NHNS (b) NS.

4. Conclusions

The results of experimental indicated to:

• Geopolymer water absorption enhanced with increase sodium hydroxide molarity.
• Increase sodium silicate content lead to reduce the geopolymer water absorption.
• The results show that the water absorption of samples cured at ambient temperature showed lower absorption compared to samples cured at elevated temperatures.
• High ratio of alkaline solution to binder effect on negative side and increase the water absorption
• Reduce the fine aggregate content lead to increase the geopolymer water absorption

5. References

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