Effect of Curing Temperature on the Mechanical Strength of Alkali Activated Laterite Geopolymeric Samples

DOI:10.36909/jer.11627

Ahmad Fahmi¹*, Alireza Babaeian amini¹, Yaser Marabi², Sohrab Rafati Zavaragh³, Ali Majnouni-Toutakhane⁴,

¹ Department of Civil Engineering, Faculty of Engineering, University of Bonab, Iran
² Graduate Student of Civil Engineering, University of Bonab, Iran
³ Researcher, University of Bonab, Iran
⁴ Department of Architecture, University of Bonab, Bonab, Iran

* E-mail: Fahmi.ahmad@ubonab.ac.ir; Corresponding Author.

ABSTRACT

A huge amount of carbon dioxide is released in the Portland cement production process. A large quantity of greenhouse gases is produced because of the significant amount of energy consumption via making bricks through firing. Using the pozzolanic sources containing the aluminosilicate and alkaline reagents, a new type of green materials called geopolymeric materials are produced with quite lower environmental hazards. The use of laterite as an iron-rich aluminosilicate material has a high potential for building materials. In this study, the effect of the curing temperature and characteristics of the alkaline reagent including the concentration of sodium hydroxide solution and the water-glass to sodium hydroxide mass mixing ratio on the mechanical strength of the laterite-based, oven-cured geopolymer samples was investigated. The results showed that the curing temperature had a significant effect on the compressive strength of the laterite-based
geopolymer samples so that with a 15°C change at the curing temperature, the compressive strength of the samples could be multiplied and a sharp increase in the mechanical strength could occur. Also, according to the results of this study, the 6 M sodium hydroxide is recommended for the construction of the laterite-based geopolymer materials with low cost and relatively high strength, and for the construction of higher-strength building materials, the 14 M sodium hydroxide is recommended.

**Keywords:** Geopolymer; Laterite; Alkali activated; Curing Temperature; Mechanical strength

**INTRODUCTION**

To produce one ton of Portland cement, a bit less than one ton of carbon dioxide is released [1, 2]. About 5-7% of the carbon dioxide produced in the world is related to the Portland cement industry [3, 4]. After concrete, brick is the second most widely used construction material [5]. Then scads greenhouse gases were produced because of the significant amount of energy consumption via making bricks through firing [6-8]. Using the chemical reaction of the alkaline reagents such as sodium hydroxide and sodium silicate solutions with solid aluminosilicate compounds, it is possible to produce the environmentally-friendly cement (or binder) called geopolymer. If geopolymeric cement and materials replaced with (some part of) the conventional building materials such as Portland cement concrete and fired brick, Greenhouse gas emissions have been significantly reduced [2, 9, 10].

The high mechanical strength; low shrinkage; high durability against acid, fire, and various environmental conditions; and low thermal exchange are among the advantages of geopolymer materials. [9, 11]. Geopolymers are strong and durable cementitious materials that are capable of hardening and polycondensation at temperatures lower than 100 °C [12]. The aluminosilicate base used to produce geopolymer can include natural resources such as
volcanic ash, kaolinite, and laterite, artificial aluminosilicates, as well as various industrial waste such as brick and concrete waste powders, fly ash, furnace slag, rice husk ash, and palm oil fuel ash [13-19].

Natural laterite is the composite material of iron, aluminum, and silicon [20]. Among others, the use of laterite as an iron-rich aluminosilicate material has a high potential for building materials.

The firing of non-geopolymer laterite bricks (without the use of alkaline reagent) at various temperatures was investigated by Mbumbia, de Wilmars [21]. The mixture of laterite with Ca(OH)₂ in the autoclave as well as the laterite mixture with Portland cement can also be used to produce the building blocks [22]. The presence of alumina and silica make it possible to use sustainable and low-cost silica sources to produce complex composites with a high fraction of ferrisilicates, through the polymerization process. Curing of samples in temperature 50–100 °C make it possible to enhance the transformation of hydrates and oxyhydrates to ferrisilicates in addition to the formation of ferrosialates and polysilanes [23].

The laterite geopolymer products were classified as green, environmentally friendly, and sustainable products [24] Laterites are abundant throughout the globe and could be assumed as a consistent precursor for geopolymers [3]. In the field of geopolymers, various studies have been conducted on the production of laterite-based geopolymer samples, and their set time and mechanical properties have been investigated [21, 25, 26]. Also, the phosphoric acid and alkaline reagents were separately studied for the production of geopolymer samples with the laterite aluminosilicate source (laterite soil) by Gualtieri, Romagnoli [25]. The improvement of laterite soil using the alkaline stabilizing solution has also been investigated [27, 28].
Temperature and time are two important parameters influencing the strength of geopolymer materials [9]. It is possible to obtain building materials such as bricks hardened at room temperature or ceramic bricks with maximum heating from 85°C to 450°C [22]. The effect of these parameters on the geopolymer samples based on the fly ash, metakaolin, and other aluminosilicate precursors has been investigated [29-31]. Oven-cured geopolymers gain their maximum strength at early ages (unlike the ambient-cured geopolymers) [32]. The present study explores the mechanical strength of the different laterite-based geopolymer samples cured in the oven. The effect of curing temperature in the range of 25 to 100 °C with different molarities of sodium hydroxide solution on the 3- and 7-day compressive strengths of the samples were considered. The research challenge in this study is the effect of increasing the curing temperature on the change of mechanical strength of the laterite-based geopolymer samples.

MATERIALS AND METHODS

MATERIALS

The aluminosilicate base material used in this study for the production of the geopolymer sample is the laterite rock powder. Laterite rock was provided from the Ghar-Yaghdi laterite-bauxite deposit of Miandoab, Iran. Based on the results of the XRF analysis, the oxide compounds existing in the laterite rock powder are presented according to Table 1. This iron-rich material as an aluminosilicate source for making geopolymer samples has relatively low silica in the comparison with other aluminosilicate custom materials. To prepare the alkaline solution for preparation of laterite-based geopolymer samples, sodium hydroxide (99% caustic soda flakes), water (drinking water from Bonab, Iran), and commercial water-glass of 2.5 ratios (specifications according to Table 2) were used. Some 50×50×50 mm (0.125 liters) cubic molds, 160×40×40 mm (0.256 Liter) prismatic molds, and standard briquette (dumbbell-shaped 0.065 liters) molds were used to prepare
geopolymer samples to evaluate the compressive, flexural and tensile strength of the samples respectively (all used molds were triplex). Test methods according to ASTM C109 / C109M – 21 and ASTM C348 – 21 standards for cement mortars were used for evaluation of the compressive and flexural strength of 50 mm cubic and prismatic geopolymeric samples. The equipment used in this study includes a laboratory chemical hood for preparing the alkaline solutions, a pan mixer, and a vibrating device (table) for preparing the samples, and an oven for the curing of them. Also, a uniaxial compression testing device for measurement of the compressive strength and a dual-purpose testing machine for measurement of the flexural and tensile strength of the cured samples were used.

**Tab 1.** Chemical composition (oxide compounds) of laterite rock powder based on XRF analysis

<table>
<thead>
<tr>
<th></th>
<th>K₂O</th>
<th>CoO</th>
<th>MnO</th>
<th>SO₃</th>
<th>P₂O₅</th>
<th>CaO</th>
<th>V₂O₃</th>
<th>Cr₂O₃</th>
<th>MgO</th>
<th>TiO₂</th>
<th>Fe₂O₃</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.04</td>
<td>0.07</td>
<td>0.08</td>
<td>0.09</td>
<td>0.47</td>
<td>0.15</td>
<td>0.13</td>
<td>1.64</td>
<td>2.89</td>
<td>32.8</td>
<td>16.7</td>
<td>32.5</td>
<td>12.36</td>
<td></td>
</tr>
</tbody>
</table>

**Tab 2.** Specifications of used water-glass

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Ratio</td>
<td>2.5</td>
</tr>
<tr>
<td>Na₂O %</td>
<td>13-13.89</td>
</tr>
<tr>
<td>SiO₂ %</td>
<td>32.59-34.64</td>
</tr>
</tbody>
</table>
TEST DESIGN OF LATERITE BASED GEOPOLYMER SAMPLES

The laterite rock was crushed in the crusher machine and the laterite rock powder passing sieve No. 30 was selected as a solid aluminosilicate base material. The NaOH flakes were dissolved in water to make the solution with the required Molarity before the preparation of the samples and casting. The 6, 10, 14, and 18 molar sodium hydroxide solution was used to produce the laterite-based geopolymer samples. To prepare the alkaline reagent solution, the water-glass to sodium hydroxide mass mixing ratio was selected as 1 or 2. For example, the G10-2 geopolymer sample indicates the use of 10 M sodium hydroxide solution and the water-glass to sodium hydroxide solution mass mixing ratio of 2.

The mix design for the geopolymer cubic samples is shown in Table 3. The mass values presented in the table are those of materials needed to produce a cubic 5×5×5 sample. For all samples, the weight ratio of alkaline solution to laterite powder was selected at 0.25. Eight alkaline solutions with different molarities of sodium hydroxide and weight ratios of water-glass to sodium hydroxide solution were used. For preparing the cubic laterite-based geopolymer samples with 8 different alkaline solutions in 6 curing temperature ranges (25, 40, 55, 70, 85, and 100 °C) and two different ages (3 days and 7 days), 8×6×2=96 test designs were planned. For each test design, three replications (for measuring the average strength of three samples for each composition design) and thus totally 288 cubic samples were prepared.
Tab3. Mix design of geopolymer cubic samples cured at 6 different temperatures (25, 40, 55, 70, 85 and 100 °C)

<table>
<thead>
<tr>
<th>No.</th>
<th>Sample</th>
<th>NaOH Solution Molarity</th>
<th>Mass mixing ratio of NaOH Solution to Water-Glass</th>
<th>Mass of materials for each cubic sample (g)</th>
<th>Weight Ratio of alkaline solution to Laterite Powder</th>
<th>Number of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Laterite Powder</td>
<td>NaOH Solution</td>
</tr>
<tr>
<td>1</td>
<td>G 6-1</td>
<td>6</td>
<td>1</td>
<td>250</td>
<td>31</td>
<td>31</td>
</tr>
<tr>
<td>2</td>
<td>G 6-2</td>
<td>6</td>
<td>2</td>
<td>250</td>
<td>21</td>
<td>41</td>
</tr>
<tr>
<td>3</td>
<td>G 10-1</td>
<td>10</td>
<td>1</td>
<td>250</td>
<td>31</td>
<td>31</td>
</tr>
<tr>
<td>4</td>
<td>G 10-2</td>
<td>10</td>
<td>2</td>
<td>250</td>
<td>21</td>
<td>41</td>
</tr>
<tr>
<td>5</td>
<td>G 14-1</td>
<td>14</td>
<td>1</td>
<td>250</td>
<td>31</td>
<td>31</td>
</tr>
<tr>
<td>6</td>
<td>G 14-2</td>
<td>14</td>
<td>2</td>
<td>250</td>
<td>21</td>
<td>41</td>
</tr>
<tr>
<td>7</td>
<td>G 18-1</td>
<td>18</td>
<td>1</td>
<td>250</td>
<td>31</td>
<td>31</td>
</tr>
<tr>
<td>8</td>
<td>G 18-2</td>
<td>18</td>
<td>2</td>
<td>250</td>
<td>21</td>
<td>41</td>
</tr>
</tbody>
</table>

For preparing the prismatic laterite-based geopolymer samples with 8 different alkaline solutions in curing temperature of 70 °C and two different ages (3 days and 7 days) 8×1×2=16 test designs were planned. For each test design, three replications and thus a total of 48 prismatic samples were prepared. Similarly, 16 test designs for briquette geopolymer samples (totally 48 samples) were prepared too. The mass of consumed materials for each prismatic
sample is 2.05-fold and for each briquette, the sample is 0.52-fold of the cubic samples. (According to table 3).

PREPARATION AND CURING OF LATERITE BASED GEOPOLYMER SAMPLES

For preparing the samples of each test design according to the mentioned composition designs, according to Table 3; the required amount of laterite powder was poured into the plastic container and the required amount of specified alkaline solution was gently added and mixed (care should be taken that the mixing of the materials should never be done in the metal containers). The materials were then poured into the pan mixer, and after 5 minutes of complete mixing, the materials were poured into the molds and vibrated on a vibrating device. All samples after the initial setting and removal from the mold, were stored in the oven at a specified temperature for 3 days.

MEASUREMENT OF THE MECHANICAL STRENGTH

After leaving the oven and cooling, the 3-day mechanical strength of the specified geopolymer samples was measured and the average mechanical strength of three samples of each test design was calculated. Other oven-cured geopolymer samples (for evaluating the 7-day mechanical strength) were kept at ambient temperature for 4 days, and the 7-day mechanical strength of the samples was measured and the average mechanical strength of three samples of each test design was calculated. The compressive strength of the cubic samples was measured by the uniaxial compression testing device. The flexural strength of the prismatic samples was measured using the 3-point-bending method. The flexural and tensile strength of the prismatic and briquette samples were measured using a special multipurpose device.
X-RAY FLUORESCENCE (XRF)

The dried laterite powder pressed to tablets with boric acid as additive to determine the elemental composition with S4 EPLORER X-ray fluorescence spectrometer.

X-RAY DIFFRACTION (XRD) ANALYSIS

XRD analysis in the angular region (2θ) of 15-80° was recorded by X’pert Pro diffractometer (Panalytical, Netherland) with Cu anode, at room temperature, step size of 0.026°, the rate of 1°/min, the wavelength of 0.154056 nm, 40 kV and 40 mA. XRD analysis were performed for laterite powder and powdered geopolymer samples including G6-1, G10-1, G14-1 and G18-1 cured at 85 °C.

FE-SCANNING ELECTRON MICROSCOPY (FESEM) AND ENERGY-DISPERSIVE X-RAY (EDX)

FE-scanning electron microscopy analysis of Laterite rock sample, Laterite rock sample and geopolymer samples (G6-1, G10-1 and G14-1, G18-1 cured at 85 °C were scanned at room temperature and 20 kV with FESEM microscope. The EDXs were recorded for samples and elements of Si, Al, Fe, Na and O were considered.

RESULTS AND DISCUSSION

COMPRESSIVE STRENGTH

The 3- and 7-day compressive strengths of the geopolymer samples are shown in Fig. 1. The compressive strength is the same as average compressive strength of 3 samples of each test setup. The 3- and 7-day compressive strengths of all samples cured at 25 and 40 °C (near the ambient temperature) are less than 10 MPa. Also, the 3- and 7-day compressive strengths of the cured samples at 70-100 °C are above 10 MPa (10 to 57 MPa). Particularly, the compressive strength above 20 MPa is often achieved at 85 and 100 °C. Therefore, in
summary, it can be concluded that to bring the compressive strength of the geopolymer samples based on the laterite (used in this research) to the values above 10 and 20 MPa, it is necessary to cure the samples at a temperature range of 70-100 °C in the oven, and at a lower temperature (near the ambient temperature), a less compressive strength is achieved. Therefore, to prepare the high-strength geopolymer building materials based on the laterite, it is necessary to use the oven, and this is also confirmed by the results of various studies performed in the field of geopolymer[33].

Also, according to Fig. 1, in the geopolymer samples produced by 6 M sodium hydroxide (G6-1, G6-2) with the minimum use of alkaline reagents at the curing temperature of 85 °C, the 3-day compressive strength above 20 MPa was obtained. Therefore, the use of the mentioned test setups (G6-1, G6-2) for the production of building materials such as engineering bricks and precast components is important from three viewpoints: low cost, relatively good mechanical strength, and early strength (compressive strength of samples reaching to desirable values in a short 3-day period).
The results of studies in the field of geopolymer produced by fly ash show that by increasing the sodium hydroxide molarity, the compressive strength of the samples is increased and the highest compressive strength of the samples was obtained for the use of 12 M sodium hydroxide, and the samples prepared with higher molarity had less compressive strength [30, 34-36]. However, according to the results of this study, for the laterite-based geopolymer samples, the maximum compressive strength of the samples was obtained using the 14 M sodium hydroxide.

As shown in Fig. 1, for the geopolymer samples produced using the alkaline reagent solution containing 14 M sodium hydroxide and the water-glass to sodium hydroxide solution mass mixing ratio equal to 1 (samples G14-1) at 85 and 100 °C, the 3-day compressive strength values of the geopolymer samples are higher than 50 MPa, and therefore, the high-strength precast components can be produced by the geopolymer samples with the specified characteristics (samples G14-1). Various studies have emphasized the necessity to use the geopolymer technology for the construction of precast components with high mechanical
strength [37, 38]. The amount of alkaline sodium hydroxide used in the construction of samples G14-1 is more than 2 times the samples G6-1, while the compressive strength of the samples is increased more than 2 times. It should be noted, however, that with higher use of alkaline reagent (samples G18-1), the compressive strength will be decreased. Therefore, for the construction of geopolymer construction materials based on the laterite (laterite used in this research) at low cost and relatively high strength, the use of 6 M sodium hydroxide is recommended, and for the construction of higher-strength building materials, it is recommended to use 14 M sodium hydroxide.

According to Fig. 1, by increasing the curing temperature from 70 to 85 °C, the 3-day compressive strength of samples G14-1 considerably increased from 12.1 to 50.8 MPa (320% increase) and the 7-day strength of the same samples was significantly increased from 13.7 to 52.1 MPa (280% increase). These sharp increases in the compressive strength values were obtained only with 15°C increase in the curing temperature.

In the studies on the geopolymer concrete produced by fly ash, the compressive strength of the samples was also increased by increasing the time and age of the samples and also increasing the curing temperature from 10 to 80 °C (as well as from 60 to 90 °C) [29, 39]. In the metakaolin-based geopolymer samples, by increasing the curing temperature from 15 to 60 °C, the compressive strength of the samples was increased, and with a further increase in the curing temperature, the compressive strength was reduced [24]. However, according to the results of the present study, for the laterite-based geopolymer samples, as shown in Fig. 1, for the prepared samples produced by sodium hydroxide concentration of 8 and 10 M, with the continuous increase in the curing temperature from 25 to 85 °C, the compressive strength of the geopolymer samples was increased, but by increasing the temperature from 85 to 100 °C, the compressive strength showed a decreasing trend. However, for the samples with the 14 and 18 M concentration of sodium hydroxide, by increasing the temperature from 25 to 100
°C, the compressive strength of the geopolymer samples is constantly increased and does not show a decreasing trend.

According to the results of this study, in the laterite-based geopolymer samples, according to Fig. 2, by increasing the curing temperature from 85 to 100 °C in some samples, the increased compressive strength is achieved, and in some other samples, the compressive strength is reduced. In other words, the mechanical behavior of laterite-based geopolymer samples varies with the concentration of the alkaline solution (sodium hydroxide molarity) and the curing temperature.

**Fig 2.** Increase in 3-day compressive strength of different geopolymer samples due to increase in curing temperature from 85 to 100 °C

Also, according to Fig. 3, by the 60°C increase in the curing temperature of the samples from 40 to 100 °C, the compressive strength of the geopolymer samples with 6 and 10 M sodium hydroxide was increased from 196 to 270%, and for the samples with 14 and 18 M sodium hydroxide, it was increased from 600 to 1570%. Specifically, this increase in the samples with the water-glass to sodium hydroxide solution ratio of 1 considerably varies between 1445 and 1570%.
Fig 3. Increase in 3-day compressive strength of different geopolymer samples due to increase in curing temperature from 40 to 100 °C

Also, comparing the 3 and 7-day compressive strength values of cured geopolymer samples in the temperature range 25-100 °C (Fig. 4) shows that the difference percentage of 3- and 7-day compressive strengths in the samples produced by the 6-14 M sodium hydroxide is insignificant, and there is only a significant difference in the samples produced by the 18 M sodium hydroxide. However, for the 25°C curing temperature in all the samples, the difference percentage of 3- and 7-day compressive strengths is not low.
The 3- and 7-day flexural and tensile strengths of the geopolymer samples (cured at 70°C) are shown in Fig. 5. Similarly to the results of the compressive strength, the highest amounts of the flexural and tensile strength are belonged to G 14-1 samples. The flexural and tensile strength of the samples G 6-1 and G 14-1 samples have not much difference, so from the aspect of economics, for making of the geopolymer samples, using the lower molarity (6M) of sodium hydroxide is preferable. For very high strength class F fly ash geopolymer mortar, the flexural strength of samples, at 72 h heat curing in 75°C, the flexural strength of samples was reported between 5.2-12.9 MPa [40]. In this research, flexural strength increases with Na(%) increment and for more increment of it the flexural strength decreases[40], so these results for F fly ash geopolymer mortar is accordance to our results for laterite based geopolymer samples.
Fig 5. 3- and 7-day flexural and tensile strengths of geopolymer samples, cured at 70 °C

FESEM, EDX AND XRD ANALYSIS

FESEM & EDAX analysis of laterite rock sample and laterite-based geopolymeric samples G10-1 and G14-1 cured at 85 °C is according to Fig. 6. For laterite rock sample no picks related to Na element was recognized. But, in geopolymeric samples Na element was recognized. Weight percent of Na in G10-1 sample and G14-1 sample were reported 9 and 19% respectively.

SEM examination of Laterite rock (A), Laterite grinded powder (B), geopolymer sample G6-1 (C), geopolymer sample G10-1 (D), geopolymer sample G14-1 (E), geopolymer sample G18-1 (F) at 10000× magnification are according to Fig. 7. Comparison of the FESEM
examination of Laterite rock (A) with Laterite grinded powder (B) shows that after grinding the structure of material was partly changed. FESEM examination of geopolymer samples (C-D) shows that the structure of geopolymer samples in the comparison the laterite base materials (A & B) completely were changed. Also for increment of sodium hydroxide molarity (C to D), the porosity of geopolymer samples were decreased.
Fig6. A,C (FESEM and EDAX of laterite rock sample), B,D (FESEM & EDAX of G10-1 sample) and E,F (FESEM and EDAX of G14-1 sample)
According to XRD analysis, the Laterite rock powder was consisted of Kaolinite (Aluminum Hydroxide Silicate), Hematie (Iron Oxide) and Cristobalite (Silicon Oxide) minerals.
According to Fig.8, in the geopolymeric samples the picks related to these minerals were changed more with increment of the concentration (molarity) of sodium hydroxide, so that in G18-1 the change of picks are significant.

**Fig 8.** XRD analysis of Laterite powder (LP) and geopolymeric samples G6-1, G10-1, G14-1 and G18-1

**CONCLUSION**

The results of the present study show that the curing temperature has a significant effect on the compressive strength of the laterite-based, oven-dried geopolymer samples, and the compressive strength of the samples can be sharply increased by the 15 °C change in the curing temperature, and a sharp increase can occur in the mechanical strength. The compressive strength of the geopolymer samples can be increased over 20 MPa and even over 50 MPa over 3 days, and thus, it is possible to be used in the building materials such as engineering
bricks and high-strength precast components. Also, the molar concentration of sodium hydroxide solution and the water-glass to sodium hydroxide solution mass mixing ratio have a significant effect on the mechanical strength of the laterite-based geopolymer samples. Similarly to the results of the compressive strength, the highest amounts of the flexural and tensile strength are belonged to G 14-1 samples. The flexural and tensile strength of the samples G 6-1 and G 14-1 samples have not much difference, so from the aspect of economics, for making of the geopolymer samples, using the lower molarity (6M) of sodium hydroxide is preferable. Laterite rock powder with high content of iron oxide in the presence of alumina and silica could use to produce sustainable, low cost and high strength geopolymeric materials.

ACKNOWLEDGMENTS

Hereby, we would like to appreciate the supports of the Scientific Student Association Office of the University of Bonab from the student members of the Scientific Student Association of the University to conduct this research.

CONFLICT OF INTEREST:

The authors declare that they have no conflict of interest.

REFERENCES


19. Adam, A.A. 2009. Strength and Durability Properties of Alkali Activated Slag and Fly Ash-Based Geopolymer Concrete, RMIT University Melbourne, Australia.


