The influence of various basalt mineral and sodium hydroxide concentration on the physical properties and characterization of geo-polymer products

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Abstract. Environment friendly product has a major concern nowadays. The use of cement as a binder to make concrete cannot be negligible since cement production has contributed to the increasing of CO₂ emissions. To produce 1 ton of cement, approximately 1 ton of CO₂ is also released from the process. Rock geo-polymer cement as one categories of cement is known to be more environmentally friendly than Portland cement. Basalt rock has an opportunity for a material substitute in geo-polymer cement production. Variability influence of basalt and sodium hydroxide concentration on the physical properties and characterization of Geopolymer products are observed in this research. The raw materials used are basalt, sodium hydroxide, sodium silicate, and water. Geo-polymer is made by adding basalt as much as 2.5 %, 5 % and 7.5 % with basalt grain size of 80, 150 and 270 mesh. This research shows that the greater variation of sodium hydroxide concentration, the smaller compressive strength produced; however the absorption considered becomes greater. Geo-polymer with addition sodium hydroxide 2.5 % with basalt grain size of 270 mesh has a highest compressive strength and the lowest absorption rate. Characterization of the sample using XRF and XRD have indicated that the sample has the highest SiO₂ compound content of 48.731 % with 2.5 % sodium hydroxide in the 270 mesh, while the phases formed were Albite Na(Si₃Al)O₈, Cristobalite (SiO₂), Forseterit (Mg₂SiO₄), Anorthite Ca(Al₂Si₂O₈) and Magnetide (Fe₃O₄).

Keywords: Basalt; geo-polymer; sodium hydroxide; compressive strength; hydride phase

1. Introduction
The development of infrastructure has grown rapidly along with the times. It is closely related to the use of construction materials that contain unfriendly environmental materials. The increasing demand for concrete in construction causes the high need for cement as one of the main materials of concrete constituent. The increased use of Portland cement annually resulted in an increase in the number of Portland cement Industries in Indonesia. The development of the Portland cement industry has another side impact to the environment [1].

The continuous cement production affects the environment. The cement Industry has become a major contributor to CO₂ emissions since to produce 1 ton of cement, approximately 1 ton of CO₂ is
also released [2]. To reduce the emission or waste produced, more friendly material is needed as a part of substitute cement material. Basalt rock has the opportunity as a friendly material.

Geo-polymer cement is one of the cement types. The main raw materials required for the manufacture of geo-polymer cement are ingredients that contain a lot of silica elements (SiO₂) and alumina (Al₂O₃), one of the raw materials containing many silica elements and alumina is a basalt stone which is have chemical composition SiO₂ = 40-55 % and Al₂O₃ = 12-17 % [3].

The geopolymer reaction is carried out by an alkaline activator in the form of sodium hydroxide and sodium silicate. Sodium hydroxide is related to the Si and Al elements contained in basal minerals. While sodium silicate accelerates polymerization during the reaction [4]. In this study, we used an alkaline sodium hydroxide activator with a variation of 2.5 %, 5 %, and 7.5 % and sodium silicate was 13 %. This refers to previous research that sodium hydroxide with concentrations below 2.5 % will produce a stronger press.

Based on data from Energy and Mineral Resources Information in 2011, basalt non-metallic mineral resources in Indonesia are around 5,571,251.56 tons. According to sources from mining and energy officers in Lampung, a number of basalt rock reserves are scattered in the area is around 318,480,000 tons [5]. With an abundant amount of basal mineral reserves, it is expected to create environmentally friendly innovations to optimize the use of basal minerals to have high economic value.

2. Methodology
Basalt powder preparation is done by crushing the basalt stone using a jaw crusher and grinding it in a ball mill for ± 3 hours. Basalt powder is then sieved using 80, 150 and 270 mesh filters. XRF Epsilon 3xle Bench Top is used for characterization in the formation of oxide content, XRD PAN analytical X'pert™ is used for characterization in the formation of minerals which are gradual in nature.

The raw materials for geo-polymer samples are basalt minerals from East Lampung, Sodium silicate, sodium hydroxide and water. Geo-polymer samples consist of 3 types of raw materials; the first is a sample with 69.5 % basalt weight composition, the second is 67 % basalt weight and the third is 64.5 % basalt weight. Each sample type has a grain size 80 mesh, 150 and 270 mesh with variations of sodium hydroxide around 2.5 %, 5 % and 7.5 %, respectively. While the sodium silicate and water used are set at 13 % and 15 %.

Preparation of the geo-polymer test specimen begins by mixing sodium hydroxide with water in each composition. The sodium silicate is added into sodium hydroxide solution and mix into a mixer that has basalt powder rather than stirring it for about ± 20 minutes to be homogeneous. The mixture is then put into a cube mold that has a size (5×5×5) cm³, is coded and stored for 24 hours. The sample is then dried at 100 °C for 8 hours and allowed to cool to room temperature. The compressive strength, absorption and XRF test, XRD characterization conducted after the sample reaches room temperature.

3. Result and Discussions
3.1. Preliminary testing on basalt
XRF characterization of basalt sample is presented in Table 1. Table 1 indicates that basalt minerals have the highest SiO₂ compound content at 48.463 %, followed by Al₂O₃ 20.143 %, Fe₂O₃ 11.510 %, CaO 9.608 % and MgO 4.269 %. These results indicate that the amount of silica (SiO₂) and elemental content of alumina (Al₂O₃) is fulfilled the chemical requirements as main raw material manufacture for geo-polymer products; whereas to manufacture geo-polymer products, the silica content should be at least 40-55% while for the element of alumina around 12-17 %.

The determination of the basalt powder sample using XRD equipment is shown in Figure 1. Basalt mineral dominated by the culmination phases among others, Anorthite at the highest peak 2θ = 27.7879°, Augite phase at the highest peak 2θ = 29.7377°, Forsteritice phase at the highest peak 2θ = 29.4753° and Quartz phase at highest peak 2θ = 26.6691°, respectively.
Table 1. XRF mineral basalt characterization results.

| No | Oxide compounds | Percentage (%) |
|----|----------------|----------------|
| 1  | SiO₂           | 48.463         |
| 2  | Al₂O₃          | 20.143         |
| 3  | Fe₂O₃          | 11.510         |
| 4  | CaO            | 9.608          |
| 5  | MgO            | 4.269          |
| 6  | Na₂O           | 3.559          |
| 7  | K₂O            | 1.266          |
| 8  | P₂O₅           | 0.605          |
| 9  | MnO            | 0.195          |

Figure 1. Diffractogram XRD pattern of basalt samples

3.2. Compressive strength sample Geo-polymer
A 5 × 5 × 5 cm³ geo-polymer samples were prepared for compressive strength tests where pressure was applied to the sample until it breaks. The pressure was then recorded, and the results of the compressive test are shown in Figure 2.

Figure 2. Correlation of sodium hydroxide concentration with compressive strength geo-polymer
Compressive strength test for sample with 2.5 % sodium hydroxide concentration and basalt mineral grain size of 80 mesh, 150 and 270 mesh have showed a value of 6.992 MPa, 7.204 MPa and 7.576 MPa, respectively. The higher rate was between 5 % and 7.5 % of sodium hydroxide concentration. Whereas, compressive strength for 5 % sodium hydroxide concentration and basalt mineral grain size of 80 mesh, 150 and 270 mesh showed a value lower than 2.5% sodium hydroxide concentration which were 3.116 MPa, 3.292 MPa and 3.484 MPa, respectively. A 7.5 % sodium hydroxide concentrations and basalt mineral grain size of 80, 150 and 270 mesh showed a lower value than 5 % sodium hydroxide concentration. The smaller size of basalt mineral with 2.5 % sodium hydroxide concentration resulted in higher the geo-polymer compactness. Whereas the greater concentration of sodium hydroxide, the lower value of compressive strength. This might be due to the higher sodium hydroxide concentration used for alkali activator that cause difficulty in stirring and casting.

3.3. Absorption of Geo-polymer Samples
Absorption test is done on size of 5 × 5 × 5 cm³ geo-polymer object test. Figure 3 shows a correlation between sodium hydroxide concentration and geo-polymer absorption. The absorption test of geo-polymer samples with 2.5 % sodium hydroxide concentration and basalt mineral grain size of 80 mesh, 150 mesh and 270 mesh showed a value of 8.89 %, 9.48 % and 11.03 %, respectively. The absorption test sample with 5 % sodium hydroxide concentration and basalt mineral grain size of 80 mesh, 150 and 270 mesh showed a value of 9.29 %, 11.24 % and 11.56 %. While the sample absorption test with 7.5 % sodium hydroxide and basalt mineral grain size of 80 mesh, 150 and 270 mesh have a value of 10.88 %, 11.46 % and 11.71 %, respectively. The chart shows the absorption on the mesh 270 slightly better than the mesh 150 and 80. The greater sodium hydroxide concentration, the higher water absorption rate. In accordance with the research of M. Amin and Suharto [3] that stated the value of absorption is associated with an addition of sodium hydroxide from geo-polymer. This might be due to the downward of sodium hydroxide absorption rate in geo-polymer. The large absorption rate of geo-polymer was a sign of lower durability due to the geo-polymer tend to absorbs water easily and effect the degradation strength of geo-polymer. Water can enter the geo-polymer not only through the pores or cavity formed on geo-polymer, but also through a diffusion and absorption [6].

Figure 3. Correlation of sodium hydroxide concentration with geo-polymer absorption

3.4. Characterization of XRF of Geo-polymer Samples
XRF geo-polymer characteristics are shown in Table 2. XRF data sample characterization shows the highest SiO2 compound of 48.731 % at 2.5 % sodium hydroxide concentration and basalt sample grain size 270 mesh. Whereas the samples with 5 % and 7.5 % sodium hydroxide and basalt mineral grain
size 150 and 270 have a lower SiO₂ content than 2.5 % sodium hydroxide concentration. This signifies that the SiO₂ contained in the basalt minerals react with existing alkali activators forming a polymer bond in the geo-polymer product. The highest compound content in the sample is SiO₂ followed by Al₂O₃, Fe₂O₃, CaO and Na₂O. Other compounds come with a percentage below 3 % such as MgO, TiO₂, P₂O₅, K₂O and MnO.

Table 2. XRF geo-polymer Characterization Results.

| No | Oxide compounds | Mesh 80  | Mesh 150 | Mesh 270 |
|----|----------------|----------|----------|----------|
| 1  | SiO₂           | 48.563   | 48.601   | 48.564   | 48.710   | 48.570   | 48.731   |
| 2  | Al₂O₃          | 20.153   | 20.205   | 20.160   | 20.305   | 20.210   | 20.503   |
| 3  | Fe₂O₃          | 10.966   | 10.052   | 10.974   | 10.749   | 10.983   | 10.522   |
| 4  | CaO            | 9.508    | 9.677    | 9.510    | 9.687    | 9.550    | 9.710    |
| 5  | Na₂O           | 5.061    | 5.061    | 5.064    | 5.520    | 5.070    | 5.541    |
| 6  | MgO            | 2.628    | 2.648    | 2.654    | 2.310    | 2.662    | 2.767    |
| 7  | Ten₂           | 1.006    | 1.112    | 1.014    | 1.265    | 1.183    | 1.335    |
| 8  | P₂O₅           | 0.966    | 0.976    | 0.950    | 0.900    | 0.951    | 0.900    |
| 9  | K₂O            | 0.651    | 0.663    | 0.500    | 0.051    | 0.060    | 0.030    |
| 10 | MnO            | 0.408    | 0.550    | 0.550    | 0.041    | 0.040    | 0.013    |

3.5. Characterization of XRD of Geo-polymer Samples

Geopolymer XRD characterization results are shown in Figure 4, 5, and 6. Based on XRD characterization from the samples with 2.5 %, 5 % and 7.5 % sodium hydroxide and basalt mineral grain size of 80, 150, and 270 mesh, the phase formed in geo-polymer sample are albite Na(Si₃Al)O₈, Cristobalite (SiO₂), Forseterit (Mg₂SiO₄), Anorthite Ca(Al₂Si₂O₈) and Magnetide (Fe₃O₄).

Figure 4. (a) The XRD concentrations of 2.5 % sodium hydroxide on 80 mesh, and (b) The XRD concentration of 7.5 % sodium hydroxide on 80 mesh
Figure 5. (a) The XRD concentration of 2.5 % sodium hydroxide on 150 mesh, and (b) The results of XRD concentration of 7.5 % sodium hydroxide on 150 mesh.

Figure 6. (a) The XRD concentration of 2.5 % sodium hydroxide on 270 mesh, and (b) The XRD concentration of 7.5 % sodium hydroxide on 270 mesh.

4. Conclusion
The greater variation of sodium hydroxide concentrate, the smaller compressive strength will be produced. Although the size of the basal mineral granule is smaller, due to the fact that the higher concentration of sodium hydroxide will cause a difficult time to be mixed with other compounds. Many pores will form in the sample which causes greater water absorption. The results of XRD characterization in geo-polymer samples showed that the phases formed were Albite, Cristobalite, Forsterite, Magnetid, and Anorthite.

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