Effect of Method and Duration of Curing on the Compressive Strength of the Lime-Fly Ash Geopolymer Concrete

Andi Arham Adam¹, Sri Nur Akifa², Atur P.N. Siregar¹, and Mustofa³

¹Department of Civil Engineering, Tadulako University
²Post-Graduate Program, Tadulako University
³Department of Mechanical Engineering, Tadulako University

Abstract. The use of lime to produce ambient cured Geopolymer concrete is one of a practical solution to avoid heat curing in the production of geopolymer concrete. The addition of lime in the geopolymer system will add another hardened mechanism, i.e. hydration in addition to condensation-polymerisation. This research aims to determine the influence of air and water curing on the strength of geopolymer concrete which uses class F fly ash and lime as raw materials. The geopolymer concrete was produced by varying the activator (sodium silicate and sodium hydroxide) with the variation of the activator to binder ratio of 0.45, 0.50, 0.55, and 0.60. Five per cent of the fly ash was replaced by slaked lime and the water added to the mix to maintain the ratio of water to solid (activator + binder) to 0.32. The test specimens were 100 mm dia x 200 mm height cylinder. Water-immersion curing was performed for 7, 14, and 21 days and the compressive strength test was done at 28 days. The results show that there is a difference in the curing process between the geopolymer concrete and the normal concrete. In the geopolymer concrete, there is a decrease in compressive strength if soaked for seven days and recovered with the increase in the age of immersion. Whereas for normal concrete, the compressive strength continues to increase with increasing age of immersion.

1. Introduction
Referring to the contribution of the cement industry to the total carbon dioxide (CO₂) emissions, it is necessary to find the solution to minimize the production of the gas that pollutes the environment. Partially substitution of cement parts in the manufacture of concrete, or total replacement of cement with other materials that are more environmentally friendly becomes a more promising choice.

One alternative solution is to use fly ash, which is an industrial waste containing amorphous silica from a coal-fired power plant station.

Fly ash-based geopolymer concrete is a further development of the use of waste ash as a substitute for cement. The term "geopolymer" was first used in 1970 by a French engineer and scientist, Prof. Joseph Davidovits [1]. Geopolymers themselves are formed from the chemical reactions of aluminium and silicon as basic chemicals which, with the help of an alkaline activator, will undergo an inorganic polymerization process, which results in a solid object that can be used as a binding material in concrete. Waste, such as fly ash, can be used as a primary material for making geopolymers. This geopolymer technology can be used as a tool to reduce fly ash waste so that it can be reused and has resale value. It also can reduce carbon dioxide levels in the atmosphere because it is used as a binder in concrete mixtures.
In addition to material variations, curing method also significantly influence the compressive strength of concrete. In normal concrete, water curing is an effective curing method resulting in a higher compressive strength concrete compared to air curing concrete. It is known that geopolymer concrete has different hardening characteristics compared to normal concrete. In pure geopolymer concrete, the hardening process occurs through the polymerization and condensation. During this process, the geopolymer does not need water to react [2] and heat curing is necessary [3–5]. Whereas in normal concrete, concrete hardening process that occurs is a hydration process which requires water to react the calcium and silicate content in normal concrete. With the addition of calcium content in geopolymer concrete, there will be two hardening mechanisms, namely geopolymerization, which does not require water and a hydration process that requires water. Therefore the effectiveness of the curing method in geopolymer concrete with the addition of slaked lime, both air and water curing is an interesting research topic.

2. Experimental Details

2.1. Materials

Class F fly ash taken from a local power plant and slaked lime were used as binder materials. The chemical composition of the fly ash and slaked lime can be found in Table 1.

| No. | Oxide  | Fly Ash (%) | Slaked lime (%) |
|-----|--------|-------------|-----------------|
| 1.  | SiO₂   | 55.54       | 3.95            |
| 2.  | Fe₂O₃  | 23.76       | 1.3             |
| 3.  | Al₂O₃  | 14.02       |                 |
| 4.  | CaO    | 2.02        | 89.47           |
| 5.  | K₂O    | 1.58        |                 |
| 6.  | SO₃    | 1.3         |                 |
| 7.  | TiO₂   | 0.92        | 0.07            |
| 8.  | MnO    | 0.291       |                 |
| 9.  | MgO    |             | 4.64            |
| 10. | SrO    |             | 0.512           |
| 11. | Other oxides | 0.556 | 0.054 |
|     | Total  | 100         | 100             |

The chemical activator was made by mixing the sodium silicate solution (15.4% Na₂O; 32.33% SiO₂) and sodium hydroxide (10 M).

The 3/8 and 3/4 inch coarse aggregate with the fineness modulus of 5.97 and 6.75 respectively were used in combination river sand with the fineness modulus of 2.48. The combined grading of aggregate was achieved by using the 38% fine aggregate and 62% coarse aggregate. The proportion of the 3/8-inch to 3/4-inch aggregate was 0.385 to 0.615.

2.2. Mix Proportion, Mixing and Testing

The curing methods used in this study were water curing with 7, 14 and 21 days of immersion and air curing in which the specimens were left at room temperature until testing. Besides geopolymer concrete, normal concrete made of OPC was used as a control test specimen. The mix design for geopolymer concrete made from fly ash and slaked lime developed in previous research (Adam, et al 2019), was adopted in this study. Four different mixes (Table 2) were chosen to study the effect of air curing and water curing on the compressive strength. The normal concrete used control specimens was designed according to SNI 2000 with characteristic strength of 20 MPa. The test specimen is a 10 cm x 20 cm cylindrical specimen. After removing from the mould, the water curing specimens were then...
immersed in a water curing tank for 7, 14 and 21 days, after that they were allowed to stand at room temperature until it reaches 28 days of age. A set of specimens were left in room temperature after without further treatment which refers to as air curing.

Table 2. The mix proportion details.

| Mix   | Binder (Kg) | Aggregate (Kg) | Activator (Kg) | Water (Kg) | Total (Kg) |
|-------|-------------|----------------|----------------|------------|------------|
|       | Fly Ash     | Slaked Lime    | Sand 3/8 in    | ¾ in       | Sodium Silicate | Sodium Hydroxide |   |
| A0.60 | 378         | 20             | 569            | 407        | 650        | 131            | 107  | 33         | 2295 |
| A0.50 | 390         | 21             | 569            | 407        | 650        | 113            | 92   | 50         | 2293 |
| A0.55 | 384         | 20             | 569            | 407        | 650        | 122            | 100  | 41         | 2294 |
| A0.45 | 398         | 21             | 569            | 407        | 650        | 104            | 85   | 59         | 2291 |

3. Results and Discussion

Compressive strength test results of geopolymer concrete mix A0.45; A0.50; A0.55; and A0.60 can be found in Table 3, and the relationship between duration of water curing and 28 days compressive strength are shown in Figure 1.

Table 3. Compressive strength with different curing length.

| Days of water curing | A0.45 | A0.50 | A0.55 | A0.60 | control |
|----------------------|-------|-------|-------|-------|---------|
| 0                    | 17.6  | 14.0  | 21.0  | 16.3  | 15.9    |
| 7                    | 11.9  | 12.3  | 13.0  | 14.9  | 18.9    |
| 14                   | 16.1  | 17.8  | 17.6  | 15.9  | 21.0    |
| 21                   | 19.1  | 19.7  | 21.4  | 23.8  | 25.1    |

In general, there was a decrease in strength as the specimens undergo 7 days of water curing, then back up from the 7 days to the 21 days immersion. Air curing has the highest concrete compressive strength in the A0.55 mix (21.0 MPa) and the lowest (14.0 MPa) was found in the A0.50 mix. At the end of 7 days water curing, compressive strength in all mixes had decreased though the decrease was not constant. The highest compressive strength at 7 days immersion was A0.60 mix (14.9 MPa), and the lowest was A0.45 mix (11.9 MPa). Compressive strength slowly raised after 14 days immersion and the highest compressive strength was found in the A0.50 mix (17.8 MPa), and the lowest was in the A0.60 mix (15.9 MPa). At the end of 21 days of water curing, the concrete compressive strength continues to increase. The highest compressive strength after 21 days of immersion was found at A0.60 mix, reaching 23.8 MPa and the lowest in A0.45 mix (19.1 MPa).
Figure 1. Compressive strength of concrete in different length of water curing.

The drastic decrease in compressive strength between air curing and the 7 days of water curing was caused by the disruption of the polymerization process due to the change of the activator concentration. The water immersion will affect the concentration of sodium contained in the geopolymer concrete activator due to the leaching process, namely the dissolution of sodium (Na) in the activator solution thereby reducing the alkali metal content in geopolymer concrete, which affects the polymerization process [6]

At the age of 14 days immersion, the compressive strength of the concrete rose again and also at the age of 21 days the compressive strength increased, this is caused by the hydration process formed between lime and the silica content contained in the concrete to form of C-S-H. Similar to normal concrete where C-S-H will fill the pores contained in concrete and led to the increase in compressive strength of geopolymer concrete. This hydration reaction was also found when granulated blast furnace slag added to the fly ash based geopolymer [7]

Previous research by Adam et al [8] showed that A0.55 mix in geopolymer concrete contained fly ash and lime has the optimum compressive strength; similar results were found in this study. The compressive strength of mix A0.55 is the highest compressive strength achieved by the air curing method compared to other mixes which reach 21.0 MPa. From Figure 1, it can be seen that for mix A0.55, compressive strength values for specimens with air curing and 21 days water curing have similar compressive strength values; therefore, the authors assume that the water curing method was not effective for A0.55 mix. As with other mixes, it can be seen in Figure 1 that the compressive strength of concrete between air curing and the 7 days water curing decrease, though not very significant.

Water curing treatment on geopolymer concrete made from fly ash and lime can stabilize the compressive strength in accordance with the increase of activator in the concrete. On the other hand, the water can interfere with the polymerization process, and this makes water curing method in geopolymer concrete made from fly ash and lime less effective. The researchers proposed another curing methods for geopolymer concrete made from fly ash and lime that is wrapped in an airtight container so that the two hardening processes in the concrete namely polymerization and hydration will continue until the compressive strength test is carried out. The curing method by wrapping the specimen in an airtight container can maintain the amount of water content in the concrete so that the hydration process can continue, and also the sodium content in the activator does not dissolve due to the addition of water. With this treatment, both processes can make geopolymer concrete made of fly ash and lime have high compressive strength.
From Figure 2, it can be seen that the air curing concrete had a dark brown colour and there were white patches on the entire surface of the concrete called efflorescence due to excess unreacted activators. Efflorescence is the formation of white salt deposits on or near concrete surfaces due to evaporation of water in the curing process bringing salt to the surface of concrete, in concrete using Portland cement this condition is not harmful rather than changing the colour of the concrete. The efflorescence is more pronounced in the ambient temperature cured samples because of the presence of the excess alkaline solution due to incomplete dissolution of the fly ash spheres and low dissolution rate[9]. Efflorescence can be a problem when the specimens are exposed to humid air or come into direct contact with water [10]. The water curing for 7 and 14 days changes the colour of the concrete to fade brown and returns to dark brown with the absence of efflorescence with 21 days water curing.

4. Conclusion

1. Pure geopolymer concrete does not need water to react, and even water only interferes with the polymerization process because the dissolved Na content in the activator. However, if slaked lime is added to the fly ash based geopolymer concrete, water is needed for the hydration process.
2. The time needed for the water curing of geopolymer concrete based containing fly ash and lime is above 21 days of immersion, and the compressive strength will continue to increase if the duration of immersion is more than 21 days.
3. The water curing for the geopolymer concrete containing fly ash and lime is less effective because though needed for hydration process, water disrupts the actual polymerization process especially at the early age. Whereas in normal concrete compressive strength values always increase with increasing immersion age, so that the water curing is the most effective treatment for normal concrete.

Acknowledgments

Authors wishing to acknowledge the ministry of research and higher education for providing funding for the research under the scheme of “Penelitian Dasar 2019”

References

[1] Davidovits PJ. 30 Years of Successes and Failures in Geopolymer Applications . Market Trends and Potential Breakthroughs . Geopolymer 2002 Conf 2002. doi:10.1017/CBO9781107415324.004.
[2] Rangan BV. Fly ash-based geopolymer concrete. Res Rep GC4 2008:1–44. doi:10.1007/s10853-006-0523-8.
[3] Adam AA, Horianto. The effect of temperature and duration of curing on the strength of fly ash
based geopolymer mortar. Procedia Eng., vol. 95, 2014. doi:10.1016/j.proeng.2014.12.199.

[4] Bakria AMM Al, Kamarudin H, BinHussain M, Nizar IK, Zarina Y, Rafiza AR. The Effect of Curing Temperature on Physical and Chemical Properties of Geopolymers. Phys Procedia 2011;22:286–91. doi:http://dx.doi.org/10.1016/j.phpro.2011.11.045.

[5] Kovalchuk G, Fernández-Jiménez A, Palomo A. Alkali-activated fly ash: Effect of thermal curing conditions on mechanical and microstructural development - Part II. Fuel 2007;86:315–22. doi:10.1016/j.fuel.2006.07.010.

[6] Sindhunata, van Deventer JSJ, Lukey GC, Xu H. Effect of Curing Temperature and Silicate Concentration on Fly-Ash-Based Geopolymerization. Ind Eng Chem Res 2006;45:3559–68. doi:10.1021/ie051251p.

[7] Kumar S, Kumar R, Mehrotra SP. Influence of granulated blast furnace slag on the reaction, structure and properties of fly ash based geopolymer. J Mater Sci 2009;45:607–15. doi:10.1007/s10853-009-3934-5.

[8] Adam AA, Ramadhan BR, Maricar S. The Effects of Water to Solid Ratio, Activator to Binder Ratio, and Lime Proportion on the Compressive Strength of Ambient-Cured Geopolymer Concrete. J Civ Eng Forum 2019. doi:10.22146/jcef.43878.

[9] Temuujin J, van Riessen A, Williams R. Influence of calcium compounds on the mechanical properties of fly ash geopolymer pastes. J Hazard Mater 2009;167:82–8. doi:http://dx.doi.org/10.1016/j.jhazmat.2008.12.121.

[10] Zhang Z, Wang H, Provis JL, Reid A. Efflorescence : A Critical Challenge for Geopolymer Applications ? Concr Inst Aust Bienn Natl Conf 2013.