Setting Time of the Geopolymer Binder with White Soil Substitution

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Abstract. This study analyzes the effect of white soil as a substitution of fly ash on geopolymer binders. Fly ash from Tanjung Jati Jepara, white soil from Kupang, water and NaOH as alkaline activators are mixed into binders and tested for setting time with a Vicat tool. The water factor of the binder was used 0.5. The white soil substitution in fly ash using a percentage of 0 to 30 of the weight of fly ash. Fly ash and white soil used had the particle size smaller than 0.074 mm or passed the number 200 sieve and had a moisture content of 0%. The results show that the setting time of geopolymer binder with substitution white soil in the range of 52.5 to 105 minutes for the initial setting time and is between 180 to 225 minutes for the final setting time. This value approaches the same as the setting time of Portland cement in SNI 15-2049-2004. From the 7 variations tested, the binder with 15% of white soil has a faster setting time compared to other variations, i.e 52.5 minutes for initial and 180 minutes for final setting time. The temperature when testing affects the setting time of the geopolymer binder

1. Introduction
White soil is natural ingredients from Kupang, East Nusa Tenggara. The white soil is a rock that has undergone changes in chemical composition, caused by weathering and the influence of underground water conditions. The white soil has brownish-white features; it is compact and rather difficult to dig with simple tools. It has chemical constituents, namely SiO2, P2O5, Al2O3, Fe2O3, FeO, MnO, MgO, CaO, Na2O, K2O, TiO2 and SO3 [1]. The content of Si and Al in white soil is one source of material that can be used for geopolymer materials [2].

More recently, fly ash has been used as a component in geopolymers, where the reactivity of the fly ash can be used to create a binder similar to a hydrated Portland cement in appearance, but with potentially superior properties, including reduced CO2 emissions [3]. Research on geopolymers made from fly ash and white soil still needs to be developed. This study will examine the setting time of geopolymer binders made from fly ash with white soil substitution.

2. Research Methods

2.1. Binder Geopolymer Composite Material
This study intends to obtain the setting time of geopolymer binder with fly ash and NaOH as an activator and compare it with geopolymer binder with the white soil substitution on part of fly ash. The amount of white soil substitution given varies and each variation will be measured when initial and final setting time. The composition of the geopolymer binder is fly ash, white soil, water and alkaline activator (NaOH) with molarity level 8 M. These materials mixed formed geopolymer binders. The material used must meet certain criteria so that it can act as a binder such as size and moisture content.
In this study, white soil is a natural product from Kupang East Nusa Tenggara was used. The white soil has several chemical compounds, including calcium, magnesium, silica, alumina and iron, expressed in the form of oxides (CaO, MgO, SiO₂, Al₂O₃, Fe₂O₃). The white soil used a size smaller than 0.075 mm or passed the number 200 sieve, as shown in Figure 1(a), while the fly ash used was class F from PLTU Tanjung Jati B Jepara. The fly ash is a mineral residue produced from the combustion of refined coal at a power plant center. It used had the particle size smaller than 0.075 mm or passed the number 200 sieve, as shown in Figure 1(b). The alkaline activator used was sodium hydroxide (NaOH) 8M[4]. NaOH used to facilitate the condensation polymerisation reaction on the geopolymer, as shown in Figure 1(c).

![Figure 1. The constituent material of geopolymer binder](image)

(a) White soil (b) Fly Ash (c) NaOH

2.2 Making Binder Test Objects
In conducting this research, several tools were needed to support the work process and testing, which are as follows a set of Vicat tools with needles Ø 1 mm, scales, ebonite ring, glass plate, stopwatch, porcelain bowl and stirrer. The Vicat tool shows in Figure 2.

![Figure 2. Vicat tools](image)

2.3 Composition of Binder Geopolymer
There are 7 variations of the test object with different compositions according to the percentage of white soil substitution, which varies 0, 5, 10, 15, 20, 25, and 30% from fly ash proportion. The composition of binder geopolymer mixtures with NaOH activator as shown in Table 1.
Table 1. Composition of binder geopolymer mixtures with NaOH activator

| Test Object | White soil substitution (%) | Fly Ash (% | White Soil (gr) | Fly Ash (gr) | Water (ml) | NaOH (gram) | (% White soil to fly ash) |
|-------------|-----------------------------|------------|-----------------|--------------|------------|-------------|---------------------------|
| 1           | 0                           | 100        | 0               | 200          | 80         | 25.6        | White soil substitution 0% |
| 2           | 5                           | 95         | 10              | 190          | 80         | 25.6        | White soil substitution 5% |
| 3           | 10                          | 90         | 20              | 180          | 80         | 25.6        | White soil substitution 10% |
| 4           | 15                          | 85         | 30              | 170          | 80         | 25.6        | White soil substitution 15% |
| 5           | 20                          | 80         | 40              | 160          | 80         | 25.6        | White soil substitution 20% |
| 6           | 25                          | 75         | 50              | 150          | 80         | 25.6        | White soil substitution 25% |
| 7           | 30                          | 70         | 60              | 140          | 80         | 25.6        | White soil substitution 30% |

Usage of 8 M NaOH

Calculation of NaOH molarity with 8 M NaOH

\[ M = \frac{\text{NaOH weight}}{\text{Mr NaOH}} \times \frac{1000}{\text{Water Volume}} \]

\[ 8 = \frac{\text{NaOH Weight}}{40} \times \frac{1000}{80} \]

NaOH Weight = 25.6 gram

2.4. Method for Making Test Objects

When cement is mixed with water, it hydrates and makes cement paste. This paste can be moulded into any desired shape due to its plasticity. Within this time cement continues with reacting water and slowly cement starts losing its plasticity and set harden. This complete cycle is called setting time of cement.

The initial setting time is the time needed for the cement from the time it starts reacting with the water to cement paste until it loses its plasticizing properties. The initial setting test method uses ASTM C 191[5] standard and uses a Vicat tool with a 1 mm diameter needle. The initial setting time of the cement is obtained when the reduction reaches 25 mm. The time of setting to cement between 45-120 mm, while the final setting time is the time where needle penetration is not visually visible within a maximum of 360 minutes[6]. The setting time test on this geopolymer binder also uses the same method as follows:

1. The white soil and fly ash used were the particles that pass sieve no. 200. The white soil and fly ash had water content of 0%.
2. The white soil, fly ash, water and NaOH were weighed as planned.
3. The water and NaOH were mixed. Because of the heat produced during the mixing process, some waiting time was needed for the sample to be cold.
4. Insert the white soil and fly ash into the bowl and stir until evenly distributed.
5. Check and adjust the equipment needed, adjust the Vicat tool so that it shows the position of 0 mm.
6. The surface of the ebonite ring smeared with the oil and then placed the ebonite ring on the glass plate with a small diameter of the ebonite ring at the top.
7. The mixture of white soil and fly ash is then mixed with a solution of NaOH and water, then stir for 3 minutes to form a paste.
8. Insert the paste mixture into the ebonite ring and tap the ebonite ring with a spoon so that the air cavity in the paste disappears, then flatten the surface of the paste.
9. Release the Vicat needle freely from the starting point 0.
10. The decrease in binder at 30 seconds after the needle was removed was noted.
11) Furthermore, every 15 minutes tested and a decrease that occurs is recorded. Created the drop graph. The initial time starts to the binder at a decrease of 25 mm. Time starts to harden at a decrease of 0 mm.

During testing the equipment must be free of vibrations. Keep the needle 1mm straight and clean because the lumps of the binder attached to the side of the needle will slow penetration and if the binder sticks to the tip of the needle will accelerate penetration.

3. Result and Analysis
The results of the setting time test on the geopolymer binder could be illustrated in the graph of penetration (mm) – time (minute). For the setting time of the geopolymer binder without white soil, substitution is shown in Figure 3, while the geopolymer binder with white soil substitution in Figure 4.

![Figure 3. Penetration-time relationship in geopolymer binders (without white soil substitution)](image)

From Figure 3 it is shown that the binder which is composed of a mixture of fly ash, water and NaOH activator and tested at a temperature of 29 °C has an initial setting time of 90 minutes and a final setting time of 210 minutes. The chemical composition of fly ash which contains Silica and Alumina almost resembles the chemical composition of Portland cement, making the setting time that occurs is still within the time span given to Portland cement.
Figure 4. Penetration-time relationship in geopolymer binders (with percentage white soil substitution)

The Figure 4 shows the graph of the setting time that occurs in the binder given substitution in the proportion of fly ash with white soil, with a percentage of 5 to 30. White soil which has several chemical constituents including Calcium, Magnesium, Silica, Alumina, and Ferron affects the setting time of geopolymer binders. From the six variations of binder with white soil substitution, it can be seen that variation 4 has a faster binding time compared to other variations, that is 52.5 minutes. The influence of higher temperatures during testing (30°C) is thought to be the cause of faster setting time while the other binders tested at 29°C have an initial setting time of 75 to 105 minutes, so it can be said that the test temperature also affects the setting time of the geopolymer binder.

The experiment on 7 variations showed that the longest initial setting time occurred in binder variations with white soil substitution of 5 and 30% i.e. during 105 minutes, which means longer than the binder without white soil as shown in Figure 5.
Whereas the final setting time was seen that variations 4 and 6 which are the binders with white soil substitutions 15 and 25% have the shortest time, which means the binder hardens the fastest. The time range of the binder begins to lose its plasticizing properties to a solid mass for all variations ranging from 105 to 135 minutes. The initial and the final setting time of the geopolymer binder, both with and without white soil substitution are in the same time span as portland cement, which is a minimum of 45 minutes and a maximum of 360 minutes.

4. Conclusion

Based on the results of the analysis in this study, it can be concluded that:

1. The initial setting time of geopolymer binders with or without white soil substitution is about 52.5 to 105 minutes, while the final setting time is between 180 to 225 minutes. This range approaches the same as the setting time of Portland cement in SNI 15-2049-2004.

2. The fastest initial setting time in binder geopolymer with NaOH activator occurs in binder 4, namely by adding 15% white soil substitution, the initial setting time occurs at 52.5 minutes and the final setting time occurs at the 180 minutes.

3. The temperature when testing affects the setting time of the geopolymer binder.

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