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PROPERTIES OF CALCINED OEBELO RED SOIL MODIFIED FLY ASH BASED GEOPOLYMER

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ABSTRAK

Research on fly ash has developed rapidly in recent years, one of which is its use as an environmentally friendly geopolymer concrete material. This research study the effect of calcination temperature and duration of red soil to compressive strength and setting time of fly ash based geopolymer paste. The red soil calcination process was carried out with variations in temperature of 400°C, 600°C, and 800°C for 4 and 8 hours of calcination. The activator solution was prepared by mixing a solution of NaOH with a solution of Na2SiO3 with a 1:1 composition. Sample treatment was carried out in 2 types, with ambient curing type and dry curing type. From the results of this study, the fastest setting time and maximum compressive strength of geopolymer paste of 23.14 MPa achieved at 800°C red soil calcination at 8 hours, and dry curing.

Kata kunci : *red soil; calcination; geopolymer; setting time; compressive strength*

1. PENDAHULUAN

Concrete is very popular and widely used, among others because the materials are easy to find locally, the price is relatively cheap, and the manufacturing technology is relatively simple. However, recently, the widely known concrete has been getting more and more criticism, especially from those who are concerned with environmental sustainability. The main thing of concern is the emission of carbon dioxide (CO_2) gas with the greenhouse effect produced in the cement production process (Poudyal and Adhikari, 2021). Production of one tonne cement, the CO2 gas produced is approximately one tonne. These gases are released into our atmosphere freely and then damage our living environment, among them causing global warming. Another issue that is often questioned is the problem of the durability of the concrete itself depends on the material chosen (Frigione, 2018). Concrete buildings in general already require repairs, maintenance and replacement with the annual costs is a significant expenditure because they have started to deteriorate when the age of the building has only reached 20 years, even though it has been well planned (Gardner et al., 2018).

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Geopolymers are said to be environmentally friendly, because apart from being able to use industrial waste, the geopolymer concrete manufacturing process does not require much energy, as does the cement manufacturing process which requires at least a temperature of up to ffl1400°C. This makes geopolymer a competitive material if the temperature processing factor was considered, since by heating at ffl60°C - 80°C for one full day, it can produce high compressive strength geopolymer (Muhammad et al., 2019). Therefore, the manufacture of geopolymer concrete is able to reduce CO_2 gas emissions caused by the cement production process up to 20% (Latawiec et al., 2017; Gomes et al., 2019; Adesina, 2020; Aranyasen et al., 2021). Research results so far have shown that geopolymer concrete has good engineering properties to use, including high strength and durability (Neupane et al., 2018; Mohammed et al., 2021; Kotop et al., 2021; Amran et al., 2021; Zailan et al., 2022).

A precast concrete company in Australia has even started to produce prototypes of precast geopolymer concrete in the form of railroad sleepers, concrete pipes for sewerage, and others. The thing that gives quite important differences between geopolymer concrete and organic polymer concrete which has been introduced earlier, is the cost of manufacture.

Geopolymer concrete can be produced at a cost equivalent to ordinary concrete, which is much cheaper than the cost of producing organic polymer concrete (Khater and Nagar, 2019; Reeb et al., 2021). Geopolymer concrete is a new hope to reduce the use of Portland cement in infrastructure development in the world. Davidovits stated that the manufacture of geopolymer cement can reduce carbon dioxide gas up to 80% of the total production of carbon dioxide produced from the cement industry. Geopolymer concrete uses coal ash (fly ash) as one of the basic materials (Singh, 2018; Yadav and Ezhilselvi, 2021).

In Kupang Regency, Indonesia there is currently a Steam Power Plant which produce large amounts of fly ash. If not utilized properly, this will actually cause pollution so that new innovations are needed for processing these wastes. One way to use it is as a base material for environmentally friendly geopolymer pastes. In addition, clay which is the basic material for making bricks is a natural material that has high plasticity in the initial manufacturing process, then hardens after drying. There are several types of clay based on color and one of them is red. Red soil is a basic material that is found in many areas on the island of Timor and one of them is the village of Oebelo. It also has strong binding properties and does not pollute the environment, so this material can be used as an environmentally friendly alternative concrete. Ceramics at low temperatures, such as Venus ceramics can be produced between 50°C and a maximum of 500°C, that will produce maximum compressive strength at a temperature of 300°C-400°C. Clay containing natural chemicals will produce a fast drying range, which is referred as a geopolymeric reaction (Khalifa et al., 2020). Clay that calcined at high temperatures will undergo changes (Kaczyńska et al., 2021; Hanein et al., 2022). This will determine the chemical properties of the soil and affect the change in shape when the soil is calcined.

Research that has been carried out previously is geopolymer paste using clay without calcination with the activator solution consists of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) at 1:1 ratio with dry curing conditions resulted in a faster setting time while samples treated under ambient curing conditions experienced a longer setting time. In order to obtain a setting time equivalent to portland cement, in this study red soil calcination time and temperature was varied with the aim of achieving a faster setting time and also producing geopolymer with higher compressive strength.

2. METHODOLOGY

Red soil was obtained from Oebelo Village, Kupang, Indonesia (Figure 1). The clay will be calcined at 400°C, 600°C, and 800°C in the Energy-Informatics Materials Laboratory, Nusa Cendana University. Calcined red soil results in very different colors, the greater the calcination temperature, the brighter the red soil color looks (Figure 2).

Figure 1. Oebelo Red Soil

Fly ash used in this study was obtained from PLTU Bolok, Kupang Regency, East Nusa Tenggara Province. The oxide composition of fly ash was presented in Table 1.

The percentage of CaO is 3.64% so the fly ash can be categorized as class F fly ash since the total CaO compound is less than 10%. Alkali activator solution consists of NaOH and Na2SiO3 at 1:1 ratio were prepared 6 hour beforehand to ensure the solution was homogenously mixed. These steps was conducted at Civil Engineering Department, Nusa Cendana University.

Table 1. Oxide composition of class F fly ash

No.	Oxide	Percentage (%)
1	SiO_2	57.47
2	Al_2O_3	21.88
3	Fe_2O_3	3.61
4	TiO_2	0.81
5	CaO	3.64
6	MgO	1.41
7	Na_2O	2.69
8	K_2O_3	1.59
9	LOI	1.77

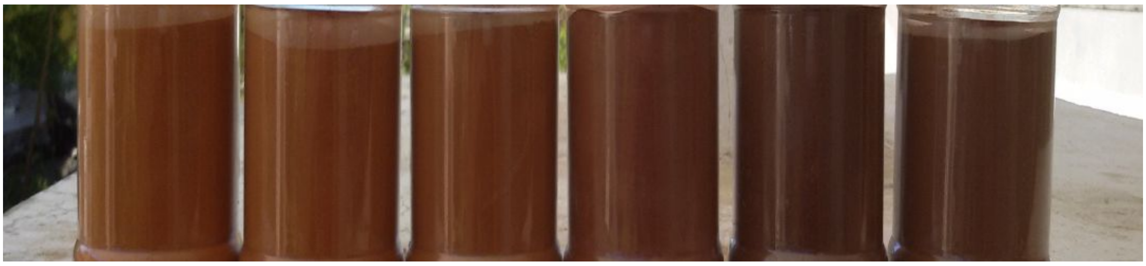


Figure 2. Calcined red soil at (a) 800°C (b) 600°C (c) 400°C

The complete mix design of this study is presented at Table 2. Geopolymer paste was made of binder consists of 95% fly ash and 5% calcined red soil and activator mixed at 2:1 ratio until become slurry form. The setting time test according to ASTM C191 was carried out using Material Basting Equiment (MBT) vicat apparatus. The slurry was casted into cyllindrical mold with a 25mm diameter and 50mm height.

Table 2. Mix Design of geopolymer with calcination temperature variation

No.	Code	Binder : Activator	Calcination Time (hour)	Calcination Temp. (°C)	Curing Method
1	A-400-4H	2:1	4	400	Ambient
2	D-400-4H				Dry
3	A-400-8H		8		Ambient
4	D-400-8H				Dry
5	A-600-4H		4	600	Ambient
6	D-600-4H				Dry
7	A-600-8H		8		Ambient
8	D-600-8H				Dry
9	A-800-4H		4	800	Ambient
10	D-800-4H				Dry
11	A-800-8H		8		Ambient
12	D-800-8H				Dry

The compressive strength test is carried out by applying a load to the test object which is installed vertically. and undergone ambient curing (room temperature) the test object until it reaches the age of 14 days and then tested its compressive strength The tested geopolymer paste can reach maximum strength when the specimen is crushed. The tool used in this compressive strength test is the Materials Testing Equipment (MTE) with a maximum capacity of 250 kN Panairsan Pratama (PP) at the Public Works Service Laboratory in Kupang City.

3. RESULTS AND DISCUSSION

3.1. Compressive strength analysis

Compressive test is carried out to analyze the relationship of calcination temperature and the strength of geopolymer. The compressive strength test for all of the mix design in Table 2 was shown in Table 3. The maximum load data is then processed to obtain the value of the compressive strength (fc) of the paste by using the following formula from equation 1.

$$fc = \frac{F}{A} \quad (1)$$

where :
fc = compressive strength (MPa)
F = maximum load (N)
A = area of sample (mm^2)

Table 3. Compressive strength of geopolymer with calcination temperature variation

No.	Code	Compressive Strength (MPa)
1	A-400-4H	7.00
2	D-400-4H	14.59
3	A-400-8H	7.76
4	D-400-8H	15.06
5	A-600-4H	9.59
6	D-600-4H	16.76
7	A-600-8H	10.35
8	D-600-8H	18.88
9	A-800-4H	9.72
10	D-800-4H	22.48
11	A-800-8H	10.56
12	D-800-8H	23.12

Figure 3 shows the relationship of curing method to compressive strength of geopolymer paste. The compressive strength of dry curing for same calcination temperature and duration were nearly doubled compared to ambient temperature. This is because the geopolymerization was accelerated when temperature is increased. Temperature of dry curing at 80°C also ensure the water from alkali activator was not evaporated as it is below 100°C, which ensure there are sufficient hydroxide to dissolve the alkali silicate in aluminosilicate binder that play major role to develop 3-D polymer network in geopolymer. It is also can be seen that calcination time of red soil only slightly increase the compressive strength, so it can be stated that 4 hour is sufficient to calcinate the red soil.

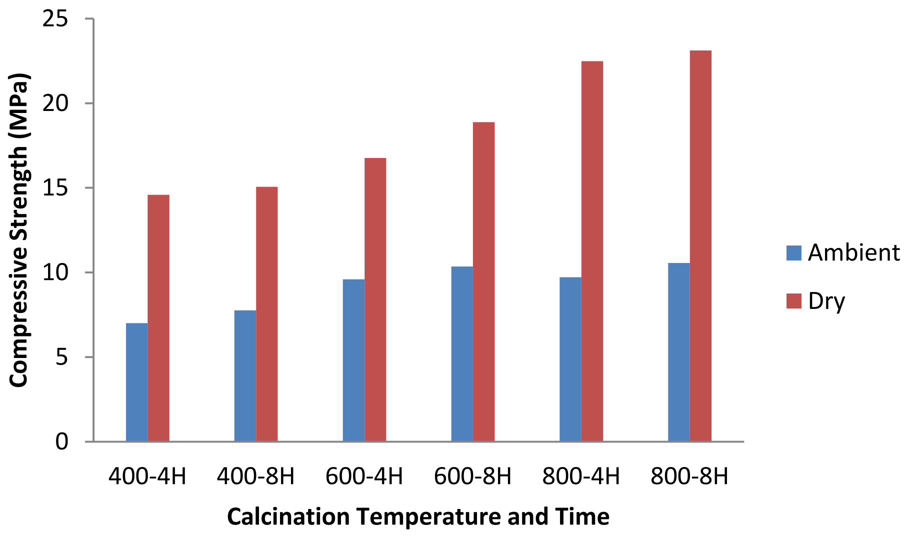


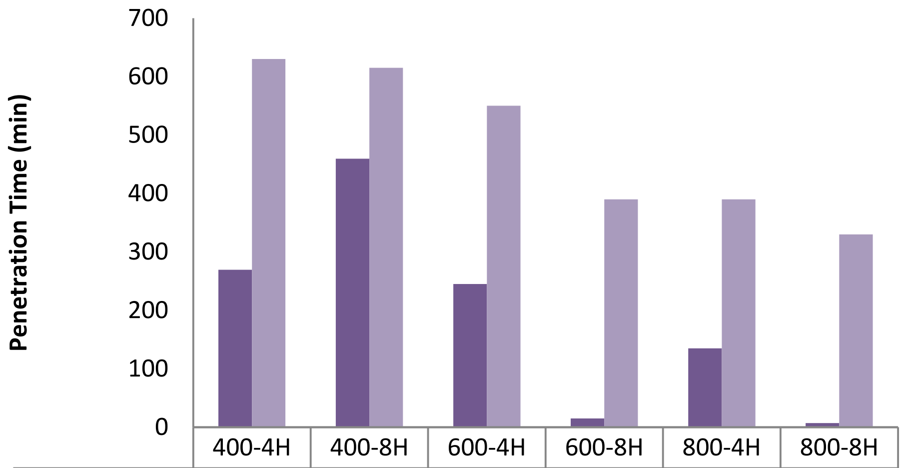
Figure 3. The effect of curing method on compressive strength of geopolymer

Temperature of dry curing below 100°C also ensure there was still small quantity of water in hardened geopolymer, hence prevents the material from undergo drying shrinkage. Particle size of precursor material of geopolymer also plays major role in strength development of geopolymer. High temperature exposure of precursor material unlock the potential of geopolymer material for elevated temperature applications.

3.2. Setting time analysis

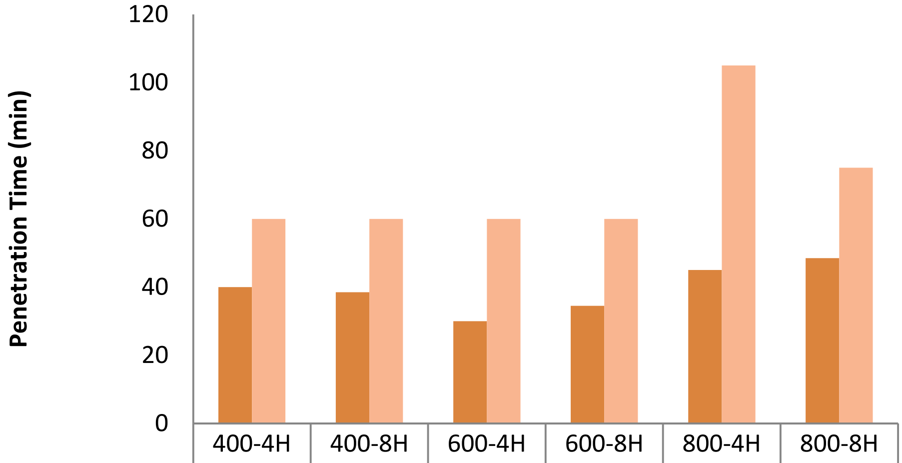
Figure 4 shows the relationship of setting time with Calcination temperature and duration of geopolymer, with increase in both parameters result in faster initial and final setting time. The mixture that close to Portland cement based concrete setting time was A-800-4H. Sample A-600-8H and A-800-8H undergoes a phenomenon called flash setting, which normally happened at Class-C fly ash based geopolymer.

Figure 5 shows initial and setting time of geopolymer in dry curing method, with different calcination duration and temperature. Compared to ambient curing, geopolymer cured at dry method reached initial setting time below 60 minute and final setting time at 120 minutes. This is due to water content in alkali activator forced to react with aluminosilicate to form geopolymer compound, results in denser material. The calcination duration and temperature did not affect initial and final setting significantly as ambient curing, which the final setting is reached not long from initial setting time. This results provide the potential utilization of geopolymer in emergency repair material, since most of the sample already reach the final setting at 60 minutes. However, the challenge is to provide the necessary temperature similar close to dry curing, since it is difficult to expose geopolymer to 80°C at closed space in field application. Exposure to hot air with altered temperature is one of the proposed method to utilize this fast setting time properties of geopolymer.



	400-4H	400-8H	600-4H	600-8H	800-4H	800-8H
Initial Setting	269.5	459.5	245	15	135	7
Final Setting	630	615	550	390	390	330

Figure 4. Setting time of geopolymer in ambient curing



	400-4H	400-8H	600-4H	600-8H	800-4H	800-8H
Initial Setting	40	38.5	30	34.5	45	48.5
Final Setting	60	60	60	60	105	75

Figure 5. Setting time of geopolymer in dry curing

4. CONCLUSIONS

From the whole series of research since preparation, sample testing, data analysis and discussion, we can concludes that calcination of red soil shorten the setting time of Geopolymer. in this case the composition of 5% red soil with calcination at 800°C, 8 hours of calcination resulted in an initial setting time of 7 minutes and a final setting time of 330 minutes. calcined red soil affects the compressive strength of geopolymer paste. The subsitution of 5% red soil to fly ash at temperature of 800°C, calcination time of 8 hours with dry curing treatment produces compressive strength of 23.12 MPa. Calcined red soil with greatly affects the color of the sample, the color transform from dark brown to red at higher calcination temperature.

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