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Original Article

A Preliminary Study of Alkali-Activated Pozzolan Materials Produced with Sodium Hydroxide Activator

Parea R. Rangan[1], M. Tumpu[2], Mansyur[3], James Thoengsal[4]

[1]*Department of Civil Engineering Department, Faculty of Engineering, University of Christian Indonesia, Toraja, Indonesia.*
[2]*Disaster Management Study Program, The Graduate School, Hasanuddin University, Makassar, Indonesia.*
[3]*Civil Engineering Department, Faculty of Engineering, Semibilan Belas November University, Kolaka, Indonesia.*
[4]*Civil Engineering Department, Faculty of Engineering, University of Technology Indonesia, Makassar, Indonesia.*

[1]*Corresponding Author : pareausanrangan68@gmail.com*

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Abstract *- Mortar that is more environmentally friendly (green mortar) is defined as concrete that at least uses waste material as one of its components or that does not cause environmental damage during the production process. Geopolymer is an environmentally friendly technology because it utilizes pozzolanic materials such as fly ash, rice straw ash, and laterite soil as waste materials. This work describes the experimental inquiry carried out to produce the geopolymer mortar based on alkali-activated fly ash, rice straw ash, and laterite soil by sodium hydroxide (NaOH). The influence of the curing technique on compressive strength and optimum mix proportion of geopolymer mortar were explored. In experiments, an alkaline activator (NaOH) was used with a binder composition at weight ratios of 4.17:1.67:4.17, rice straw ash, fly ash, and laterite soil were combined. To examine the workability and mechanical properties of the final geopolymer, experiments on mortar flow and compressive strength were performed. Fresh geopolymers subjected to flow testing reveal that all components are tightly linked, and segregation does not occur. The hardened test specimen was treated in two different ways—in the open air and by being submerged in water solutions—for up to 7 and 28 days, respectively, to ascertain its resistance. The 12 M alkali-activated geopolymer mortar may reach compressive strengths of 1.72 N/mm^2 and 3.22 N/mm^2 for air curing and 1.63 N/mm^2 and 1.68 N/mm^2 for water curing, respectively, after 7 and 28 days of casting when cured for 24 hours. The compressive strength was shown to rise with an increase in curing method, curing time, and alkali activator concentration.*

Keywords *- Fly ash, Rice straw ash, Laterite soil, Sodium hydroxide, Geopolymer.*

1. Introduction

Currently, environmental issues are a sensitive matter. Global warming and the effect of Green House Gases (GHG) in the form of CO_2 are in the spotlight in various industries [1-3]. Portland cement, one of the components of mortar, can emit greenhouse gases throughout the manufacturing process [4,5]. Its use cannot be separated from the use of Portland cement, an expensive and energy-intensive material whose production process requires burning up to 1500ºC. The calcination of lime material and burning coal or fuel to sustain high temperatures in rotary machines during the production of 1 ton of cement will produce around 1 ton of CO_2 emissions [6]. In conventional concrete, using Portland cement produces CO_2 emissions equivalent to the amount of cement used (1 kg cement = 1 kg CO_2) [7].

Apart from that, coal is needed to generate electricity, which is used as a fuel for electricity generation in many countries. The process of burning coal produces a byproduct in the form of fly ash, which is a waste. With the increasing electricity demand, coal burning is increasing, so coal waste is increasing. Only a small part of the absorbed coal waste is used to make blended cement; most of it is disposed of in a disposal pond. The ever-increasing coal waste creates environmental disturbances due to the lack of landfills [8].

Currently, the use of additional minerals from byproducts or waste minerals (fly ash, slag, and silica fume) in concrete and straw and rice husk combustion products (rice straw ash and rice husk ash) as a substitute for cement has been widely used in practice [9, 10]. Fly ash used in concrete enhances strength qualities, promotes workability in both fresh and hardened states, lowers the temperature that happens, raises the concrete's resistance to abrasion, and lessens the issue of storing and disposing of fly ash [11, 12].

In addition, fly ash can be mixed with Portland cement clinker to make mixed cement, one of which is Composite Portland Cement produced in Indonesia [13]. There have been many studies using fly ash in large quantities with Portland cement as a material for forming concrete and mortar. Based on the laboratory research, using class F fly ash in the concrete mixture produces satisfactory results [10, 12, 14]. C. D. Atis, 2005 [12], replaced cement with fly ash by up to 70% by weight, and Huang et al., 2013 [14], increased the replacement of cement with fly ash by up to 80% in concrete. These experiments aim to replace cement entirely with fly ash, which can be done by employing fly ash as a component of geopolymers.

One of the initiatives to lower cement usage and reduce coal waste and rice straw waste is the development of geopolymer materials, which can be applied to both concrete and mortar. Numerous studies have demonstrated the ability of geopolymer binders to create mortar and concrete, with mortar and concrete created using geopolymer derived from fly ash having properties similar to those of mortar and concrete created using cement. Geopolymerization is based on alumina-silica chains.

The polymerization reaction occurs between a number of alumina and silica together with a broad alkaline solution (such as NaOH, KOH, Na_2SiO_4, or a combination thereof, etc.). Geopolymerization is usually defined as alkali activation, which converts an amorphous mineral content into a composite having strong strength characteristics [15,16,18,38].

Although the color of laterite soil varies, it is typically vivid. Pink, red, and brown hues are the most prevalent. The laterite soil's physical qualities are influenced by its mineralogical makeup and particle size distribution. Depending on the origin, granulometry can range from the finest to gravel, which can affect geotechnical characteristics like strength in compression and plasticity. The fact that laterite soil does not readily swell with water and is not particularly sandy is one of its main advantages [19,20].

The aim of this research was to exploit the full potential of geopolymer and binder fly ash and rice straw ash to produce strong mortar, and a parametric study was carried out to achieve that goal. Activator Sodium hydroxide (NaOH) with a molarity of 12 M and laterite soil without the need for activation temperature and oven heating was chosen as the novelty of this study compared to previous studies that used activation temperature and oven heating as the influencing parameters. Tests performed were compressive strengths along with stress value studies that occurred on mortar specimens treated in open air or submerged water (in this study, surface water) within 7 and 28 days.

2. Materials and Method

The materials used to form geopolymers are fly ash, rice straw ash, laterite soil, and an alkaline activator in the form of sodium hydroxide (NaOH). In this study, we did not use oven heating to make the geopolymerization reaction take place.

Table 1. Physical characteristics of fly ash

No.	Type of Examination	Result of examination
1	Specific gravity	2.66
2	Fine Aggregate water absorption	26.43%
3	Sieve analysis	> 51 % pass sieve no. 50

2.1. Fly Ash

According to ASTM C618-03 (2003) [21], fly ash is a fine byproduct of coal combustion. Typically, the main chemical components of fly ash are silica (SiO_2), alumina (Al_2O_3), and ferric oxide (Fe_2O_3). Calcium oxides (CaO), magnesium (MgO), sulfur (SO_3), alkali (Na_2O, K_2O), phosphorus (P_2O_5), manganese (Mn_2O_3), and titanium (TiO_2) are some additional chemical components. Fly ash is divided into three groups by ASTM C618-03, namely class N, class F, and class C. Class N and class F require a minimum of 70% SiO_2, Al_2O_3, and Fe_2O_3 compounds, whereas class C requires between 50% and 70%. Therefore, compared to class C fly ash, where the CaO level is larger than 10%, the CaO content in class N and F fly ash is relatively low.

The findings of a flying ash character inspection. Testing the physical characteristics of fly ash from burning coal at PLTU Punagaya, Jeneponto, South Sulawesi, Indonesia, was carried out in the laboratory. The physical characteristics reviewed were specific gravity, water absorption, and sieving analysis with reference to ASTM C 618-05, 2005 [22]. The results of testing the physical characteristics of fly ash are shown in Table 1.

2.2. Rice Straw Ash

According to Rosello J. et al. (2017) [23], rice straw waste biomass has various management issues, such as field fire, which causes severe air pollution, and biological decomposition caused by natural processes emitting methane. After being turned into ash, this waste can be used again to make geopolymers. The chemical composition of ashes from several portions of the rice plant (Oryza sativa) was defined for the first time. Rice stem, leaf, and leaf sheath ashes were among the ashes that were present. Studies of onashes at the microscopic level show that the residual cellular structures' chemical element distribution is heterogeneous (spodograms). Dumbbell-shaped phytoliths (% SiO_2 > 78%) have the highest SiO_2 content.

SiO_2 is also the dominant oxide in the overall chemical makeup of ash. The Frattini test confirms the pozzolanicity of the RSA-mixed cement. The results of this reactivity are highly encouraging in terms of the possibility of reusing ash as a raw material for creating geopolymers. The rice straw ash used in this study was obtained from cutting rice straw in paddy fields in Gowa, South Sulawesi, Indonesia.

Table 2. Physical characteristics of rice straw ash

No.	Type of Examination	Result of examination
1	Specific gravity	2.37
2	Fine Aggregate water absorption	173.80%
3	Sieve analysis	< 11 % pass sieve no. 200

Rice straw ash is burned at a temperature of 500ºC. Then it is filtered until it passes sieve no. 50. Testing the characteristics of rice straw ash was carried out based on the Indonesian National Standard, which consisted of specific gravity, absorption of water, and sieving analysis. Table 2 displays the outcomes of evaluating rice straw ash's properties.

2.3. Laterite Soil

The weathering thickness of lateritic soils in the tropics is influenced by heavy rainfall, high temperatures, intense eluviation, and an effective drainage system [24]. Lateritic soils are heterogeneous and anisotropic rocks composed of strong formations of hard iron-containing minerals impregnated with soft clay materials [25]. Lateritic soils are soils rich in oxides, iron, aluminum, or both [26].

Laterite soils are widely used as building materials because they are abundantly available over many regions of the globe. There has been a great deal of research done on the use of laterite soil as a replacement for fine aggregate in concrete [27,28,30,31,39]. Laterite soils are soils that form in tropical or sub-tropical regions with a high degree of weathering from alkaline to ultramafic rocks, which are dominated by iron content.

The laterite soil used in this study comes from Gowa, South Sulawesi, Indonesia. Physical characteristic tests were carried out on lateritic soils to determine soil classification based on USCS and AASHTO. Table 3 displays the findings of tests conducted on the laterite soil's physical qualities. The Unified Soil Classification System (USCS) claims that (ASTM 2011) and this soil is classified as heavy clay (CH) and A-7-5, respectively, by the AASHTO (ASTM 2009).

Table 3. Physical characteristics of laterite soil

No.	Type of Examination	Result of examination
1	Specific gravity	2.65
2	Plastic limit (PL)	34.89%
3	Liquid limit (LL)	64.47%
4	Plastic index	32.49%

Table 4. Geopolymer mortar mixtures (1m^3)

Water (kg)	NaOH (kg)	Rice straw ash (kg)	Fly Ash (kg)	Laterite Soil (kg)
655.71	7548.95	7548.95	18872.37	18872.37

2.4. Alkaline Activator

The use of alkaline activators in the manufacture of geopolymers, both in geopolymer mortar and geopolymer concrete, has a very important role as a binding agent for aluminum and silicate elements contained in fly ash so that a polymerization bond is formed and accelerates the reactions that occur between fly ash and other elements. An alkaline activator is a substance or element that causes other substances or elements to respond. A hydrated alkaline element is employed as the activator in producing geopolymer fly ash mortar, namely sodium hydroxide (NaOH). The polymerization processes of alumina and silicate monomers require an activator. A solution of sodium hydroxide (NaOH) with a 12 Molar concentration was used as the liquid alkaline activator.

2.5. XRF Analysis

Chemical composition of laterite soil, fly ash and rice straw ash by XRF (X-Ray Flourence) test.

2.6. Mixtures Design

A mixture of laterite soil, rice straw ash, fly ash, and sodium hydroxide activator makes up the Laterite soil, rice straw ash, and fly ash geopolymer. In experiments, an alkaline activator (NaOH) was used with a binder composition of rice straw ash, fly ash, and laterite soil at weight ratios of 4.17:1.67:4.17. The early mixed experiments determined the composition of the mortar geopolymer design. Table 4 shows the components of the mortar mixture design.

2.7. Mixing Method

Combining fly ash material with rice straw ash, laterite soil, NaOH, and water. The amount of water utilized is also taken into consideration to get the ideal water content for the best compaction of laterite soil. Following is the mixing technique employed in this study:

1. Synthesis of materials with a certain content;
2. Straw and fly ashes are added to the mixing bowl;
3. After 60 seconds of slow-speed mixing, stop the mixer and manually stir the fly ash and straw ash until they are thoroughly combined;
4. While adding the previously dissolved alkaline activator (NaOH), stir the combination of fly ash and straw ash using a mixer. Mix at a slow pace for one minute. After that, shut off the mixer.
5. Manually stir the mixture to ensure even distribution. Afterwards, use a mixer to whisk the mixture for 1 minute at high speed;

6. Using a tamping rod, the resulting mixture is printed into a mold consisting of three layers and tamped 25 times each.
7. Leave the mixture alone for 24 hours so that it may set up in the mold;
8. After removing the specimen from the mold, it is cured for 7 or 28 days in water or air, respectively.

2.8. Flow Consistency Test

Flow consistency testing aims to determine the optimum amount of water to produce easy-to-work-with mortar. The amount of water used for mortar mix is closely related to workability. The ease with which a mixture can be worked is referred to as traceability or workability.

The mortar traceability test was carried out using a melting table and melting ring in accordance with SNI 03-6825-2002 [32]. The mortar was fed into a melting ring that was set on a melting table (300 mm in diameter and 20 mm in thickness) and had dimensions of 100 mm on the bottom, 70 mm on top, and 50 mm on the height. The greater the spread value, the thinner the mortar mix. The formula shown in Equation 1 is used to determine the mix flow's consistency.

$$K = \frac{Di}{D_0} \, x \, 100 \, \% \qquad (1)$$

Where:
K = consistency of mix flow (%)
Di = diameter of mortar after lifting troun conique (cm)
D0 = Diameter in troun conique (cm)

2.9. Preparing Sample Method

Through trials on mortar flow and compressive strength utilizing cube molds measuring 5 x 5 x 5 cm, this study was created to assess the workability and mechanical qualities of the final geopolymer. The mixing method used in this study is based on various pieces of literature contained in scientific journals, both national and international, and on preliminary research that has been done.

Curing is done for all test objects, including water, air, and water-air curing (immersion lift). After printing, the test object for air curing is kept at room temperature in the test object storage chamber. The air-cured test samples were kept at ambient temperature until the samples arrived at the testing laboratory. In water curing, the test sample is first removed from the mold and submerged in fresh water until it reaches the testing time. Figure 1 depicts the two curing techniques that were applied in this study: air and water curing.

2.10. Compressive Stress-Strain Test

SNI-03-6825-2002 [32] describes compressive strength testing, which involves placing a specimen between two loading rods and constantly applying a continuous monotone load to create compressive stress.

Fig. 1 Treatment process (curing) of the test object

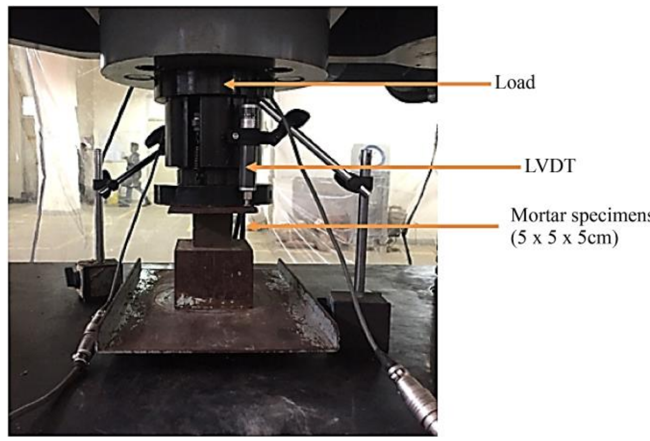

Fig. 2 Test of compressive stress-strain

In the compressive strength test, the position of the cube-shaped specimen, when loaded, is in a standing/upright state. Over time, the compressive load on the specimen will cause it to collapse or destroy.

The compressive stress at the greatest loading that causes the test object to collapse is hence the compressive strength. The specimen's vertical deflection is measured as part of the compressive strength test. The results of the compression strength and deflection tests are displayed in Figure 2. The procedure, The Universal Testing Machine Tokyo Testing Machine Inc, was used to conduct the compressive strength test with a capacity of 1000 kN and an LVDT, two 10 mm LVDTs installed vertically, along with a collection of data logging devices linked to a number of PCs. A compressive force is applied, and the LVDT is used to measure the displacement that results. This displacement value is examined to determine the strain caused by the compressive stress.

3. Results and Discussion

3.1. Chemical Characteristics of Geopolymer Mortar

Rice straw ash, fly ash, and laterite soil are the components of the materials used to make geopolymer mortar that have the following chemical properties. The purpose of checking these materials' chemical characteristics is to find the dominant compounds and enable these materials to bind and accelerate the geopolymerization reaction process.

Table 5. Chemical characteristics of geopolymer material

Oxide Content	Concentration (%)		
	Fly ash	Rice straw ash	Laterite soil
Fe_2O_3	20.06	2.35	12.58
Al_2O_3	20.16	-	50.47
SiO_2	35.53	70.92	35.51
MnO	0.27	-	0.16
TiO_2	1.27	-	1.42
K_2O	1.34	15.91	0.39
CaO	12.77	5.35	0.88
P_2O_5	-	3.62	0.47
V_2O_5	-	-	0.08
ZrO_2	-	-	0.07
SrO	0.15	-	0.05
Cr_2O_3	0.09	-	0.03
CuO	-	-	0.04
ZnO	-	-	0.04
MgO	8.19	-	-
SO_3	1.83	-	-
CoO	0.08	-	-
BaO	0.23	-	-
Pr_6O_{11}	0.06	-	-
Nd_2O_3	0.08	-	-

Table 5 displays the oxide concentration of laterite soil, fly ash, and rice straw ash. Because the fly ash used in this study contains a total of more than 70% of Ferroxide (Fe_2O_3), Alumina (Al_2O_3), and Silica (SiO_2), it is classified as class F fly ash. This is in line with several sources that state that class N and class F require a minimum of 70% SiO_2, Al_2O_3, and Fe_2O_3 compounds, whereas class C requires between 50% and 70% [20]. J. Temuujin et al., 2009 [33] also stated that the minimum content of SiO_2, Al_2O_3, and Fe_2O_3 compounds that each type of fly ash must have 70% for class N and class F fly ash and 50%–70% for class C fly ash. As a result, as compared to class C fly ash, which has a CaO level of more than 20%, the CaO percentage of class N and F fly ash is relatively low.

3.2. Flow Testing

The behavior of fresh geopolymer mortar is shown by the flow test results on a geopolymer mortar mixture with a molarity concentration of 12 M, shown in Figure 3. The flow of fresh geopolymer mortar at each molarity concentration was the same, namely 112.50 mm; the standard fresh condition flow mortar used was 110 ± 5% [34, 35].

The workability and strength of geopolymer mortar prepared from high-calcium fly ash and coarse lignite were studied by P. Chindaprasirt et al. (2014) [40]. The materials used were class F fly ash, NaOH (10, 15, and 20 M), a 2.65 specific gravity river sand at SSD, and water conditions. The obtained results show geopolymer mortar flow ranging from 110 ± 5% to 135 ± 5%.

Additionally, the geopolymer mortar's fresh specific gravity is 1901.3 kg/m^3. The laterite soil can be bound by the geopolymer mortar mixture, allowing the new mortar to flow and spread evenly without clumping or building up in the circle's nucleus.

3.3. Compressive Stress-Strain Testing

Figure 4 displays the test sample findings for air and water curing at 7, and 28 days, as well as the geopolymer's average stress. According to Figure 4 above, the test sample had an average compressive strength of 1.72 N/mm^2 and 1.63 N/mm^2 after 7 days of air and water curing, whereas 3.22 N/mm^2 and 1.68 N/mm^2 were obtained after 28 days of air and water curing and from 7 to 28 days, the air and water cure times increased by around 87.20% and 3.06%, respectively. Additionally, it demonstrates that the test sample's compressive strength grew over the course of 7 and 28 days without oven curing.

Fig. 3 Flow testing of geopolymer mortar materials

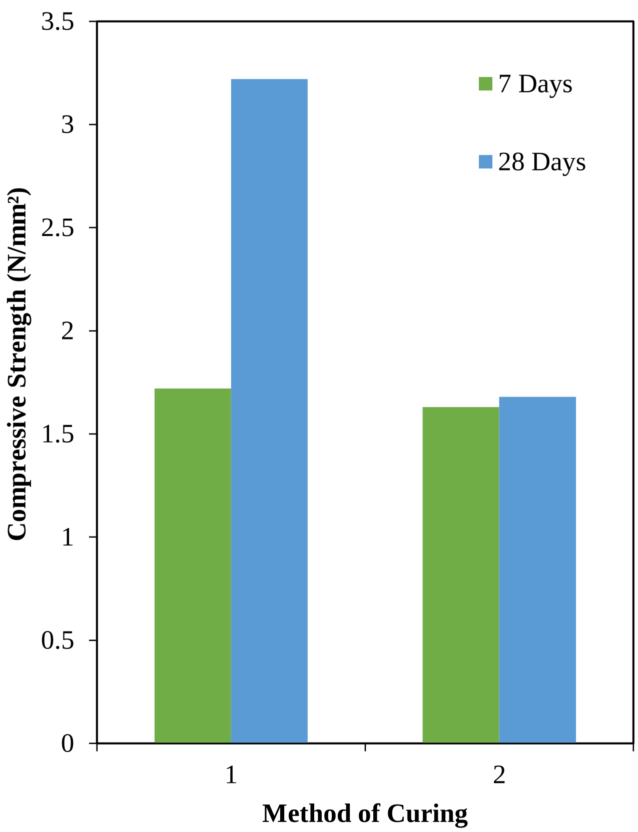

Fig. 4 Compressive stress-strain of geopolymer mortar materials

The time it takes for mortar geopolymers containing rice straw ash to transition from air curing to water curing reduces the material's compressive strength. The proportional decrease in compressive strength after 7 and 28 days of water curing. The specimens' compressive strengths are 5.23% and 47.82%, respectively, after 7 and 28 days of air-to-water cure.

Due to the heat produced by the presence of rice straw ash in this mortar combination, the fly ash geopolymer mortar with this laterite soil material can still provide strength without the curing of the oven temperature. This research also showed that compressive strength increased without oven curing similarly because the oxide content of rice straw ash, laterite soil, and fly ash could bond well and produce amorphous silica.

This demonstrates that the greater the compressive strength value, the smaller or finer the geopolymer material used. The size of the material will be directly proportional to the resulting compressive strength value. Materials that pass sieve no. 200 are one of the factors that affect the properties of geopolymer mortar, including its compressive strength [37]. The physical properties of the mortar are closely related to its durability. The damage caused by internal and external factors in the mortar itself demonstrates its durability [38-40]. Mortar usually has various pore distribution characteristics that can affect its transport properties, such as absorption, diffusion, and sorptivity, which determine the quality of the mortar.

4. Conclusion

- The mortar geopolymer may bond effectively in its fresh state without segregation or bleeding.
- The development of geopolymer's compressive strength gets stronger with time.
- The silica and alumina are leached by sodium hydroxide (NaOH) and act as a binder.
- The findings of this study can be utilized to encourage the use of waste materials, such as fly ash and laterite soil, as well as local materials, such as straw ash, as components of geopolymer mortar.
- Furthermore, it can aid in the development of eco-friendly (environmentally friendly) national infrastructure by eliminating the need for oven heat to initiate the polymerization reaction.
- This research can also be developed to increase the compressive strength of geopolymer mortar, which resembles conventional concrete in general.

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