SINERGI Vol. 29, No. 2, June 2025: 375-384
http://publikasi.mercubuana.ac.id/index.php/sinergi
http://doi.org/10.22441/sinergi.2025.2.009

Investigation of mechanical properties and microstructural characteristics of rice husk ash-based geopolymer mortar as patch repair

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Abstract
The rapid expansion of the construction sector has escalated cement use, significantly impacting the environment due to CO_2 emissions. Geopolymers are eco-friendly construction materials designed to reduce cement use and have the potential to be a patching material to rehabilitate concrete structures due to corrosion damage. Among these, pozzolanic materials like rice husk ash, rich in aluminosilicate, are abundant and suitable for geopolymer binders. This study explored the use of rice husk ash and alkali activators ($NaOH/Na_2SiO_3$), with different activator percentages (40%, 45%, and 50%), to evaluate their mechanical properties and potential applications as patch repair materials. This research involved formulating an optimal mix design through trial and error in a laboratory setting, followed by curing at 70 °C and testing at room temperature. XRF and SEM-EXD analyses were performed to determine the chemical composition and microstructure of the specimens. The activators, NaOH and Na_2SiO_3, were employed in a 1:3.5 ratio, with 14M molarity and 2% superplasticizer, to enhance workability. The test yielded the geopolymer mortar's highest compressive strength of 8.14 MPa at a 40% activator variation. In comparison, the highest split tensile and flexural strengths were 2.50 MPa and 1.00 MPa, respectively, both at a 50% variation. These findings demonstrated the suitability of the mortar for patch repair on concrete substrates with compressive strengths below 8 MPa. The mechanical properties of the rice husk ash geopolymer mortar were influenced by the silica, calcium, and alkali activator content, affecting the mortar's strength and density.

Keywords:
Compressive strength;
Durability;
Microstructure;
Patch repair;
Rice husk ash;

Article History:
Received: July 19, 2024
Revised: September 9, 2024
Accepted: October 19, 2024
Published: May 1, 2025

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INTRODUCTION

The construction sector in Indonesia has expanded at an annual rate of 7% to 8%, driven by rising infrastructure demands due to rapid population growth. However, this expansion has exacerbated environmental issues, particularly due to the significant carbon dioxide emissions associated with cement production [1][2]. Cement manufacturing is a major contributor to global greenhouse gas emissions, with growing demand driven by escalating concrete consumption [3, 4, 5, 6, 7]. Accordingly, it gives rise to a pressing need to reduce cement's environmental impact. Geopolymers, made from aluminosilicate materials like industrial by-products and agricultural waste, offer an eco-friendly alternative, preserving strength and stability in construction while minimizing environmental harm [4, 8, 9, 10, 11, 12]. The materials mentioned, including fly ash [5, 9, 13, 14], rice husk ash [9, 15, 16], sugarcane bagasse ash, and eggshells, contain significant amounts of silica (Si) and alumina (Al), which are crucial elements for the synthesis of geopolymers.

Geopolymers provide a significant benefit in mitigating the release of carbon dioxide during construction. Research has unveiled that geopolymers have the potential to decrease carbon dioxide emissions by 80% when compared to conventional Portland cement [6]. This reduction is accomplished using industrial and agricultural waste materials, which not only redirects this waste from landfills but also reduces the dependence on energy-intensive cement manufacturing [6]. Geopolymers provide a sustainable answer to the environmental difficulties presented by the building sector [17], [18].

Indonesia's large agricultural sector presents an opportunity to create sustainable building materials. Rice husk ash (RHA), a byproduct rich in silica, is abundant in rice production, offering the potential for eco-friendly construction material development [7, 19, 20]. The high silica concentration of RHA makes it highly suitable for usage as a pozzolan. Pozzolans are substances that, when combined with lime, have cementitious characteristics. To be classified as a pozzolan, a material must have a collective amount of silicon dioxide (SiO_2), iron oxide (Fe_2O_3), and aluminum oxide (Al_2O_3) exceeding 70%. Due to its composition, RHA fulfills these requirements and can be efficiently utilized in geopolymer compositions.

Although there are plenty of pozzolan resources in Indonesia, their use in the building sector is still restricted [21][22]. The main reason for this underutilization is a lack of research and understanding of the possible usage of these environmentally beneficial materials. This research intends to investigate the feasibility of using agricultural waste, primarily rice husk ash, as a pozzolan material in geopolymer binders. The research examined several combinations of agricultural waste and activators using three ratios (60%:40%, 55%:45%, and 50%:50%). This study performed a sequence of tests to examine the chemical composition (using X-ray fluorescence, XRF), microstructure (using scanning electron microscopy with energy-dispersive X-ray spectroscopy, SEM EDX), and mechanical properties of the geopolymer mortar. These properties include flowability, compressive strength, splitting tensile strength, and flexural strength. This study seeks to establish the feasibility of using rice husk ash as an environmentally friendly building material by analyzing the characteristics and effectiveness of geopolymer mortar.

Currently, academic study has yet to explore the use of rice husk ash (RHA) as a pozzolan in geopolymer binders for building applications. While many studies have demonstrated the benefits of using waste materials for eco-friendly construction, data on the mechanical properties of RHA as a cement substitute in geopolymers remain limited. This study aims to address this gap by analyzing the physical properties of RHA-based geopolymers, specifically for patch repair mortar formulations [23, 24, 25, 26, 27]. It presents a unique method for reducing the ecological impact of the building sector.

MATERIAL AND METHODS

The experimental procedure is summarized in Figure 1, and the details are explained in the following sub-section.

Material

This study employed an experimental method involving direct experimentation to collect data linked to the variables under investigation. The research aims to analyze the potential of agricultural waste, specifically rice husk ash, as a substitute in mortar mixtures, with the cement replacement ratios being 40%, 45%, and 50%. The mixtures also included tap water and chemical compounds such as NaOH and Na_2SiO_3. The experimental tests were designed to determine the compressive strength, splitting tensile strength, and flexural strength of the geopolymer mortar specimens.

The fine aggregate utilized for testing was Merapi sand, obtained from the Sand Mine of Merapi Mountain in Yogyakarta. The rice husk ash employed as a cement alternative was produced from burnt and crushed rice husk trash. It was sourced from the Pajangan District, Bantul Regency, Yogyakarta Province, and went through a No. 200 sieve. For this experiment, tap water was deployed as the mixing water to prepare the mortar. The alkali activator is a chemical substance combined with silica (Si) and alumina (Al). It is usually composed of a mixture of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3). NaOH was utilized as an activator for the alumino-silicate (Al-Si) reaction in geopolymers, whereas Na_2SiO_3 acted as a catalyst to speed up the polymerization in geopolymer mortar. The superplasticizer in this test functioned as a water reducer, allowing the mixture to achieve a thinner consistency with minimal water addition. Figure 2 (a to d) exhibits the fine aggregate, rice husk ash, Na_2SiO_3, and NaOH.

Mix proportion and specimen design of mortar

The mix design calculation involved a comparative analysis of rice husk ash, sand, water, and superplasticizer materials within a 1 m^3 volume. This test utilized mixed design calculations to determine the optimal composition for creating the test specimens. The results of these mixed-design calculations are detailed as follows. The composition possessed a fine aggregate to binder ratio of 70%:30%, while the binder materials consisted of three variations with ratios of 60%:40%, 55%:45%, and 50%:50%. Additionally, alkali activators, NaOH and Na_2SiO_3, were utilized in a 1:3.5 ratio, with a molarity of 14 M (NaOH 14M). The amount of water was 25% of the binder weight. Table 1 presents the mix design proportions for three ratios: (a) 60%:40%, (b) 55%:45%, and (c) 50%:50%. Subsequently, extra water, amounting to 25% of the volume of the alkali activator, was added to the mixture.

Curing method

The process of retaining the moisture content of the concrete surface occurred throughout the curing phase, which began with the compaction phase and continued through the hydration phase. The minimum curing time for conventional concrete was seven days, whereas the minimum curing time for concrete with high early compressive strength was three days. This information was derived from the standards established by SNI 03-2847-2019. There was a significant relationship between the hydration process and the risk of concrete cracking, and the humidity and temperature of the concrete had a substantial effect on the hydration. Curing was carried out for 28 days.

In the beginning, the concrete was exposed to room temperature after being oven-cured for one day at a temperature of 70 °C. The concrete mortar was coated in aluminum foil and plastic wrap to keep it moisturized during this room temperature exposure.

Fresh and hardened properties tests of mortar

The flow table test was run to evaluate the fresh qualities of the mortar. This test examined the consistency of the material and its ability. The technique involved depositing a predetermined quantity of mortar onto a flow table, followed by raising and lowering the table, following the procedure specifications. To determine the flowability of the mortar, the spread of the mortar was measured. In the case of geopolymer mortar, this test is necessary to evaluate the comfort level with which the mixture can be manipulated and applied to guarantee that it contains the proper consistency for practical application.

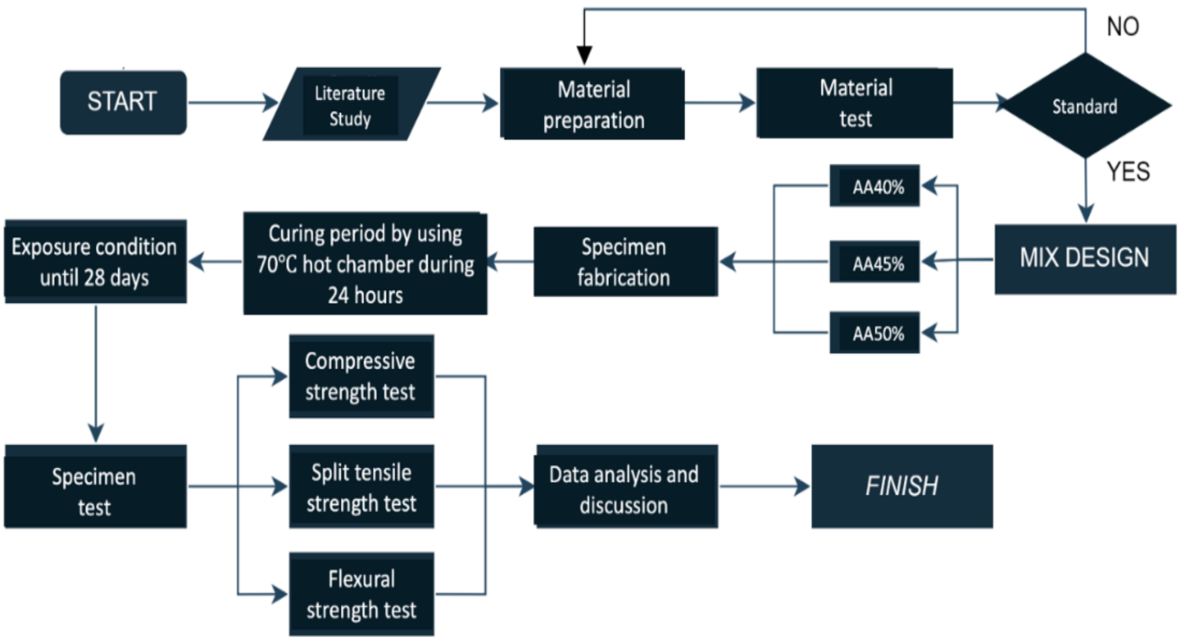

Figure 1. The experimental flowchart

Table 1. Mix design of geopolymer mortar specimens

Material	AA40 Variation	AA45 Variation	AA50 Variation
Fine aggregate (grams)	1,680	1,680	1,680
Rice husk ash (grams)	432	468	432
NaOH (grams)	44.8	50.4	56
Na_2SiO_3 (grams)	80	176.4	196
Water (grams)	59.7	90	100
Superplasticizer (grams)	6	6	6

Figure 2. The materials of the experiment: (a) fine aggregate, (b) rice husk ash, (c) Na_2SiO_3, and (d) NaOH

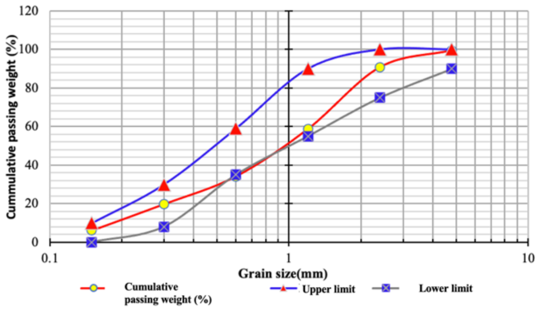

Figure 3. Grain size distribution of the fine aggregate

The compressive strength, split tensile strength, and flexural strength of the mortar were evaluated as part of the testing for the mortar's hardened characteristics. It is possible to determine the material's maximal load-bearing capacity before it fails by measuring its compressive strength (the ability of the material to endure axial loads). Split tensile strength was adopted to evaluate the mortar's resistance to tensile pressures and its potential to resist breaking under load. The capacity of the mortar to withstand bending or flexural stress was assessed using flexural strength, determining how well the mortar performs when subjected to structural stresses. These tests provided insights into the mechanical qualities and durability of the hardened mortar, conveying complete information.

RESULTS AND DISCUSSION

Characteristics of the mortar constituent materials

The analysis results of the grain gradation test adhere to ASTM C136/C136M-19, outlining the test methods for sieve analysis of fine and coarse aggregates. The fine aggregate material tested, sourced from Merapi Mountain, complies with the ASTM C136/C136M-19 standard requirements, specifying a value range of 1.5 to 3.8. x

To determine the flowability of the mortar, the spread of the mortar was measured. In the case of geopolymer mortar, this test is necessary to evaluate the comfort level with which the mixture can be manipulated and applied to guarantee that it contains the proper consistency for practical application.

The results of the moisture content test for the fine aggregate from Merapi Mountain yielded a value of 4.715%. This average moisture content falls within the specified range according to SNI 1971:2011. The mud content test on fine aggregate Progo sand generated a value of 3%. SNI 03-4142-1996 sets the requirement for mud content in aggregates to be less than 5%. Thus, Merapi sand also meets the specified requirements.

Furthermore, the X-ray fluorescence test of rice husk ash discovered that the material contained chemical elements such as silica (SiO_2), calcium oxide (CaO), phosphorus pentoxide (P_2O_5), potassium oxide (K_2O), ferric oxide (Fe_2O_3), sulfur trioxide (SO_3), and others. The results disclosed that rice husk ash had a high silica (SiO_2) content of 85.6%. Therefore, rice husk ash was utilized as the primary reference material in this study.

Flowability of mortar

Flowability testing was conducted using the flow table test. This test assessed the ease of workability for different variations of pozzolan geopolymer mortar, categorized as good due to the use of a 2% superplasticizer. The test results unveiled that the flow diameters for samples with 40%, 45%, and 50% alkali activator variations were 14.7 cm, 13.65 cm, and 13.5 cm, respectively. These values fall within the normal and good workability range for field applications.

Compressive strength

The compressive strength test was conducted in accordance with SNI 1974:2011, outlining the procedures for assessing the compressive strength of geopolymer mortar. The objective of the test was to determine the compressive strength of the geopolymer mortar samples. Figure 4 illustrates the results, showcasing an average compressive strength ranging from 2.88 MPa to 8.18 MPa, with 8.18 MPa being the highest recorded value.

It was observed that the variant with 60% rice husk ash demonstrated the highest compressive strength. This higher strength was likely attributed to the greater proportion of binder and alkali activator, particularly rice husk ash, compared to the other variants. Previous studies indicated that rice husk ash contains higher concentrations of silica (SiO_2) and calcium oxide (CaO) due to its production process [12, 20, 28]. Silica, a major component of rice husk ash, significantly contributed to the mortar's compressive strength. When silica reacted with water, it formed silica gel, which had excellent binding properties and enhanced the mortar's density by filling its pores.

Split tensile strength

The split tensile strength test was carried out to determine the split tensile strength of geopolymer mortar by following SNI 03-2491-2002, specifying the method for testing the tensile strength of concrete. The test yielded the highest split tensile strength of 2.45 MPa and the lowest of 1.49 MPa. Figure 5 depicts these results.

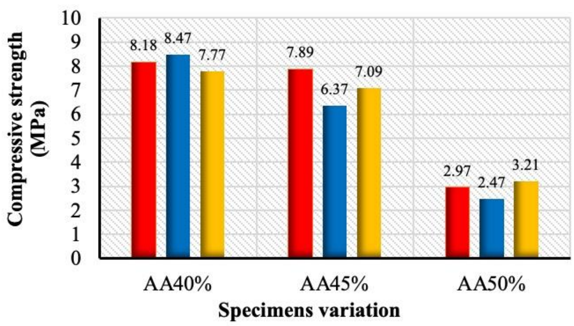

Figure 4. Compressive strength test result of geopolymer mortar

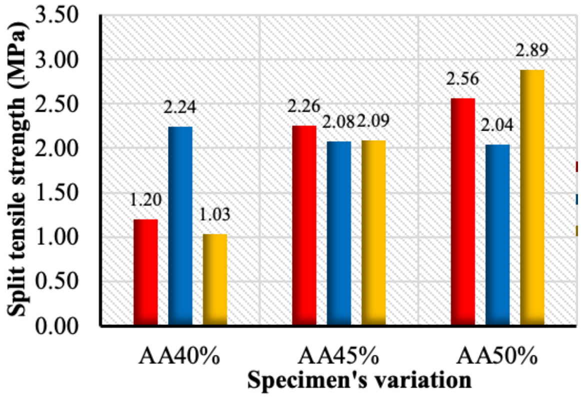

Figure 5. Split tensile strength test result of geopolymer mortar

The chemical composition of the pozzolan materials influenced the split tensile strength of geopolymer mortar. The highest split tensile strength of 2.4959 MPa was observed in the AA50% variant, attributed to the increased concentration of alkali activator, enhancing the split tensile strength by forming more robust silica-alumina polymer bonds. Conversely, the lowest split tensile strength of 1.49 MPa was discovered in the AA40% variant, where an excess of rice husk ash reduced strength due to excessive silica release, leading to a decrease in strength.

Flexural strength

The flexural strength test was performed using a central point loading method on the midpoint of the mortar beam span, applying maximum load until optimal failure was achieved. This test followed the guidelines of SNI 4154:2014, specifying the method for testing the flexural strength of mortar with a central point load. The highest flexural strength recorded was 1.00 MPa for the AA50% variation, while the lowest was 0.52 MPa for the AA45% variation. Figure 6 exhibits these results. The higher flexural strength in the AA50% variation was attributed to the higher content of the alkali activator compared to other variations. Increased alkali activator content was directly proportional to the density of the test specimens due to the enhanced binding. Higher specimen density resulted in improved strength and durability. However, excessive alkali content could lead to overreactions, causing expansion or cracking in the test specimens and declining flexural strength. Additionally, an excessive amount of rice husk ash particles in the mixture negatively impacted flexural strength due to their relatively brittle amorphous nature.

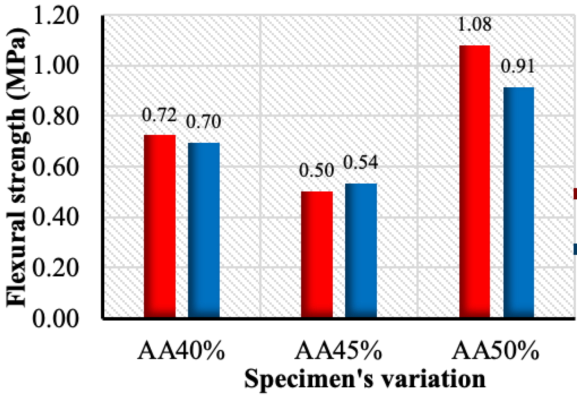

Figure 6. Flexural strength test result of geopolymer mortar

Microstructural appearance

Microstructural element testing was run using a scanning electron microscope with energy dispersive X-ray (SEM–EDX) on geopolymer mortar specimens to reveal the microstructure of rice husk ash. This test examined the microstructure of rice husk ash-based geopolymer mortar, as displayed in Figure 7. The optimal SEM-EDX test analysis focused on identifying microstructures such as pores, micro-voids, and air bubbles within the rice husk ash geopolymer mortar.

Figure 8, at 10,000x magnification, clearly demonstrates large pores and microcracks in the rice husk ash geopolymer mortar, with sizes not exceeding 10 µm. Additionally, needle-shaped Ettringite particles are visible, caused by the deposition of unreacted alkali with the precursor. At 1,000x magnification, the SEM-EDX analysis revealed a homogeneous microstructure, indicating uneven mixing in some microstructures. Figure 8 also portrays the elemental analysis at 10,000x magnification, revealing the highest elements of oxygen (O), carbon (C), silica (Si), and sodium (Na). Moreover, aluminum (Al), calcium (Ca), potassium (K), and iron (Fe) were present in smaller quantities as summarized in Figure 9. These elements resulted from the polymerization, acting as binding agents between the rice husk ash and the aggregate.

The low calcium (Ca) content in the geopolymer mortar tended to affect the mechanical strength properties, as the presence of calcium aided in hardening and escalating the density of the geopolymer mortar.

Comparison to the mechanical properties of the normal mix design

This study included a comparison of the mechanical properties of geopolymer mortar with normal mortar.

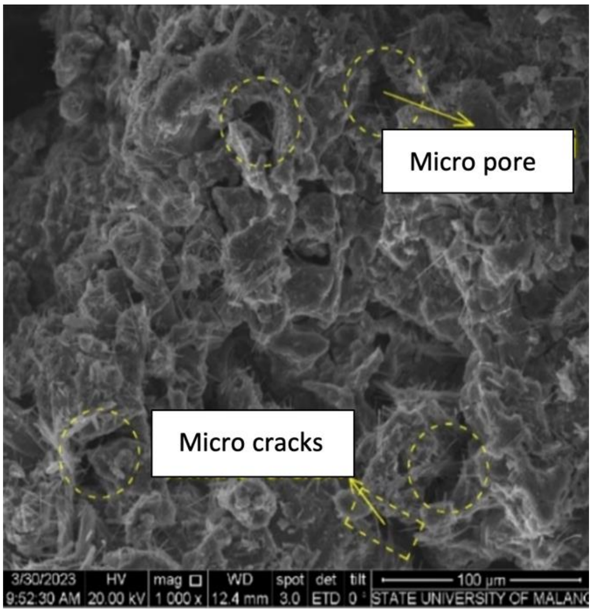

Figure 7. The microstructure of mortar geopolymer using rice husk ash based on the SEM-EDX at 1,000x magnification

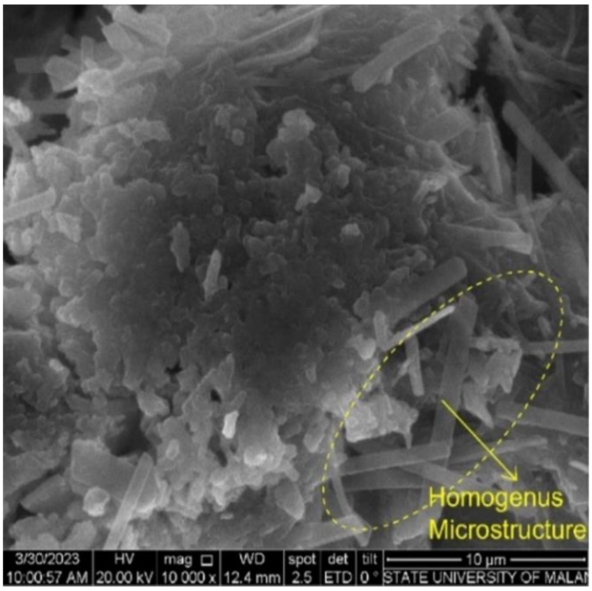

Figure 8. The microstructure of mortar geopolymer using rice husk ash based on the SEM-EDX at 10,000x magnification

This comparison intends to comprehend the differences in the mechanical strength properties between geopolymer mortar and normal mortar. The results disclosed that rice husk ash-based geopolymer mortar had lower mechanical properties than normal mortar. The mechanical properties of normal mortar were obtained from previous research [29]. This comparison could be seen in the values of compressive strength, split tensile strength, and flexural strength. The results of the mechanical property tests are depicted in Figures 10 (compressive strength graph), 11 (split tensile strength graph), and 12 (flexural strength graph).

Based on Figures 10, 11, and 12, rice husk ash-based geopolymer mortar exhibits lower mechanical properties compared to normal mortar. It was apparent in each test conducted. The compressive strength test yielded the highest value of 8.14 MPa for the AA40% variation, whereas normal mortar achieved 22.8 MPa.

The split tensile strength test generated the highest result of 2.5 MPa for the AA50% variation, while normal mortar acquired 5.27 MPa. The flexural strength test produced the highest value of 1.00 MPa for the AA50% variation, in contrast to normal mortar’s 4.18 MPa.

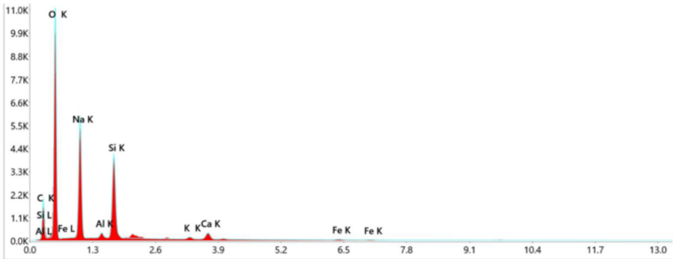

Figure 9. The chemical component of the mortar based on SEM-EDS mapping analysis

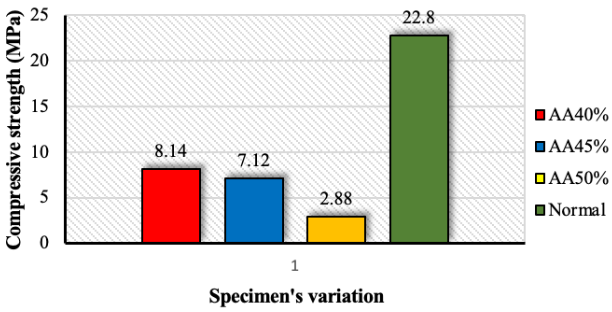

Figure 10. Comparison of the compressive strength of geopolymer mortar to that of normal mortar

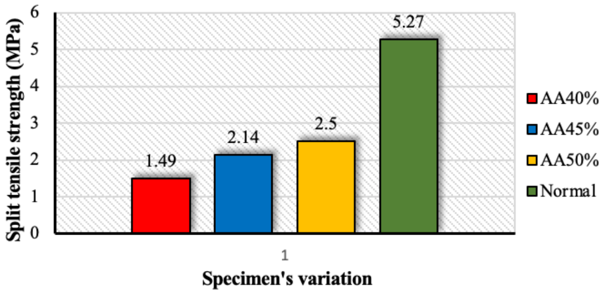

Figure 11. Comparison of the split tensile strength of geopolymer mortar to that of normal mortar

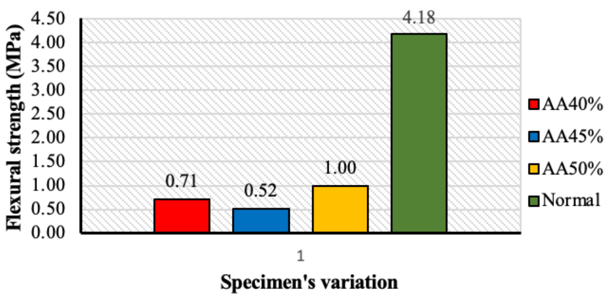

Figure 12. Comparison of the flexural strength of geopolymer mortar to that of normal mortar

The lower mechanical properties of geopolymer mortar were attributed to incomplete hydration reactions within the mortar mix, leading to cracks in the mortar paste and resulting in weaker structural bonds [30]. Additionally, rice husk ash had lower reactivity, affecting the effectiveness of the chemical reactions. Several factors influenced the variability in the quality of rice husk ash and, consequently, the quality of geopolymer mortar. These factors included the burning technique of the rice husk ash and the type of rice employed. Therefore, it is crucial to optimize these processes to ensure high-quality rice husk ash for geopolymer mortar production.

The results of rice husk ash-based geopolymer mortar exhibited lower mechanical properties compared to normal mortar, it was in good agreement with the previous research [6][7], suggesting potential for improvement. Optimizing the burning techniques and selecting appropriate types of rice could enhance the quality of rice husk ash, leading to better performance of geopolymer mortar.

The novelty of this research lies in developing a patch repair material using rice husk ash in geopolymer mortar. This approach leverages a sustainable agricultural byproduct, potentially enhancing performance while reducing costs and environmental impact. The study introduces a new, eco-friendly formulation for effective, cost-efficient repair solutions. With further research and development, rice husk ash-based geopolymer mortar could become a viable alternative to traditional mortar, offering a sustainable and eco-friendly option for construction repair due to corrosion deterioration. Additionally, rice husk ash-based geopolymer mortar could help repair structures where only low-quality concrete, equivalent to the quality of rice husk ash-based mortar, is required. This application could utilize the available material while still adhering to the structural needs of specific low-demand projects.

CONCLUSION

This study highlights that rice husk ash, with a high silica content of 85.6%, played a crucial role in the mechanical properties of geopolymer mortar. The mechanical tests revealed the highest compressive strength of 8.14 MPa, achieved with a 40% alkali activator. In comparison, the optimal split tensile and flexural strengths of 2.50 MPa and 1.00 MPa, respectively, occurred with a 50% alkali activator. These strengths were linked to the high silica content and effective formation of silica-alumina polymer bonds. Current limitations of rice husk ash-based geopolymer mortar included incomplete hydration and lower reactivity, leading to weaker bonds. Factors like burning technique and rice type affected the quality. Although it may not match traditional mortar strength, it has offered a sustainable, eco-friendly option for low-strength applications such as repair and large-scale structure in aggressive conditions.

ACKNOWLEDGMENT

The author would like to extend their gratitude to the Majelis Diktilitbang Pimpinan Pusat Muhammadiyah through RisetMu Research Grant under contract 0258.803/I.3/D/2025 and Research and Innovation Institute, Universitas Muhammadiyah Yogyakarta (LRI UMY) for their assistance with the research as well as the journal publishing.

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