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FLEXURAL STRENGTH OF PINEAPPLE LEAF FIBER REINFORCED METAKAOLIN ZIRCONIA CARBONATE APATITE GEOPOLYMER COMPOSITE

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ABSTRAK

Perkembangan di bidang material mendorong penggabungan mineral seperti kaolin, zirkonia, dan karbonat apatit, dan material alam. Indonesia yang beriklim tropis banyak ditumbuhi tanaman nanas. Geopolimer merupakan material keramik yang getas sehingga perlu ditingkatkan nilai kuat lentur untuk aplikasi komposit. Serat daun nanas dapat digunakan sebagai penguat dalam komposit Geopolimer. Penelitian ini bertujuan untuk menganalisis pengaruh penambahan serat daun nanas (Ananas comosus (L.) Merr.) terhadap sifat mekanik dan karakteristik morfologi komposit Geopolimer berbasis metakaolin-zirkonia-karbonat apatit. Penggunaan coupling agent kitosan sebagai pengikat antara matriks dan filler. Sampel penelitian terdiri dari 4 kelompok dengan jumlah 5 sampel pada setiap kelompok. Serat divariasikan 0-4% pada Komposit Geopolimer. Sampel Komposit Geopolimer diuji kuat lentur dan Scanning Electron Microscope (SEM). Hasil pengujian menunjukkan bahwa penambahan serat daun nanas meningkatkan kuat lentur komposit Geopolimer. Komposit tanpa penambahan serat memiliki kuat lentur 11.24 MPa, sedangkan penambahan 1%, 2.5%, dan 4% serat nanas menghasilkan kuat lentur sebesar 20.71 MPa, 11.57 MPa, dan 11.01 MPa. Hasil pengujian SEM menunjukkan gambaran celah dengan ukuran yang bervariasi. Sampel dengan penambahan serat nanas 4% menunjukkan pembentukan Na₂CO₃ yang menjelaskan penurunan kuat lentur pada sampel tanpa serat nanas.

Kata kunci: Geopolimer, Zirkonia, Karbonat Apatit, Serat Daun Nanas, Kuat Lentur, SEM

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ABSTRACT

Developments in the materials engineering have encouraged the incorporation of minerals such as kaolin, zirconia, and carbonate apatite, and natural materials. Indonesia, which has a tropical climate, is overgrown with pineapple plants. Geopolymer is a brittle ceramic material so it is necessary to increase its flexural strength value for composite application. Pineapple leaf fiber can be used as reinforcement in geopolymer composites. This study aims to analyze the effect of adding pineapple leaf fiber (Ananas comosus (L.) Merr.) to the mechanical properties and morphological characteristics of metakaolin-zirconia-carbonate apatite-based geopolymer composites. The use of chitosan coupling agent as a binder between the matrix and filler. The research sample consisted of 4 groups with a total of 5 samples in each group. Fiber varied 0-4% in Geopolymer Composites. Geopolymer Composite samples were tested for flexural strength and Scanning Electron Microscope (SEM). The test results showed that the addition of pineapple leaf fiber increased the flexural strength of the geopolymer composite. The composite without the addition of fiber had a flexural strength of 11.24 MPa, while the addition of 1%, 2.5%, and 4% pineapple fiber resulted in a flexural strength of 20.71 MPa, 11.57 MPa, and 11.01 MPa. The results of the SEM test show a picture of the void with varying sizes. The SEM Images of composite with the addition of 4% pineapple fiber showed the formation of Na₂CO₃ which explained the decrease in flexural strength compared to the sample without pineapple fiber addition.

Keywords: Geopolymer, Zirconia, Carbonate Apatite, Pineapple Leaf Fiber, Flexural Strength, SEM

INTRODUCTION

Commonly used ceramic posts are made of zirconia (ZrO₂). Zirconia ceramic post has high fracture resistance and good aesthetic value but is brittle and has a relatively expensive price (Grech and Antunes, 2019). These deficiencies encourage the development of alternative materials that can be used for the manufacture of dental posts. The alternative material used consists of a mixture of several materials that produce new materials with different compositions and properties are called composites (Ngo, 2020). Composite has been developed with the addition of fiber as reinforcement called fiber reinforced composite (FRC). FRC can be used as dental posts which have a high aesthetic value, are more flexible and have a modulus of elasticity that resembles dentin (Fragkouli, 2019; Sofiani and Natasya, 2022). Types of dental posts with good esthetics such as pegs FRC and ceramic posts can support good optical properties of crown restorations above the post compared to natural teeth (Bajraktarova-Valjakova, 2018; Alshouibi and Alagil, 2019).

Geopolymer is a potential material for composite matrix because the polymerization can be occurred at room temperature.

One of the raw material for Geopolymer is Metakaolin, an abundant natural material in Indonesia which composed of aluminosilicate. Another advantage of Geopolymers composite matrix are high hardness, compression strength as well as good high temperature resistance (Zainal et al., 2016; Qu et al., 2020). To obtain the biocompatibility properties, zirconia and carbonate apatite (CO3Ap) can be added to Geopolymer. Zirconia is a biocompatible and stable ceramic in human body, has good hardness and fracture toughness. In addition, zirconia is not corrosive and has low thermal conductivity (Kim and Kim, 2021).

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Carbonate apatite is an apatite containing a considerable amount of carbonate in form of calcium phosphate carbonate predominates over other components. Calcium phosphate has a chemical structure similar to bone and teeth and has good biocompatibility properties so it is effective for repairing damaged bones and hard tooth tissue (Eliaz and Metoki, 2017).

Natural fibers is a polymer that can be used as filler in composite. It consists of polysaccharides which have several advantages such as having good biocompatibility, flexibility and high tensile strength, low density.

Natural fibers are easy to obtain and inexpensive (Thyavihalli et al., 2019). One source of natural fiber is pineapple leaf fiber. Pineapple is one type of plant that grows in tropical climates such as Indonesia. In one harvest, the waste of pineapple leaves produced can reach 90%. Pineapple leaf fiber have the potential to be utilized because it has a chemical composition that is quite potential as a source of cellulose because it contains up to 81.27% (Asim et al., 2015). Cellulose is divided into three types based on the length of the chain, namely α -cellulose, β -cellulose and γ -cellulose. The α -cellulose type has long chains and a high degree of polymerization while β -cellulose and γ -cellulose have long chains and low degree of polymerization. The high content of α-cellulose indicates the level of purity of cellulose and can provide high strength properties (Shaikh et al., 2021). Pineapple leaf fiber has relatively large mechanical properties, especially its elasticity and flexural stiffness (Sena Neto et al., 2015). Based on their size, cellulose fibers are divided into three, namely macro, micro, and nano sizes. In general, the smaller the size of a fiber, the greater its mechanical strength. Cellulose fiber with nano size was developed in line with advances in the field of nanotechnology that uses cellulose fiber as a reinforcing material in nanocellulose nanometer size called (Gopakumar et al., 2019).

Composite matrix and filler binding abilities can be enhanced by using coupling agent constituent particles. One of the coupling agents that can be used is chitosan. Chitosan is a chitin substance that has been deacetylated (Zivanovic et al., 2015). The use of chitosan is expected to reduce the void and increase the uniformity distribution of the materials in the composite which can be seen by morphological characteristics test so as to produce better mechanical properties.

One of the common parameter in composite is flexural strength, which is defined as materials resistance to elastic deformity when it is subjected to a load until the sample breaks. This mechanical strength is the combination of tensile and compression.

Morphological characteristization of composite can be studied using Scanning Electron Microscope (SEM), which has high magnifications.

Based on the description above, this study is conducted to determine the addition effect of pineapple leaf fiber (PLF) to the flexural strength and morphological characteristics of metakaolin-zirconia-carbonate apatite Geopolymer composite. SEM characterization is needed to study the generated geopolymer distribution in composite materials.

RESEARCH METHODS

Synthesis of Chitosan 2% solution

2 ml of acetic acid was mixed with 98 ml of distilled water to obtain 100 ml of 2% acetic acid as solvent. Chitosan weighing 2 g was dissolved in 100 ml of 2% acetic acid solution and then stirred using a magnetic stirrer and magnetic bar until homogeneous. The solution was cooled in room temperature for about 3 hours.

Synthesis of Metakaolin

Technical grade of Kaolin powder was obtained from Bangka, Indonesia. The material was calcined in an oxygen furnace for 6 hours at 850°C to produce metakaolin powder (Figure 1). Table 1 indicates the oxide compositions of Metakaolin in this study using X-Ray Fluorscence (XRF) method.



Figure 1. Metakaolin powder after calcination at 850°C

Table 1. Chemical compositions of Metakaolin using XRF

Oxide	Percentage (wt%)
SiO_2	65.00
Al_2O_3	33.00
CaO	0.08
Fe_2O_3	0.56
Na_2O	0.06
SO_3	-

Synthesis of Zirconia

3.5 g of zirconium chloride (ZrCl₄) was dissolved in 150 ml of distilled water and stirred using a magnetic stirrer and magnetic bar. The solution was added with 0.09 g of CaCl₂.2H₂O then stirred until homogeneous. The solution was ultrasonicated for 10 minutes followed by heating in an oven at 150°C for 24 hours to remove the solvent until a white crust was formed. The formed crust is crushed using a mortar and pestle. It was then calcined at 900°C for 2 hours using an oxygen furnace. The calcined results (Figure 2) are cooled and pulverized using a mortar and pestle.



Figure 2. Zirconia powder after calcination at 900°C

Synthesis of Carbonate Apatite

Calcium nitrate tetrahydrate (Ca(NO₃)₂.4H₂O) 0.1M solution was synthesized by dissolving 2.3615g of calcium nitrate tetrahydrate in 100 ml of distilled water.

Diammonium hydrogen phosphate $((NH_4)_2HPO_4)$ 0.06M solution was carried out by mixing 0.7923g of diammonium hydrogen phosphate dissolved in 100 ml of distilled water.

Sodium hydrogen carbonate (NaHCO₃) 0.06M solution was synthesized by dissolving 0.50406g of sodium hydrogen carbonate was dissolved in 100 ml of distilled water.

All solutions of calcium nitrate tetrahydrate, diammonium hydrogen phosphate, and sodium hydrogen carbonate which has been prepared is added with 25% ammonia per drop to pH 9 in separate occasions. The solutions was precipitated for 1 day and then ultrasonicated for 10 minutes. It was then centrifuged at 4000 rpm for 10 minutes. Resulting precipitate material was put in a petri dish, dried in the oven for 10 minutes at 80°C to form a paste. It was put in a combustion boat and calcined at 700°C for 2 hours (Figure 3).

After cooled at room temperature, it was mashed with a mortar and pestle.

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Figure 3. Carbonate apatite powder after calcination at 700°C

Synthesis of PLF

Pineapple leaf fiber was shredded to obtain 5mm size (Figure 4). It was mashed and weighed as much as 75 g. It was then dissolved in 1 liter of a mixture of 3.5% HNO₃ and 10mg NaNO₂ then heated for 2 hours at 90°C. The pineapple leaf fiber was filtrated and then washed using distilled water until the pH is neutral.



Figure 4. Shredded Pineapple Leaf Fiber

15g of NaOH was added into 750 ml of distilled water to form 2% NaOH solution. Isolation of pineapple leaf fiber cellulose was carried out by diluting pineapple leaf fiber into 2% NaOH solution that had been mixed with 10 g of Na₂SO₃ while stirring with a magnetic stirrer and heated using a hot plate. The solution was stirred for 60 minutes at 50°C. It was then cooled then filtered and washed with distilled water to obtain pH value of 7 (neutral value). 1.75% NaOCl solution was prepared by dissolving 140 ml of NaOCl into 860 ml of distilled water.

The pineapple leaf fiber which has been neutralized was put into the NaOCl solution while stirring with a magnetic stirrer and heated using a hot plate. The solution was stirred for 30 minutes at 90°C, after that it was cooled and distilled water was added to pH 7. The pineapple leaf fiber suspension was separated by filtering it using filter paper and a funnel. 45% H₂SO₄ solution was prepared by dissolving 230 ml of H₂SO₄ into 270 ml of distilled water. Cellulose extraction was carried out by dissolving 60 g of cellulose in H₂SO₄ solution. It was stirred for 45 minutes at 45°C. After cooled, and 270 ml of distilled water was added. The solution was allowed to stand for 24 hours until a precipitate formed. The precipitate was centrifuged at 10,000 rpm for 10 minutes and distilled water was added to neutralize the pH. The precipitate was put into a beaker and then ultrasonicated for 10 minutes.

Synthesis of Alkali Activator Solution

40 ml of distilled water was mixed with 28g sodium hydroxide (NaOH) flakes, stirred using a magnetic stirrer until homogeneous and then cooled to obtain a 14M NaOH solution. It was mixed with 10M sodium silicate (Na₂SiO₃) in a ratio of 1:2 to make an activator solution (Figure 5).



Figure 5. Synthesis of Alkali Activator Solution

Manufacture of Geopolymer Composite

The matrix ratio of metakaolin: zirconia: and carbonate apatite was kept at 3:1:1. It was mixed and grounded with mortar and pestle. PLF was added to geopolymer slurry according to mix design of Geopolymer composite in Table 2.

Table 2. Mix Design of PLF reinforced Geopolymer composite

Code	PLF (wt%)
PLF-0	0.00
PLF-1	1.00
PLF-2.5	2.50
PLF-4	4.00

Rectangular-shaped sample mold was prepared with a length of 30 mm, a width of 5 mm, and a height of 2 mm according to the ASTM D-790. The schematic of the sample mold can be seen in Figure 6 below.



Figure 6. Mold for composite according to ASTM D-790

The composite mixture consists of metakaolin, zirconia, and carbonate apatite as matrix and PLF cellulose as filler was mixed with 3 drops of 2% chitosan solution then stirred using a spatula until homogeneous. After that, the mixture was dried using oven with the temperature of 50°C for approximately 10 minutes and then mashed. The activator solution was added and then stirred until homogeneous. The nanocomposite mixture that has been added to the activator solution is put into the mold and then heated again in the oven at 80°C for 20 hours (Figure 7).



Figure 7. Geopolymer composite sample after heated in the oven at 80°C for 20 hours

Flexural Test

The flexural test was conducted using three point bending method. The sample is placed on a support device that is 20 mm apart. Each sample is marked with a center line as a marker for placing the load. The universal testing machine type TENSILON UCT-5T type with an initial load of 2 kgf and a deformation rate of 5 mm/minute is turned on and the sample is pressed against the center line until the sample fractures. Universal testing machine shows maximum force value after fracture sample. Tests were carried out on 4 groups of samples with each group consisting of 8 samples. Flexural strength was calculated using formula (1) below.

$$\sigma_{FS} = \frac{3FL}{2hd^2} \tag{1}$$

Note:

 σ_{FS} = Flexural strength (MPa); F= Total Load (N); L= Distance between two support (mm); b= Sample width (mm); d= Sample height (mm).

Scanning Electron Microscope Test

The morphological characteristics of the samples using SEM were tested at the Scanning Electron Microscopy Laboratory, Center for Research and Development of Marine Geology, Bandung. The sample used was the debris collected from flexural test. Samples characterized by SEM must have good electrical conductivity. Since Geopolymer composites are not conductive, the samples was coated with gold using an ion sputtering method.

RESULT AND DISCUSSIONS Flexural Strength Analysis

The flexural strength of PLF-reinforced Geopolymer composite was presented in Table 3 below.

Table 3. Flexural test results of Geopolymer Composite

Code	Flexural Strength (MPa)
PLF-0	11.24 ± 2.22
PLF-1	20.71 ± 7.42
PLF-2.5	11.57 ± 2.88
PLF-4	11.01 ± 3.17
	11.01 = 0.17

Based on the data in Table 3, the flexural strength value of composite without PLF addition (PLF-0) was 11.24±2.22 MPa. Addition of 1%, 2.5%, and 4% Pineapple Leaf Fiber resulted in 20.71±7.42 MPa, 11.57±2.88 MPa, and 11.01±3.17 MPa, respectively. The test results showed that addition of PLF can increase the flexural strength when used up to 2.5%. The significant increase in flexural strength only occurred in PLF-1, while the flexural strength PLF-2.5 did not increase much compared to composite without filler addition. PLF-4 showed a decrease in flexural strength. These results prove that the addition of PLF has a certain limit on the mechanical properties to be applied as filler in Geopolymer composite.

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Statistical Data Test Analysis

Statistical data analysis was carried out using the Software Statistical Package the Social Sciences (SPSS) version 17. The normality test was carried out using the Saphiro-wilk test because the number of samples was less than 50 while the homogeneous test used the Levene test. The results of the Saphiro-wilk test showed that the flexural strength values were normally distributed (p>0.05) and the Levene test results showed homogeneous data (p>0.05), so the analysis method of this study used parametric analysis, namely One Way ANOVA. The results of the One Way ANOVA test resulted in a p-value of 0.008 (p<0.05) indicating that there was a significant difference between the four sample groups, so a further test was carried out with post-hoc LSD. Normality test was performed using the Saphiro-wilk test, showing that the data were normally distributed (p>0.05). The next test was homogenity test using Levene's test which resulted in a p value of 0.066 (p>0.05), which indicates that the data used were Data homogeneous. that are normally distributed and homogeneous can be analyzed by parametric statistics using One Way ANOVA which aims to determine the significant difference in the flexural strength value of each sample group. The results of the One Way ANOVA test showed a p-value of 0.008 (p<0.05), so it can be concluded that there is a significant difference between the flexural strength values in PLF-0, PLF-1, PLF-2.5, and PLF-4.

SEM Analysis

SEM Images of Geopolymer composite with PLF addition was shown in Figure 8-11

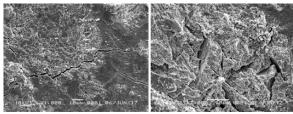


Figure 8. SEM Images of PLF-0 with magnification of 1000X (left) and 2000X (right)

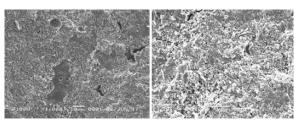


Figure 9. SEM Images of PLF-1 with magnification of 1000X (left) and 2000X (right)

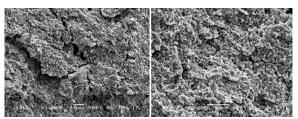


Figure 10. SEM Images of PLF-2.5 with magnification of 1000X (left) and 2000X (right)

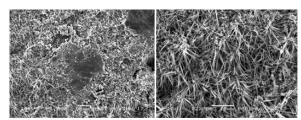


Figure 11. SEM Images of PLF-4 with magnification of 1000X (left) and 2000X (right)

The morphological behaviour of composites can explain the fluctuations in flexural strength results. Figure 8 which is the composite without PLF addition showed there are cracks and voids. The mechanical properties of composites are influenced by the interaction between the matrix and filler.

The interaction of matrix and filler can be influenced by the amount and composition of the material used (Feng et al., 2016). The perfect interaction between metakaolin and filler will affect the amount and distribution of the geopolymer structure formed. This is proven by results in Figure 9 and Figure 10 showed that the length of the cracks was smaller than Figure 8. It showed that PLF can fill the gaps in composite, resulted in increase of Flexural Strength.

Figure 9 and 10 also showed more evenly distributed geopolymer structure. This will improve the mechanical properties of the material. The Geopolymer formation can be disrupted by the carbon (C) ions present in PLF. Further increase in filler means more C ions will bind to the activator solution to form sodium bicarbonate (Na₂CO₃) compounds. This is in line with the morphological picture in Figure 11 which shows the formation of Na₂CO₃ are only formed in composites with the highest concentration of PLF. The needlelike structure almost covers all area of composite. Na₂CO₃ are not expected to form in geopolymer because it tends to decrease the mechanical properties.

CONCLUSIONS

Based on the research that has been conducted, the conclusions that can be drawn are as follows. The highest average flexural strength value of 20.71 ± 7.42 MPa was obtained in PLF-1 which has addition of 1% Pineapple Leaf Fiber, this value was increased by 84% compared to PLF-0 that has compressive strength value of 11.01 ± 3.17 MPa.

The addition of PLF has an effect on morphological characteristics of Geopolymer composite.

The sample without Pineapple Leaf Fiber (PLF-0) shown large void and cracks are presented. PLF helps the composite to become denser. This explains the flexural strength increase in PLF-1 and PLF-2.5. Further increase in PLF formed Na₂CO₃ compound as shown in PLF-4. This compound can disrupt the Geopolymerization which resulted in flexural strength decrease. It shows that there are limitations on PLF utilization in Geopolymer based composite.

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