Improved mechanical properties with the soaking time of NaOH in composites made from sugarcane bagasse fibers for future windmill blades material

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Abstract. The purpose of this study was to determine the effect of 5% NaOH alkalization and soaking time for 1 hour, 2 hours, and 3 hours on mechanical properties by tensile and impact testing and scanning electron microscope (SEM) on composites with sugarcane bagasse fiber reinforcement and epoxy matrix which have the potential as future wind turbine blades material. The method used in the manufacture of composites was a hand lay-up method with a volume fraction ratio of 60% epoxy matrix and 40% waste sugarcane bagasse fiber. The optimum results of this composite study showed the impact strength value on the composite with 5% NaOH soaking for 2 hours was 0.120 Jmm⁻², the tensile strength value was on the composite with 5% NaOH soaking for 2 hours at 10.80 MPa. SEM analysis results on the tensile test fracture pattern showed that composites have failed in the form of fiber pull out and debonding which can reduce mechanical properties, especially in strength. In this study, it was concluded that the alkalization process of 5% NaOH for 2 hours had a significant influence on improving the mechanical properties of sugarcane-epoxy fiber composites.

Keywords: composites, waste sugarcane bagasse fiber, epoxy, alkalization, wind turbine blades

1. Introduction
Due to the new era, the manufacturing industry is experiencing very fast development. The need for material in this field is also increasing. Materials with certain characteristics such as strength, ductility, and other mechanical properties as needed are highly demanded. Various types of materials have been widely developed and are also being researched in order to obtain new, effective, and environmentally friendly material. One material that is currently being researched and developed is a composite material.

Basically, a composite is a new type of engineered material consisting of two or more materials in which the properties of each material differ from one another both chemical and physical properties, and remain separate in the final result of the material. Composite materials began to
be widely used because it has several advantages including lighter weight, higher strength, corrosion resistance, easy to form, and cheaper than other materials such as metal materials. Polymer-based composite materials are the most commonly used composite materials. This material uses a polymer made from resin as a matrix and fiber as a reinforcement. Some examples of polymers are phenol-formaldehyde resins, polyester, epoxy, and others. While the fibers that can be used as reinforcement are synthetic fibers and natural fibers. Synthetic fibers such as glass fiber and carbon fiber, while natural fibers such as water hyacinth fiber, hemp fiber, pineapple fiber, bagasse fiber, and other natural fibers. Synthetic fibers can be used in the manufacture of wind turbine blades because of their strong, lightweight, and corrosion-resistant properties. But the weaknesses are availability, health factors, expensive costs, and non-biodegradable. Therefore, the presence of natural fibers is expected to replace and cover the weakness of synthetic fibers. Natural fibers can be combined with polymers called natural fiber-reinforced polymers (NFRP) [1].

The type of natural fiber composite has its own advantages compared to other alternative engineering materials including strong, lightweight, corrosion-resistant, low density, non-abrasive, biodegradability, abundant raw material availability, and low cost [2]. One of the natural fibers is the sugarcane bagasse fiber which is usually used for animal feed ingredients. Scientifically, the utilization of bagasse fiber can be further developed. Sugarcane bagasse has strong fiber with soft parenchyma tissue and was chosen because of its abundant availability in nature. In addition, this type of wood has good strength and durability. This is an added value for sugarcane bagasse to be a higher value application.

In previous studies, sugarcane bagasse and epoxy had been used as a composite material with KMnO4 treatment and hand lay-up technique, where the maximum tensile strength value obtained at 30% fiber volume fraction was 45 MPa [3]. Sugarcane bagasse is a complex composite that composed of cellulose around 40-50%, hemicellulose around 25-35%, and lignin around 15-35% and other impurities [4]. Hemicellulose is a complex polysaccharide, hydrophilic, high-branched, and amorphous which consisting of about 30% by weight of biomass. It has a low polymerization rate of around 200 and is chemically heterogeneous, their side chains can be acetylated [5]. Whereas lignin can absorb moisture and is considered a waste product. Lignin has a complex network of various similar chemicals that have varying molecular weights and their structures are not uniform [6]. The content of those substances can affect the mechanical properties of composite materials. Thus, to remove lignin and hemicellulose substances, an alkaline treatment process is carried out. One of them is the process of alkalizing sugarcane bagasse to improve the properties of the fiber, reducing lignin, producing better mechanical interlocking with the matrix, and separating contaminants contained in the fiber [7]. Alkalization is one of the methods of modifying natural fibers to improve compatibility between matrices [8]. The soaking time of the alkaline solution also affects the strength of the resulting composite. In previous studies, analyzed the flexural properties and impact strength of glass fiber/epoxy composites after being soaked in a sodium hydroxide (NaOH). Independently, flexural strength and flexural modulus decrease with soaking time. However, alkaline solutions show a decrease in flexural properties. The same tendency was observed for impact strength [9]. The focus of this study was to determine the effect of 5% NaOH alkalization on composite materials made from natural fibers with a volume fraction ratio of 60% epoxy matrix and 40% waste sugarcane bagasse fiber. The variables in this study were without treatment of bagasse fiber, alkalization treatment with 5% NaOH for 1 hour, 2 hours, and 3 hours each with a fiber length of 10 cm. In making composites for this study using the hand lay-up method. Tests carried out were tensile testing (ASTM D-638), impact resistance testing (ASTM D-6110) as well as analysis of the interface between the polymer matrix and natural fibers of the fracture form used by using SEM (scanning...
electron microscope). The future goal in this research was to find out the maximum tensile strength and impact strength of sugarcane bagasse-epoxy composite materials to later be developed as future wind turbine blades.

2. Materials and methods

2.1 Preparation of sugarcane fibers

Yellow sugar cane for this research was found in the city of Balikpapan, East Kalimantan province, Indonesia. Sugar cane stems cut to lengths of 25 cm. Furthermore, the sugar cane was peeled and squeezed using a press to make pulp. Sugarcane bagasse fiber was cut into uniform lengths of 10 cm. Then the sugarcane bagasse fiber was soaked with 70% ethanol solution for 3 hours (Figure 1a) to maintain metabolism and remove impurities contained in the bagasse fiber. Drying of sugarcane bagasse was done by forced convection for 6 hours and using an electric oven at a temperature of 110 °C for 2 hours to reduce water content. The final step was weighing the bagasse fiber mass as in Figure 1b.

2.2 Moisture testing

This test serves to determine the moisture content of waste sugar cane fiber, which was used as a composite filler. Testing of moisture content was intended to determine the parameters of drying of waste fiber. In this study, a comparison was carried out in the test data of water content on waste sugar cane fiber as a standard and an actual using an electric oven with a temperature of 110 °C for 2 hours so that data obtained in the form of initial weight and dry weight varies. Tests were conducted at the Faculty of Forestry, Mulawarman University, Samarinda City, Indonesia. The obtaining data was processed with the equation 1 [10].

\[ WC = \frac{Iw - Fw}{Fw} \times 100\% \]  

Where:

- WC = Water Content (%)
- Iw = Initial Weight
- Fw = Final Weight

2.3 Variations in fiber treatment

![Figure 1. Stages of making bagasse fiber composites: (a) Soaking of sugarcane bagasse with 70% ethanol, (b) Weighing sugarcane bagasse, (c) Pouring epoxy into aluminum foil-coated molds, (d) Preparing orientation of sugarcane bagasse fiber composites, (e) Bagasse fiber composite molds](image-url)
There were four variations of treatment, namely A: without treatment, B: alkalization with 5% NaOH for 1 hour, C: alkalization with 5% NaOH for 2 hours, and D: alkalization with 5% NaOH for 3 hours. The alkalization process was carried out by soaking of 50 grams of sugarcane bagasse with 5% 500 ml NaOH solution each time soaking variation of 1, 2, and 3 hours. After soaking, the fiber was washed by soaking the bagasse fiber with amides solution and then squeezing it. For drying, an electric oven was used at a temperature of 80 °C for 30 minutes. Then the dried bagasse fiber was stored in a container to reduce humidity.

2.4 Composite manufacturing
Sampling was done using the hand lay-up method (Figure 1c). The process of making composites was finished by mixing the epoxy matrix with bagasse fiber using a volume fraction ratio of 40% fiber and 60% epoxy matrix with the density of fiber using a value of 1.25 gr cm$^{-3}$ [11]. The results of volume and mass calculations are shown in Table 1. A mixture of epoxy and catalyst ± 30% was poured into a mold evenly coated with aluminium foil and then given bagasse fiber with a length of 10 cm fiber arranged in a uniform orientation angle 0° or unidirectional (Figure 1d) to cover a mixture of epoxy and catalyst. After that, the top of the fiber was repeated cover a mixture of epoxy and the catalyst until the mold was full. The formation of air bubbles during the process needs to be avoided. The next process was the curing process for 9-10 hours and then the composite can be removed from the mold (Figure 1e). The molds of composite test specimens were finished in dimensions to conform to ASTM standards of tensile and impact test samples. The tensile test sample was in accordance with the ASTM D 639 standard (Figure 2a), while the impact test sample was in accordance with D 6110-4 standard as shown in Figure 2b.

| Sample     | Mold in Volume (cm$^3$) | Fiber in Volume (cm$^3$) | Fiber Mass (gram) |
|------------|-------------------------|--------------------------|-------------------|
| Impact Test| 11.65                   | 4.65                     | 5.81              |
| Tensile Test| 23.10                  | 9.24                     | 11.55             |

2.5 Mechanical testing and characterization
The testing stage was divided into several parts namely impact testing, tensile testing, and scanning electron microscope (SEM) testing. For impact and tensile testing were carried out at the Mechanical Engineering Laboratory, Department of Mechanical Engineering, Faculty of Engineering, Muhammadiyah University of Malang, Indonesia. Impact testing requires samples with ASTM D 6110-4 standard provisions including notches as deep as 0.25 ± 0.05 mm. The method used in testing was the Charpy method. The sample was placed horizontally on the support until it was on the surface opposite the notch. While tensile testing requires samples in
accordance with ASTM D 638-04 standards. First, the sample was placed on the grip of the test equipment in harmony with the long axis of the sample, then slowly withdrawal was interpreted by the tensile testing machine during withdrawal until the sample was fractured. Then observed and recorded the force at the yield point and its ultimate point to increase in length until it fractured. For the analysis of the microstructure interface, it was carried out after tensile testing using SEM 300x magnification to observe the morphology of the fiber surface interface and analyze the fracture patterns on the fiber as well as the interaction between the epoxy matrix on the surface of the bagasse fiber.

3. Results and discussion

3.1 Moisture test results
The data obtained are shown in Table 2 about the average moisture content obtained of 7.69%. These results serve as the standard content of sugarcane bagasse fiber content in the manufacture of composites. However, the results of actual water content testing conducted using an electric oven at a temperature of 110 °C for 2 hours obtained an average decrease in water content of 11.99%. The results of the actual test data stated that the water content of sugarcane bagasse has decreased in water content that is greater than the standard water content results. The water content of natural fibers greatly determines the strength of bonds between cellulose and fiber resistance to the environment. The amount of water content that is too large will reduce the binding capacity between cellulose and lignin making up the fiber. In addition, the content of water content greatly affects the binding capacity (adhesion) between the fiber and the matrix [12]. It is indicated if the fiber with high water content is applied with a matrix, the interface bond becomes weak, whereas if the fiber with low water content is applied with the matrix, the interface bond becomes more optimal. Therefore, it can be concluded that the drying of the fibers with the results of the actual data is free of water content. Therefore, sugarcane bagasse used with the results of actual data drying can be applied in making composites.

| Data Type | Average Initial Mass (gram) | Average End Mass (gram) | Average Water Content (%) |
|-----------|-----------------------------|-------------------------|---------------------------|
| Standard  | 0.14                        | 0.13                    | 7.69                      |
| Actual    | 149.5                       | 133.5                   | 11.99                     |

3.2 Impact test results
From the tests carried out, the value of absorbed energy or fractured energy and the value of impact strength on composites with a 40% volume fraction of bagasse fiber and 60% epoxy matrix owned by each specimen treatment was shown in Figure 3 and Figure 4. The upward and downward trends in both graphs are the same. Statistical analysis used standard deviations to sort data that had a range or a considerable distance. The test data showed that the composite without alkali treatment has an impact strength value of 2.17 Joules which is smaller than the epoxy matrix impact test sample with a value of 2.50 Joules. While the test sample with 5% NaOH soaking for 1 hour has higher values for absorbing energy and impact strength values of 9.50 Joule and 0.074 J mm$^{-2}$ respectively than epoxy matrix impact sample and composite impact test sample without treatment. Thus, it indicates that the sugarcane bagasse-epoxy fiber composite by giving an alkalization treatment with 1-hour time has an effect on increasing strength and ductility. However, interesting things happened in composite samples with the soaking of 5% NaOH for 2
hours which has the highest values both in absorbing energy and impact strength, namely 12.00 Joule and 0.096 J mm
\(^{2}\). While the test sample with 5% NaOH soaking for 3 hours is decreased with a value of 4.50 Joules for absorbing energy and 0.035 J mm
\(^{2}\) for impact strength even lower than the composite test sample with 5% NaOH soaking for 1 hour.

With the results of impact testing and data processing carried out, it was found variations of 5% NaOH alkali treatment in 40% sugarcane bagasse composite specimens and 60% epoxy matrix had a significant effect in strengthening sugarcane bagasse-epoxy composite material. From the data obtained that the value of the energy absorbed and the impact strength value shows the variation of alkali treatment with soaking for 2 hours is the best variation among other variations. From the data on the impact test, it can be assumed that the longer soaking variation on the composite can cause a decrease in the value of absorbed energy or fracture energy and the impact strength value due to alkalization and penetration processes in excessive cellulose chains [13] thereby causing damage to the bagasse fiber and reducing the value of fracture energy and the impact toughness of the composite.

**Figure 3.** Average impact energy absorption of control samples (epoxy matrix) and various types of treatment with a volume fraction of 40% waste sugar cane fiber and 60% epoxy matrix

**Figure 4.** Average impact strength of control samples (epoxy matrix) and various types of treatment with a volume fraction of 40% waste sugar cane fiber and 60% epoxy matrix
3.3 Tensile test results

From the results of tensile testing of composite test samples, obtained a graph of the relationship between force load and length increase. Data on the force load and length increase can be processed and graphed for stress, strain, and elastic modulus. Tensile test results on composite test samples were taken on average from each experiment on the control sample (epoxy matrix) and variations without treatment and 5% NaOH alkali treatment for 1 hour, 2 hours, and 3 hours as shown in Figure 5, Figure 6, and Figure 7. It was found that the epoxy matrix test sample has a low strain of 2.00. On the other hand, this value is almost the same in untreated composite tensile test specimens which have a strain of 2.04. Based on Figure 6, composite tensile test specimens with 5% NaOH soaking for 1 hour were decreased strain to 1.92. And the reduction in strain occurs until the composite tensile test specimen at 5% NaOH soaking for 2 hours with a value of 1.17. Furthermore, there was an increase in the composite sample with 5% NaOH soaking for 3 hours, namely 1.92. This allows the soaking for 2 hours to have the lowest decrease or brittleness compared to other composite specimens. Decrease in strain value on composites with 5% NaOH alkali treatment for 2 hours is supported by the theory that plant fibers, in general, have higher stiffness and strength when given an effect based on the extraction method [14]. In this case, the type of plant fibers was bagasse with the alkalization extraction method.

![Graph showing tensile strength](image)

**Figure 5.** Average tensile strength of control samples (epoxy matrix) and various types of treatment with a volume fraction of 40% waste sugar cane fiber and 60% epoxy matrix

From the tensile strength resume and shown in Figure 5, all samples are directly proportional to the elastic modulus shown in Figure 7. It can be analysed that variations with the 5% NaOH alkali treatment and soaking time of sugarcane bagasse make the composite stronger and have an increase in soaking limit 2 hours. Tensile test results with soaking for 2 hours produced the highest tensile strength and modulus of elasticity, which were 9.46 MPa and 809 MPa, respectively. The decrease in the value of the tensile strength and modulus of elasticity occurs at 3 hours soaking. This reasoning is also supported by the results on impact strength in the same sample, which is a 3-hour soaking which is no longer effective which is likely due to the dissolution of the cellulose element so that its strength starts to decrease. The increase in soaking time will cause the strength of sugarcane bagasse to decrease due to alkalization and cellulose dissolution [13] resulting in weakness or damage to the bagasse fiber composite.
Figure 6. Average strain of control samples (epoxy matrix) and various types of treatment with a volume fraction of 40% waste sugar cane fiber and 60% epoxy matrix

Based on the modulus of elasticity shown in Figure 7 proves that the composite has the ability to influence the amount of stress compared to the control specimens or epoxy matrix specimens. From the modulus of elasticity obtained that the composite with 40% reinforcing sugarcane bagasse fiber and 60% epoxy matrix has the characteristics of more brittle material. Because of the higher the modulus of elasticity the higher the stiffness value [15]. The greatest possibility of a decrease in strength in composites with variations in specimens without alkali treatment was caused by less optimal distribution of stresses received by the matrix to the fiber due to using the simple method of hand lay-up.

Figure 7. Average modulus of elasticity of control samples (epoxy matrix) and various types of treatment with a volume fraction of 40% waste sugar cane fiber and 60% epoxy matrix

3.4 Macroscopic fractographic
Macroscopic fractographic is a physical test carried out by analyzing the macro surface image of a test specimen which aims to determine differences in the shape of fractures and composite structures. Macro testing was carried out on impact test specimens and tensile tests.

3.5 Impact-tested specimens
The following macrographic photos of all impact test specimens after the test are shown in Figure 8. The fracture in the epoxy matrix impact test specimen (Figure 8 a) was fractured into 2 parts
and the fracture that occurred showed that the epoxy matrix used in this study had the brittle properties. In the untreated composite sample shown in Figure 8b, failure occurred dominated by debonding and fiber pull out. Debonding the fiber-matrix interface starts with random fiber termination known as one of the main damage mechanisms in the unidirectional (UD) composite direction. The growth of the fiber-matrix interface debonding leads to a decrease in stiffness and ultimately a failure of the UD composite by combining several deboned gaps [16]. That is why the modulus of elasticity is low in untreated samples. In addition, the failure occurs because the fiber didn’t absorb energy optimally during the shock loading thus the interface between the bagasse fiber and the epoxy matrix is not so strong which results in the release of the bonding amplifier with the matrix. This sample fracture is brittle.

Failure in alkaline soaking for less than 1 hour in observation occurs in Figure 8 (c). Failures that occur were dominated by fiber pull-out which means the fiber absorbs more energy so that the interface between the bagasse fiber and the epoxy matrix was stronger. Failures were also seen to be less on the 2 hour alkaline soaking and the interface bond density between the fiber and epoxy was good (Figure 8d). However, fiber pull-out failures began to reappear in the 3 hour alkaline soaking sample (Figure 8e). Alkaline soaking samples of 1-3 hours showed brittle properties.

**Figure 8.** Macroscopic aspect of impact-tested epoxy composite Charpy specimens with (a) epoxy matrix, (b) composite without treatment, (c) composite of soaking for 1 h alkaline, (d) composite of soaking for 2 h alkaline, and (e) composite of soaking for 3 h alkaline

### 3.6 Tensile-tested specimens

The following Figure 9 is a macrographic photo of all the tensile test specimens after the test. Overall, the form of failure is almost similar to the impact test specimen dominated by debonding and fiber pull-out. The different thing from the observation of this tensile test sample is the fracture pattern that resembles a cup cone due to the direction of the load in the direction of the fiber. So that it is more likely to be ductile. In Figure 9 d, it is seen in a macro that there is a high level of interface density between fibers and epoxy that occurs in composites with an alkaline NaOH 5% treatment with soaking for 2 hours, making this composite has the highest tensile strength value.
Figure 9. Macroscopic aspect of tensile-tested epoxy composite Charpy specimens with (a) epoxy matrix, (b) composite without treatment, (c) composite of soaking for 1 h alkaline, (d) composite of soaking for 2 h alkaline, and (e) composite of soaking for 3 h alkaline

3.7 Microscopic fractography

Figure 10 is a photograph of SEM observations with 300x magnification of all specimens from without treatment and the 5% NaOH alkali treatment. The 5% NaOH treatment in the fiber of Figure 10b to 10d causes a partial loss of hemicellulose, lignin, which is on the fiber surface. The surface of the fiber becomes coarser and results in better mechanical bonding with the matrix. With the loss of lignin on the surface of the fiber, the reaction and chemical bond between the fiber and the matrix will improve its mechanical properties [7]. The fiber widens because the longer soaking time is shown in Figures 10b to 10d because more and more water molecules are absorbed by the fiber. Loosening of the bonding of fibers with polyester or so-called debonding on the fiber results in mechanical damage or a decrease in the mechanical strength of the composite. In the composite soaking fracture section shown in Figure 10a, it is clear that some points have defects or failures. This is the reason why it can reduce tensile strength. In the test sample with 5% 1 hour, NaOH soaking in Figure 10b shows the type of failure that is debonding and fiber pull-out. But the percentage of failure is smaller than the sample without soaking. Debonding failure is more caused by weak bonds between the fiber and the matrix. Whereas the type of fiber that experienced more failure due to fiber damage caused by not being able to bear the load was accepted. In the test sample with 2 hours soaking of 5% NaOH in Figure 10 (c), there was almost no debonding occurring which resulted in a strong bond occurring between the matrix and the fiber. In contrast to Figure 10d the 3-hour soaking composite sample which has the lowest tensile strength value for the 5% NaOH treatment. That is because fibers are seen to begin to appear again debonding defects and fiber pull-out. The breaking of the bond between the fiber and the matrix is caused by damage from the fiber caused by too long soaking.

It can be seen clearly from SEM testing that the dominant failure known to occur in this composite is debonding. Specifically, for composites with 2 hours alkaline soaking (Figure 10c), it appears that it is clear of debonding compared to other composite specimens. In addition, the density of the interface bond between the fiber and epoxy is good and makes the composite withstand the greater load when it is withdrawn. Due to the high level of interface density between the fibers and epoxy that occurs in composites with 5% NaOH alkali treatment for 2 hours, making the composites in this treatment have the highest tensile strength values in this study. The results of this study prove that natural fiber, especially sugarcane bagasse, can be optimized by the influence of alkali treatment. Further research needs to be done in the form of variations in fiber orientation, matrix selection, merging biaxial and triaxial directions, as well as more advanced methods of hand lay-up such as vacuum bagging process, vacuum infusion
process, and compression molding. With the development of this research, it is convinced that it can produce wind turbine blades that are environmentally friendly, biodegradable, and potential candidate substitute materials.

![SEM Observation](image)

**Figure 10.** Observation of SEM with 300x magnification of composite tensile test specimens on (a) composite without treatment, (b) composite of soaking for 1 h alkaline, (c) composite of soaking for 2 h alkaline, and (d) composite of soaking for 3 h alkaline

4. **Conclusion**

This study can be concluded that from each impact test results and the tensile strength reinforced composite bagasse with an epoxy matrix with various variations, it was found that for the energy absorbed, the impact strength value, tensile strength, and the optimal tensile modulus elasticity value were found in composite specimens with alkali treatment for 2 hours. This value in the average there is absorbed impact energy obtained 12.00 J and the value of the impact strength obtained 0.096 J mm$^{-2}$, the value of the tensile strength obtained 9.46 MPa, and the modulus of elasticity obtained 809 MPa. Reviewed from the results of the fracture pattern of tensile testing which was then carried out structural morphological analysis with SEM testing of each variation of the specimens, it appears that the composite experienced a failure in the form of fiber pull-out and debonding which can reduce mechanical properties, especially in its strength. But in the results of the interface bond density between sugarcane bagasse and the epoxy matrix, the best is in the test specimens with 5% NaOH alkali treatment with soaking for 2 hours. From the results of this study, there is optimism that the bagasse fiber research can be developed to become a future wind turbine blade material.
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