Analysis of the fracture area of tensile test for natural woven fiber composites (hibiscus tiliaceus-polyester)

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Abstract. The natural fiber is currently widely applied in aerospace applications and the transportation sector. The problem that often arises is the area of failure can be hardly predicted. This condition making it difficult to use fiber in a more complex application. The purpose of this study is to investigate the fracture area of woven natural fiber composites. Hibiscus tiliaceus bast fibers, as reinforcement, were prepared with alkali treatment using NaOH solution and then were added by the coupling agent. The composite was formed using a woven fiber arrangement with 60:40 fiber-matrices mass ratio using a vacuum pressure resin infusion method. The tensile test on the specimens was done on the ASTM D638-03 standard and fracture analysis was performed based on tensile tests. The results of the study show that the narrowing of the fracture area in composites was better and more predictable.

1. Introduction
Since early 1960, there has been an increase in demand for stronger, stiffer, and lighter materials to be used in the aerospace, transportation, and construction industries. In recent years, natural fiber seems to be an extraordinary material that has emerged as a substitute for synthetic fibers [1]. Natural fibers such as recycled sisal, banana, hemp, oil palm, kenaf, hemp, and coir have been used as reinforced composites for sophisticated applications such as aircraft and aerospace structures. Besides, natural fibers are also applied in ordinary applications such as consumer goods, furniture, low-cost housing, civil structures [2]. The use of natural fibers is also widely applied in various automotive industries such as bumpers and dashboards.

The availability of cheap lignocellulosic fiber in tropical countries provides a promising future to explore its possible uses for various applications. The use of natural fiber-based composite materials is an effective innovation step compared to synthetic materials [3]. Besides its environmentally friendly nature, a reinforced composite made of natural fiber has various advantages, such as lightweight, low price, abundant amount, easy to obtain, biodegradable, and has high mechanical properties [4].
In tropical countries like Indonesia, opportunities to use natural fibers as composite materials are certainly high. One of the examples is the use of fiber of hibiscus tree bast (Hibiscus tiliaceus). This fiber is widely used as nets, bags, ropes, and various handicrafts in daily life. Fibers from the hibiscus tree (Hibiscus tiliaceus) have strong and tough fibers. Based on the results of tensile testing of a single fiber, hibiscus fiber has a tensile strength of 207.30 MPa. Besides having high strength, this fiber is unique in the form of woven fabrics. So this fiber is very potential as a reinforcing material in composite materials [5].

Because of their plentiful amount, the use of natural fibers in polymer composites is still a quite popular material to be developed. The incorporation of rigid fibers in soft matrices can promisingly lead to new materials with extraordinary mechanical properties which include the advantages of fibers and matrices [6]. However, the use of composites with natural fiber is lacking on the bond between the fiber and the matrices [7], as well as the delamination [8]. These problems make it hard to predict and direct the faults of composites made of natural fibers (are unpredictable/not as desired). β

According to various literature, to improve bond quality, the fiber can be chemically modified using a silane coupling agent as a binding agent between natural fibers and polymer matrices. Silane coupling agents can react with cellulose to reduce hydroxyl groups that are hydrophilic in natural fibers [9]. Silane coupling agents can improve interfaces on the fiber surface, hence fibers and matrices compatibility will be increased [10] [11]. Many techniques have been investigated to prevent delamination from occurring. Woven material has shown potential in preventing delamination [12]. The locking technique is meant to cause friction between bundles of fibers, preventing fiber flakes from passing each other easily because of the locking, for example on a knotted tie on a rope. The high tension held on the fiber during the weaving process affects the overall architectural appearance and mechanical properties. Less stress will result in higher strength in the material but causes greater damage to the area and a reduction in some mechanical properties [12].

This research discusses the fracture area in composites reinforced with hibiscus fiber based on tensile testing. The Hibiscus tiliaceus bast was previously given treatment to form chemical and mechanical binds. Through this step, it is expected the composite to have maximum strength and fracture area as desired. The benefit of this research is to utilize fiber sourced from hibiscus leather (Hibiscus tiliaceus) in the form of natural woven which will potentially substitute synthetic fibers.

2. Method

2.1. Material
The fiber material was obtained from the Hibiscus tiliaceus tree farmer, Tulungagung, Indonesia. The fiber was then extracted from the bast by soaking them in water for 12 days. Before being experimented, the fiber was previously heated at a room temperature of 27°C for 1 week. The polyester resin was obtained from PT. Justus Kimiaraya, Surabaya, Indonesia, as the matrices. The materials had low viscosity with a weight capacity of 1.4 gr/cm³, a hot distortion temperature of 70°C and curing for 24 hours according to the manufacture recommendation.
The fiber used for reinforcement was in filament form, woven in both ways forming a 45/45 degree. This design brought better energy distribution by lessening the crimp, creating an interlaminate mattress. This method also formed an interlocking mechanism between fibers and matrices both in micro and macro.

2.2. **Alkalization**
The alkalization was done by soaking the Hibiscus tiliaceus bast fiber in 6% NaOH at a room temperature of 27 °C for 120 minutes. The soaked fibers were then rinsed using running water until they reached a pH of 7. The wet fibers were dried a room temperature for 24 hours, followed by a heating process in the oven with 40 °C heat for 2 hours.

2.3. **Modification of the Fibers Surface**
Modification of the surface was done by doing Alkyl silane treatment. After NaOH treatment was done, the fibers were soaked in methacryloxtrimetoxysilane solution with 0.75% concentration of distilled water (Aquadest) mass. The solution’s pH was set around 3-4 by adding acetic acid. The Hibiscus tiliaceus bast fibers were soaked for 4 hours. Afterward, the wet fibers were dried at room temperature for 24 hours, followed by the heating process in the oven with 40 °C heat for 2 hours.

2.4. **Composite Forming Process**
Composite was formed by combining the vacuum method and VAPRI (vacuum pressure resin infusion) pressure using a vacuum machine 3 x 10-1 Pa, 0.003 mbar, 15 microns. This method is very effective to diminish the void in the composite specimen after it is dried. The size of the composite mold was based on ASTM D638-03 with fiber and resin weight ratio 60:40.
2.5. **The Observation of Fibers and Composite**

In this research, observation was done to find out the composite's fracture and crack using pictures taken with a digital camera Canon D700 macro lens. The pictures were analyzed using ImageJ software. SEM observation was done to find out the difference of fibers treatment using Scanning Electron Microscope desktop Phenom XL 5kV with 500x magnification.

2.6. **Tensile testing**

Tensile testing of the specimen was done in testing machine Hydraulic Servo Pulser with 0.02 mm/s speed and 10 kN load. Each composite was tested five times to evaluate its mechanical characteristic. The test was stopped when the failure occurred.

3. **Result and discussion**

3.1. **Scanning Electron Microscope Analysis**

SEM test analysis findings are shown in figure 3. NaOH treatment resulted in the changes in the fibers' surface form. *Hibiscus tiliaceus* bast fibers without treatment had a smooth surface with a wax-like coating so the fibers and matrices could not bind well. The interface of fibers with alkali treatment using NaOH 6% has proven to make the fibers cleaner and form a rough fiber texture. Alkali treatment could wash some lignin and cellulose in the *Hibiscus tiliaceus* bast fiber surface. The rough surface produced a tighter mechanical bond between fibers and matrices. With the less lignin and cellulose in the fibers, the chemical reaction between fibers and matrices became stronger, which will consequently improve the mechanical characteristic of the composite. Furthermore, the observation finding using ImageJ software showed the difference between fibers without treatment compared to those with NaOH treatment. Without treatment, the fibers tend to have much lignin covering the fiber’s surface, weakening the interlocking between fibers and matrices. It causes debonding which will reduce the mechanical characteristic of composite material.
Figure 3. Analysis of SEM and ImageJ (a) Untreated (b) NaOH Treated (c) Silane Treated

Fibers with added silane coupling agent show better surface morphology. The surface of *Hibiscus tiliaceus* bast fibers becomes clean, flat, tight, and smooth. With a better interface, compatibility between *Hibiscus tiliaceus* bast fibers and polymer matrices was formed. Matrices and *Hibiscus tiliaceus* bast fibers could bind well so the composites could take the load equally, giving a significant effect on improving the tensile strength of the *Hibiscus tiliaceus* bast fibers composites.

3.2. Fracture Area Analysis

Fracture area analysis on the composites was observed along with the increase of the load given in the tensile test. According to the graphic showing the test and observation of the fracture area, treatment to the fibers affects the mechanical characteristic of the composite.
Figure 4. Fracture Analysis of Composite Untreated Fiber

The analysis of the fracture on the composite using fibers without treatment can be seen in Figure 4. According to the composite tensile test result, fibers without treatment had the lowest strength of 45.897 MPa. Stretches on the composite were very low, only around 0.017. The fracture phase occurred indicating that when given load to the fibers, it was not distributed evenly, so the spreading crack occurred. The fracture area of the composites spread widely because the fibers and the matrices could not bind perfectly. Macro observation on the object showed that there was fiber pull out and debonding.

Figure 5. Fracture Analysis of Composite NaOH Treated

Composite formed by fibers with NaOH treatment can be seen in Figure 5. The mechanical characteristic of the composite was improving by 57,888 MPa followed by the increase of composite stretching level by 0.019. Seen from its phase, the fracture of the composite started by the complete crack on the center of the specimen altogether. NaOH treatment was suspected to create an interlocking mechanism, so the mechanical bonding between fibers and matrices was improving. *Hibiscus tiliaceus* bast fiber with NaOH treatment was proven to increase the strength of the mechanical composite and consequently narrow the fracture area, so it will be easier to predict the composite. The macro picture of the specimen showed a decrease of fiber pull-out and debonding.
Figure 6 describes that the modification of *Hibiscus tiliaceus* bast fiber using a silane coupling agent was proven to improve the mechanical strength and narrow the fracture area well. The mechanical composite strength increased by 65.589 MPa. The phase when composites fracture occurred can be completely seen, in which the composite was stretched, cracked, and then the fracture occurred. Composites with added silane had the most stretching level of 0.024. Adding silane to *Hibiscus tiliaceus* bast fiber was proven to make the fiber surface more hydrophobic so the resin could be absorbed and then formed chemical bonding. Compatibility between fiber and matric created an affinity effect. Matrices spread was even in the composites, so the load from matrices can be distributed completely and tend to be more homogenous. The macro picture from the fracture area proved that the fracture area could be predicted as desired.

4. Conclusion

According to the study findings, it can be concluded that NaOH-Silane treatment on the Hibiscus tiliaceus bast fibers affects the fracture area on the composites. SEM analysis shows the difference in which fiber quality is better, resulting in the improvement of the compatibility between fibers and matrices. Analysis of the macro picture of the composite shows there is a narrowing of fracture area making it easier to predict as desired. The composite tensile strength level of Hibiscus tiliaceus bast fiber with added silane has the highest level of 65.589 Mpa and a stretching level of 0.024.

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