The characterization tensile and thermal properties of hibiscus tiliaceus cellulose fibers

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Abstract. In our study, hibiscus tiliaceus (HT) fibers were treated with potassium hydroxide (KOH) 8% for two hours at ambient temperature. The tensile strength and thermal properties of the treated fibers were examined by using tensile test and Thermogravimetric analysis (TGA). The surface structure of the hibiscus tiliaceus fibers were analysed using Scanning Electronic Microscopy (SEM). Experimental results revealed that under KOH treatment, the tensile strength increased up to 5144.9 MPa, resistance thermal and cellulose of the fibers become increased; due to hemicellulose content were reduced of the fibers. SEM images show that after alkali, the hibiscus tiliaceus fibers surface becomes clear, fibril and rougher.

1. Introduction

One source of fiber that is cheap, eco–friendly, abundant, and not harmful to human health is fiber from plants such as corn husks, Jute, Pandanus Amarylliosus, and Kapok [1,2]. Natural fibers have been used as a filler for composite resin [3–6], and they have been applied in the field of building construction, and automotive [4].

However, the use of natural fiber as filler in polymer materials is less preferred due to a number of hydroxyl groups and impurities attached to the surface of cellulose fibers, which causes them to be incompatible with resin [7,2]. Surface modification of natural fibers using chemical and physical treatments is a method to produce surface roughness and the reactive hydroxyl groups to form bonds with polymeric materials [1,7,8].

Many researchers investigated the effect of alkali treatment using sodium hydroxide (NaOH) on the surface of natural cellulose fibers. Most of the reported works focused on the mechanical properties of the fibers and its composites [1,9]. After NaOH treatment, the elongation, Young’ moduli and tensile strength of natural fiber become increased [10,11,1]. On the contrary, investigation related to the effect of potassium hydroxide (KOH) on natural fiber properties still limited. Abdul Khalil et al. [12] analyzed the effect of activated carbon from oil palm empty fruit bunch, bamboo stem and coconut shell treated by potassium hydroxide and phosphoric acid on the flexural properties of epoxy–nanocomposites. They reported that potassium hydroxide (KOH) activated carbon-based epoxy nanocomposites had a higher flexural strength value compared to phosphoric–acid activated carbon. Furthermore, Pan et al. [13] reported the effect of Ca(OH)₂, KOH and NaOH as alkalinity on deacetylation of a konjac–glucomannan. These previous studies have shown that the influences of KOH treatment on the properties of the natural fibers still had not investigated.

Therefore, this paper has been done to provide comprehensive data and discussion on the thermal, and tensile properties of hibiscus tiliaceus fibers treated with potassium hydroxide (KOH). SEM, TGA and tensile strength analysis of the fibers were performed to expand an understanding of the single fiber constituent behaviors. These constituent behaviors were used to systematically analyze treatment effects on fiber tensile strength and thermal properties. The study suggests that KOH treated hibiscus tiliaceus (HT) fibers can be as an alternative reinforce in polymer composites fabrication.
2. Material and Methods

2.1 Raw materials
Hibiscus tiliaeus (HT) skins were obtained from around West Lombok, West Nusa Tenggara, Indonesia. For alkalization, a commercial grade of Potassium hydroxide (KOH) with concentrate 8% was used.

2.2 Extraction of fibers
HT skins were soaked in water for one week to undergo microbacterial. Then, they were combed with a wood brush to retained a bundle of fibers and dried through wind convection.

2.3 KOH treatment of fibers
In this process, HT fibers were immersed in 8% KOH solutions at ambient temperature (32°C) for 2 hours. After KOH treatment, the fibers were thoroughly washed six times with distilled water in order to remove the KOH adhering to the surface of the HT fibers and dried in an oven maintained at 70°C.

The physical properties of fiber as shown in Table 1.

| Fiber types   | Diameter (cm) | Density gr/cm³ | Moisture content (%) |
|---------------|---------------|----------------|----------------------|
| Untreated     | 0.41 ± 0.02   | 0.76 ± 0.01    | 11.32 ± 0.01         |
| Treated KOH   | 0.27 ± 0.02   | 0.68 ± 0.01    | 7.49 ± 0.01          |

2.4 Characterization
Tensile strength, the morphology and thermogravimetric (TGA) of the surface of untreated and KOH treated HT fibers were characterized.

2.4.1. Tensile strength
Individually fibers (untreated and treated fibers) prepared. All the samples were stored in a chamber at 21°C and 63% relative humidity for 24 h for proper conditioning as determined in ASTM D 1776–04 [14] before testing. The maximum load of 20 N was applied and a crosshead speed of 5 mm/min with a gage length of samples of 25 mm. In each treatment, there are five fibers samples were tested.

2.4.2 Morphology of fiber Surface
The surfaces of KOH treated and untreated HT fibers were evaluated using scanning electron microscopy, SEM (FEI Inspect S50) and has been compared with untreated fibers. The samples were coated with gold using a plasma sputtering–apparatus and accelerating voltage 10 kV.

3. Results and discussion

3.1 Fiber diameter
The diameter of HT fibers was decreased by 34 ± 0.2% upon 8% KOH treatment at 32°C (see in Table 1). After KOH treatment, the diameter reduction was caused by the deletion of lignin and hemicellulose in the fiber. These results indicate that 8% KOH of the alkaline treatment can eliminate noncellulosic materials (hemicellulose and lignin) more effective than using 8% NaOH as reported by Sari et al.[1]. They reported a 22% reduction of the cornhusk fibers diameter upon treatment with 8% NaOH at 31°C for 2 h.

3.2 Chemical analysis
The content of fiber constituents of treated fibers is presented in Table 2. From Table 2 it can be noted that after 8% KOH treatment, the hemicellulose, and lignin in the fiber were decreased compared to
the untreated HT fibers (0% KOH); consequently, the cellulose content was increased because KOH treatment has no effect on cellulose molecules. These results indicated that the treatment of KOH dissolved most of the hemicellulose and lignin from the fiber.

Table 2. The chemical content of untreated and treated hibiscus tiliaceus fibers.

| Fiber types   | Cellulose (%) | Hemicellulose (%) | Lignin (%) |
|---------------|---------------|-------------------|------------|
| Untreated     | 64.0          | 27.1              | 17.3       |
| Treated KOH   | 71.4          | 20.2              | 9.8        |

3.3. Tensile strength
Under 8% KOH treatment at 32°C, the tensile strength of HT fibers was observed to increase by 700% (5144.9 MPa) compared to untreated HT fibers (650 ± 110 MPa) (Figures 1 and 2) and the elongation was noted to increase by 61.3% (Figures 3a and 3b). The increase in strength was related to the decrease in diameter and roughness surface on HT fibers; due to lignin, or hemicellulose from the more effective interfibrillar region removed.
This result is significantly higher than the value obtained by NaOH treated cornhusk fibers developed by Sari et al. [1], which showed that the tensile strength of the 8% NaOH treated CHFs was 368.25 ± 78.97 MPa. It proves that the KOH–treatment on fiber surface can be a good alternative to the filler in the composite material.
3.4. Surface morphology

The SEM photo exhibited that after 8% KOH treatment, the surface structure of the HT fiber become rougher and fibril. It is attributed to the removal of noncellulosic (hemicellulose and lignin) substances and impurities on the HT fibers (Figures 4a and 4b). These investigations indicated that the strength improvement of the HT fibers related to defibrillation. When the fibers were used as a filler in composite resins, the surface of chemically treated fiber allowed them to form chemical bonds between the fibers/matrix [2].

3.5 Thermogravimetric Analysis

From Figure 5 it can be observed that at about 220 °C weight loss was very small. The large weight loss occurred at temperature ranges 280–370 °C. Kabir et al. (2013) stated that the cellulose was decomposed (250–350 °C) caused molecular structures of cellulose broken down. Then, Placet
(2009)[16] stated that cellulose has a crystalline structure containing strong intermolecular hydrogen bonding and intramolecular, so it takes a lot of energy to break the molecular structure of cellulose [16–18].

The decomposition of hemicellulose presented three steps of decomposition. The first step (25–180 °C) exhibited the loss of moisture from hemicellulose. In the second step (180–280 °C), the majority of the hemicellulose constituents decomposed with fast weight losses (water evaporated). Then, at the last step (280–600 °C), they decomposed leaving a residue of charcoal.

Thermal decomposition of lignin constituents occurred at 150–550 °C. At the heating temperature, lignin formed aromatic hydrocarbons hydroxyphenolics, and syringyl [19]. At higher temperatures, all components of lignin which contain –OH groups became unstable; this is because they experience a radical reaction between themselves which results in the structural rearrangement. The existence of these molecular activities causes degradation of lignin at prolonged temperatures and a smaller weight loss.

Figure 5 also shows alkaliize fiber samples. Under alkali treatment, the decomposition temperature of the HT fiber is higher than untreated fibers. In the range of 250–350°C, weight reduction of KOH treated HT fibers is smaller than untreated fibers; due to the reduction of hemicellulose and lignin constituents. Consequently, the decomposition process occurs in cellulose, so that the degradation temperature of the KOH treated HT fiber increases.

4. Conclusions

On KOH treatment, the tensile strength of the HT fiber increased by up to 700%. The increase of elongation of 4.75 mm exhibited that the KOH treated fibers maintained their flexibility. Tensile and thermal characterization of HT fibers noted that the increase in strength and thermal resistance were due to the removal of hemicelluloses. This treatment has a potential and viable structural application.

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