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Characterization of Tensile Properties of Alkali-treated Kenaf/Polypropylene Composites

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Abstract. The composite of alkali-treated kenaf/polypropylene (PP) with 6 mm fiber length and 16 wt. % fiber content was fabricated by hand lay-up technique using a compression molding. Kenaf fiber were alkali-treated with 6% NaOH at room temperature for different durations of 0, 4, 10, 24, and 36 h to verify the effect of alkali treatment duration on tensile properties of the composites. Hemicellulose contained in kenaf fiber was wholly removed due to alkali treatment, but lignin was gradually reduced as an increase of alkali treatment duration, leading to increasing the composite tensile strength and modulus. The tensile fracture surface morphologies showed the existence of brittle fibers, fiber pullout and voids in the composites without and with alkali treatment for 4 h. Alkali-treatment duration longer than 4 h made the fiber brittleness disappeared and tended to be ductile with long fiber structure which showed relatively strong interface bonding, especially for the period of 36 h. The results clarified that alkali-treatment at 6% NaOH for 36 h would be an advantage for improving the tensile properties of the natural fiber composites which were also supported by an excellent fiber dispersion in all the composites.

INTRODUCTION

Natural fibers reinforced polymer composites are used for interior and exterior components of the vehicle as an alternative to replace glass fibers as a light-weighting solution because natural fibers come from nature, renewable resources and thus have a lower environmental effect in comparison with glass fiber. The use of natural fiber composites in automotive has some benefits such as reducing fuel consumption due to its lightweight, leading to weight reduction 10-30% and also energy saving, no emission of toxic compounds, favorable eco-balance, satisfactory mechanical properties and good acoustic properties [1]. Natural fibers used in the automotive industry are predominantly jute, kenaf, hemp, and flax. They are mostly combined with polypropylene (PP) or polyethylene (PE) by compression and injection molding. Toyota Boshoku Corporation, Japan has firstly utilized kenaf as a raw material for the interior automotive components [2]. Besides, Germany is a leader in the use of natural fiber composites. The German auto-manufacturers, BMW, Audi, and Volkswagen have taken the initiative to introduce natural fiber composites for interior and exterior applications [3].

However, the low impact resistance of natural fibers and the moisture degradation have been the problems in the composite fabrication [4] due to an inherent characteristic of hydrophilic fiber. Fiber selection including fiber treatment and content, matrix selection, interfacial strength, fiber dispersion, fiber orientation, composite manufacturing process, and porosity are the main factors influencing the mechanical properties of natural fiber

reinforced composites [5]. Thus, efforts to improve the mechanical properties of natural fiber reinforced composites have been extensively carried out.

Studies of the natural fiber composites have discussed how to improve the interface bonding between the fiber surface and the matrix because it plays a crucial factor in determining the mechanical properties of the composite. The physical or chemical method has usually carried out to overcome the problem. An example of a physical method has been the heat treatment of kenaf fiber [6] which can improve the tensile strength of fiber due to enhancing the degree of crystallinity of fiber.

On the other hand, alkali treatment may be the most straightforward chemical method and gives a better effect on the fiber mechanical strength than by physical method. Alkali treatment at room temperature can completely remove hemicellulose, but not for lignin [7]. Therefore, optimization of chemical treatment on the natural fibers to improve the fiber mechanical properties and its related composite has been still investigated, because the aspect of fiber origin including its harvest time and cultivation area will affect the treatment condition of the fiber surface.

Chemical treatment of kenaf fiber with NaOH in the concentration of 3, 6, 9 and 12% and soaking time for 24 h has indicated that 6% has been an optimum concentration for alkali treatment. By which has been resulted in clear fiber surface and higher surface roughness than with NaOH in the concentration less than 6%. An increase of alkali concentration reduces the fiber tensile strength [8] Kenaf fiber treatments with NaOH in the concentration of 6 and 9% at room temperature and 100°C for an hour have been reported and indicated that hemicellulose was entirely removed at all conditions [9], but residual lignin was identified. The highest tensile strength (612 MPa) has been achieved by soaking the fibers in the 6% NaOH at room temperature for 1 h. However, the composites alkalinized with NaOH in the concentration of 6% and 9% at 100°C resulted in fiber surface damage which drastically decreased tensile strength to be ~200 MPa and 98 MPa for 6% and 9% NaOH concentrations, respectively. Besides, alkalization of kenaf fiber with 6% NaOH for 24 h and a fiber loading with various concentrations from 10% to 50% has shown that the highest tensile strength and modulus of kenaf/PP composite at 40% fiber loading were around 25 MPa and 2450 MPa, respectively, [10]. In addition, the characterization of tensile properties of kenaf/PLA affected by unbleached and bleached fibers using H₂O₂ [11] has shown that the highest tensile strength and modulus obtained at 30% fiber content: i.e. 45 MPa and 1400 MPa, respectively. Use of 6% alkali concentration seemed to be an optimum value for surface modification of kenaf fiber, but the period of fiber treatment is undefined because non-cellulosic components contained in each fiber origin is not similar. Therefore, optimization of alkali treatment duration is necessary.

The present work has investigated the influence of alkali treatment duration from 4 h to 36 h on the tensile properties of kenaf/PP composites with 20% fiber content and discussed from the fiber properties, the morphology of fracture surface and fiber dispersion. Our earlier study had verified that the kenaf/E-glass/PP hybrid composites with different fiber content from 20% to 30% resulted in the composite tensile properties with 20% fiber content was slightly higher than with 30% fiber content [7].

EXPERIMENTAL METHODS

Materials and Fiber Treatments

Kenaf fiber and PP sheet were purchased from Balittas-Malang, Indonesia and PT. Sumber Jaya, Indonesia, respectively. Kenaf fibers were washed with flushing water to remove the contaminants present on the fiber surface and dried with an electric fan for 2 days. The fibers were then soaked in the NaOH solution at 6% concentration at room temperature (RT) for various durations 4 h, 10 h, 24 h, and 36 h to eliminate the non-cellulosic components such as hemicelluloses and lignin to make a better strength of the fiber surface and the matrix interface. Subsequently, the fibers were washed with flushing water and dried in an oven at 100°C for 30 min. Alkali-treated kenaf fibers were then chopped into 6 mm length, and PP sheets were cut into 17 mm length and 2.2 mm width.

Composite Fabrication and Tensile Test

The kenaf/PP composites were fabricated using hand lay-up technique in a hot compression molding, as described elsewhere [7]. Kenaf fiber and PP sheets were arranged manually into 13 laminates with 20% fiber loading in a Fe-mold with the dimension of 17 cm length, 2.2 cm width, and 2 cm depth and hot pressed at around 175°C and 3.5 MPa for about 10 min.

The tensile test of composite specimens was prepared using the computer numerical control (CNC) machine in accordance to ASTM D638-02. At least 6 composite specimens were used for each variation of alkali treatment duration. The tensile tests were conducted at a maximum load cell of 2 kN, a crosshead speed of 500 mm/min and a gauge length of 50 mm.

Characterization

The chemical structure of kenaf fibers before and after alkali treatments was qualitatively examined by Fourier transform infrared (FTIR) spectroscopy (Shimadzu). Scanning electron microscopy (SEM, TESCAN VEGA3 LMU) was utilized to characterize the morphology of the tensile fracture surface of the composite specimens. Before SEM examination the specimen surface was metallic coated with Au-Pd. Optical microscopy for characterization of fiber dispersion in the matrix was obtained from the cross-section surface on a small piece near the fracture area (see Fig. 1, see arrow).

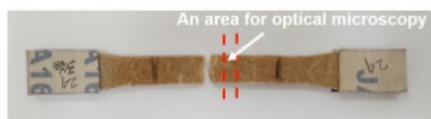


FIGURE 1. The tensile tested specimen is showing an area for optical microscopy.

4 RESULTS AND DISCUSSION

FTIR of Kenaf Fibers

FTIR spectra of the untreated and alkali-treated kenaf fibers for different alkali treatment durations (Fig. 2) illustrate a different chemical structure of the fiber qualitatively. The broad peaks identified at 3400 cm^{-1} are attributed to the presence of free OH groups in the cellulose molecules [10]. The peaks positioned between 2800 cm^{-1} and 2950 cm^{-1} correspond to C-H stretching vibration. The absorbed band at 1734 cm^{-1} observed in the spectrum of untreated kenaf is related to the existence of a carboxylic group ($=\text{C}=\text{O}$) which is associated with hemicelluloses

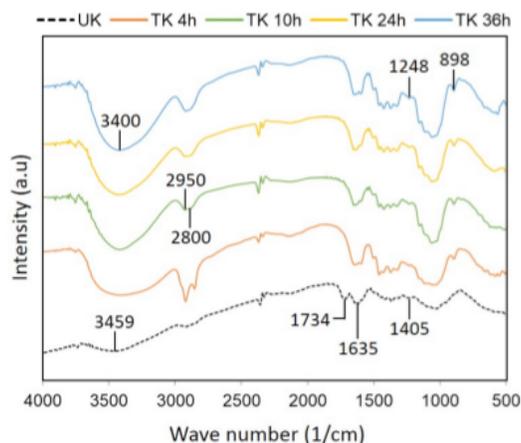


FIGURE 2. FTIR spectra of untreated kenaf (UK) and alkali treated kenaf (TK) fibers.

[12, 13]. However, this peak completely disappeared in all alkali-treated kenaf fibers due to hydrolysis of hemicellulose in alkali medium. This result confirmed that the disappearance of hemicelluloses occurred after alkali treatment either for short or relatively long duration. The peak at 1635 cm^{-1} showed water absorption in all

conditions. The peaks at the wave number from 1248 cm^{-1} to 1405 cm^{-1} showed the characteristic of $-\text{O}-\text{CH}_3$ and $\text{C}=\text{C}$ bond of lignin, respectively.[8] which are identified in the untreated and also alkali-treated kenaf fibers. However, it was considered that lignin was gradually removed by increasing alkali treatment duration. The peak at the wave number of 898 cm^{-1} identified the characteristic of the absorption of the CH_2 group present in cellulose and β -glycosidic linkages [14].

Tensile Properties of Kenaf/PP Komposites

Tensile strength and modulus of kenaf/PP composites increase with an increase of the duration of alkali treatment (Fig. 3). These results are significantly higher compared to the results reported by Akhtar *et al.* [10] who have investigated tensile properties of kenaf/PP composites affected by varying fiber content from 10% to 50% using untreated and alkali-treated kenaf in 6% NaOH for 24 h. However, unlike this study, the composites were fabricated by an injection molding. With similar fiber content of 20%, tensile strength and modulus of the composite resulted in this study are 44 MPa and 1.8 GPa, respectively, (Fig 3a) which are much higher than those reported by Akhtar *et al.* [10]: i.e. 22.8 MPa and 1.7 GPa, respectively. These results indicated that the natural composite fabrication using a compression molding technique is also recommended, but the shortcoming manual manufacturing might be the formation of voids because the chance of air inclusion formed inside the composite is high.

Related to FTIR analysis results Akhtar *et al.* [10] indicated that lignin and hemicellulose were removed after alkali treatment for 24 h. Removal of lignin and hemicellulose resulted in a good chance for fibers to be separated with each other which can also be said that the degree of fibrillation becomes higher, leading to increasing the number of interfacial bonding sites. In the natural fiber system, lignin works as a connecting component between the fibers. So that, if the presence of lignin is gradually removed, the degree of fibrillation also gradually increases. The longer the alkali treatment duration, the amount of lignin will remove progressively. Therefore, it can be explained that a gradual increase of tensile strength and modulus are related to the gradual elimination of residual lignin in the fiber as confirmed by FTIR analysis result (Fig. 2). In addition, an increase of elongation of the composite due to the longer of alkali treatment duration (Fig. 3b) is discussed corresponding to the following results.

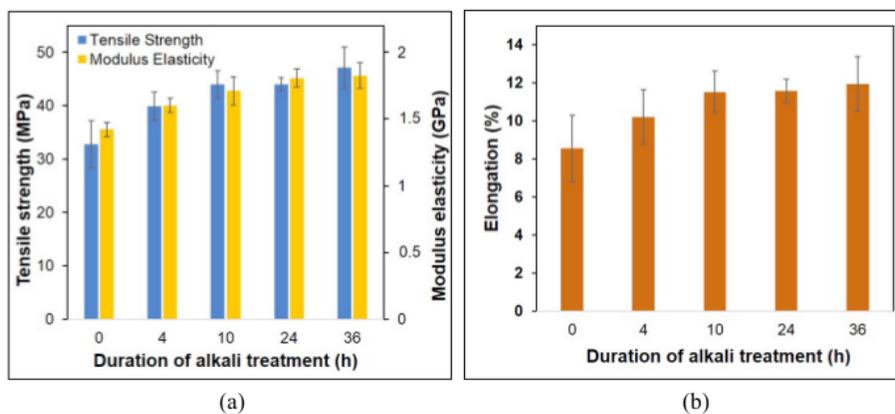


FIGURE 3. Tensile strength and modulus (a) and elongation (b) versus duration of alkali treatment.

Tensile Fracture Surface and Fiber Dispersion

SEM images of the fracture surfaces (Fig. 4) show the gradual alteration of surface morphology with alkali treatment durations. Composite with the untreated fiber (Fig. 4a) revealed that the fibers tend to be brittle. Many fibers were broken (see white arrows), and some fiber pullout (see, yellow arrows) were also formed during the tensile test, explaining that the fiber and the matrix interface bonding is weak.

At the alkali treatment duration of 4 h (Fig. 4b), the number of broken fibers and fiber pullout decrease, but, debonding (see blue arrows) was observed. The fiber brittleness was not found in the specimen with the alkali

treatment duration longer than 4 h, but some fiber pullout exists. The presence of some voids (see red arrows) can be understood due to the manual fabrication carried out in this work.

Surface morphologies of the composite specimens with alkali treatment duration longer than 4 h (Fig. 4c, Fig. 4d and Fig. 4e) shows different from those in Fig. 4a and Fig.4b due to gradually increase of elongation or tensile strain of the composites as depicted in Fig. 3. The results suggest that the fibers would be more ductile after alkali treatment duration longer than 4 h. The elongation of the specimen with alkali treatment durations for 10 h, 24 h, and 36 h, however, was almost similar. These are reflected in the surface morphology with long-fiber structure, indicating an excellent interface bonding between the fiber and the matrix. The interfacial bonding between the fibers and the matrix of those specimens seemed to be relatively good. SEM examination on the other side of the fracture surface of a composite specimen with the duration of 36 h has also been performed to verify the surface morphology. The result indicated that both sides showed a similar morphology with long-fiber structure, confirming an excellent bonding strength between the fibers and the matrix.

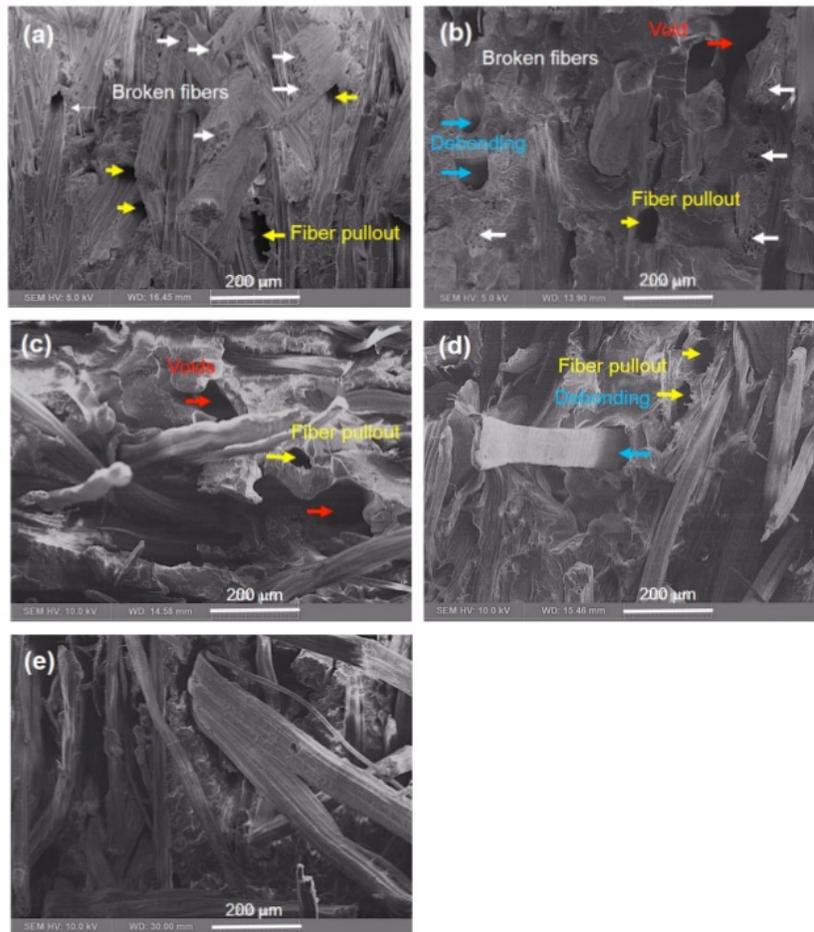


FIGURE 4. SEM images of tensile fracture surface obtained from the composite specimens with different alkali treatment durations of kenaf fibers. (a) Untreated, (b) 4 h, (c) 10 h, (d) 24 h, and (e) 36 h.

Besides, fiber dispersion characterized by a digital optical microscope revealed relatively good fiber dispersion in all composite specimens as represented in Fig. 5. These optical microscope images demonstrated kenaf fibers as

the particle-like structure which homogeneously distributed within the matrix, resulting in relatively high tensile properties.

The overall microstructural characteristics obtained from the composite specimens in this work showed relatively good results, because the tensile properties of this composites are comparable with those resulted by Abdul Razak et al. [11] and higher than those reported by Akhtar et al. [9] although they used an injection molding for fabricating the composites.

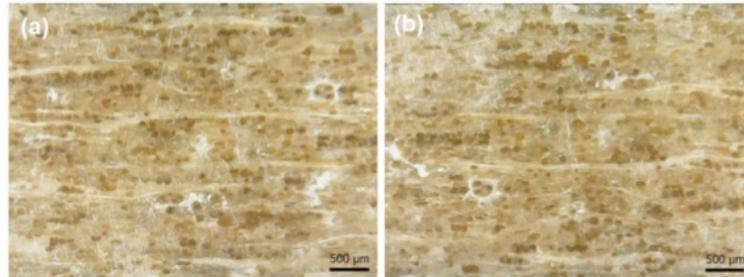


FIGURE 5. Optical micrographs of kenaf fibers dispersion in the PP matrix obtained from the specimen with untreated (a) and treated fibers for 4 h (b).

CONCLUSIONS

The kenaf/PP composites using different alkali treatment durations of fiber from 4 h to 36 h have successfully manufactured with relatively high tensile properties. The composite with the duration of fiber alkali treatment for 36 h showed the highest values: i.e., 48 MPa and 1.8 GPa for tensile strength and tensile modulus, respectively.

An increase of the tensile properties has been affected by a gradual removed non-cellulosic component of lignin due to increasing alkali treatment duration which leads to enhancing the fiber/matrix interface bonding sites. Homogeneous fiber dispersion in all composite specimens also supported in improving the tensile properties.

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