EFFECT OF ALKALI TREATMENT AND MAPP ADDITION ON TENSILE STRENGTH OF SISAL/POLYPROPYLENE COMPOSITES

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ABSTRACT: Mechanical properties of composite are strongly influenced by surface treatment of fibers and fiber content. The study aimed to verify the influence of a modified fiber surface using an alkali solution and, the addition of maleic anhydride grafted polypropylene (MAPP) and both (alkali solution + MAPP) on increasing the tensile strength of sisal/polypropylene (PP) composites. The composites were fabricated using hot compression molding with a fiber content of 30 and 50 wt%. Untreated and alkali-treated sisal fibers with a fiber length of ~ 2 mm were used. Various amounts (5, 10 and 15 wt%) of MAPP were added to the untreated sisal/PP and alkali-treated sisal/PP composites. The results indicated that the tensile strength of the untreated sisal/PP composite containing 50 wt% of fiber was higher than that of 30 wt% fiber content. The addition of 5 wt% MAPP into both untreated and alkali-treated sisal/PP composites significantly increased tensile strength compared to that of 10 and 15 wt% MAPP addition. Good interfacial bonding between sisal fiber and the PP matrix was observed at 5 wt% MAPP loading. Excessive MAPP content could form a barrier that tends to inhibit the stress transferred across the matrix-fiber interfaces.

KEYWORDS: *Sisal Fiber; Polypropylene; Alkalization; MAPP; Composite*

1.0   INTRODUCTION

Natural fibers are biodegradable, lightweight, renewable, abundant, inexpensive and have good mechanical properties and therefore have

good potential for composite reinforcement. Natural fiber-reinforced composites cause less environmental impact than composites reinforced with synthetic fibers. Based on fiber structure, each fiber type shows different physical and mechanical behaviours [1] for example the tensile strength of the sisal fiber (*Agave sisalana*) (637 MPa) [2] is reported to be higher than that of coir (90 MPa), jute (249 MPa) [1] and kenaf (502 MPa) [3]. Sisal fiber reinforced-polypropylene (PP) composites have potential applications in railways and automotive parts as well as construction and building materials [4].

Most polymer matrix especially thermoplastics (polypropylene/PP, polyethylene/PE, polyvinyl chloride/PVC, etc.) are hydrophobic (non-polar). Conversely, natural fibers are hydrophilic (polar). Natural fiber has a hygroscopic nature, leading to poor wettability and inadequate bonding strength between the fiber and the matrix which results in the formation of debonding and pull-out in fibrous composites. This inappropriate behavior leads to a reduction in the mechanical properties of the composites [5-6]. To overcome these problems, a coupling agent is often used to enhance the mechanical properties of the composite. Coupling agents generally consist of bonding agents and surface active agents (surfactants) that include compatibilizers and dispersing agents [7].

Tests with various fiber treatments such as sodium hydroxide, isocyanates, maleic anhydride grafted polypropylene (MAPP), benzyl chloride and permanganate have indicated that MAPP is the best coupling agent for enhancing mechanical properties [8]. The addition of MA (maleic anhydride) or MAPP into several composites of sisal/PP [7, 9], sisal/PP+PLA [10], sisal/rHDPE [11] to improve mechanical properties has been reported. The mechanical properties of polymer composite seem to not only depend on the addition of MAPP but also on the fiber content. A small amount of MAPP (0.55 wt%) added into sisal/PP composites with a fiber content of 10-30 wt% showed a slight increase of tensile and impact strengths [7]. Significant improvement of the mechanical properties was attained by adding 1% MAPP into sisal/PP composites with a fiber content of 21 wt% [12]. Our prior work [9] has revealed that by 3 wt% of MAPP loading, the flexural strength of untreated sisal/PP composite with the fiber content of 50 wt% increased by 19%; from 23.75 MPa to 29.38 MPa. Several studies have also shown that mechanical properties in the range 20 - 50 MPa have mostly been achieved by a natural fiber composite with the fiber content ranging from 15 to 30 wt%, with and without MAPP [7, 10, 11, 13]. Our earlier results, however, indicated that treated kenaf/PP composites with a fiber content of 40 wt% [14] resulted in 20 MPa flexural strength; 50

wt% fiber loading and the addition of 5 wt% MAPP [3, 15], increased the tensile strength to 40 MPa. Zampaloni et al. [16] also showed that 30 wt% (kenaf) and 40 wt% (sisal) could have relatively high tensile strength, 47 MPa and 35 MPa, respectively. The mechanical properties of sisal fiber reinforced composites are higher compared to other types of natural fiber composites. Sisal fiber surfaces are relatively rougher than those of other fibers resulting in good interfacial bonding properties between sisal fiber and the polymer matrix [17].

Many studies indicated that treatment on the fiber surface and the fiber content strongly affect the mechanical properties of natural fiber composites. A defined correlation effect between these properties, however, has not been adequately demonstrated.

In this study, short sisal fiber reinforced PP composite laminates were fabricated with fiber contents of 30 wt% and 50 wt%. A composite with an untreated sisal fiber loading of 30 wt% was prepared and compared with a 50 wt% loading. The untreated and alkali treated sisal/PP composites with 50% fiber content were fabricated with the addition of three MAPP concentrations (5, 10 and 15 wt%). The effects of hot alkali-treatment, MAPP addition and both (alkali-treatment + MAPP addition) on enhancing the tensile strength of the sisal/PP composites were studied. The tensile strength of all produced composites was compared to that found in previous studies [7, 8, 25].

2.0   METHODOLOGY

2.1   Fibers and Polymers

Sisal fibers purchased from Balittas Malang, Indonesia, PP sheets with a density of 0.92 g/cm³ and MAPP (Mw ~9,100) supplied by Sigma Aldrich were used as reinforcement, polymer matrix, and a coupling agent, respectively. Untreated sisal fibers were washed with water and dried in an oven at 70°C for 30 min. Other sisal fibers were chemically treated in 6% NaOH at 100 °C for an hour and neutralized in 1% CH_3COOH to remove a part of non-cellulosic components and other contaminants present at the fiber surface. The fibers were then washed in flushing water and dried under the same condition as the untreated fibers. Untreated and treated sisal fibers were chopped into ~2 mm length (Figure 1(a)). The alkali-treated sisal fibers (Figure 1(b)) appeared to agglomerate unliked the untreated sisal fibers (Figure 1(a)). The PP sheet was cut into 17 mm length and 2 mm widths as shown in Figure 1(c).

Figure 1: Photographs of short sisal fibers (a) before, (b) after alkalization and (c) PP sheets used at the polymer matrix

2.2 Fabrication of Sisal/PP Composites

Short sisal fibers and blended MAPP were manually mixed and fabricated as components of the sisal/PP composites using a hand lay-up technique in a hot compression molding as described elsewhere [9]. Untreated sisal fibers reinforced composites were prepared with fiber loadings of 30 wt% and 50 wt%, and designated as (US-30-0) and (US-50-0), respectively. Various amounts of MAPP (5, 10 and 15 wt%) were added to US-50-0 and designated as US-50-5, US-50-10 and US-50-15, respectively. Alkali-treated sisal fibers reinforced composites with a fiber content of 50 wt% were prepared without and with 5 wt% MAPP loading and designated as TS-50-0 and TS-50-5, respectively. Specimen codes corresponding to the fiber treatment condition are summarised in Table 1.

Table 1: Sample codes related to fiber content

Sample codes	Untreated	Alkali-treated (6% NaOH, 100C, 1h)	Fiber content (Wt %)	MAPP (Wt %)
US-30-0	√	–	30	–
US-50-0	√	–	50	–
US-50-5	√	–	50	5
US-50-10	√	–	50	10
US-50-15	√	–	50	15
TS-50-0	–	√	50	–
US-50-5	–	√	50	5

US: untreated sisal, TS: treated sisal.

2.3 Tensile Test and Characterization of the Sisal Fibers

Tensile specimens were tested following the ASTM D638-02 standard using a universal testing machine (Torsee AMU-5-DE) at a maximum load cell capacity of 2 kN, a crosshead speed of 2 mm/min and a gauge length of 50 mm. Characterisations of chemical composition in the

untreated and alkali-treated sisal fibers were carried out using Fourier transform infrared (FTIR, Shimadzu) spectroscopy in the wave number range of $4000 - 500\,cm^{-1}$. The morphology of untreated and alkali-treated sisal fiber surfaces and the tensile fracture surface of the composites were examined by scanning electron microscopy (SEM) operating at 10 kV. Before SEM examination, the specimen surfaces were metallic coated with platinum (Pt) to increase their electrical conductivity.

3.0 RESULTS AND DISCUSSION

3.1 Morphology of Sisal Fiber Surface

Natural fiber consists mainly of cellulose and non-cellulosic components of hemicellulose and lignin in which they form a natural composite. The hemicellulose interacts with the cellulose forming network with microfibrils [19]. Lignin is a connecting cement between the microfibrils that can highly cross-link to each other (Figure 2).

In the untreated fibers, three fiber bundles in which each bundle consists of sub-micron scaled fibers strongly bonded to each other due to the presence of lignin (Figure 3(a)). The fiber bundle surface was generally covered by waxes, pectin and some inorganic contaminants as non-cellulosic constituents. The untreated fibers used in this study may be acceptable as fibers only when washed in flushing water and dried. Thus, some pollutants remained visible on the fiber surface (Figure 3(a)).
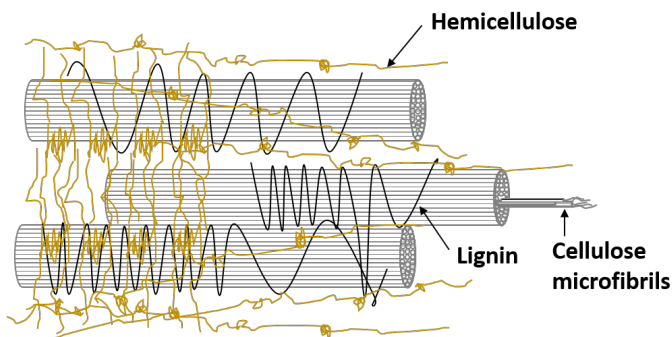

Figure 2: Illustration of the relationship between cellulose, hemicellulose and lignin

Those contaminants were removed using a hot alkali treatment (6% NaOH, 100°C, 1h) resulting in fibrillation of part of fiber bundles into individual fibers (Figure 3(b)). The dissolution of some lignin in hot

alkali (100˚C for ≥ 1 h) increased the volume fraction of the hydroxyl groups on the fiber surface. As a result, the fibers tended to be more hydrophilic and agglomerate with each other (Figure 1(b)), confirming that alkalization at high temperatures led to higher water absorption of the fibers.
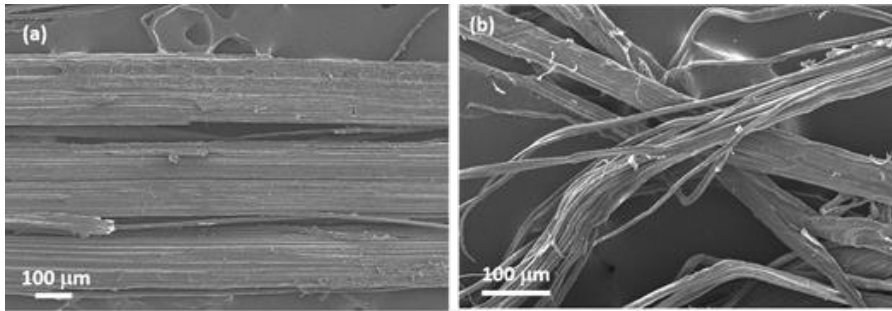

Figure 3: SEM micrographs of sisal fibers morphology: (a) untreated sisal and (b) alkali-treated (100 ˚C, 1 h) sisal

3.2   FTIR of the Sisal Fibers

FTIR spectra of untreated and alkali-treated sisal fibers (Figure 4) showed a slight discrepancy. The broad peak at 3440 cm^{-1} was characteristic of –OH stretching vibration of the hydroxyl band, whereas the peak at 2900 cm^{-1} corresponded to C-H stretching vibration. A strong peak at 1734 cm^{-1}1 was observed in untreated sisal fiber, but not in alkali-treated sisal fiber. The absorbance peak at 1734 cm^{-1} was related to the carboxylic group (=C=O) corresponding to hemicellulose [20-21], indicating that hemicellulose was removed after hot alkalization. OH bending of absorbed water was identified as relatively strong peaks in both spectra at around 1637 cm^{-1} [22]. The band around 1248 cm^{-1} is attributed to the C-O ring of both lignin and pectin [23]. The existence of this peak was not found after alkalization, verifying that lignin is dissolved in a hot alkali. The peak near 1420 cm^{-1} and 898 cm^{-1} are characteristic of the absorption of the CH$_2$ group that presents in cellulose and of β–glycosidic linkages [24-25].
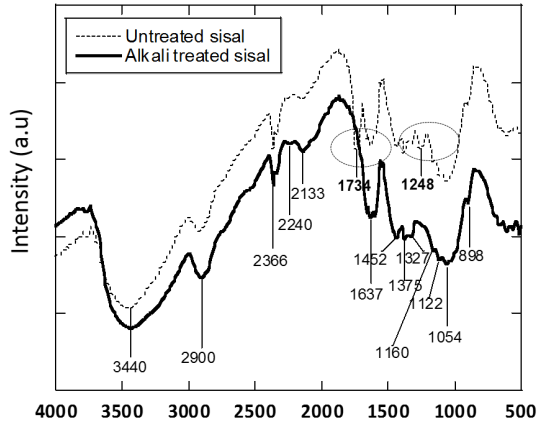
Figure 4: FTIR spectra of untreated and alkali treated sisal fibers

3.3 Tensile Strength and Fracture Surface of the Sisal/PP Composites

The tensile strength of the untreated sisal/PP composite with the fiber content of 30 wt% (US-30-0, 30 MPa) was lower than that of 50 wt% (US-50-0, 34 MPa) (Figure 5(a)). This result is in support with the result reported by Kaewkuk et al. [7]: for example, a sisal/PP composite with similar fiber length and fiber content to those of this study, added with 0.55 wt% of MAPP and fabricated with an injection molding machine showed a tensile strength around 28 MPa. The composite manufactured with an injection molding [7] often produced more uniform fiber distribution within the matrix in comparison to that with a hot compression molding (this work). The addition of MAPP by less than 1 wt% might have an insignificant effect in enhancing the tensile strength of the composite. The result indicated that the hot press machine used in the current work had a relatively good performance. Besides, the tensile strength of the alkali treated sisal/PP composite with the fiber content of 50 wt% (TS-50-0) was lower than that of untreated sisal/PP composite (US-50-0) (Figure 5(a)). The peaks related to hemicelluloses (1734 cm⁻¹) and lignin (1248 cm⁻¹) were not identified in the alkali-treated sisal fiber, as confirmed by the FTIR result (Figure 4). Elimination of lignin led to an increase in the volume fraction of the hydroxyl group on the fiber surface and the fibers tended to agglomerate. As a result, fiber dispersion uniformity level within the matrix became low and decreased the tensile strength of the composite.
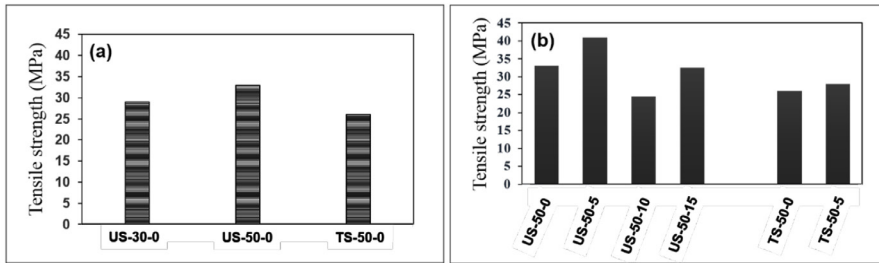
Figure 5: Tensile strength of sisal/PP composites (a) without MAPP and (b) with MAPP compared to that without MAPP

Our study also found that tensile strength of untreated sisal/PP composites with 50 wt% fiber loading increased by about 20% by the addition of 5 wt% MAPP (US-50-5) (Figure 5b). Increasing the MAPP concentration to 10 wt% (US-50-10) and 15 wt% (US-50-15), decreased the tensile strength. This trend is similar to that reported by Fuqua et al. [24] where the tensile strength of the corn fiber/PP composite increased from 27 MPa to ~32 MPa with 5 wt% of MAPP loading and drastically decreased to ~25.5 MPa at 10 wt% of MAPP loading. Besides, Figure 5(b) also shows that the tensile strength of US-50-5 specimen is higher in comparison to that of TS-50-0 and TS-50-5 specimens. The addition of MAPP on the alkali-treated sisal fibers, however, had less effect than that on untreated sisal fibers. Agglomeration of the fiber may cause this condition due to hot alkalization (100 °C, 1h). Our study, therefore, has verified that in enhancing the tensile strength of the composite, the role of MAPP addition is more efficient than the modification of fiber surface by hot alkalization both for untreated and treated fiber composites.

The changes in the tensile strength of the composites were influenced by different concentrations of MAPP (Figure 5(b)) and this can be explained by the morphology of their fracture surface (Figure 6). At 5 wt% MAPP loading (Figure 6(a)), the fracture surface of the composite appeared to be more flat with a relatively low volume fraction of micro-voids (see long arrows) and fibers pull-out (see small arrows). Moreover, Figure 6 exhibited better fiber distribution in the matrix and lower volume fraction of fiber pull-out (see short arrows) at 5 wt% of MAPP loading (Figure 6(a)) compared to composites with 10 wt% (Figure 6(b)) and 15 wt% (Figure 6(c)) of MAPP loading. The lower the volume fraction of fiber pull-out, the higher the tensile strength. The strong bonding between the fiber and the PP matrix due to the 5 wt% of MAPP loading could be seen clearly in a magnified image (Figure 6(a'), see arrows) of a small rectangular area in Figure 6(a). In contrast, from Figures 6(b)-

(c), it is evident that more fibers were not fully bonded to PP matrix which caused a decrease in the tensile strength of the composites.

The strong bond between the fiber and the matrix can be obtained if the polarity of the two phases matched each other. During the fabrication process of the composite, MAPP reacted with hydroxyl groups on the fiber surface to form ester bonds [26-27] resulting in lower hydroxyl groups or decreasing hydrophilicity of the fibers. This could improve the compatibility between the fiber and the matrix and also the mechanical properties of the composite. High concentrations of MAPP addition may impede the uniformity of fiber dispersion in the matrix causing a decrease in the mechanical properties and there might be an interaction between the loading concentration of MAPP and the fiber content in the composite.
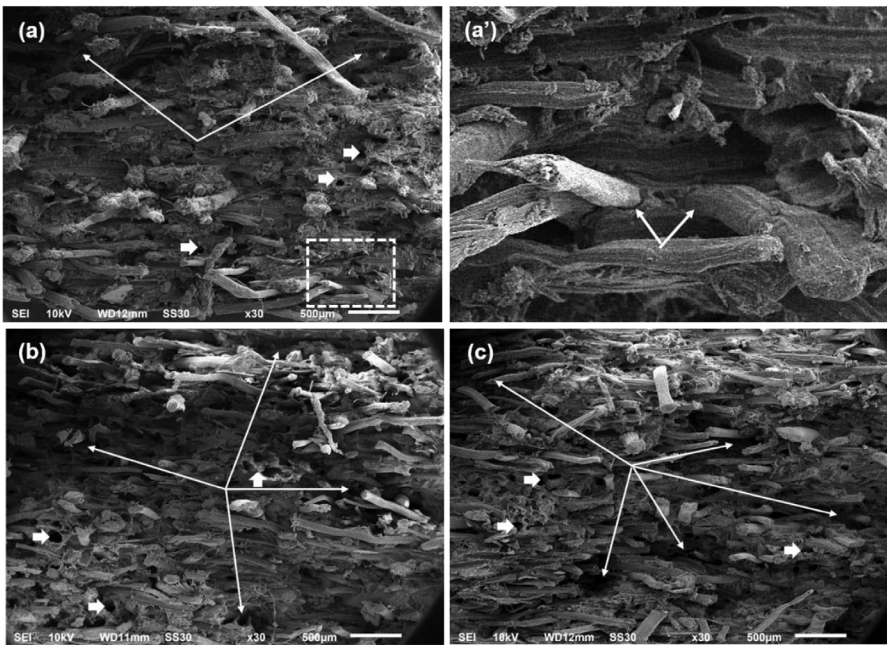

Figure 6: SEM micrographs of the tensile fracture surface of sisal/PP composites with the addition of (a) 5 wt% MAPP, (b) 10 wt% and (c)15 wt%. A magnified image of a small rectangular area in (a) indicates a strong bonding between the fibers and the matrix (see arrows) (a')

3.4 Effect of MAPP Addition on the Tensile Strength of the Sisal/PP Composites

The addition of MAPP associated with the change in tensile strength can be explained by the reaction between MAPP and the hydroxyl group occurring on the fiber surface (Figure 7). Thus, there was a strong interfacial bonding between the fiber and the matrix that led to the presence of the media to transfer stress from the matrix to the fiber. The stronger the interfacial bonding, the more easily stress was transferred. An increased tensile strength of untreated sisal/PP and alkali-treated sisal/PP composites with the addition of MAPP described above verified the vital role of MAPP in enhancing the tensile strength of the composites and being an efficient way to improve the mechanical properties. This trend was in line with our preliminary results indicating that there is an increase in the flexural strength achieved by 3 wt% of MAPP loading [8].
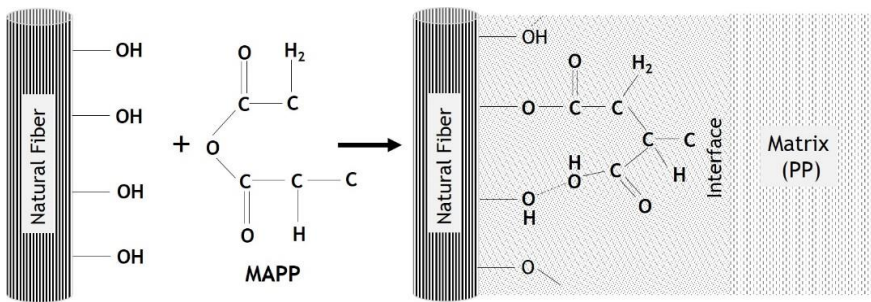

Figure 7: Illustration of the mechanism of an interfacial reaction between hydroxyl groups formed on the fiber surface and MAPP

4.0 CONCLUSION

The present study has verified that the addition of ≤ 5 wt% MAPP is an efficient way to improve the mechanical properties of natural fiber reinforced composites using either untreated or alkali treated fibers with a fiber content of ≤ 50 wt.%. Alkali treatment at $100°C$ for ≥ 1 h makes the sisal fibers more hydrophilic and decreases the compatibility between the fibers and the matrix,leading to reduced mechanical properties of the corresponding composites.

ACKNOWLEDGMENT

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