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Influence of chemical treatment on the tensile properties of kenaf fiber reinforced thermoplastic polyurethane composite

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Abstract. In this study, the effect of polymeric Methylene Diphenyl Diisocyanate (pMDI) chemical treatment on kenaf (*Hibiscus cannabinus*) reinforced thermoplastic polyurethane (TPU/KF) was examined using two different procedures. The first consisted of treating the fibers with 4% pMDI, and the second involved 2% NaOH + 4% pMDI. The composites were characterized according to their tensile properties, Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The treatment of the composite with 4% pMDI did not significantly affect its tensile properties, but the treatment with 2% NaOH + 4% pMDI significantly increased the tensile properties of the composite (i.e., 30 and 42% increases in the tensile strength and modulus, respectively). FTIR also showed that treatment with 2% NaOH + 4% pMDI led to the strongest H-bonding. Additionally, the surface morphology of specimens after tensile fracture confirmed that the composite treated with 2% NaOH + 4% pMDI had the best adhesion and wettability.

Keywords: *polymer composites, thermoplastic polyurethane, kenaf fibers, pMDI chemical treatment, FTIR*

1. Introduction

Natural fiber composites are having a strong influence in the field of composite materials. Cost effectiveness, a lighter weight, lower abrasion of equipment and renewability are amongst the benefits of natural fiber composites. However, natural fiber composites face some difficulties that prevent their widespread use. Fiber-polymer incompatibility has been the subject of previous studies [1–4]. This incompatibility is caused by the hydrophilic nature of the fibers and the hydrophobic nature of many polymers used in this field. The presence of hemicellulose, lignin and other impurities also causes a lack of adhesion between fibers and polymers.

The kenaf plant has a single, straight, unbranched stem. It has a short growing period, high biomass output and good mechanical properties. It reaches 3–4 meters in height in 4–5 months. It is reported that kenaf can yield three times a year in Malaysian climates [5], and in terms of quantity it can yield 2 to 25 ton/acre, depending on many factors such as soil type, variety and the month of planting [6]. Kenaf is traditionally used in cordage, canvas and sacking [7]. Kenaf is a good alternative to wood in the pulp and paper industry [3].

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Kenaf reinforced thermoplastic polyurethane was developed by the authors in a previous study [8]. Kenaf bast fiber was used because of its fast growth and good mechanical properties. Kenaf also absorbs nitrogen and phosphorus from the soil and accumulates carbon dioxide at a considerably high rate [9]. Thermoplastic polyurethane (TPU) was chosen due to its good mechanical properties and rubber-like elasticity. Another reason for selecting TPU is that polyurethane is polar, which reduces the fiber-polymer incompatibility.

Previously, the effect of various treatments on TPU/KF composites has been considered. The pretreatment of fibers with different concentrations of NaOH resulted in deterioration of its mechanical properties [10]. This result was supported by FTIR, thermogravimetrical analysis TGA and SEM studies.

Using pMDI isocyanate as an additive also showed no significant effect on the properties of the composite when pMDI was placed into the mixer directly before adding the fibers [11]. The NCO active group of isocyanate might have reacted with the matrix before the fibers were added to the mixer, which most likely resulted in the negligible effect on the properties of the composite.

Alkali treatment of natural fibers has several advantages for the removal of undesired substances; it affects the properties of the fibers and fiber-matrix interlocking [12]. Isocyanate has also shown a positive effect when used as a coupling agent between natural fibers and polymers. Previous studies have used isocyanates as coupling agents and reported their positive effect on fiber-matrix interfacial bonding [13–16]. Therefore, in this study, the same treatments (i.e., alkalization and isocyanate) were used with some modifications. Instead of charging the isocyanate into the mixer directly, it was used to pretreat the natural fibers, and in this case, it is referred to as pMDI chemical treatment. Alternatively, the fibers were first mercerized with NaOH and then treated with pMDI. Details on the chemical treatments are given in the methodology. The aim of this research is to investigate the effect of pMDI chemical treatment on TPU/KF composites.

2. Experimental

2.1. Materials

TPU (Texin®) and pMDI (Desmodur® 44V10L) were obtained from Bayer Co. (Malaysia) Sdn Bhd, Petaling Jaya, Selangor, Malaysia. The TPU used in this study was polyester based, and the properties are shown in (Table 1). pMDI isocyanate has an NCO content of 30.5 to 32.5 wt%. The kenaf V36 bast fiber used in this study was supplied by KEFI (Malaysia) Sdn Bhd, Setiu, Terengganu, Malaysia.

Table 1. Characteristics of TPU

Specific gravity	1.21
Tensile strength	48 MPa
Melting temperature	210°C
Hardness	55D

2.2. Fiber preparation

Mechanical decortication was used to extract the bast fibers. The fibers were pulverized using a Fritsch Power Cutting Mill PULVERISETTE 25 (Fritsch GmbH, Laborgeraetebau, Germany). The fibers were subsequently sieved using an automatic shaker sieve with meshes of 50 to 120 to a size range of 125–300 μm. This fiber size exhibited the optimum mechanical properties for TPU/KF composites [8].

2.3. Fiber treatments

2.3.1. Alkali treatment

The fibers were treated using a 2% aqueous solution of NaOH (by weight). The fibers were soaked in the solution for 3 hours. Subsequently, the fibers were washed 6 times with distilled water and oven dried at 80°C for 24 hours.

2.3.2. pMDI chemical treatment

Kenaf was pre-treated with 4% pMDI by weight of the fiber as follows. First, kenaf was mixed with toluene in a flask. Then, pMDI was mixed with toluene in a dropping funnel and then dropped into the flask over 20 minutes. The flask was heated at 50°C on a flat heater with continuous mixing for one hour. After the treatment, the treated fibers were separated by filtration, washed several times with toluene and dried in an oven at 70°C for 2 hours.

2.4. Composite preparation

2.4.1. Internal mixer

The TPU/KF composite was mixed using a Haake Polydrive R600 internal mixer at 190°C for 11 minutes at 40 rpm. These parameters were used because they led to the optimum tensile strength of the TPU/KF composite [8]. A 30% fiber loading was used

Table 2. The four different formulations prepared in this study

Formulation	Treatment	
	NaOH [%]	**pMDI [%]**
COMP1	–	–
COMP2	2	–
COMP3	–	4
COMP4	2	4

throughout the study because it resulted in the optimal strength of the composite [17]. TPU was charged into the mixer until the torque was stabilized, and then the fiber was added into the mixer. This procedure is explained in detail in a previous study [8]. Four different formulations were prepared in this study, as shown in (Table 2).

2.4.2. Compression molding

The sample was pre-heated for 7 minutes at 190°C. Then sample was hot-pressed using a Vechno Vation 40 ton compression molding device for 10 minutes at 190°C. Afterwards, the sample was cold pressed at 25°C for 5 minutes.

2.5. Tensile testing

The tensile properties were measured using an Instron 3365 electronic universal testing machine (Instron Co., Norwood, MA, USA) according to ASTM D 638(2010). The specimens were prepared by cutting them into dumbbell shapes using a hydraulic cutter machine. Five specimens were tested with a crosshead speed of 5 mm/min.

2.6. FTIR spectra characterization

The FTIR spectra of raw fibers, treated fibers and composites with and without treatment were recorded in the range of 4000 to 400 cm^{-1} using an FTIR machine Model Perkin-Elmer Spectrum 100 FT-IR Spectrometer (Perkin-Elmer, Norwalk, CT, USA). The samples were compacted into thin pellets with potassium bromide.

2.7. Surface characterization

The morphology of the tensile fracture surfaces of the treated and untreated composites were investigated using a scanning electron microscopy (SEM) on (S-3400 SEM, Hitachi Ltd., Ibaraki, Japan). The voltage used in the test was 5 kV.

3. Results and discussion

3.1. Effect of chemical treatment on the tensile properties of TPU/KF

Figure 1 shows the effect of various treatments on the tensile properties of TPU/KF. NaOH treatment of COMP2 decreased the tensile strength and strain; however, it slightly increased the tensile modulus. The pMDI chemical treatment of COMP3 resulted in a slight increase in the tensile strength and modulus and a slight decrease in the strain. The tensile strength of COMP4 showed a significant increase from 33 MPa for the untreated composite (COMP1)

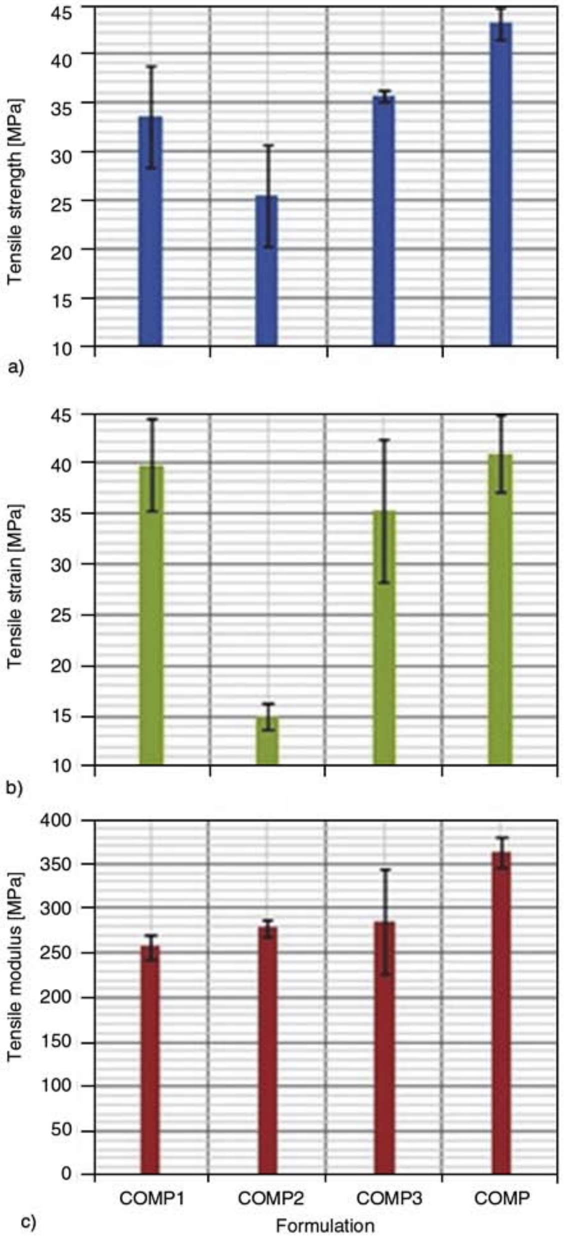

Figure 1. Effect of various treatments on the tensile properties of TPU/KF. (a) tensile strength, (b) tensile strain, (c) tensile modulus.

to a mean value of 43 MPa after treatment, which is an increase of approximately 30%. The modulus also increased from 257 MPa for the untreated composite to 364 MPa after treatment, about a 42% increase, while strain to failure increased only slightly.

In COMP1, the fibers were untreated, and the fiber bundles were still strongly bonded to each other with high stacking. Mixing the fibers with TPU led to interfacial bonding with the surface without reaching the inside of the structure of the fiber bundles. The strength of the COMP1 was most likely a result of the natural cohesion of the untreated fiber bundles.

In COMP2, the fibers were treated with NaOH, which cleaned them and provided a rougher surface; however, it softened the inter-febrillar matrix, which negatively affected the stress transfer in the fibers [12]. Alkali treatment uncovers the fibrils and gives the fibers a rough surface topography [18]. Alkali treatment also changes the fine structure of the native cellulose I to cellulose II by a process known as alkalization [18–20]. The reaction of NaOH with cellulose is shown in Equation (1). Figure 2a shows the surface of an untreated fiber. The fiber surface is smooth as a result of oils and waxes. Figure 2b shows kenaf fibers treated with 2% NaOH. It is clear that the treatment removes a certain amount of hemicellulose, lignin, wax, oils, and other impurities, and the surface becomes rougher. Similar observations were found in a previous study [21]. It has been demonstrated that TPU is not compatible with NaOH treatment [10]. NaOH treatment was used in this study to compare COMP2 with COMP4, which was treated with pMDI at the same alkali dosage (Equation (1)):

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O}^{-}\text{Na}^{+}\_\text{H}_2\text{O} + (\text{Surface impurities}) \quad (1)$$

In COMP3, the fibers were treated with pMDI. The NCO groups are believed to react with the OH groups of the cellulose; therefore, the composite exhibited better interfacial bonding between the treated fibers and the matrix. However, the increment of the tensile strength was not significant, which is attributed to the availability of other materials, such as hemicellulose, lignin, waxes, and oils, which may have prevented the full effect of the isocyanate treatment. Therefore, in the next formulation, the fibers were first treated with NaOH to remove the undesirable impurities that reduce the ability of pMDI to form effective crosslinks during the treatment of fibers.

In COMP4, the fibers were treated with NaOH, which removed undesirable materials; thus, more reactive sites (i.e., hydroxyl groups) of cellulose were revealed [12]. Then, the fibers were treated with pMDI. In general, polyurethane consists of two main components: isocyanates and polyols. The main reaction that forms polyurethane is the reaction between the isocyanate NCO groups and the OH groups of the polyols. Isocyanate represents the rigid segment while polyol represents the soft component [22]. It is believed that isocyanate played the same role when utilized as a chemical treatment for the NaOH pretreated kenaf fibers; thus, by adding isocyanate to the fibers, they became more rigid. More rigid fibers have a better ability to transfer the load to the matrix. In other words, the NCO groups could react more effectively with the OH groups after NaOH treatment because the fibers had more

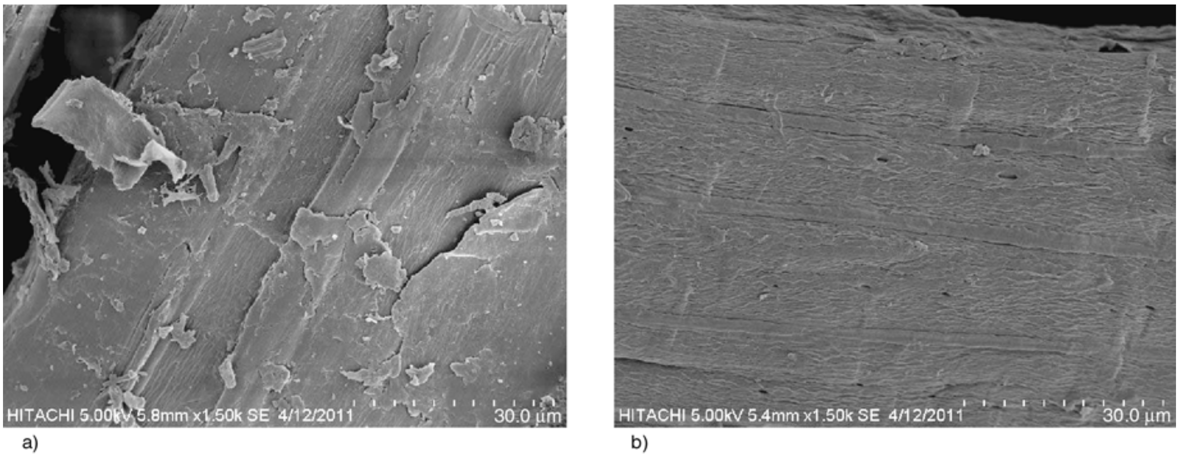

Figure 2. SEM of (a) untreated kenaf fiber and (b) kenaf fiber treated with 2% NaOH [10]

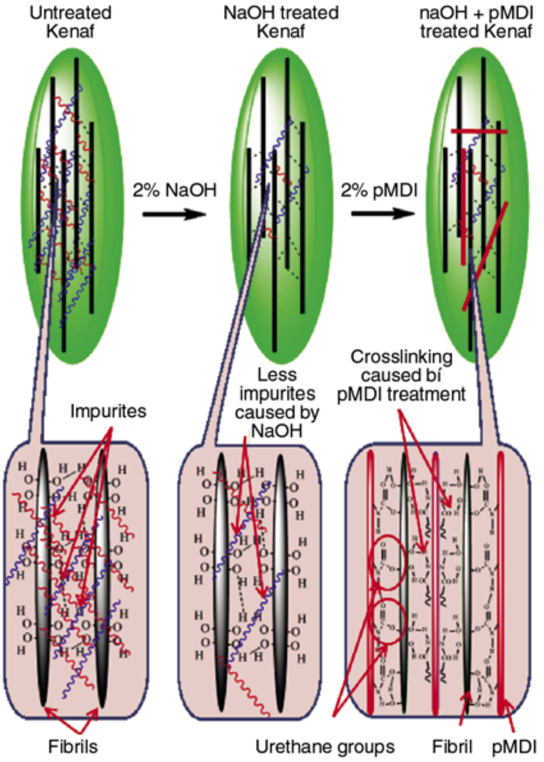

Figure 3. Schematic hypothetical chemical structure of kenaf fibers treated with 2% NaOH + 4% pMDI

available reactive sites for the NCO groups. The crosslinking reconstructed the fibers and made them stronger; thus, they were able to transfer more load. In addition, the increase of strength in COMP4 could be ascribed to the increased strength of the interface between the fibers and the matrix, thereby allowing the fibers to positively contribute.

A schematic hypothetical chemical structure of the fibers treated with 2% NaOH + 4% pMDI is shown in Figure 3. In this drawing, untreated kenaf is covered with undesirable materials. However, after treatment with NaOH, the fibers were coated with fewer impurities. After treatment with pMDI, crosslinking can be observed between the OH groups of the fibrils and pMDI to form urethane groups.

3.2. FTIR spectra of treated and untreated fibers and composites

Figure 4 shows the FTIR spectra of curve (a) untreated kenaf fibers, curve (b) kenaf fibers treated with 2% NaOH, curve (c) kenaf fibers treated with 4% pMDI, curve (d) kenaf fibers treated with 2% NaOH + 4% pMDI, and curve (e) pMDI. Figure 5 shows the FTIR spectra of COMP1, COMP2, COMP3, COMP4, and pure TPU. The main IR bands

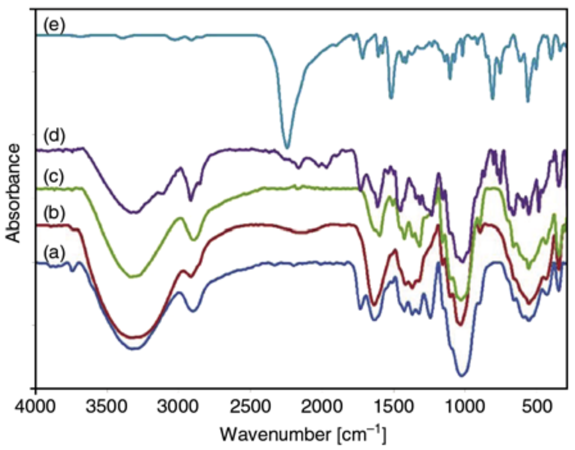

Figure 4. FTIR spectra of (a) untreated kenaf fibers, (b) kenaf fibers treated with 2% NaOH, (c) kenaf fibers treated with 4% pMDI, (d) kenaf fibers treated with 2% NaOH + 4% pMDI and (e) pMDI

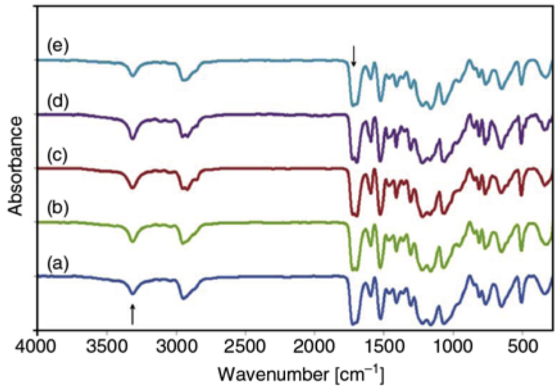

Figure 5. FTIR spectra of (a) COMP1, (b) COMP2, (c) COMP3, (d) COMP4 and (e) TPU

for all of the previous materials are summarized in (Table 3).

In the untreated kenaf spectra, the absorption peak was observed at approximately 1736 cm^{-1}, corresponding to the C=O stretching of hemicellulose, but this peak was almost missing in the spectrum of the 2% NaOH treated fibers, indicating the elimination of hemicelluloses and lignin as a result of alkali treatment. The elimination of this peak was observed in a previous study [23]. It is observed again at 1730 cm^{-1} in some materials after alkali treatment such as kenaf treated with 2% NaOH + 4% pMDI, TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% pMDI, and TPU/KF treated with 2% NaOH + 4% pMDI as a result of the formation of ν(C=O) of free urethane.

The strong absorption at 2250 cm^{-1} corresponds to the –N=C=O stretching of pMDI. This peak was

Table 3. Main FTIR bands of kenaf, pMDI, TPU, kenaf treated with 2% NaOH, kenaf treated with 4% pMDI, kenaf treated with 4% pMDI, kenaf treated with 2% NaOH + 4% pMDI, COMP1, COMP2, COMP3, and COMP4

Main peaks	Materials	Kenaf	pMDI	TPU	Kenaf + NaOH	Kenaf + pMDI	Kenaf + NaOH + pMDI	COMP1	COMP2	COMP3	COMP4
Chemical structure	**P L**										
NH^a	3420–3200	–	–	3324	–	3351	3346	3325	3322	3325	3324
OH^a	3300–3400	3340	–	–	3347	3313	3300	–	–	–	–
C–H^a	3100–3000	–	3040	–	–	–	–	–	–	3060	–
CH$_2$^{a} and CH$_3$^{a}	3000–2800	2924	2927	2985, 2936	2931	2910	2910	2958	2961, 2935	2961, 2927	2961, 2925
–N=C=O^b	2250	–	2250	–	–	–	–	–	–	–	–
NH^c	1590–1650	–	1603	–	1602	1603	1603	1603	1601	1600	1598
C=O^d	1690	1736	–	1729	–	–	–	1728	1729	1729	1730
C=O^e	1740	–	–	1715	–	–	–	1713	1703	1705	1702
C=C^a	1600	1647	1610, 1521	–	–	1641	–	–	–	–	–
H–N–C=O Amide II^f	1550–1510	–	–	1530	–	–	–	1531	1532	1531	1531
CH$_2$^{c} and CH$_3$^{c}	1500–1300	–	–	–	–	1428, 1323	1426, 1321	–	–	1415	1414, 1312
C–O^a	1300–1000	1042	–	–	1044	1040	1038	1075	1078	1076	1076

P L; peak location [cm^{-1}], ^{a}stretching, ^{b}isocyanate, ^{c}bending, ^{d}non-bonded urethane stretching, ^{e}associated urethane, ^{f}combined motion

almost missing in the spectrum of the pMDI treated fibers (Figure 4) and composites (Figure 5), which confirms that presence of the urethane group formed between kenaf and pMDI in pMDI chemically treated fibers and between pMDI chemically treated fibers and TPU in TPU/KF. The elimination of the peak at 2250 cm^{-1} indicating –N=C=O stretching of pMDI, with a corresponding increase in the peak at 1730 cm^{-1}, mentioned in the previous paragraph, suggests the formation of carbamate esters as a result of the reaction between –N=C=O and OH [24].

The following observations were made for untreated kenaf, kenaf treated with 2% NaOH, kenaf treated with 4% pMDI, and kenaf treated with 2% NaOH + 4% pMDI. Untreated kenaf and kenaf treated with 2% NaOH have alcoholic O–H stretching absorptions at approximately 3340 cm^{-1}. However, kenaf treated with 4% pMDI and kenaf treated with 2% NaOH + 4% pMDI have overlapping signals of alcoholic O–H stretching with H-bonded absorption and N–H stretching with H-bonded absorption at approximately 3340 cm^{-1}.

TPU, TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% pMDI, and TPU/KF treated with 2% NaOH + 4% pMDI were studied. All of these samples include urethane N–H stretching and N–H bending absorptions at approximately 3324 and 1530 cm^{-1}, respectively, and C=O stretching absorptions at approximately 1701 cm^{-1}. The stretching absorption of an ether linkage (C–O–C) was observed at approximately 1040 cm^{-1}. In addition, absorption bands arising from –CH$_2$ stretching were found between 2910 and 2985 cm^{-1}, and bending –CH$_2$ band were observed at approximately 1428 and 1323 cm^{-1}.

The main factor contributing to the mechanical properties of the natural fiber composites is H-bonding. It causes shifts and differences in the intensities of the absorptions; all of these shifts are shown in Figure 6. For example, the stretching H-bonding of NH at approximately 3324 cm^{-1} [25] showed the highest absorption intensity from TPU/KF treated with 2% NaOH + 4% pMDI and the lowest absorp-

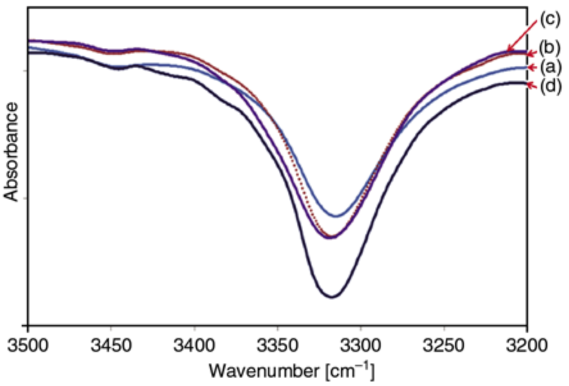

Figure 6. FTIR spectra in the range between 3500 and 3200 cm^{-1} for (a) COMP1, (b) COMP2, (c) COMP3 and (d) COMP4

tion intensity from untreated TPU/KF, which indicates that the H-bonding increased in the following order: TPU/KF treated with 2% NaOH + 4% pMDI, TPU/KF treated with 4% pMDI, TPU/KF treated 2% NaOH and untreated TPU/KF.

TPU/KF treated with 2% NaOH + 4% pMDI yielded the highest H-bonding and thus, the best tensile properties because undesirable materials were removed from the kenaf by the NaOH treatment, and then the fibers were treated with pMDI to create chemical crosslinking between the fiber bundles. By following these steps, the degree of polymerization might have increased. In addition, urethane groups were formed, which made the fibers capable of transferring greater loads. Therefore, the tensile properties were enhanced.

3.3. SEM of the fracture surface of tensile specimens

Both fiber breakage and fiber pull-out are shown on the fracture surface of the untreated TPU/KF (Figure 7a), which indicates that the fiber-matrix adhesion is quite good. Figure 7b shows the fracture surface of TPU/KF treated with 2% NaOH, in which poor fiber-matrix adhesion is confirmed by the presence of gaps between the fibers and the matrix and fiber pull-outs.

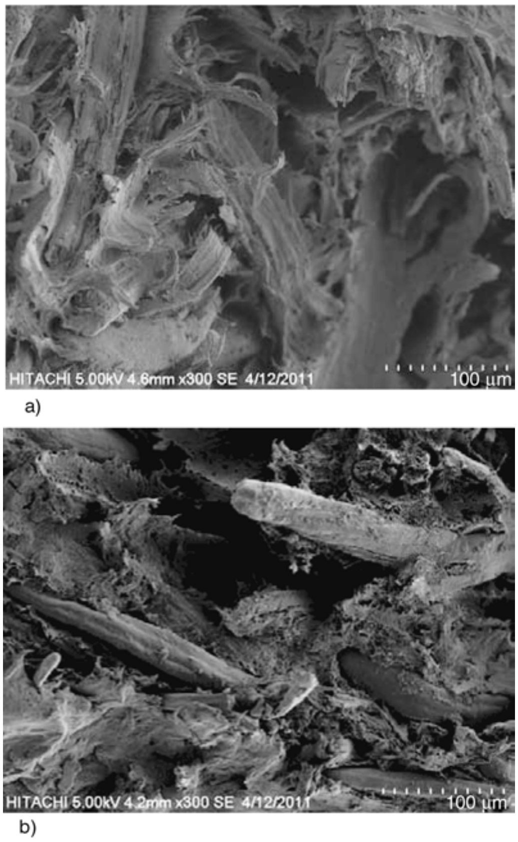

Figure 7. SEM of (a) untreated TPU/KF and (b) TPU/KF treated with 2% NaOH [10]

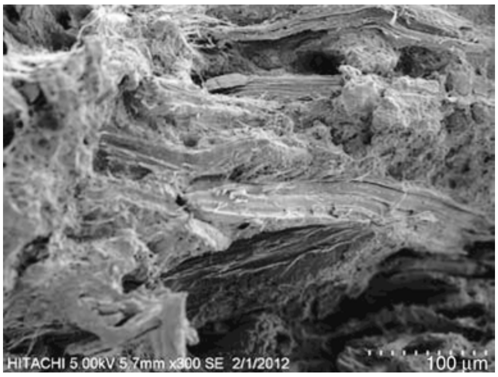

Figure 8. SEM of TPU/KF treated with 4% pMDI chemical treatment

Treatment of TPU/KF with 4% pMDI also led to a mix of fiber breakage and pull-outs Figure 8. However, when TPU/KF was treated with 2% NaOH + 4% pMDI, fiber breakage became dominant, as shown in Figure 9a, which confirms the strong fiber-matrix adhesion. Furthermore, no gaps were seen between the fibers and the matrix in Figure 9b, which is a high magnification image of Figure 9a,

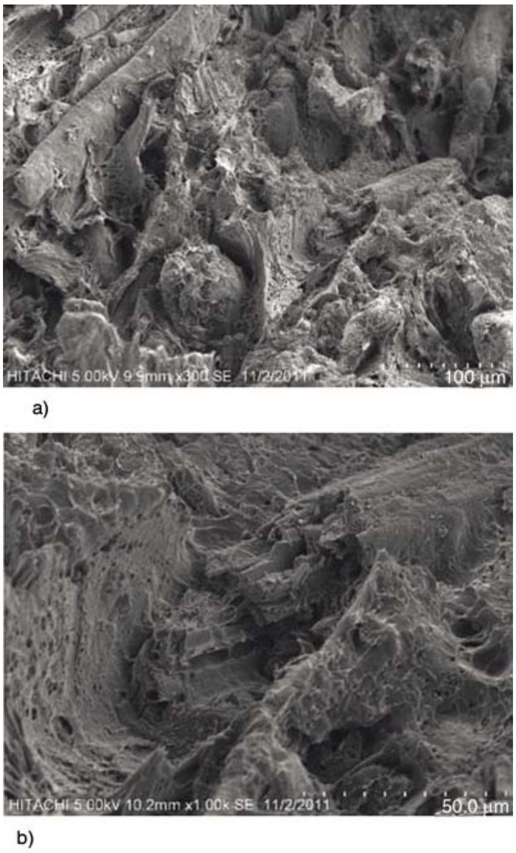

Figure 9. SEM of TPU/KF treated with 2% NaOH + 4% pMDI; (a) low magnification, (b) high magnification

indicating better wetting and adhesion between the fibers and matrix.

4. Conclusions

While the treatment of composites with NaOH alone led to a deterioration of their properties, pMDI chemical treatment increased the properties of the composite only slightly. However, the treatment of kenaf fibers with NaOH followed by pMDI had a significant effect on the composite, which was evident in the 30% increase in the tensile strength and approximately 42% increase in the tensile modulus. The FTIR spectra also provided evidence that the NCO groups of pMDI covalently linked with the OH groups of kenaf fibers and that there was strong H-bonding in the composite treated with NaOH + pMDI. The SEM morphological study shows that the composites treated with NaOH + pMDI have better fiber-matrix adhesion and wettability.

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