

Effect of alkali treatment on mechanical and thermal properties of Kenaf fiber-reinforced thermoplastic polyurethane composite

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Abstract In this study, a composite of thermoplastic polyurethane reinforced with short Kenaf fiber (*Hibiscus cannabinus*) was prepared via melt-blending method using Haake Polydrive R600 internal mixer. Effect of various sodium hydroxide NaOH concentrations, namely 2, 4 and 6% on tensile, flexural and impact strength was studied. Mean values were determined for each composite according to ASTM standards. Tensile, flexural and impact strength negatively correlates with higher concentrations of NaOH. Scanning electron microscope (SEM) was used to examine the surface of both treated and untreated fibers as well as fracture surface of tensile specimens. Morphology of treated and untreated fibers showed a rougher surface of treated fibers. It also showed that some of high concentrations of NaOH treated fibers have NaOH residues on their surface. This was confirmed by energy dispersive X-ray point shooting performed on the same SEM machine. Morphology of surface of fracture indicated that untreated composite had a better adhesion. Treated and

untreated fibers as well as composites were characterized using Fourier transform infrared spectroscopy (FTIR). FTIR of treated fibers showed that NaOH treatment resulted in removal of hemicelluloses and lignin. FTIR also showed that untreated composite has more H-bonding than all treated composites. Thermal characteristic studies using thermogravimetry analysis and differential scanning calorimetry showed that untreated composite was more thermally stable than treated composites.

Keywords Thermoplastic polyurethane · Kenaf bast fibers · Alkali treatment · FTIR · TG · DSC

Introduction

Efficiency of composite materials depends mainly on the properties of the individual components as well as interface between fibers and matrix. Incompatibility is among the main obstacles facing today's very promising natural fiber composites industry. Many studies have been conducted to address this problem and overcome this obstacle. Various fiber pre-treatments and coupling agents have been used to enhance the fiber-matrix interface. Bledzki et al. [1] have reviewed physical and chemical methods of surface modification of natural fibers. The mechanisms of chemical modifications of natural fiber-reinforced composites i.e. alkalization, acetylation, and silane treatments have been reviewed by Jone and Anandjiwala [2]. Use of pre-treated natural fibers in polymer matrix-based composites has been reviewed, and effect of surface modification of natural fibers on the properties of fibers and fiber-reinforced polymer composites has also been discussed by Kalia et al. [3].

NaOH is the most common chemical treatment used to clean fiber surface and enhance fiber properties. Untreated

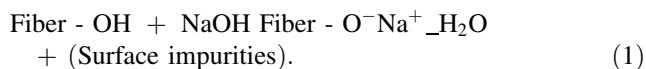
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fibers are covered with hemicellulose, lignin, pectin, waxy substances, and natural oils. Treatment with NaOH reveals the fibrils and gives fibers a rough surface topography [4]. It also changes the fine structure of the native cellulose I to cellulose II by a process known as alkalization [2, 4, 5]. The reaction of NaOH with cellulose is shown in Eq. (1). Alkali treatment can increase the compatibility of nonpolar polymer interface with natural fibers [6].



The importance of natural fiber-reinforced polymers is becoming more apparent due to their numerous benefits such as being less abrasive to equipment, and for their renewability, biodegradability, and their cost-effectiveness. Fiber polymer incompatibility is among the difficulties that face natural fiber polymer composites. While most polymers used in this field are hydrophobic, natural fibers are hydrophilic, which causes lack of adhesion and wettability. Therefore, fiber treatments, coupling agents and binders in the interface are used for a better adhesion. In this study, the researchers exploit the hydrophilicity of polyurethane that is polar in nature. Polyurethane thermoset has been compounded with different natural fibers [7–11]; however, thermoplastic polyurethane-reinforced natural fiber composites have not been used. Thermoplastics are reformable, have shorter processing time and lower energy, are tougher, and recyclable. They are also excellent abrasion resistant and have good mechanical properties with rubber-like elasticity and tear resistance.

Kenaf is planted commercially in China, Myanmar, India, Bangladesh, and Thailand. Kenaf plant has good mechanical properties, high biomass output, and a short growing period. It reaches 3–4 meters in 4–5 months. It can yield two to three harvests a year in tropical climates [12]. It can produce 5–10 tons of dry fiber per acre. Two types of fibers can be extracted from this plant: bast fiber, which is the outer layer, and core fiber, which is the inner layer. Morphological and chemical analysis by Ashori et al. [13] indicated that bast and core fibers were significantly different. Bast fiber represents nearly 1/3 of the plant, and the core represents the rest. Malaysia has put more efforts in planting Kenaf. Since 2000, more than RM48 million has been spent by Malaysian government for the research of Kenaf plantation and utilization [14].

Therefore, the objective of this study is to investigate the effect of NaOH on the fiber-matrix adhesion of TPU/KF 70/30 wt%. Mechanical properties (tensile, flexural, and impact) of the composites were measured. Surface characterization was performed. SEM and FTIR spectroscopy were carried out to observe the effect of NaOH treatment on fibers and on composites as well. TG and DSC thermal studies have been conducted to further investigate the effect of NaOH on the composite. To the best of the

Table 1 Characteristics of TPU

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

researchers' knowledge, TPU/KF composites have not yet been investigated by other researchers.

Materials

Polyester-based thermoplastic polyurethane (TPU) was obtained from Bayer Co., (Malaysia) Sdn Bhd. Characteristics of TPU are shown in Table 1. Kenaf V36 bast fiber was obtained from Lembaga Kenaf Negara, Terengganu, Malaysia.

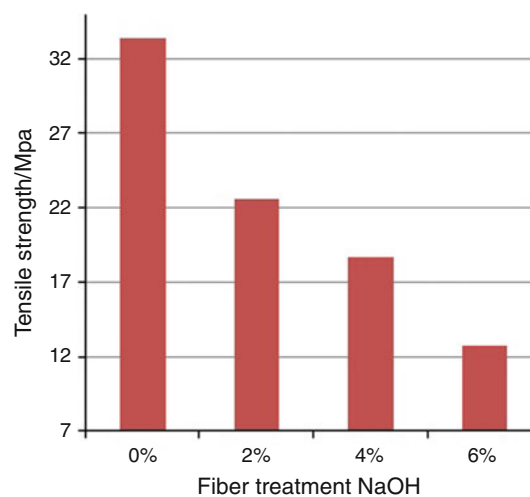


Fig. 1 Effect of NaOH on tensile strength of TPU/KF

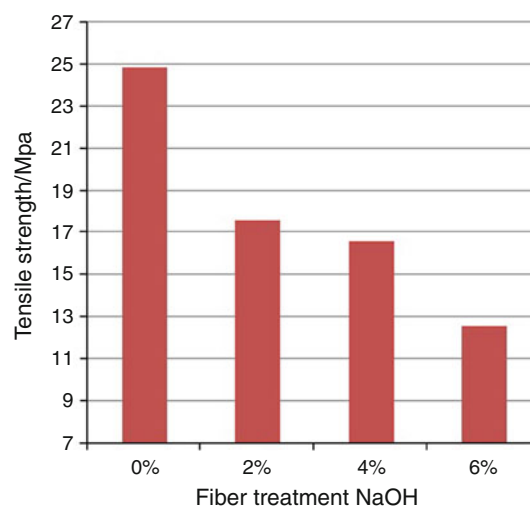


Fig. 2 Effect of NaOH on flexural strength of TPU/KF

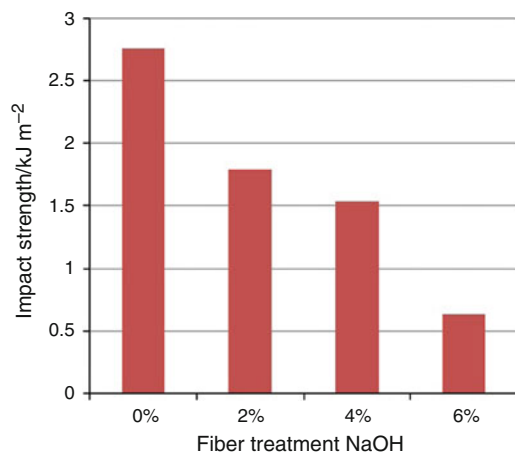


Fig. 3 Effect of NaOH on impact strength of TPU/KF

Methods

Fiber preparation

Bast fiber was extracted by mechanical decortications and then pulverized using Fritsch Pulverisette mill. Pulverized fiber was sieved using an auto shaker sieve into size range between 125 and 300 μm using mesh 50–120.

Fiber treatment

Fibers were soaked in 2, 4 or 6% NaOH solution for 3 h and then washed 6 times with distilled water. Fibers were then oven dried at 80 $^{\circ}\text{C}$ for 24 h.

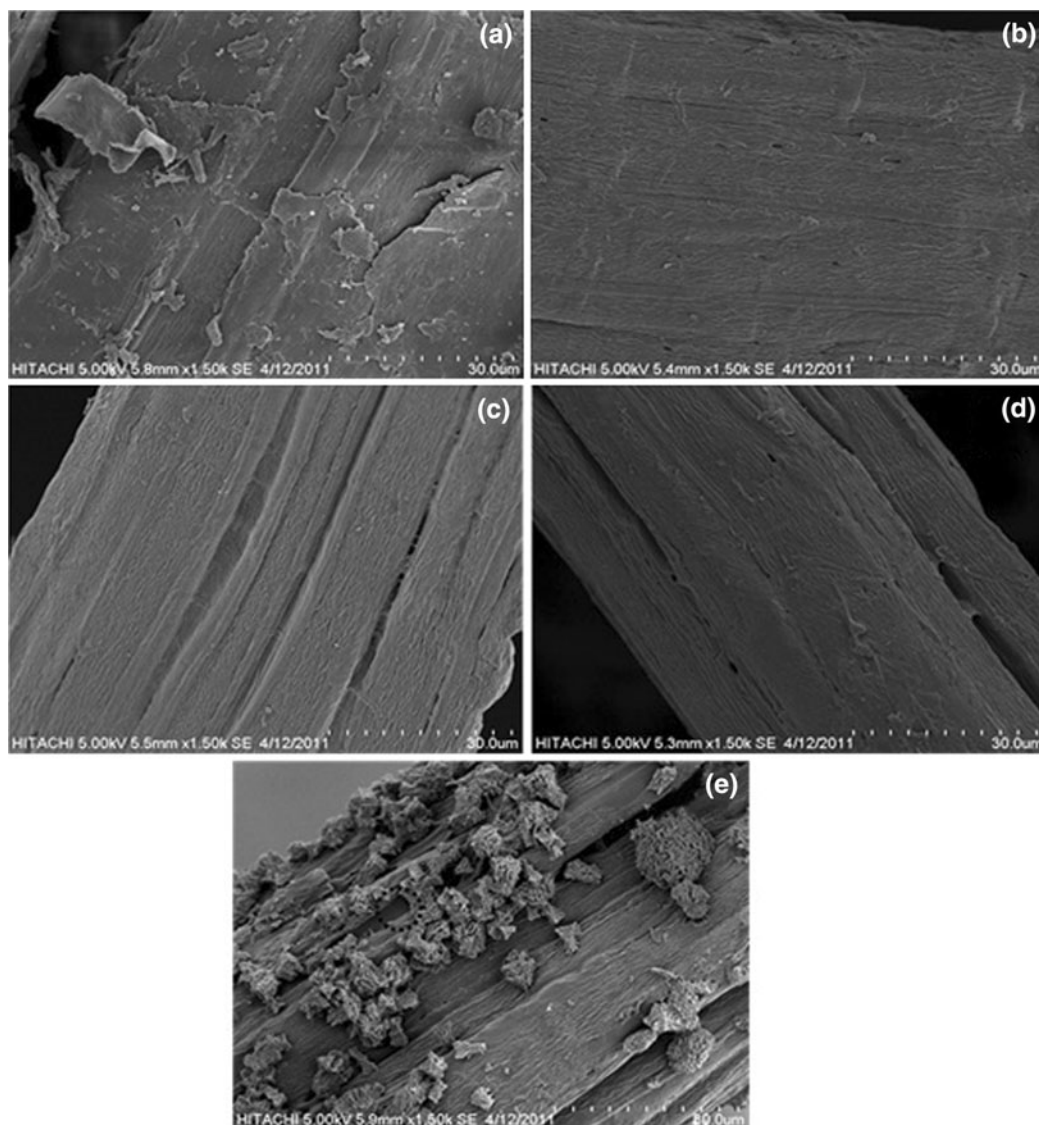


Fig. 4 SEM of **a** untreated Kenaf fiber, **b** Kenaf treated with 2% NaOH, **c** Kenaf treated with 4% NaOH, **d** Kenaf treated with 6% NaOH, and **e** Kenaf treated with 6% NaOH

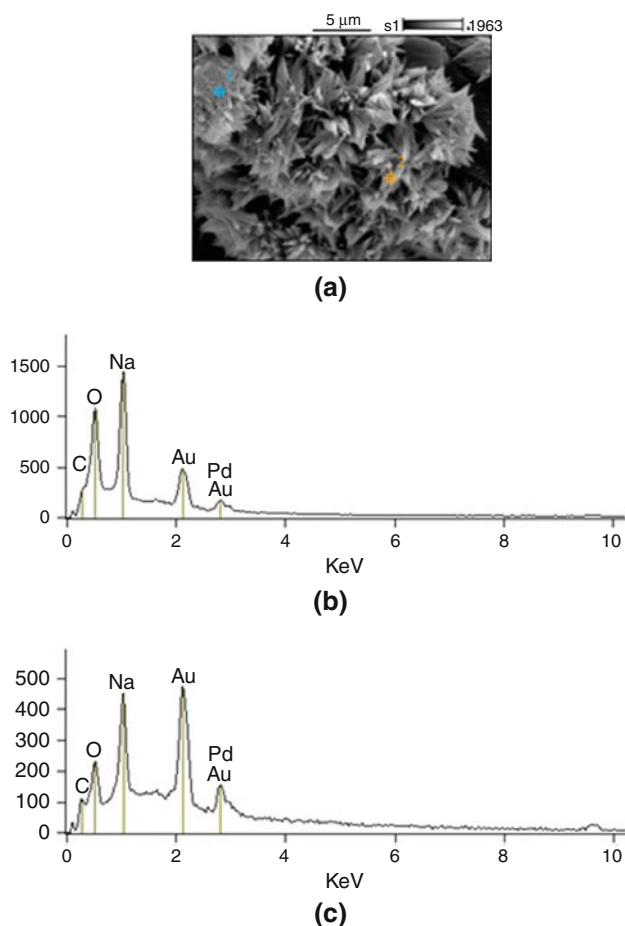


Fig. 5 EDX for substance adhered to the fibers **a** two shoots on the substance, **b** point 1, and **c** point 2

Composite preparation

TPU/KF was prepared by melt-blending method using Haake Polydrive R600 internal mixer. Matrix was charged into the mixer until torque was stabilized. Then fiber was added into the mixer. A 30% by mass fiber loading was fixed throughout the study.

The sample was hot pressed using Vechno Vation 40 ton compression molding for 10 min at 190 °C. Prior to full-press, the sample was pre-heated for 7 min at 190 °C. The sheet was then cold pressed between two plats for 5 min at 25 °C.

Mechanical characterization

Tensile testing

Tensile properties were measured using Instron 3365 machine, according to ASTM D 638. The specimens were prepared by cutting them into dumbbell shapes using a hydraulic cutter machine. Five specimens were tested with crosshead speed of 5 mm min⁻¹.

Flexural testing

A three-point bending flexural test was conducted using Instron 3365 machine, according to ASTM D 790. The specimens were cut using a band saw machine with dimensions of 130 × 13 × 3 mm. The specimens were tested with a crosshead speed of 2 mm min⁻¹.

Impact testing

Notched impact strength was measured by 43-02-01 Monitor Impact Tester according to ASTM D 256. Dimensions of the samples were 63 × 13 × 3 mm. At least five samples were tested. The impact strength (kJ m⁻²) was calculated by dividing the recorded absorbed impact energy with the cross-section area of the samples.

Surface and chemical characterization

Scanning electron microscope

The surface morphology of raw fibers, treated fibers and fracture of tensile specimens were investigated using a scanning electron microscope (SEM), (SEM HITACHI S-3400 N).

Fourier transform infrared spectroscopy

FTIR spectra of raw fibers, treated fibers, and composites with and without treatment were recorded in PerkinElmer Spectrum 100 FT-IR Spectrometer.

Thermal characterization

Thermogravimetric analysis

TG tests were carried out using a Mettler Toledo TGA/SDTA851^e analyzer. These tests were performed in the temperature range between room temperature and 600 °C at a heating rate of 10 °C min⁻¹ in an atmosphere of nitrogen that flowed at 10 mL min⁻¹. A sample of 5–20 mg of the materials was heated in a sample pan.

Differential scanning calorimetry

DSC was carried out using a Mettler Toledo DSC823^e analyzer. The tests were performed in the temperature range between room temperature and 300 °C at a heating rate of 10 °C min⁻¹ under nitrogen atmosphere with flow rate of 10 mL min⁻¹. Around 5 mg of the materials were heated in the sample pan, and an empty pan was used as a reference.

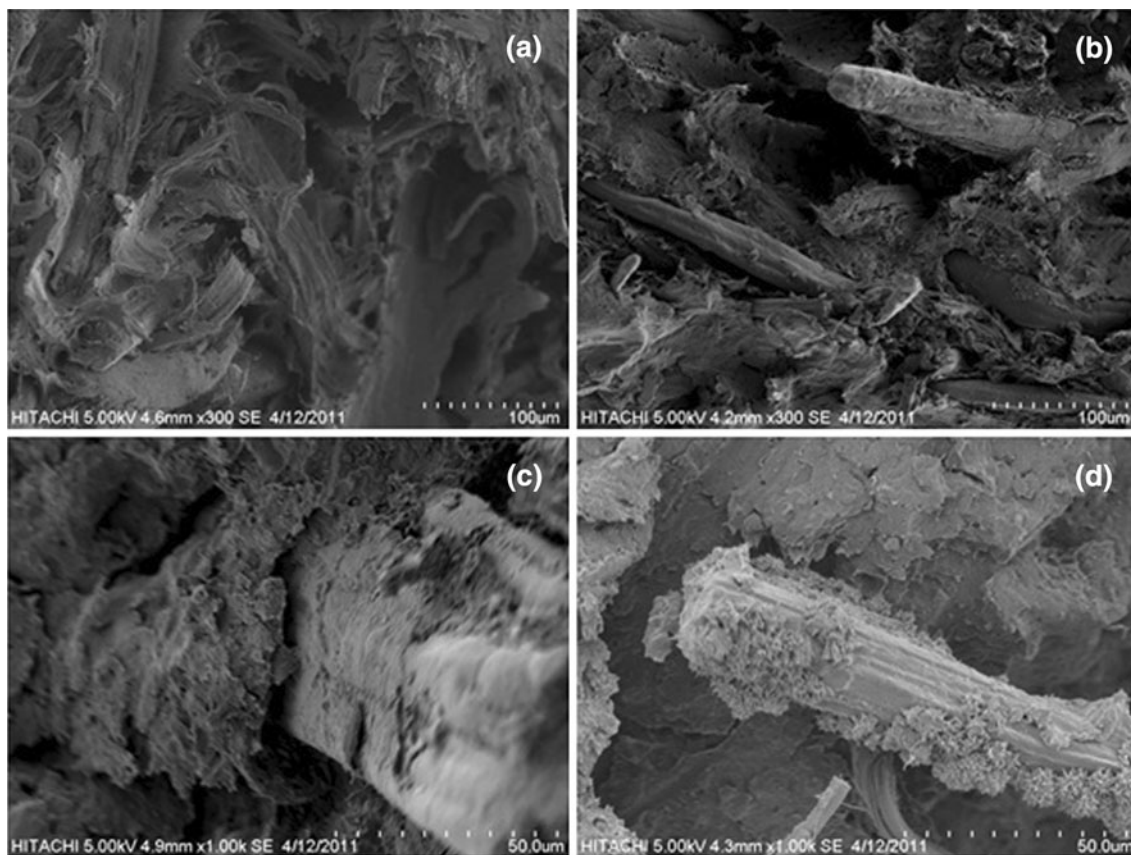


Fig. 6 SEM of **a** untreated TPU/KF, **b** TPU/KF treated with 2% NaOH, **c** TPU/KF treated with 4% NaOH, and **d** TPU/KF treated with 6% NaOH

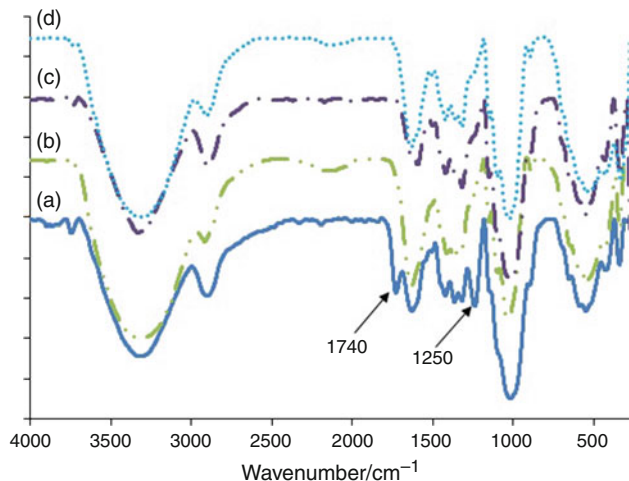


Fig. 7 FTIR for **a** untreated Kenaf fibers, **b** Kenaf treated with 2% NaOH, **c** Kenaf treated with 4% NaOH, and **d** Kenaf treated with 6% NaOH

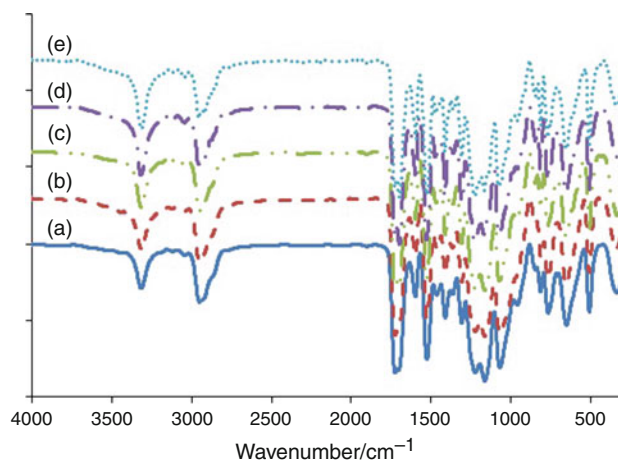


Fig. 8 FTIR of **a** pure TPU, **b** untreated TPU/KF, **c** TPU/KF treated with 2% NaOH, **d** TPU/KF treated with 4% NaOH, and **e** TPU/KF treated with 6% NaOH

Results and discussion

Effect of NaOH on mechanical properties of TPU/KF

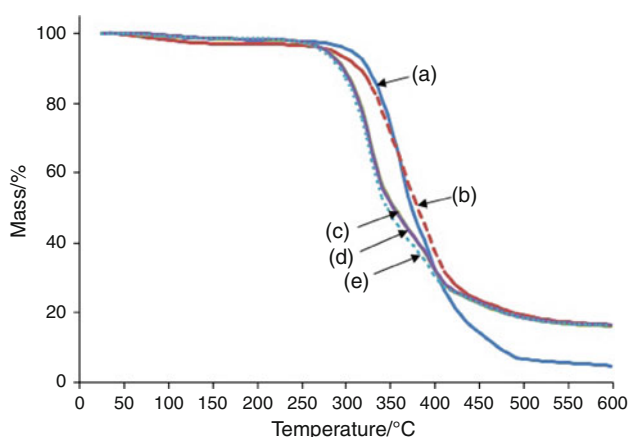
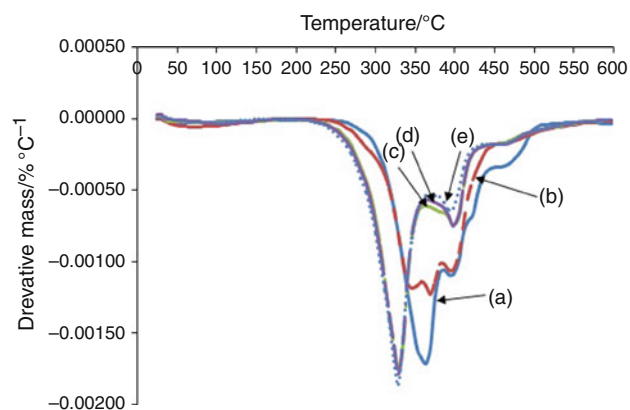
Treatment of TPU/KF composite with increasing concentrations of NaOH led to decrease in its tensile (Fig. 1), flexural (Fig. 2), and impact (Fig. 3) strengths.

SEM of treated and untreated Kenaf surfaces

The treatment of Kenaf with NaOH cleaned the fibers from impurities and produced a better surface roughness as shown in (Fig. 4). The untreated fiber (Fig. 4a) is smooth as a result of oils and waxes. However, Fig. 4b–d shows

Table 2 Main FTIR bands of pure TPU, untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH and TPU/KF treated with 6% NaOH

Peak location (cm ⁻¹) [20–22]	Chemical structure	Motion	Pure TPU	Untreated TPU/KF	TPU/KF treated 2% NaOH	TPU/KF treated 4% NaOH	TPU–KF treated 6% NaOH
3,420–3,200	N–H	Stretching	3,324	3,325	3,322	3,323	3,323
1,590–1,650	N–H	Bending	1,603	1,603	1,601	1,599	1,599
3,000–2,800	CH ₂ and CH ₃	Stretching	2985, 2936	2958	2961, 1935	2962, 2945	2963, 2954
1,740	C=O	Non-bonded unurethane stretching	1,729	1,728	1,729	1,731	1,731
1,690	C=O	Associated urethane	1,715	1,713	1,703	1,702	1,702
1,550–1,510	H–N–CO amide II	Combined motion	1,530	1,531	1,532	1,533	1,533

**Fig. 9** TG of **a** pure TPU, **b** untreated TPU/KF, **c** TPU/KF treated with 2% NaOH, **d** TPU/KF treated with 4% NaOH, and **e** TPU/KF treated with 6% NaOH**Fig. 10** DTG of **a** pure TPU, **b** untreated TPU/KF, **c** TPU/KF treated with 2% NaOH, **d** TPU/KF treated with 4% NaOH, and **e** TPU/KF treated with 6% NaOH

that treatment of fibers with 2, 4, or 6% alkali, respectively, removes a certain amount of wax, oils, and other impurities, and the surface becomes rougher. Fig. 4e shows that

some of the 6% alkali treated fibers contain other substances adhered to their surface. This is probably due to inability to wash-off NaOH residues from the surface of fibers treated with the higher concentration of alkali. However, washing with water cleared these residues from surface of fibers treated with 2 and 4%. To determine that the substance observed under SEM was NaOH residue, energy dispersive X-ray (EDX) point shooting was performed on the same SEM machine. Figure 5 shows the point shooting for two points. Figure 5b and c show the presence of NaOH residue. These graphs also show the presence of gold and palladium that were used for coating the samples to have a conductive surface that will prevent the electrostatic charge from accumulating on the sample.

SEM of fracture surface of tensile specimens

A combination of fiber breakage and fiber pull-out is shown on the fracture surface of untreated TPU/KF (Fig. 6a). This indicates that fiber-matrix adhesion is quite good. Figure 6b shows the fracture surface of TPU/KF treated with 2% NaOH. Treatment of TPU/KF with 2% NaOH (Fig. 6b) and 4% NaOH (Fig. 6c) shows poor fiber-matrix adhesion, as there are gaps between fibers and matrix and fiber pull-outs. Treatment of TPU/KF with 6% NaOH also shows poor adhesion as well (Fig. 6d). Fiber pull-outs, voids, and NaOH residues are also observed. SEM morphology study demonstrates that alkali treatment has a negative effect on the fiber-matrix adhesion that explains the lost mechanical properties.

FTIR spectra of treated and untreated Kenaf fibers

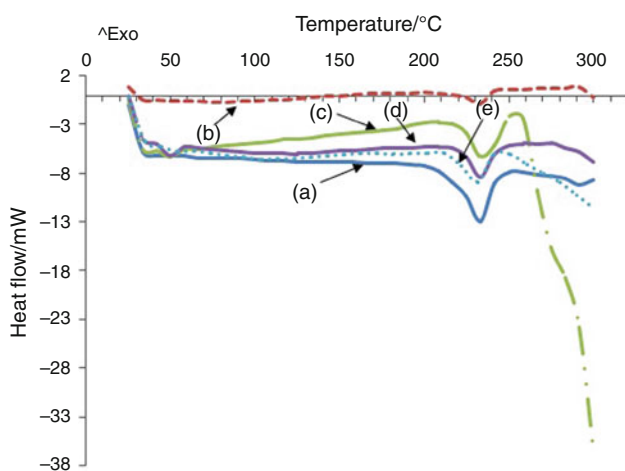
Figure 7 shows that there are absorption bands at $\sim 1,740$ and $1,250$ cm⁻¹ for the untreated fibers that disappear in the treated fibers. The hemicellulose contains groups that absorb

Table 3 Mass loss percentage of pure TPU, untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH, and TPU/KF treated with 6% NaOH

Sample	Mass loss/%						
	$T_{100.83}/^{\circ}\text{C}$	$T_{252.5}/^{\circ}\text{C}$	$T_{299.16}/^{\circ}\text{C}$	$T_{351.66}/^{\circ}\text{C}$	$T_{398.33}/^{\circ}\text{C}$	$T_{450.83}/^{\circ}\text{C}$	$T_{503.33}/^{\circ}\text{C}$
Pure TPU	0.96	2.24	4.26	28.93	66.79	86.05	93.45
Untreated TPU/KF	1.91	3.35	7.1	30.59	61.63	76.49	81.03
TPU/KF treated 2% NaOH	0.71	2.65	10.93	49.25	67.32	77.57	81.15
TPU/KF treated 4% NaOH	0.5	2.44	11.43	49.84	67	77.17	80.09
TPU/KF treated 6% NaOH	0.76	2.66	13.03	53.04	69.08	77.48	77.5

Table 4 DSC of pure TPU, untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH, and TPU/KF treated with 6% NaOH

	Pure TPU	Untreated TPU/KF	TPU/KF treated 2% NaOH	TPU/KF treated 4% NaOH	TPU/KF treated 6% NaOH	Notes
E_m	-12.92	-8.17	-6.25	-8.4	-8.86	E middle of peak
T_m	233.33	233.33	233.33	233.33	231.5	
E_f/mJ	-597.99	-134.74	-383.77	-296.96	-300.22	

**Fig. 11** DSC of *a* pure TPU, *b* untreated TPU/KF, *c* TPU/KF treated with 2% NaOH, *d* TPU/KF treated with 4% NaOH, and *e* TPU/KF treated with 6% NaOH

in the carbonyl region and ester groups on the surface of the fiber. Since hemicellulose is soluble in alkaline solutions, a substantial portion of uronic acid and fatty substances is removed by alkali treatment resulting in the disappearance of these peaks at $\sim 1,740$ and $1,250\text{ cm}^{-1}$ [15]. This is consistent with findings reported by others [4, 15, 16].

FTIR spectra of pure TPU and composites treated and untreated

FTIR spectra of TPU, untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH, and TPU/KF

treated with 6% NaOH are shown in Fig. 8. The main IR bands are summarized in Table 2.

TPU's carbonyl absorption displayed two bands at $1,730$ and $1,715\text{ cm}^{-1}$. The network structure formed between TPU and KF in the untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH and TPU/KF treated with 6% NaOH is confirmed by the difference in the intensity of the absorption [17]. More specifically, it is observed by an increase in the intensity of C=O absorption at $1,715\text{ cm}^{-1}$, perhaps this network is attributed to intermolecular hydrogen bonding.

The hydrogen bonding between TPU, untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH and TPU/KF treated with 6% NaOH causes a slight increase in the absorption frequency of the carbonyl group C=O (ester or associated urethane). Virgin TPU C=O absorbs at $1,715\text{ cm}^{-1}$, while untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH and TPU/KF treated with 6% NaOH at 1713 , 1703 , 1702 , and 1702 cm^{-1} , respectively. This is an indication that H-bonding degree decrease with increase in the concentration of NaOH [18]. In higher alkali concentration, excess delignification of natural fiber occurs, resulting in a weaker or damaged fiber [19].

Thermogravimetric analysis

Thermogravimetric analysis is one of the thermal analysis techniques used to measure the mass change, thermal decomposition, and thermal stability of composite material [23]. Figures 9 and 10 demonstrate TG and DTG for the pure TPU, untreated TPU/KF, TPU/KF treated with 2% NaOH, TPU/KF treated with 4% NaOH and TPU/KF

treated with 6% NaOH. Mass loss percentage of TPU, untreated and treated composites, is summarized in Table 3. For all formulations, a slight mass loss is observed below 100 °C, which may be attributed to evaporation of absorbed moisture. TPU/KF untreated composite showed more mass loss than the other formulations up to 1.9% at 100 °C compared to less than 1% in the other formulations. The composites decompose at a temperature less than pure TPU, and the untreated composite is more thermally stable than the treated composites due to decrease in decomposition temperature indicating that the interaction of fiber and matrix is decreased. Table 3 shows that untreated composite has a less mass loss in the temperature range between 300 and 500 °C than all treated composites. The decrease in thermal stability of natural fiber composite after alkali treatment is in line with the findings of Alvarez et al. [24].

Differential scanning calorimetry

DSC is a thermal analysis technique that measures the temperature and heat flow associated with transition in materials as a function of temperature and time. One of the most widely used applications of DSC is the determination of glass transition (T_g), melting point (T_m), and energy of fusion [25, 26]. T_m and energy of fusion are summarized in Table 4.

From the curves in Fig. 11, T_m of all materials was around 233.33 °C except for 6% NaOH treated TPU/KF that showed a T_m of 231.50 °C. The energy of fusion is determined by comparing the areas under the curve of the five different materials (Fig. 11). The untreated material show the smallest area, while the rest of materials show almost equal areas implying that they all have almost the same energy of fusion.

Conclusions

This study has yielded interesting results. NaOH fiber treatment resulted in enhancement of fiber morphology. This was supported by FTIR spectra of treated and untreated fibers. SEM and FTIR have shown that hemicellulose, lignin, and some undesirable components are removed from the surface of the fibers by NaOH treatment. Theoretically this may increase mechanical interlocking between fibers and matrix—as fibers become rougher after treatment.

In contrast, the mechanical characterization (i.e., tensile, flexural and impact) of the composite has shown a decline in mechanical properties with the increase in NaOH concentration. This was supported by FTIR spectra and the thermal study (i.e., TG and DTG) of the composites. FTIR

has shown that H-bonding degree decreased with increasing NaOH concentration. Untreated TPU/KF was shown by TG analysis to have a better thermal stability, indicating that the interaction of fiber and matrix decreased after NaOH treatment. NaOH treatment is a standard protocol for treatment of natural fibers; however, it is not suitable for the TPU/KF composite material probably due to the inherent amorphous behavior of TPU/KF treated composites.

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