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Polyester–Kenaf Composites: Effects of Alkali Fiber Treatment and Toughening of Matrix Using Liquid Natural Rubber

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ABSTRACT: In this study, polyester–kenaf fiber composites were prepared by adding various percentages of kenaf fiber in unsaturated polyester resin and subsequently cross-linked using methyl ethyl ketone peroxide and the accelerator cobalt octanoate. Liquid natural rubber (LNR) (3%) was added as a toughening agent. Kenaf fibers were treated with sodium hydroxide solution to improve the interfacial bonding between the fiber and the matrix. The mechanical properties of the composites were evaluated by impact and flexural testing. Environmental stress cracking resistance (ESCR) of composites in acid and base medium was also studied. Bonding mechanisms were assessed by scanning electron microscope and Fourier transform infrared analysis. It was found that the addition of LNR increased the impact strength and fracture toughness. Alkali fiber treatment was found to provide better impact and flexural strengths to the composites. Measurement of ESCR shows that the composite with acid medium has the fastest diffusion rate, followed by that with base medium, and then without medium.

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Figures 6 and 7 appear in color online: <http://jcm.sagepub.com>

KEY WORDS: kenaf, alkalization, polyester, toughened, liquid natural rubber.

INTRODUCTION

IN RECENT DECADES, growing environmental awareness has resulted in renewed interest in the use of natural materials for different applications. Increasingly, more stringent environmental policies have forced industries, such as the automotive, packaging, and construction industries, to search for new materials that can substitute traditional composite materials consisting of a plastic matrix and inorganic reinforcement [1]. The availability of bio-based structural composites offers the opportunity for environmental gains, reduced consumption of more expensive materials, lighter weight, improved insulation and sound absorption properties, and reduction in dependence on petroleum-based and forest product materials. The production of synthetic fibers, such as glass, aramid, and carbon fibers, is energy intensive and petroleum based. Kenaf (*Hibiscus cannabinus*) L., fiber is obtained from renewable resources, which is biodegradable, environment friendly, and offer CO₂ neutral life cycle [2]. The most rapidly expanding application for kenaf at present is as reinforcement in composites. The popularly used matrix materials in kenaf fiber composites ranges from thermoplastics, such as polypropylene and polyethylene, to thermosets, such as polyester and epoxy [3,4].

Unsaturated polyester resins are used in thermosetting system due to their low cost and adaptability to be transformed into large composite structures. The most frequently used reinforcement in this system is provided by sized glass fibers available as fabrics of various orientation, shapes, and densities. More recently, the possibility of using cellulosic fibers as reinforcement in composites has brought forth several studies. For instance, Dash et al. [5] reported the mechanical properties of jute fiber–unsaturated polyester composites. The authors optimized the fiber loading (60 wt%) that has the maximum tensile strength of 132 MPa and Young's modulus of 2.9 GPa for composites. Acha et al. [6] used jute fabric to reinforce unsaturated polyester resin. The results showed that the Young's modulus of all the composites increased significantly (fiber content 46–56 wt%); however, tensile strength of the composite was not improved.

Although polyester resins are widely used as a matrix in polymer composites, curing of these resins results in brittleness due to their high cross-linking level. The addition of a low molecular weight reactive liquid rubber can improve fracture toughness and impact strength of the cured polyester resin. The purpose of introducing dispersed rubber particles into the primary phase in these systems is to enhance their toughness. It is known that both size and volume fraction of the rubber particles affect the degree to which the polyester is toughened [7]. Dahlan et al. [8] has extensively studied the utilization of liquid natural rubber (LNR) as a compatibilizer on various natural rubber–polyolefin blends. They found that the LNR has the same microstructure as NR but with short chains of polyisoprene and a different molecular weight (M_w). The M_w for LNR is around 5000, whereas for natural rubber (NR) it is 900,000. The LNR with some active terminals like –OH is expected to react with the plastic particles and thereby bond the plastic particles to the NR matrix. Studies on NR and various polyethylene (PE) with LNR as a compatibilizer have successfully shown that LNR can act as a plasticizer or cross-linking agent, depending on its M_w [7,9].

Cellulose fibers contain many hydroxyl groups (–OH) and readily interact with water molecules by hydrogen bonding. In contrast to glass fibers, where water adsorption only occurs at the surface, cellulose fibers can interact with water throughout their bulk. It is difficult to entirely eliminate the absorption of moisture in composites without using expensive surface barriers [10]. Good wetting of the fiber by the matrix and adequate fiber–matrix bonding can decrease the rate and amount of water absorption in the interface region of the composite [11]. Removal of lignin from fiber has the potential to increase fiber–matrix adhesion within a composite due to the more open or rougher fiber surface, allowing for increased physical bonding with the matrix [12,13]. Alkali fiber treatments have been found to remove noncellulosic compounds and improve the strength of natural fiber composites [14,15].

The objective of this research is to determine the influence of alkali treatment of kenaf fibers and addition of LNR in matrix on the mechanical properties of composites.

MATERIALS AND METHODS

Preparation of Fibers

The kenaf fibers used had been harvested at 4 months of age. Fibers were then cut and ground with an Ika Werke MF10 heavy duty grinder (Staufen, Germany), and finally sieved into 300–500 micron sizes.

Surface Treatment of Kenaf Fiber

Sodium hydroxide (NaOH) solution (6%) was used to treat the surface of kenaf fiber. The kenaf fibers were soaked in NaOH solution for 2 h at 70°C and rinsed with distilled water to reduce the pH to 7. Then, the fiber was heated at 70°C for 24 h in an oven to remove all moisture and kept in a dessicator.

Preparation of Matrix

Unsaturated polyester Reversol UN 8100 P-8542CSH from Revertex Sdn. Bhd., Malaysia, was used in this research. For the composites without LNR, the polyester resin was mixed with the initiator methyl ethyl ketone peroxide (MEKP) and the hardener, 1% cobalt octanoate. For the composite with LNR, polyester resin and LNR were stirred for 30 min before mixing with the initiator and the hardener.

Preparation of Composites Board

Dried kenaf fibers were measured into 5%, 10%, 15%, 20%, and 25% (by weight) and mixed with polyester resin and LNR. The mixture was put into the mold and pressed at 10,000 psi pressure at 90°C for 15 min. The resulting board was cured at room temperature for 24 h and post cured at 60°C for 2 h, 90°C for 90 min, 120°C for 1 h, and 150°C for 30 min. The mechanical properties of each type of board were then evaluated after cutting the samples into the required shape for different tests.

Composite Characterization

Flexural strength was measured under a three-point bending approach using a universal testing machine according to ASTM D 79s0-91. The dimensions of the samples were $120 \times 20 \times 3 \text{ mm}^3$. The distance between the spans was 100 mm, and the strain rate was 5 mm/min. Impact testing was carried out with a Ray-Ran Pendulum Impact System (Warwickshire, UK) according to ASTM D 256. Fracture toughness was determined according to ASTM E 399 using a three-point bending flexural test. The strain rate was 2 mm/min. The K_{Ic} was determined by the following equation:

$$K_{Ic} = \frac{SPc}{BW} \cdot f(\Sigma),$$

where

$$f(\Sigma) = \frac{3 \Sigma^{1/2} \left\{ 1.99 - \Sigma(1 - \Sigma)(2.15 - 3.93 \Sigma + 2.7 \Sigma^2) \right\}}{2(1 + 2 \Sigma)(1 - \Sigma)^{3/2}},$$

$$\Sigma = \frac{a}{W},$$

where S is the span length, P_c the maximum load, B the sample thickness, and W the sample width. For mechanical testing, five specimens were tested for each batch to get the average values. Environmental stress cracking resistance (ESCR) tests were carried out according to ISO standard 22088-3:2006. Samples of the ASTM E 399 were prepared with the dimensions of $80 \times 4 \times 10 \text{ mm}^3$. The specimen was bent using a three-point bending jig mounted in a chemical environment chamber. The test was undertaken in control medium, 1% NaOH (pH 13.5) and 1% hydrochloric acid (pH 2.5) medium. Three environmental conditions were used in this test as:

- without medium (om)
- with acid medium (am)
- with base medium (bm)

After t_0 , defined as the time necessary to stabilize the loading condition, the chemical was rapidly introduced into the chamber under stress. The variation of the stress was recorded using a sensitive load cell 100 N connected to multimeter incrementally at $t_0 = 120 \text{ s}$, $t_1 = 120 \text{ s}$, $t_2 = 180 \text{ s}$, $t_3 = 360 \text{ s}$, $t_4 = 360 \text{ s}$, $t_5 = 600 \text{ s}$, and $t_6 = 600 \text{ s}$. When the stressed composite is exposed to a chemical environmental, polymer resistance will vary with time. Since micro-crazes, crazes, or cracks weaken the chemically exposed polymer, it will tend to have both a shorter time to rupture and a lower stress value, compared to the values for a control specimen (without medium). The test was performed in air, 1% NaOH for base medium (pH 13.5), and 1% hydrochloric acid for acid medium (pH 2.5). The test was carried out under laboratory conditions in an air-conditioned room, controlled at $23 \pm ^\circ\text{C}$ and 50% relative humidity. The E factor is defined as the load (F) in an acid or base media at the time after t_0 which is (t_1 , t_2 , t_3 , t_4 , t_5 , and t_6) divided by the load (f) in without medium and this equation is divided with the load in acid or base medium at t_0 which is divided with load at t_0 without medium.

$$E = \frac{F_{t_n}(\text{am@bm})/F_{t_n}(\text{om})}{F_{t_0}(\text{am@bm})/F_{t_0}(\text{om})}.$$

Morphological observation of the flexural fracture surface of the composite with different compositions was carried out by a scanning electron microscope (SEM) (Philips XL30, USA). Fourier transform infrared (FTIR) spectrum of samples was recorded using FT-IR spectroscopy from 4000 to 400 cm^{-1} using KBr disk.

RESULTS AND DISCUSSION

Impact Properties

The impact strength of matrix and composites are shown in Figure 1. It can be seen that the impact strength of the matrix was higher than that of the composites with lower fiber content, then the impact strength increased in general with increasing fiber loading. The lower values of impact strength at lower fiber content could be due to the introduction of flaws created by the fiber ends. These flaws act as stress concentrations and cause the bond between fiber and matrix to break. At higher volume fractions, the matrix is sufficiently restrained and the stress is more evenly distributed. This results in reinforcing effects outweighing the effects of stress concentrations and results in an increase in impact strength at higher fiber loading [16]. Although benefit was seen in higher fiber loading, the fiber loading was limited to 25 wt% due to the limited wet ability of the resin; and higher than 25 wt% (for example 30 wt%), the polyester resin could not wet and cover the fiber sufficiently. The impact strength of composites may also be influenced by many other factors, including the toughness of the reinforcement, the nature of the interfacial region,

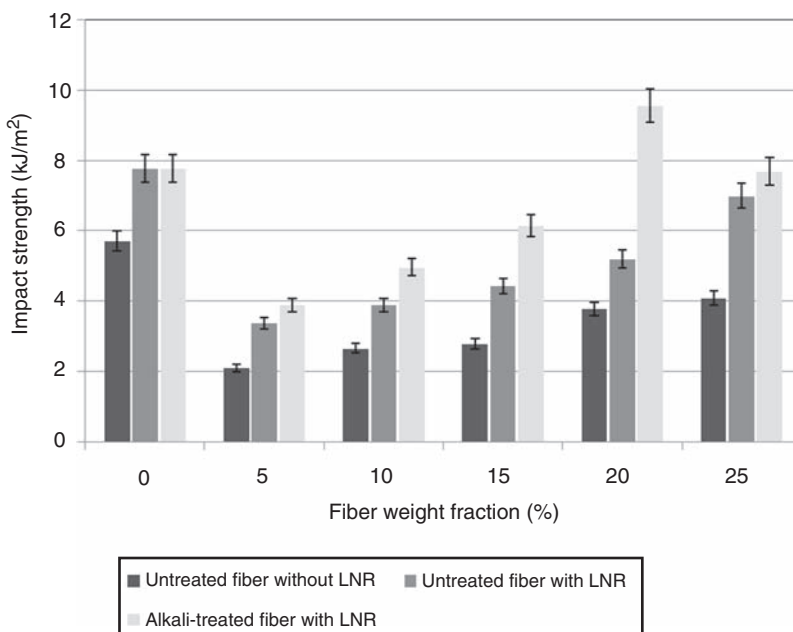


Figure 1. Impact strength of polyester–kenaf composite consisting untreated fiber, and unmodified matrix, untreated fiber with LNR modified matrix, and alkali-treated fiber with LNR modified matrix.

and the frictional work involved in pulling the fibers from the matrix [17]. Addition of LNR was found to improve the impact properties of composites probably due to the presence of rubber particles in the polyester which may have acted as an impact modifier in the composite. It can also be seen that alkali-treated fiber composites provided higher impact strength than that of untreated fiber. This behavior could be due to the difference in surface properties of the fiber. It has been evaluated elsewhere that the number of hydroxyl groups present at the surface of the natural fiber is only approximately 1% of the total number in cellulose. In the case of untreated fiber, more of these groups are covered by lignin and hemicelluloses, present in the primary wall of the fiber. During alkali treatment, alkali reacts with the cementing materials of the fiber, particularly hemicelluloses and lignin. Alkalization led to the removal of the outer surface of the fiber, which resulted in increased destruction of the mesh structure of the fiber, and splitting of the fibers into finer filaments. The breaking down of the fiber bundle increases the effective surface area available for wetting by the matrix. Thus, bonding between the fiber and the matrix at the interface may be improved, and resulted in an improved impact strength [18,19]. Removal of resinous lignin which is considered to be more brittle than cellulose [20] could well explain the increase in impact strength. The result shows that the optimum kenaf loading to produce the highest impact strength is 20% for alkali-treated fiber and 25% for untreated fiber. The maximum impact strength was found to be 9.6 kJ/m^2 for 20% treated kenaf–polyester composite.

Flexural Properties of Composite

Figures 2 and 3 show the effects of fiber loading, addition of LNR, and alkali fiber treatment on flexural properties of the polyester–kenaf composites. Although both

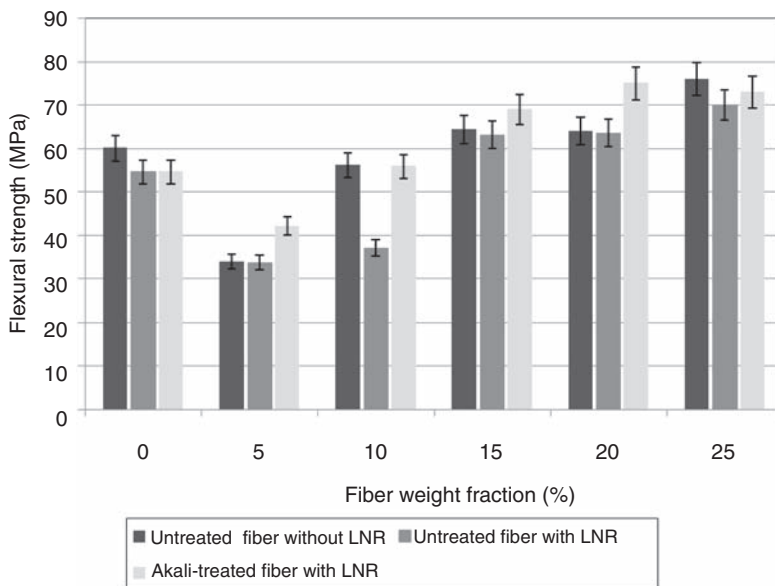


Figure 2. Flexural strength of polyester–kenaf composite consisting untreated fiber and unmodified matrix, untreated fiber with LNR modified matrix, and alkali-treated fiber with LNR modified matrix.

flexural strength and modulus was higher than that of composites containing lower amount of fiber loading, both flexural strength and modulus was found to increase significantly as the fiber volume fraction increased. As discussed earlier, at lower fiber content, below the critical fiber volume fraction, fiber ends act as stress concentrations and cause the bond between fiber and matrix to break. At higher volume fractions, the matrix is sufficiently restrained and the stress is more evenly distributed. This results in reinforcing effects outweighing the effects of the stress concentrations and results in an increase in flexural strength and modulus [9]. Although the flexural behavior of composites without LNR tended to behave like composite with LNR, there was generally a slight reduction of strength and modulus was found for composites with LNR. This reduction of flexural properties could be due to the plasticizing effect of rubber particles as evident from the impact properties where impact strength was found to be higher for composites with LNR. Strength of the thermoset material was also affected by the degree of cross-linking process where strength of the material will decrease if the cross-linking process is disturbed. Alkali-treated fiber composite provided higher flexural strength and modulus than untreated fiber composites. This is likely to be due to better mechanical interlocking and chemical bonding between fiber and the matrix as discussed earlier. The increase in flexural modulus is also thought to be due to an increase in crystallinity of the fiber cellulose making the fiber more rigid [21,22].

Fracture Toughness of Composite

The crack growth behavior of composites is compared in Figure 4 based on K_{Ic} values. Fracture toughness of composites both with and without LNR increased with increasing fiber volume; however, the values dropped after 15% of fiber loading for composites

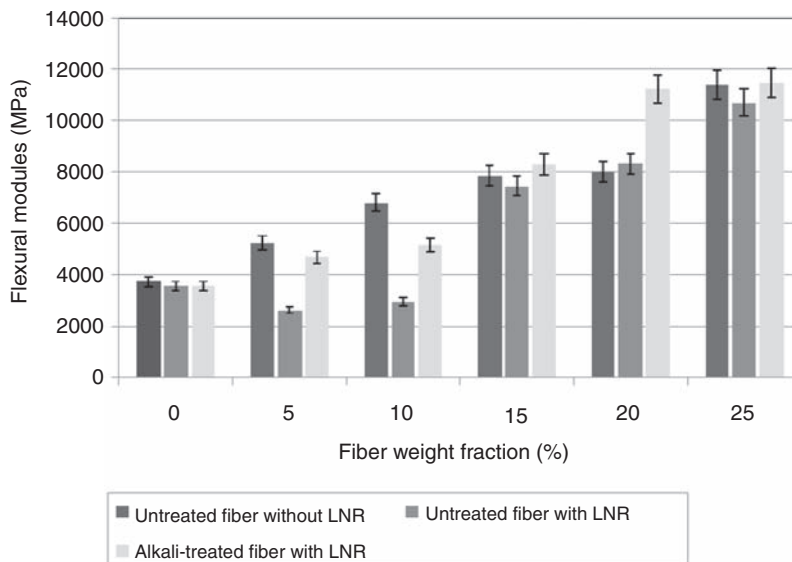


Figure 3. Flexural modulus of polyester–kenaf composite consisting untreated fiber and unmodified matrix, untreated fiber with LNR modified matrix, and alkali-treated fiber with LNR modified matrix.

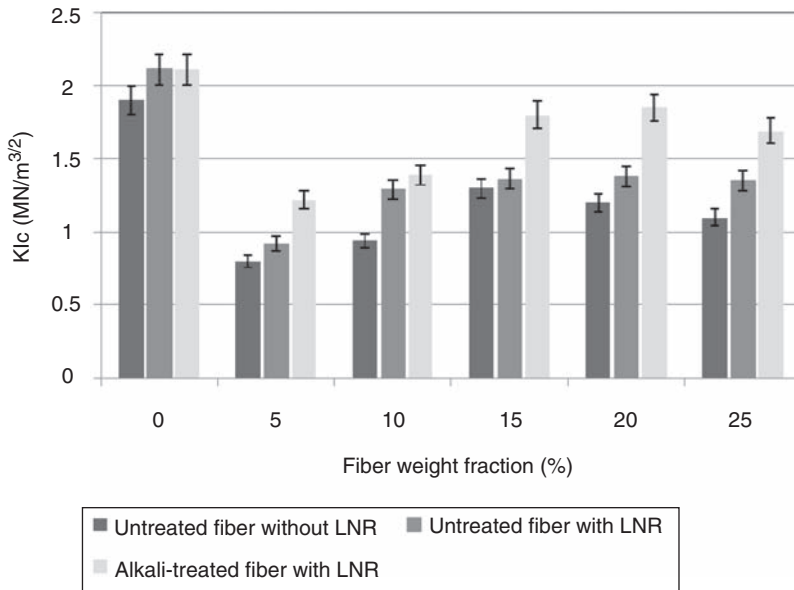


Figure 4. Fracture toughness (K_{Ic}) of polyester–kenaf composite consisting untreated fiber and unmodified matrix, untreated fiber with LNR modified matrix, and alkali-treated fiber with LNR modified matrix.

without LNR and 20% fiber loading for composites with LNR. The reduction of K_{Ic} at higher fiber content composites may be due to increased fiber–fiber contacts which would make it comparatively easier for the fiber to debond from the resin than for composites at lower fiber fraction composites. Therefore, larger force could be required for the fiber to be debonded from the matrix at lower fiber content. The addition of higher percentages of kenaf also could interfere with the cross-linking process and decrease the K_{Ic} values [3,22]. It was also noted that the values of fracture toughness were higher for composites with LNR, which could be due to the plasticizing effect of rubber particles as evident from the impact properties where impact strength was found to be higher for composites with LNR. Alkali-treated fiber composite provided higher fracture toughness than untreated fiber composites. Removal of resinous lignin which is considered to be more brittle than cellulose [20] could well explain the increase in fracture toughness.

Environmental Stress Cracking Resistance

ESCR is the premature initiation of cracking and embrittlement of a plastic due to the simultaneous action of stress and strain contact with a specific fluid. This phenomenon is also described as environmentally assisted cracking [23]. Figures 5 and 6 show the ESCR result on polyester–kenaf treated composite with LNR (PKTL) and polyester–kenaf composite with LNR (PKL). Figures 5 and 6 show that the stress values rapidly decreased whether the composites had acid or base media. The stress values of composites without medium declined over time, but the reduction was found to be less than that of composites with medium. Both composites, without LNR and with LNR, showed the same trend across the three different medium conditions. However, acid medium weakened the composite more rapidly than the base medium. Figure 5 indicates that over time, polyester

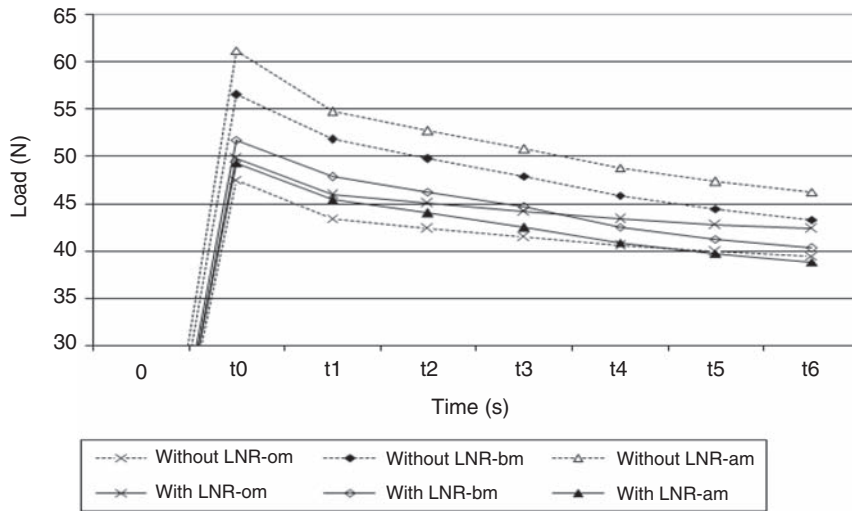


Figure 5. ESCR of composites with and without LNR.

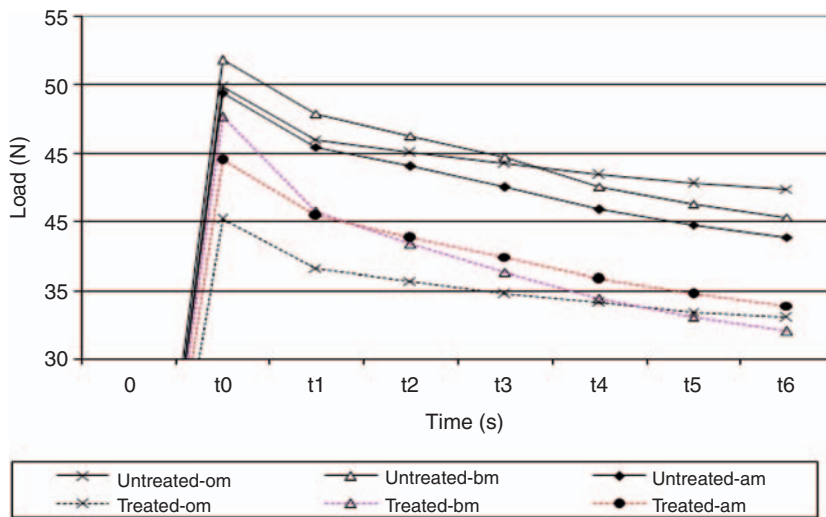


Figure 6. ESCR of composites consisting untreated and alkali-treated fiber composites.

composite with LNR decreased the stress by 25% with acid medium and 23% with base medium. Figure 6, however, shows that the stress value of polyester composite without LNR decreased 16% in acid medium and 20% in base medium. The stress value of composites with LNR declined about 17% with no medium, while that without LNR declined by about 14%. Figures 5 and 6 reveal that when the stressed composite is exposed to a chemical environment, the polymer resistance to acid and base varies with time. Since micro-crazes, crazes, or cracks weaken the polymer exposed to chemicals, it will tend to have both a shorter time to rupture and a lower stress value, compared to those of a

Table 1. E-factor value of untreated kenaf composite with LNR.

	PKTL (om)	PKTL (am)	PKTL (bm)
<i>t1</i>	1	1	1
<i>t2</i>	1	0.98	0.9
<i>t3</i>	1	0.97	0.86
<i>t4</i>	1	0.95	0.85
<i>t5</i>	1	0.94	0.83
<i>t6</i>	1	0.93	0.82

Table 2. E-value of treated kenaf composite with LNR.

	PKL (om)	PKL (am)	PKL (bm)
<i>t1</i>	1	0.98	1
<i>t2</i>	1	0.97	0.98
<i>t3</i>	1	0.95	0.97
<i>t4</i>	1	0.94	0.95
<i>t5</i>	1	0.92	0.93
<i>t6</i>	1	0.91	0.93

control specimen without medium. Results showed that both types of composites were affected more by the acid medium over time than by the base medium. Although the trend of ESCR was found to be similar for treated fiber compared to untreated fiber composites, treated fiber composites was less affected by the acid and base medium compared to untreated fiber composites, which could be due to better interfacial bonding and removal of lignin. *E* factor values are shown in Tables 1 and 2 and can be seen that all composites showed good resistance in different environments from *t1* to *t6* where the values were near to 1.

Infrared Spectroscopy

A series of FT-IR spectra have been obtained to detect the occurrence of any chemical interaction between polyester and kenaf in the formation of polyester–kenaf composite. The characteristic IR absorption peaks observed for neat polyester, untreated kenaf fiber, alkali-treated kenaf, and treated fiber composites system are shown in Figure 7. The strong IR absorption peak due to the presence of hydroxyl group appeared at 3436.02, 3435.44, 3430.10, and 3428.57 cm^{-1} . Decreasing intensity of hydroxyl group shows that there are interactions between the OH group and polyester resin. The peaks at 2929 and 2924 cm^{-1} represent the aliphatic C–H stretching of neat polyester and polyester–kenaf treated composite, respectively. The carbonyl groups of polyester C=O bonds was determined from the peak at 1731.1 cm^{-1} , while that of C=O bonds of composite was determined from the peak at 1730 cm^{-1} . The carbonyl region (1800–1600 cm^{-1}) reveals probably the presence of carbonyl group from the isoprene chain of LNR. The carbonyl absorption was observed from the both spectrums with difference in intensity. The network structure formed between polyester and treated kenaf to become polyester–kenaf treated composite is confirmed by the decrease in the intensity of C=O bond and appearance of band at

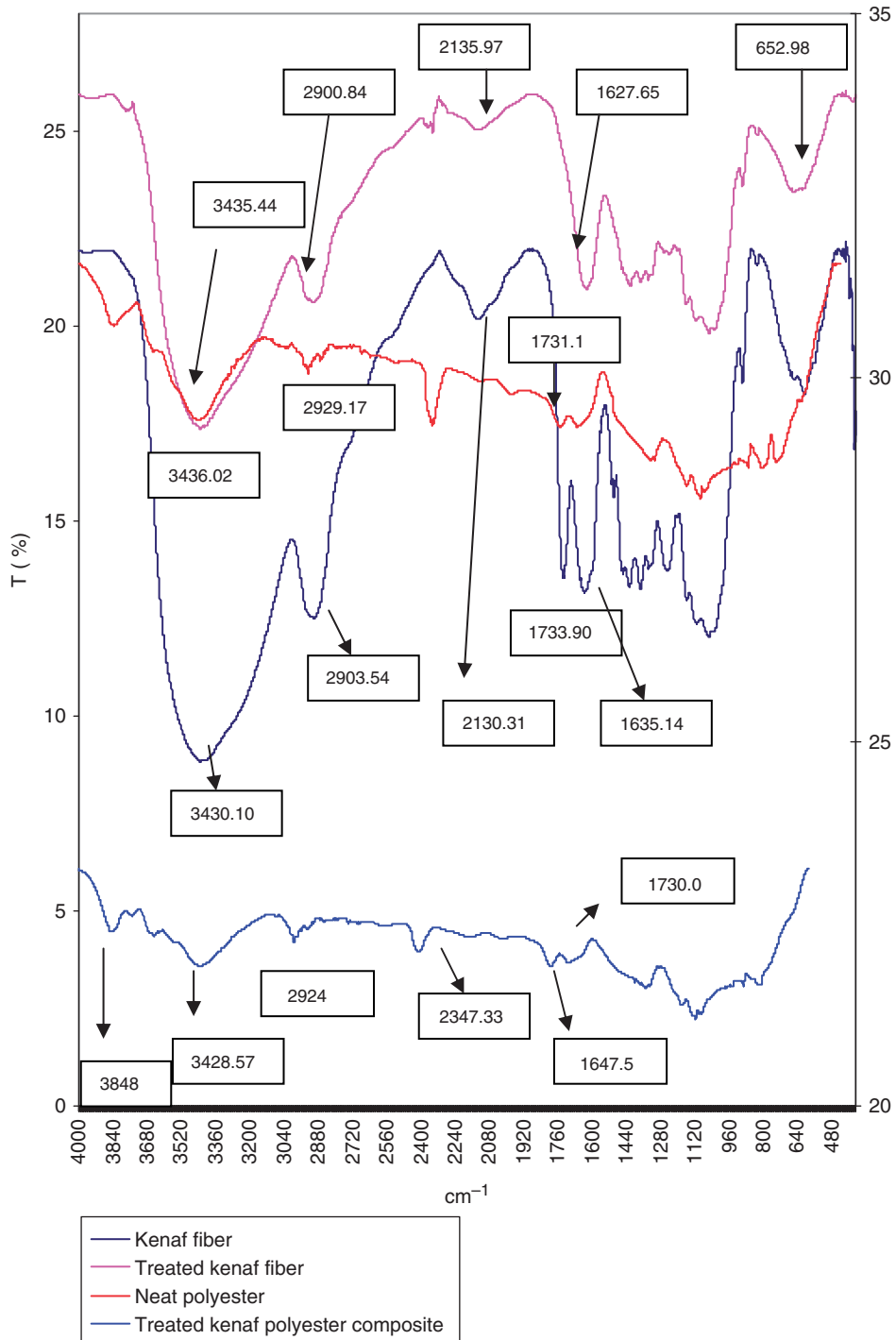


Figure 7. FT-IR peaks of treated kenaf–polyester composite.

1647.5 cm^{-1} , where conjugation of esters $\text{C}=\text{O}$ occurred. An aromatic functional group ($\text{C}-\text{C}$ stretch in ring) was observed from the absorption bands at 1600 and 1475 cm^{-1} and an alkane group (CH_3) from the absorption band at 1376 cm^{-1} for the composites with LNR. It can be noted that the aromatic functional group can also appear from matrix polymer. In this study, absorbance at 1730 cm^{-1} (stretching mode of the carbonyl groups) was chosen as an internal standard. Stretching mode of the carbonyl group ($\text{C}=\text{C}$) was observed from the absorption bands at 1600 and 1700 cm^{-1} . Hydroxyl group was observed for both peaks from the absorption bands at 3600–3300 cm^{-1} .

Raw fibers show the absorption band of carbonyl stretching at 1733.90 cm^{-1} and the band totally disappears with alkali treatment. Alkali treatment of kenaf fiber may induce two types of modifications: a more or less rapid swelling of various polymers, including pure cellulose parts, and a partial dissolution of ester containing polysaccharides, such as pectin.

The band at 1648 cm^{-1} assigned to absorbed water exhibits no difference regardless of the treatment, indicating that the amount of water in treated and untreated fibers is roughly identical [24].

Morphological Analysis

Figure 8 shows the flexural fracture surface of the composites. Good bonding was evident for both composites with and without LNR. Fiber fracture can be seen and there is an absence of hole and fiber pullout. However, for composites without LNR, a gap between the fiber and the embedded resin can be seen (Figure 8(a)). It is well known that unsaturated polyester networks shrink considerably after curing. This shrinkage reduces the specific volume of the matrix, and it results in free spaces between the matrix and the fiber. On the other hand, for composites with LNR, the region surrounding the cellulosic filler seems to be continuous with the matrix phase (Figure 8(b)). Figure 8(c) shows the SEM micrograph of a composite with treated kenaf fiber. The fiber surface shows a rough surface morphology which can be attributed to the removal of hemicellulose and lignin due to a strong interaction between the fiber and the matrix. The strong interface between fiber and matrix can prevent interfacial slippage, while surface roughness increases the adhesive bonding [25,26]. The morphological examinations of samples provide a strong evidence of good adhesion between the matrix and the filler.

CONCLUSIONS

The result shows that LNR is a good potential toughening agent for polyester resin. Impact, flexural, and fracture toughness properties were found to be improved significantly with increasing fiber volume fraction; however, optimum mechanical properties were found to be in the range of $\pm 20\%$ fiber loading. Fracture toughness and impact strength of polyester–kenaf composite noticeably increased with the addition of a small amount of LNR in composites. ESCR shows that the composites are more affected in acid medium followed by base medium. Alkalization of kenaf shows good properties on impact, flexural, and fracture toughness compared to untreated kenaf composite. Morphological analysis shows that alkalization on kenaf provided a good interaction between kenaf and polyester matrix and improved the interfacial bonding between the fiber and matrix,

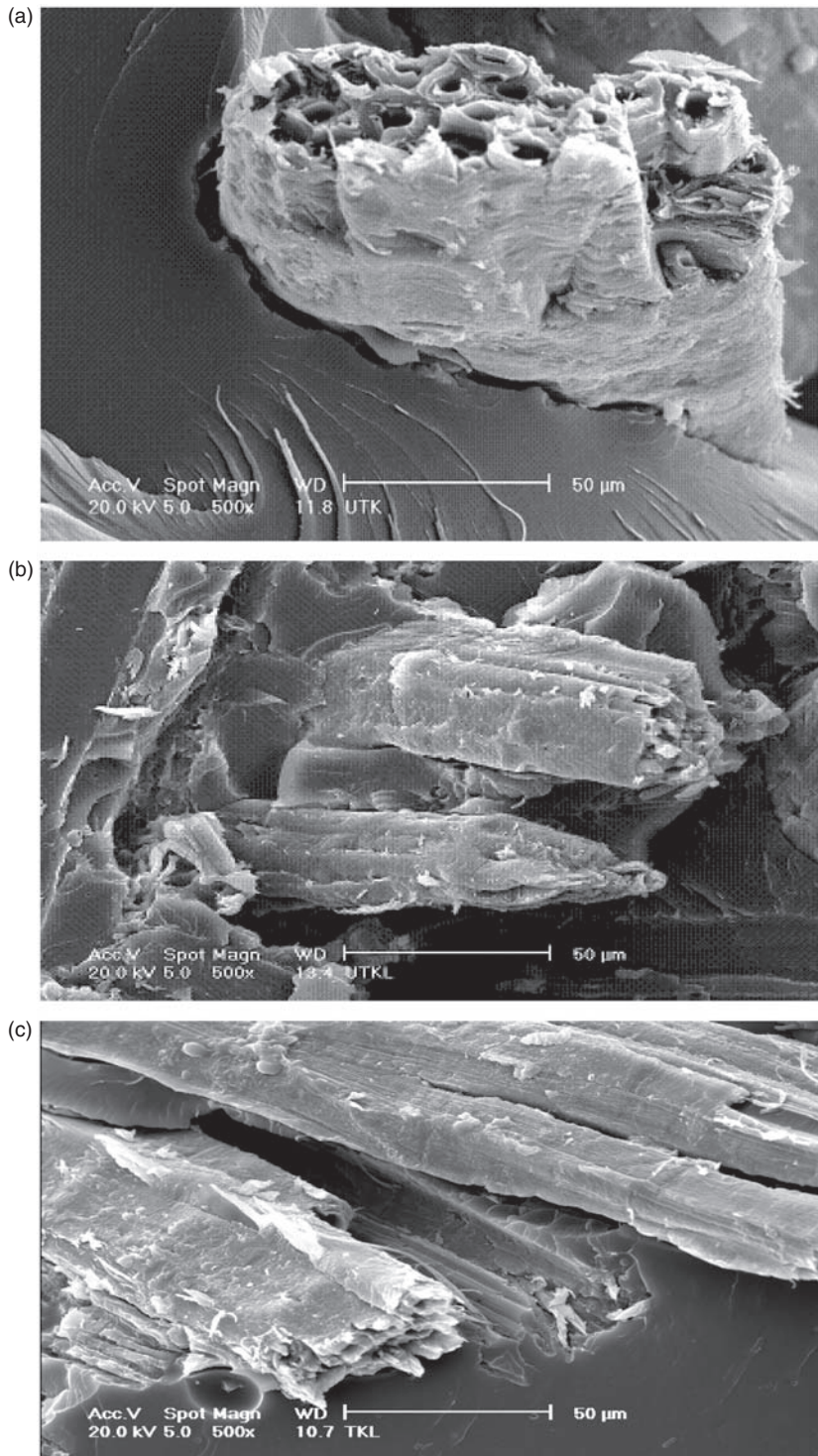


Figure 8. SEM micrographs of fracture surface of the composites (magnification 500×): (a) untreated fiber without LNR; (b) untreated fiber with LNR; and (c) treated fiber with LNR.

and thus, increased the mechanical properties of the composite. All composites showed good environmental behavior as shown by the E factor value from $t1-t6$ of almost 1.

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