

Effect of Chemical Treatment on Mechanical and Thermal Properties of Kenaf Fibre Mat Reinforced Unsaturated Polyester Biocomposites

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ABSTRACT: Kenaf fibre mat (KFM) reinforced unsaturated polyester (UPE) biocomposites have been prepared using hand lay-up and compression moulding technique. Sodium hydroxide solution (NaOH) with three different soaking time range from 0 to 6 hours were used to improve fibre-matrix adhesion properties of the KFM-UPE composites. The thermal stability and chemical changes of fibre constituents of the KFM-UPE composites was characterized with the thermogravimetric analysis (TGA) and Fourier transform infrared red (FTIR). Mechanical properties study revealed that, strength and modulus properties were increased with the NaOH treatment. The incorporation of the alkali KFM resulted in composites better tensile and flexural properties and 3 hours soaking time treatment showed the best results. Further investigation on morphological properties was also done to correlate the efficiency of chemical treatment used to improve the mechanical properties of the KFM reinforced UPE biocomposites.

Keywords: Mechanical properties, Thermal properties, Kenaf fibre mat, Unsaturated polyester, Biocomposites;

1. INTRODUCTION

For centuries, all natural fibres such as sisal, banana, jute, oil palm, kenaf, recycled jute and coir has been used globally in thermoset matrix natural fibre-reinforced composites [1]. The natural fibres are low cost, light weight, high specific strength, renewability, and biodegradability and these can increasingly promise their use for the production. Kenaf (*Hibiscus cannabinus*) is a warm season annual crop native to Africa and selected as an attractive alternative instead of man-made fibres such as glass and carbon [2]. Kenaf fibre (KF), obtained from processing the bark of the kenaf plant, offers advantages such as low density, ease of chemical modification, high toughness, good

thermal properties, acceptable specific strength, and biodegradability [3]. Kenaf have been used as non-woven mats in the automotive, textiles, fibreboard, civil and electronic industries [4]. All-natural fibres are thermally unstable compared to most synthetic fibres, and are limited to processing and working temperatures of 200 °C. If natural fibres do high performance biocomposite, they have decreased thermal stability; therefore, alkali treatment has been shown to slightly enhance the thermal stability of biocomposite. Alkali treatment also significantly improves the fibre surface properties but their usage might be limited for certain concentration and soaking time [5].

The present investigation dealt with alkali treatment effect on the mechanical and thermal properties of KFM reinforced UPE biocomposites. The influence of alkali soaking time was investigated. The result from this study is expected to inspire other researchers to further investigate the use of KFM as reinforcement in biocomposites industry.

2. MATERIALS AND METHODS

Reinforcement materials used for this work included KFM and unsaturated polyester (UPE) resin were supplied by Kenaf Agro Vet Sdn. Bhd, The UPE was cured with 2% methyl ethyl ketone peroxide (MEKP) from Merck as catalyst and 1 wt.% cobalt naphthalene (CN) from Sigma Aldrich as an accelerator. KFM was treated with immersion time of 0-6 hours range in 6% alkali (NaOH) solution at room temperature. Infrared spectra of each samples were

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Competing interests

The authors have declared that no competing interests exist.

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obtained in the range of 4000 - 400 cm^{-1} using a Fourier transform infrared spectrophotometry (FTIR) model NICOLET iZ10. FTIR was employed to determine functional groups presents in KF before and after alkali treatment. Dried untreated and alkali-treated KFM were cut in dimension of 150 mm x 150 mm and fabricated in the form of plate with thickness of 3 mm. The matrix-fibre ratio is 90:10 for KFM-UPE composites. Tensile and flexural properties were conducted by a universal testing machine

(UTM), model Instron 3366 with load capacity of 50 kN according to ASTM D3039 and D790, respectively. TGA study was carried out on a Mettler Toledo TGA/DSC2 at a constant heating rate of 10 $^{\circ}\text{C}/\text{min}$ under pure nitrogen flow at 20 ml/min. While, scanning electron microscopy (SEM) was used to characterize morphology study. Table 1 and 2 show typical properties of Reversol P9565 UPE resin and sample designation of KFM-UPE composites with different alkali soaking time (0, 3 and 6 hours).

Table 1 Properties of UPE (Reversol P9565)

Viscosity (Cp)	Density (g/cm^3)	Volume shrinkage (%)	Gel time at 25 $^{\circ}\text{C}$ (Min)	Tensile strength (MPa)	Tensile Modulus (GPa)	Elongation (%)
200 - 300	1.2	8.7	12-15	63.9 - 72	3.40 - 3.59	2.5 - 3.1

Table 2 Preparation of composites for untreated and treated KFM groups

Designation	Sample Justification
UK C	Untreated KFM-UPE
ALK3 C	Alkali-treated KFM-UPE in 3 hours soaking time
ALK6 C	Alkali-treated KFM-UPE in 6 hours soaking time

3. RESULTS AND DISCUSSION

Fig. 1 shows the FTIR spectra in the 4000 - 400 cm^{-1} range for untreated and alkali-treated KFM from 3 hours to 6 hours. A broad absorption band at 3369.14 cm^{-1} is due to O-H stretching vibrations of cellulose and hemicelluloses. The region 3500 - 2500 cm^{-1} for the untreated and treated kenaf fibres is related to OH and CH_2 groups. As for alkali-treated kenaf fibres, the 3369.14 cm^{-1} band assigned to the OH group decreased after this peak. This disappearance is a consequence of the hemicelluloses are removed from the fibre and the formation of ionic carboxylates in the incompletely extracted samples, in which instance the corresponding peak appears at lower frequencies (1597.31 cm^{-1}). It is interesting to observe that the peak at 1243.61 cm^{-1} disappears in alkali-treated KFM. Alkali treatment removes the waxy epidermal tissue, adhesive pectins and hemicelluloses that bind fibre bundles to each other [6]. It can thus be summarized that the NaOH chemical treatments remove most of the lignin and hemicellulose components, which help to improve the mechanical properties of KFM-UPE composites.

Fig. 2 shows TGA curves of UPE matrix and KFM-UPE composites under nitrogen in the 25-600 $^{\circ}\text{C}$ range. From the curve, the first stage of weight loss started from 30-110 $^{\circ}\text{C}$ was due to the release of moisture content by the fibre. The second stage major degradation occurred with the temperature range at about 200-300 $^{\circ}\text{C}$, which was related to the degradation of lignin and hemicelluloses of the fibre. The last stage of weight loss temperature ranges at about 340-380 $^{\circ}\text{C}$, indicates the degradation of α -cellulose and other non cellulosic materials from the fibre [7]. From the Table 3, it was examined that, the final degradation temperature (T_{peak}) for alkali-treated KFM-UPE composites has shifted to higher range of temperature between 475 and 483 $^{\circ}\text{C}$ as compared with untreated composites which is 384 $^{\circ}\text{C}$. The result proved that, the presence of alkali KFM in UPE composite has improved their thermal properties compared to untreated KFM filled UPE composites. The increase in thermal stability after alkali treatment can be attributed to the removal of the amorphous structure of hemicellulose which was more aware of heat than other crystalline cellulosic components [8].

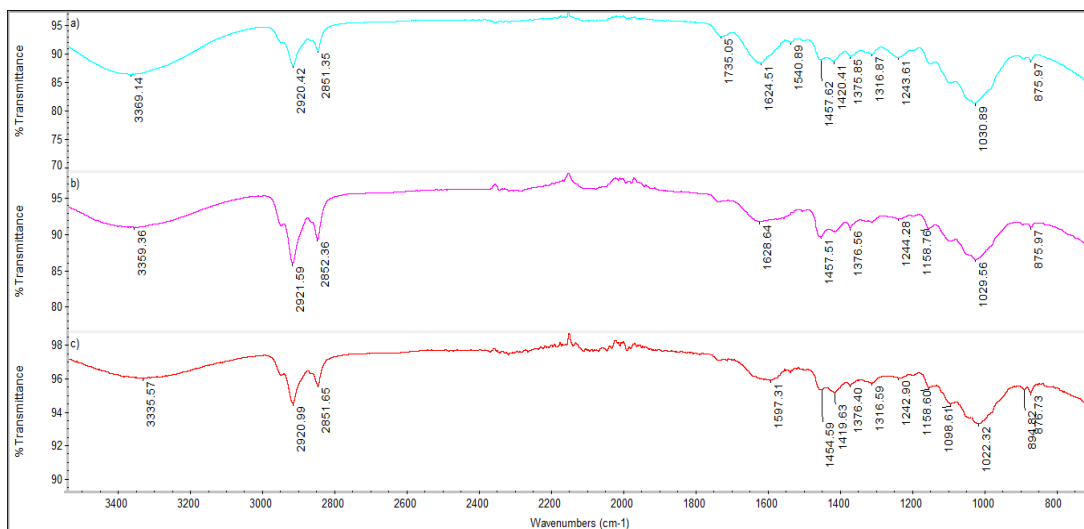


Fig. 1 FTIR spectra of KFM (a) raw and with NaOH treatment in (b) 3 hours and (c) 6 hours

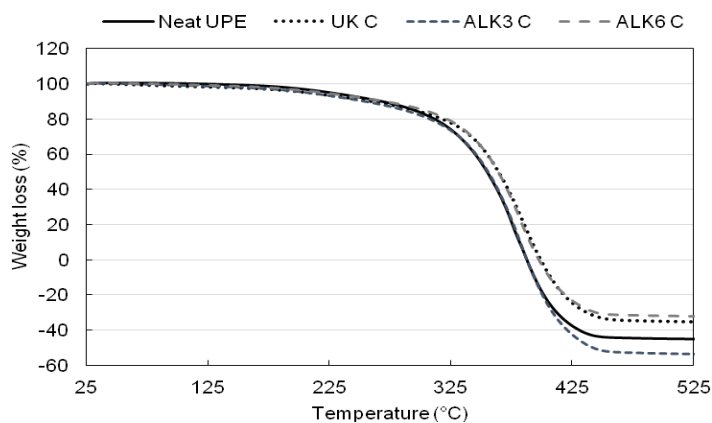


Fig. 2 TGA thermograms of UPE and its composites after alkali treatment in different soaking times

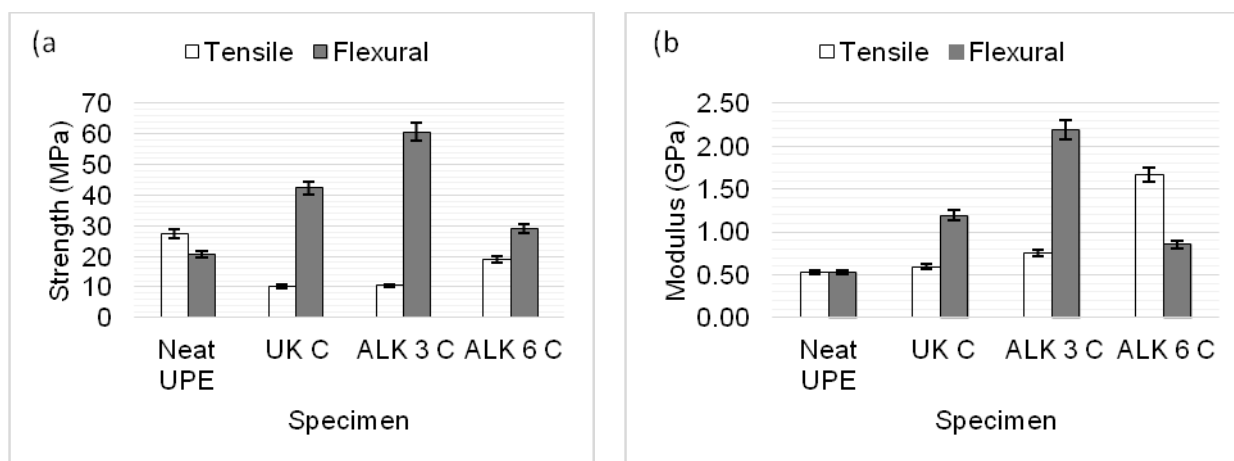


Fig. 3 Mechanical properties of of UPE and its composites after alkali treatment in different soaking times (a) Tensile and flexural strength and (b) Tensile and flexural modulus

Table 3 Thermal properties and char residue data of neat UPE and KFM-UPE composite by TGA

Sample code	T_{initial} (°C)	R_{peak} (%/min)/ T_{peak} (°C)	Char residue (%)	
			450 °C	600 °C
Neat UPE	150	0.2957/378	44.2	45.6
UK C	170	0.2598/384	33.9	36.0
ALK3 C	150	0.3111/483	52.5	54.3
ALK6 C	150	0.2853/475	31.1	32.5

Tensile and flexural properties of UPE and its composites are shown in Fig. 3. The strength and modulus properties (tensile/flexural strength and modulus) for all alkali-treated KFM composites were enhanced compared to the untreated KFM composites. At 3 hours soaking time, the alkali-treated KFM composite showed 42% improvement in strength and 83% in modulus properties.

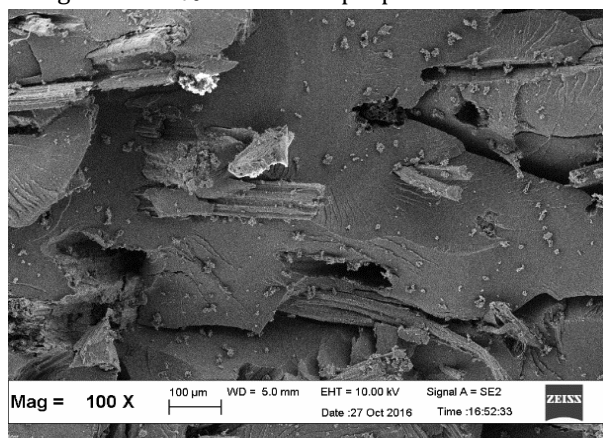


Fig. 4 SEM micrograph of tensile fractured surface of untreated KFM-UPE composite

This was possibly due to the removal of hemicelluloses, lignin and cellulosic constituents from the fibre after alkali treatment. As a result fibres became more hydrophobic and increased adhesion at the interface between the fibre and matrix. Several authors reported similar observations on improved flexural strength properties after alkalization of reinforcing fibres [9,10]. It was interesting to observe that, the alkali-treated KFM-UPE in 3 hours is good enough to achieve the highest tensile and flexural strength compared to untreated and 6 hours alkali-treated KFM. The increase in strength properties could be attributed to a greater fibre-matrix interaction and physical bonding as a result from removal of cementing materials and increased the surface roughness of KFM by application of NaOH [11,12].

Figs. 4 and 5 showed SEM micrographs of tensile fractured surface of untreated and alkali-treated KFM-UPE composites. SEM observations indicated that there was a considerable difference in the fibre-matrix interaction between 3 hours (Fig. 5a) and 6 hours (Fig. 5b) of giving treatment. Some gaps between fibre and matrix were clearly found for 6 hours alkali-treated composites which are responsible for the low mechanical properties.

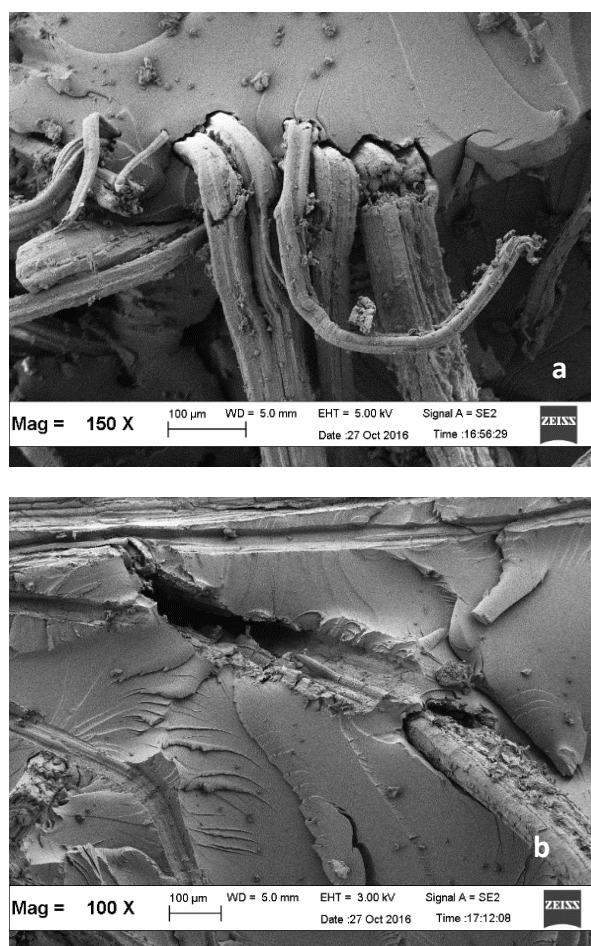


Fig.5 SEM micrographs of tensile fractured surface of (a) 3 h alkali-treated KFM-UPE composite and (b) 6 h alkali-treated KFM-UPE composite

Meanwhile, 3 hours alkali-treated composites showed better fibre-matrix adhesion and small gaps between fibre and matrix are clearly observed which are responsible for higher mechanical properties. Without the presence of chemical treatment, there are a lot of fibre pull out and fibre splitting were clearly observed as shown in Fig. 4.

4. CONCLUSIONS

In this work, the effect of alkali treatment on mechanical and thermal properties of KFM reinforced UPE biocomposites has been investigated. Alkali soaking time at constant concentration was evaluated. From the result analysis, it can be concluded that, the application of 3 hours of alkali-treated KFM is good enough in contributing the highest mechanical and thermal properties of KFM-UPE composites. It was believed that, a good fibre-matrix adhesion which was observed by using SEM is responsible for the highest mechanical properties.

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