ANHYDRIDE MODIFICATION OF CULTIVATED KENAF BAST FIBERS: MORPHOLOGICAL, SPECTROSCOPIC, AND THERMAL STUDIES

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Kenaf bast fiber was chemically modified by using propionic and succinic anhydrides. Five retention times were compared: 30, 60, 120, 180, and 240 minutes at 100°C. Confirmation of anhydride modification was established by the weight percent gain (WPG) and was further confirmed by Fourier Transform Infra-Red (FT-IR) spectroscopy. Based on WPG, succinylated fibers exhibited higher WPG than propionylated fibers. The results of WPG for both succinylated and propionylated fibers showed that 180 minutes was an optimum time for modification, yielding the highest WPG. The thermal stability of modified fibers was characterized with Thermal Gravimetric Analysis (TGA). Succinylated fibers showed better thermal stability than propionylated fibers. Anhydride modification also enhanced the fiber smoothness, as demonstrated by Scanning Electron Microscopy (SEM) analysis. Succinylated fibers showed a much smoother surface as compared to propionylated fibers and untreated fibers. Anhydride modification significantly decreased the contact angle of kenaf bast fibers, thus imparting good adhesion characteristics to the fibers.

Keywords: Kenaf bast fibers; Succinylation; Propionylation

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INTRODUCTION

Natural fibers have become well established recently as sustainable resources used to substitute glass fiber. Recyclability of natural fibers is an added benefit to make them very useful resources towards the establishment of a sustainable world. When natural fibers are closely compared to inorganic fibers, they present some well-known advantages such as lower density and cost. They are less abrasive to processing equipment, harmless, biodegradable, renewable, and their mechanical properties can be comparable to those of inorganic fibers. Furthermore, they are recyclable, easily available in most countries, subject to easy fiber surface modification, and relatively non-abrasive (George et al. 2001; Corrales et al. 2007; Li et al. 2008).

Natural fibers, especially bast fibers such as kenaf, flax, hemp, jute, and many others have been applied by some researchers as fiber reinforcement for composites in recent years (Li et al. 2007). Table 1 shows the mechanical and physical properties of various natural fibers.

Table 1. Mechanical and Physical Properties of Various Natural Fibres (Hattalli et al. 2002; Hoareau et al. 2004)

Fiber	*Tensile Strength (MPa)*	*Young's Modulus (GPa)*	*Elongation at Break (%)*	*Density (g cm^{-3})*
Abaca	400	12	3–10	1.5
Alfa	350	22	5.8	0.89
Bagasse	290	17	–	1.25
Bamboo	140–230	11–17		0.6–1.1
Banana	500	12	5.9	1.35
Coir	175	4–6	30	1.2
Cotton	287–597	5.5–12.6	7–8	1.5–1.6
Curaua	500–1,150	11.8	3.7–4.3	1.4
Date Palm	97–196	2.5–5.4	2–4.5	1–1.2
Flax	345–1,035	27.6	2.7–3.2	1.5
Hemp	690	70	1.6	1.48
Henequen	500 ± 70	13.2 ± 3.1	4.8 ± 1.1	1.2
Isora	500–600	–	5–6	1.2–1.3
Jute	393–773	26.5	1.5–1.8	1.3
Kenaf	930	53	1.6	–
Nettle	650	38	1.7	–
Oil Palm	248	3.2	25	0.7–1.55
Piassava	134–143	1.07–4.59	21.9–7.8	1.4
Pineapple	1.44	400–627	14.5	0.8–1.6
Ramie	560	24.5	2.5	1.5
Sisal	511–635	9.4–22	2.0–2.5	1.5

Natural fibers such as kenaf are getting a lot of attention in the composites industry, since they can be applied as reinforcement or fillers in polymer composites. Kenaf (*Hibiscus Bcannabinus* L.Malvaceae) is one of the fast-growing plants classified taxonomically in the *Fucaria* section of *Hibiscus*, with heights of 5 to 6 m in about 4-5 month (Alexopoulou et al. 2000; Amaducci et al. 2000; Webber and Bledsoe 2002). It is a traditional third-world crop, after wood and bamboo, which is poised to be introduced as a new annually renewable resource for industrial purposes in the so-called developed economies.

Kenaf is a warm-season annual fibre crop growing in temperate and tropical areas. Some varieties of kenaf can be found in Malaysia. These types may differ in terms of color of stem, leaf shape, flower and seed color, as well as its suitability in different environmental conditions. Kenaf sp.V36 is a kenaf variety that is widely planted in Malaysia. It was found to be the most suitable for commercial-scale production due to the climate in Malaysia.

Realizing the diverse possibilities of commercially exploitable derived products from kenaf, the National Kenaf Research and Development Program in Malaysia has been formed in an effort to develop kenaf as a possible new industrial crop. The government has allocated RM12 mil for research and further development of the kenaf-based industry under the 9th Malaysia Plan (2006-2010) in recognition of kenaf as a commercially viable crop (Edeerozey et al., 2007).

Kenaf is composed of two distinct fibers, bast and core. It is a fibrous plant, consisting of an inner core fiber (60 to 75%), which produces low quality pulp, and an outer bast fibres (25 to 40%), which produces high quality pulp, in the stem (Abdul Khalil et al. 2010). Kenaf bast and core are quite different with respect to their chemical compositions. The chemical analysis data for the kenaf core and bast are illustrated in Table 2. Kenaf bast was chosen in this research due to many advantages found on its properties. As shown, the kenaf core fibers are higher in holocellulose and lignin, while kenaf bast fibers are higher in α-cellulose. The α-cellulose content in bast fiber, at 55%, is higher than in the core, at 49%. High α-cellulose content is believed to provide high strength in paper formation and other fiber-based end products (Abdul Khalil et al. 2010). Besides that, in a study of the fiber dimensions of annual plants and agricultural residues Ververis et al. (2004) found that the slenderness ratio (fiber length/fiber diameter) of kenaf bast fibers is comparable to those of softwood fibers and clearly higher than that of kenaf core fibers. Moreover, according to Villar et al. (2009), the success of kenaf has been attributable to the quality of its bast fibers, which have low lignin content.

Table 2. Chemical Composition of Kenaf Core and Bast Fibers (Tsoumis 1991)

Chemical Composition	Kenaf Core (%)	Kenaf Bast (%)
Holocellulose	87.2	86.8
α-Cellulose	49.0	55.0
Lignin	19.2	14.7

However, the major limitations of using these fibers as reinforcements in such matrices include poor interfacial adhesion between polar, hydrophilic fibers and a non-polar, hydrophobic matrix, as well as difficulties in mixing due to poor wetting of the fibers with the matrix (John et al. 2008). The role of the matrix in a fiber-reinforced composite is to transfer the load to the stiff fibres through shear stresses at the interface. This process requires a good bond between the polymeric matrix and the fibers. Poor adhesion at the interface means that the full capabilities of the composite cannot be exploited, and leaves the structure vulnerable to environmental attacks that may weaken it, thus reducing its life span (Wambua et al. 2003). Thus, in order to overcome this problem, chemical modification of fibers is required. Chemical modification can be an alternative to alter the cell wall structure and surface chemistry of the fibers (Ismail et al. 2001).

EXPERIMENTAL

Materials

The kenaf (sp.V36) used in this research was collected from a local plantation source, Nibong Tebal Paper Mills (NTPM), Seberang Prai, Penang, Malaysia. Propionic anhydride, succinic anhydride, sodium formate, and N,N-dimethylformamide were purchased from Mega Makmur (Malaysia).

Fiber Treatments

Kenaf bast fibers were separated manually from the inner core, and then the fibers were refined using a Sprout Bauer refiner. Refining was conducted with the disc gap at 0.1mm. After that, the mat fibers were formed into a sheet by using a decker box. The bast fibers were dried first because the presence of moisture in fibers will disturb the reaction. Five sets of kenaf mats were soaked in the solution of the propionic anhydride or succinic anhydride in N,N-dimethylformamide in the presence of the catalyst sodium formate (10:1). The reaction was carried out at 100 °C. Five different retention times were set: 30, 60, 120, 180, and 240 minutes. After the modification process, the samples were washed using dry ethanol for another 3 hours to rinse away any remaining acid. The samples were air dried for a few hours before being oven dried for 24 hours with a temperature of 80°C. The weight was measured and recorded. Statistical analysis was carried out for kenaf fibre treatment with succinic and propionic anhydride by using SPSS 15 software. Mean and standard deviations of weight percent gain(WPG%) were calculated for succinic and propionic anhydride vs treatment time by using SPSS 15, and results are reported on a line graph (Fig. 1).

Structure and Thermal Analysis

Fourier Transform Infra-Red (FT-IR)

An FT-IR spectroscope, Nicolet Avatar 360, (USA), was used to examine the functional groups present in the fibers. A Perkin Elmer spectrum 1000 was used to obtain the spectrum of each sample. 1 mg of powdered fiber samples was mixed with 100 mg of KBr powder. The powder mixtures were then pressed into transparent thin pellets. FT-IR spectra of each sample were obtained in the range of 4000 to 400 cm^{-1}. Spectral outputs were recorded in the transmittance mode as a function of wavenumber.

Thermal Gravimetric Analysis (TGA)

Kenaf fibre samples were put in oven for 24 hrs at 80 ^{o}C before TGA. The fiber samples were characterized for their thermal stability using a thermogravimetric analyzer (TGA), model 2050, (TA Instruments, New Castle, DE). All three samples were scanned from 30 ^{o}C to 800 ^{o}C at the rate of 20 ^{o}C/ min. All TGA tests were carried out in a nitrogen environment.

Scanning Electron Microscope (SEM)

Studies of the morphology of the treated and untreated fibers were investigated using an SEM model S 360, Leica Cambridge Ltd, at an accelerating voltage of 5 kV. Surfaces of the specimen samples were sputter-coated with gold prior to their observation.

Contact angle

Contact angle was measured with a KSV CAM 101 Series Drop Shape Analysis Contact Angle Meter. Each non-woven mat of kenaf bast fibers was pressed at 200 kg/cm^2 to make sure the surface of the kenaf mat was flat. The fibers sample surface was targeted. A dropper was pressed to place a desired droplet of distilled water on the sample, and the drop was observed on a computer screen until it touched the surfaces. Tests were at room temperature. The value of the contact angle was calculated by the computer.

RESULTS AND DISCUSSION

Weight Percent Gain (WPG)

The weight percent gain was increased after modification with propionic or succinic anhydride. In preliminary trials, five sets of kenaf mat were soaked in the chemical solution with the temperature of 100°C. Five different retention times were set, which were 30, 60, 120, 180, and 240 minutes. The preliminary results obtained by statistical analysis are illustrated in Fig. 1.

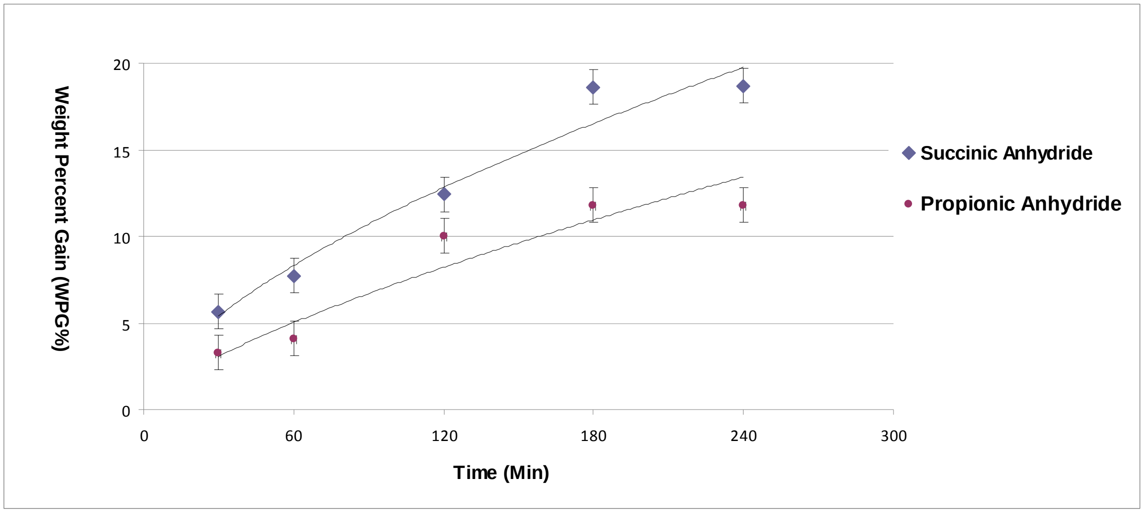

Fig. 1. Weight Percent Gain (WPG%) of succinylated and propionylated kenaf bast fiber at 100°C

From these preliminary results (Fig.1), it can be seen that the optimum time, in order to achieve the highest WPG of each sample tested, was 180 minutes. The effectiveness of treatment increased in the form of weight percent gain (WPG) until 180 min, and after that it remained constant. The sample from optimum retention time, which showed the highest WPG, was chosen for further evaluation. According to Farahani and Taghizadeh (2010), the esterification with higher WPG increased all the parameters significantly. WPG should be considered to provide sufficient improvement in the desired properties of fibers. Besides that, proof of whether modification had taken place depended on the WPG value (Abdul Khalil et al. 2001).

Based on the results, kenaf bast fiber treated with succinic anhydride gave much higher WPG as compared to fiber treated with propionic anhydride. The reason was due to adduction of a 4-carbon chain with carboxyl group to the succinylated fibres, whereas propionylation adducted only a 3-carbon hydrophobic group during modification. Succinylation is able to swell the fiber structure more effectively, making reactive chemicals sites more accessible and therefore, enhancing the reaction rate of modification which result in higher WPG. Besides, the succinylation reaction does not produce a byproduct, whereas the propionylation reaction produces propionic acid.

Therefore, the lower WPG of propionylated could be explained as being due to solubilization of the fiber component by propionic acid (Abdul Khalil et al. 2010). Figure 2 (a and b) displays the reaction schemes for succinic and propionic anhydride modifications, respectively.

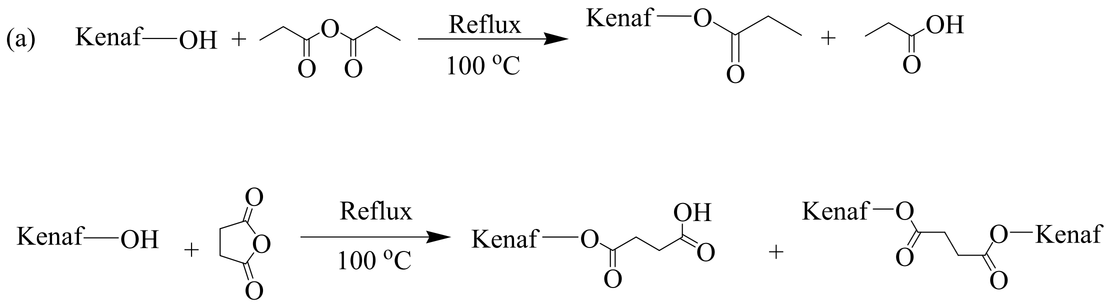

Fig. 2. Reaction of kenaf bast fiber with (a) propionic anhydride, and (b) succinic anhydride

Fourier Transform Infra-Red (FT-IR) Analysis

Based on Fig. 3, the treatment of kenaf bast fibers with propionic and succinic anhydride led to increases in their peak frequencies in the two major regions. The first region was between 1726 and 1741 cm^{-1}, which proved the existence of the carbonyl group (C=O) (Abdul Khalil et al. 2001). The increment of this peak demonstrated the evidence of the anhydride modification, which entails esterification of the hydroxyl groups, and which resulted in an increased stretching vibration of the carbonyl group present in ester bonds. Besides that, the high-frequency region of carbonyl absorption at around 1700 cm^{-1} for untreated fibers can be attributed to the presence of wax esters (Dubis et al. 1999). The second major region was around 1370 to 1383 cm^{-1}, which showed an increase of the carbon-hydrogen (C-H) bond bending region. According to Abdul Khalil et al. (2010), a decrease in the intensity of the hydroxyl groups (O=H) absorption band around 3500-3100 cm^{-1} indicated that the OH groups were reduced, which also was observed in the present research. The untreated fibers exhibited a broad peak of the OH at 3411 cm^{-1}, and peak frequencies were then been shifted after modification indicated the decrease of OH in modified fibers. Table 3 provides guidance for typical interpretation of several major absorption bands for agricultural fibers.

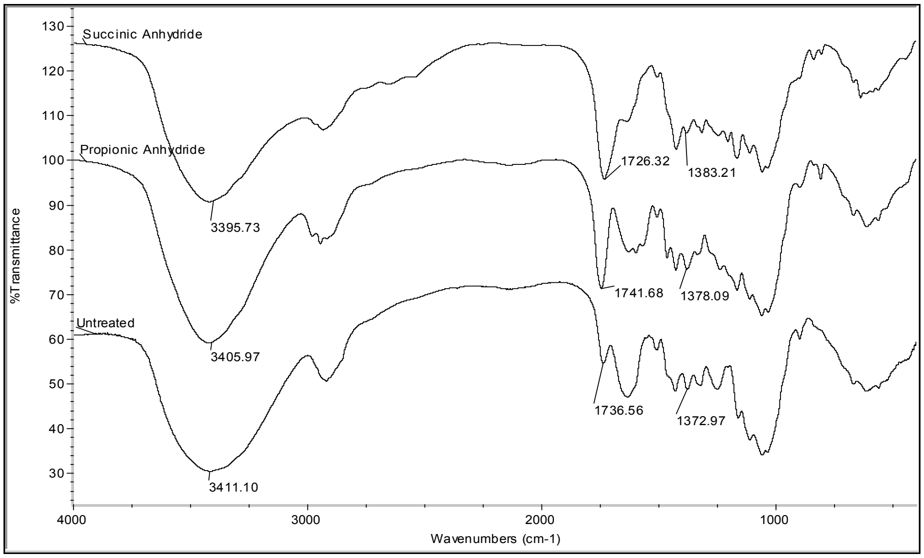

Fig. 3. FT-IR spectra of untreated, propionylated and succinylated of kenaf bast fibers

Table 3. Peak Assignments for Several Major Absorption Bands Typical for Agricultural Fibers (Ismail et al. 2001)

Frequency (cm^{-1})	Assignments
3500-3100	OH stretching
3090-2600	CH stretching of CH_2 and CH_3
1750-1700	C=O stretching
1600-1400	Aromatic ring stretching in lignin
1400-1300	CH deformation of CH_2 and CH_3
1300-1000	C-O stretching
1280-1070	C-O-C stretching
1300-1030	OH deformation

Thermal Gravimetric Analysis (TGA)

Thermal degradation studies are necessary, as many applications depend on thermal stability of cellulosic components. The TGA of untreated, propionic anhydride treated, and succinic anhydride treated samples of kenaf bast fiber are shown in Fig. 4. The first stage of mass loss was assigned to the evaporation of water from the samples (Xiao et al. 2001). The initial weight loss was found to be around 95 °C to 100 °C, and the results were similar in all samples. This was probably due to dehydration of the samples and degradation of hemicelluloses (Ndazi et al. 2007). The lignocellulosic materials decompose thermochemically between 150 °C and 500 °C: hemicellulose, mainly between 150 °C and 350 °C, cellulose between 275 °C and 350 °C, and lignin between 250 °C and 500 °C (Kim et al. 2004).

The temperature at which specific weight losses occurred are summarized in Table 4 and shown to be consistently higher for treated fibres compared to untreated fibres. The initial degradation temperature for treated fibres shifted to a higher temperature well over 255 °C to 268 °C, compared with the untreated fibres (247 °C), indicating the higher thermal stabilities of the treated fibres. Final degradation (T_{FDT}) occurred at 455 °C for untreated and between 430 °C to 471 °C for treated fibres due to complex reactions. Considering the intermediate weight loss (%) in untreated kenaf bast fibers, the mass fell rapidly as compared to propionylated and succinylated fibers. This is due to the hemicellulose degradation occurring in untreated fibers and hemicellulose and cellulose degradation in treated fibres; these results clearly proved that untreated fibers have weak bonding, which leads to faster degradation.

Table 4. Thermal Properties of Untreated, Succinylated, and Propionylated Kenaf Bast Fibres

Fibers	Degradation temperature (°C)		Char residue (%)
	T_{IDT} *	T_{FDT} **	
Untreated	247	455	11.41
Propionylated	255	431	19.66
Succinylated	268	470	12.19

* IDT= Initial decomposition temperature; ** FDT= Final decomposition temperature

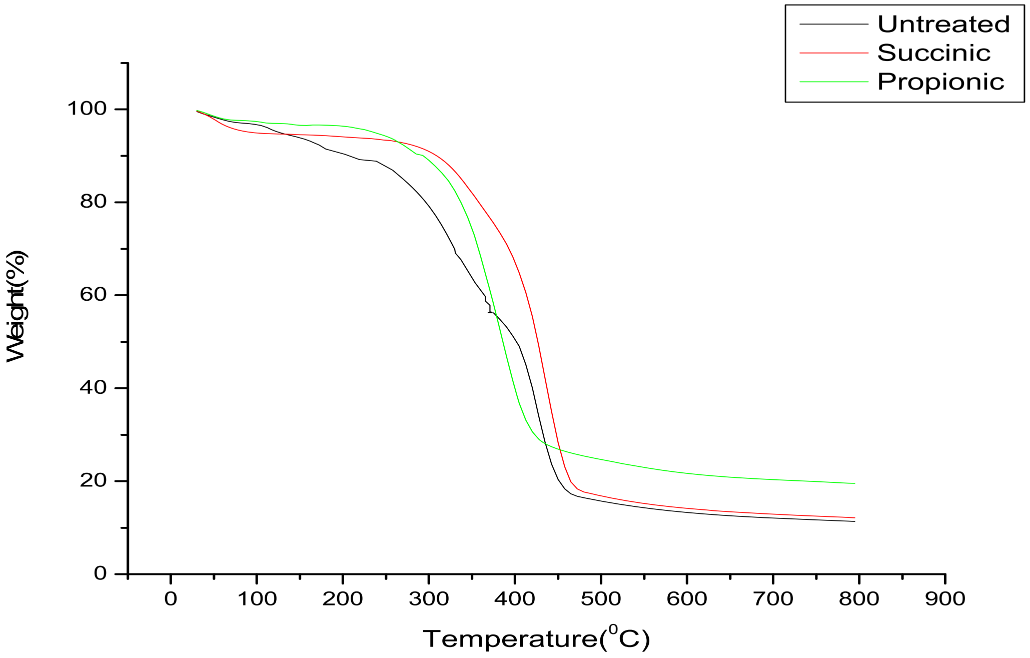

Fig. 4. TGA curves of treated and untreated kenaf bast fibers under N_2 atmosphere at 20 °C/min

Scanning Electron Microscopy (SEM)

Results of Scanning Electron Microscopy (SEM) of untreated, succinylated, and propionylated kenaf bast fibers are shown in Figs. 5 to 7. Figure 5(a) shows the structure of untreated kenaf bast fibers, where it can be seen that the surface layer exhibited coarse morphology. At high magnification, the microscopy also showed the presence of silica nodules (Fig. 5b). The untreated fibers are also covered with a layer, whose composition is probably mainly waxy substances (Tserki et al. 2005). Figures 6 and 7 show the surface of kenaf bast modified with succinic anhydride and propionic anhydride, respectively. Succinylated fibers gave a much smoother surface (Fig. 7) compared to propionylated fibers (Fig. 6) due to presence of carboxylic groups resulting from the succinic anhydride treatment, which can react with waxy substances and silica from fibers without producing by-products. This was also confirmed by previous research from Xiao et al. (2001), which indicated that the modification results in a much smoother surface due to removal of surface impurities such as silica and carboxylic compounds, which block reactive chemical groups.

Contact Angle

The measured contact angles are shown in Table 5. A significant difference was found between the contact angles of chemically untreated and treated fibers. Untreated fibers gave a high value of contact angle (98.86°) compared with propionylated (60.77°) and succinylated (43.80°). If the contact angle is less than 90° the liquid is said to wet the solid, but if it is greater than 90° it is said to be non-wetting. It is clear from the results that treated fibres had increased wettability compared to untreated fibres.

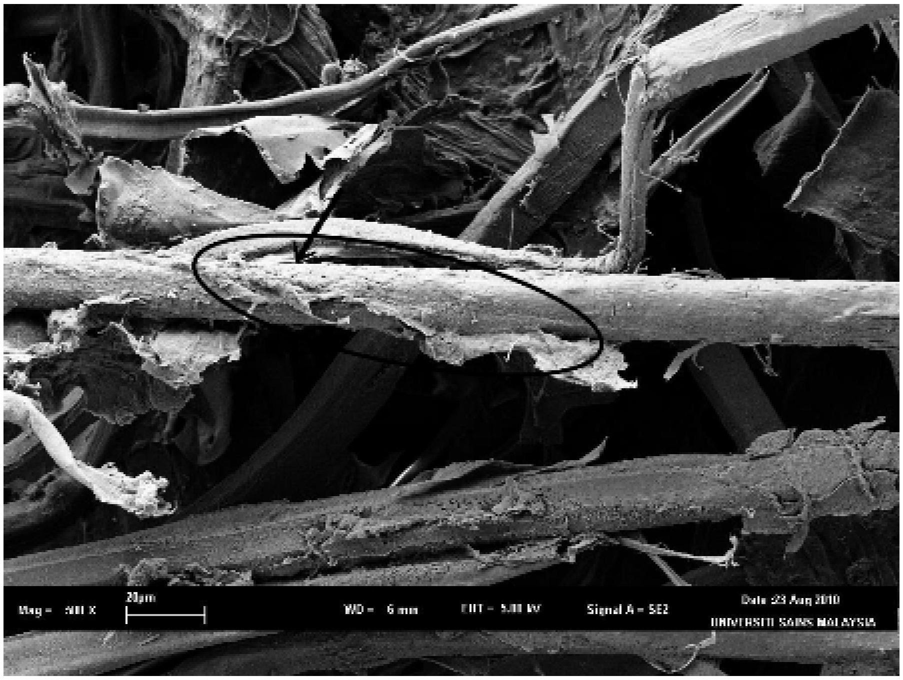

Fig. 5 (a). SEM micrograph of untreated kenaf bast fibres, showing surface of untreated fibre (x 500)

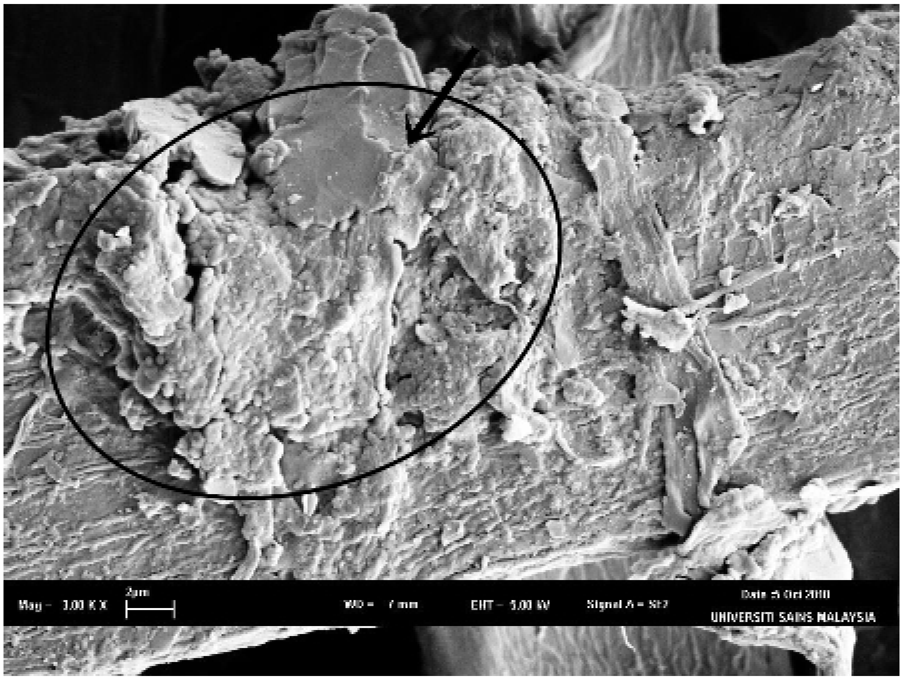

Fig. 5(b). SEM micrograph of untreated kenaf bast fibres, showing close-up of waxy substances (x3000)

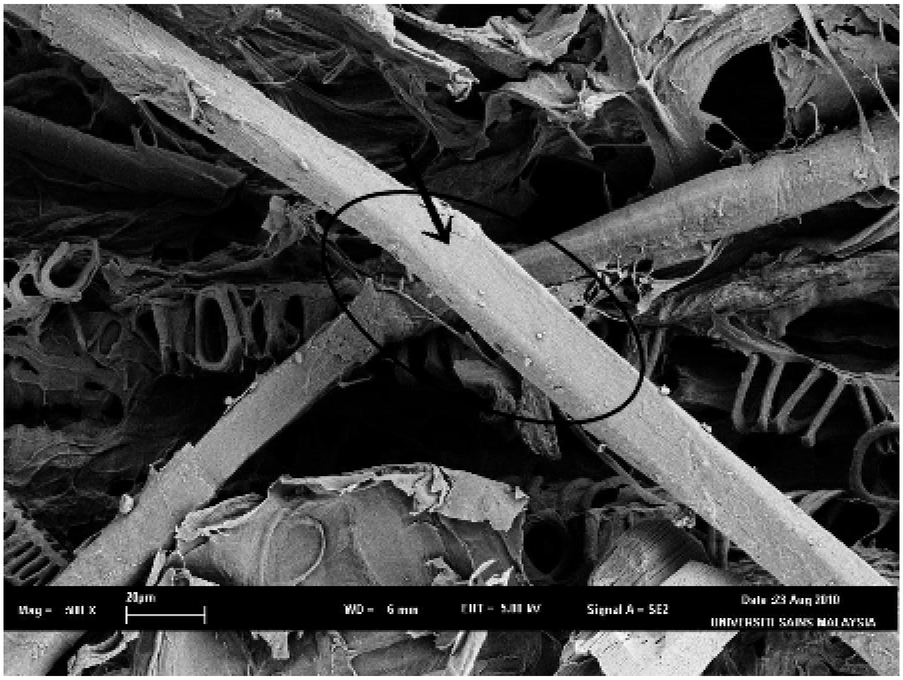

Fig. 6(a). SEM micrograph of propionylated kenaf bast fibres, showing surface of propionylated fibre (x500)

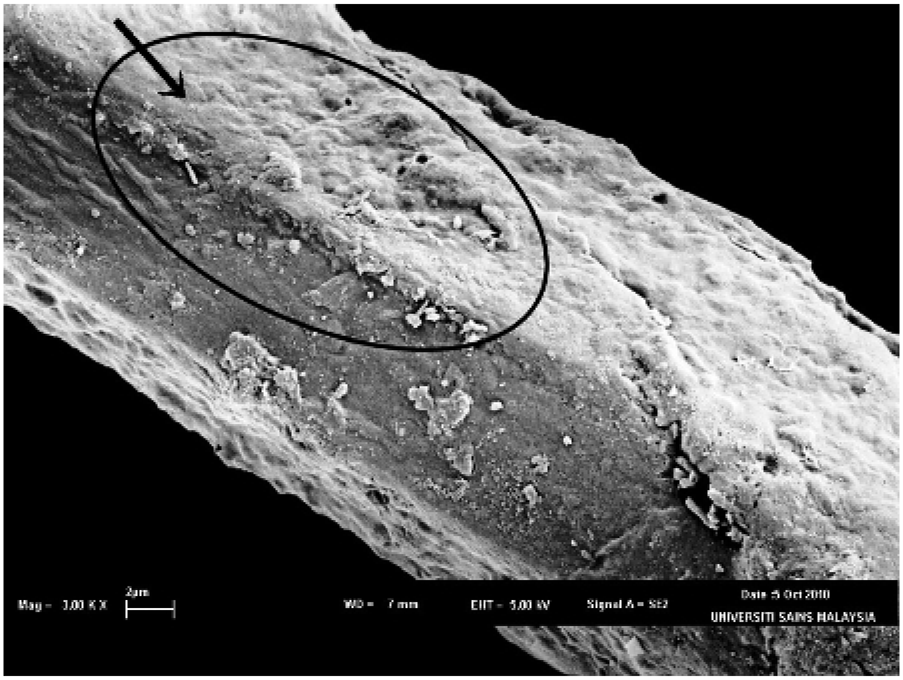

Fig. 6(b). SEM micrograph of propionylated kenaf bast fibres, showing close-up of a surface of propionylated fibre (x3000)

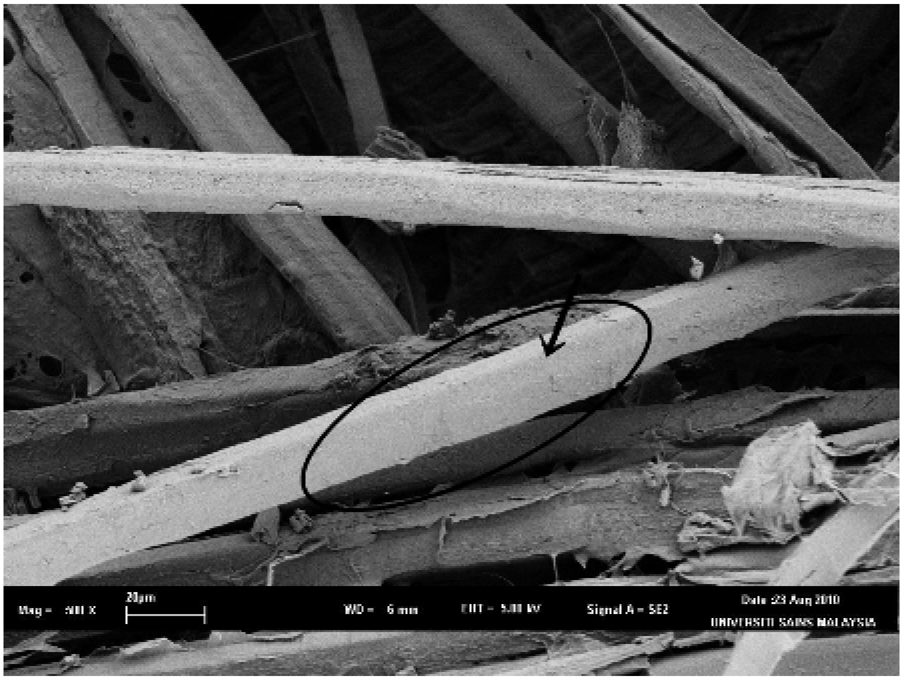

Fig. 7(a). SEM micrograph of succinylated kenaf bast fibres, showing surface of succinylated fibre (x500)

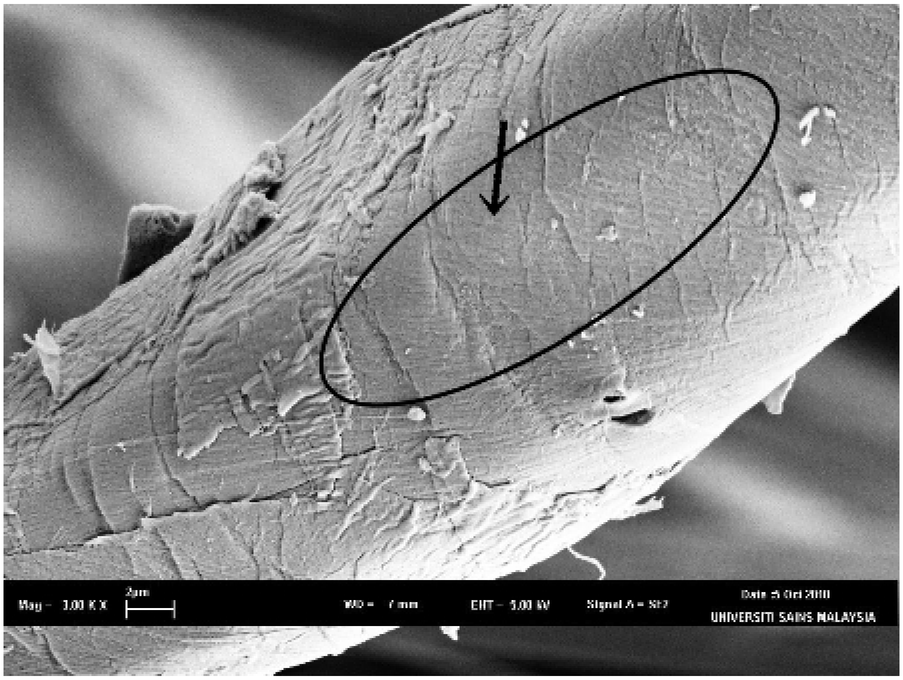

Fig. 7(b). SEM micrograph of succinylated kenaf bast fibres, showing close-up of a surface of succinylated fibre (x3000)

Previous work has shown that after chemical modification of fibres, its contact angles decreases (Wu et al., 2006); such results can be related to the theory of contact angle measurements, which predicts that if the values of contact angles are low, the liquid will spread or wet well, while high values indicate poor wetting. This means that untreated fibers have poorer wettability than treated fibres. Reactivity of the surfaces also plays an important role in wettability. During succinylation, the attachment of a carboxylic moiety, a dispersive aliphatic chain (–CH2–CH2–) is also introduced at the surface. This group will thus affect the wetting properties of the fibers (Gellerstedt and Gatenholm 1999). Based on these results, it can be concluded that a well wetted surface will have a good adhesion characteristics (Abdul Khalil et al. 2004).

Table 5. Calculated Values of Contact Angle

Samples	Mean Contact Angle (degrees)
Untreated	98.86
Propionylated	60.77
Succinylated	43.80

CONCLUSIONS

1. The chemical modification of kenaf bast fibers by succinic anhydride improved morphological, thermal, and wettability properties of fibers.
2. The weight percentage gain (WPG) of succinylated fibers was higher as compared to propionylated fibers.
3. FT-IR spectroscopy confirmed that the esterification process had occurred in treated fibers by referring to increment in peak absorbances at specific frequencies.
4. Succinylated fibers showed better thermal stability compared to propionylated fibers.
5. SEM analysis showed that treatment of the fibers by succinic anhydride gave the smoothest surface.
6. Anhydride modification significantly decreased the contact angle of kenaf bast fibers, thus giving good adhesion characteristics to the fibers.

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