Optimisation of Processing Variables of Kenaf Derived Cellulose Reinforced Polylactic Acid

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Optimisation of composite processing variables (temperature and time) was carried out by monitoring both the stabilisation zone (to ensure composite is well mix at sufficient duration) and the maximum mixing temperature (below 200 °C to avoid fibre degradation) by analysing mixing torque curves upon compounding 5 wt % KDC/PLA using Brabender internal mixer at 160-180 °C for 10, 20 and 30 min., respectively. The composites were pressed and cut into tensile test specimens prior to testing. The 5 wt % KDC/PLA composite demonstrated an optimum tensile strength at three combinations of variables, however the best condition was chosen at 170 °C for 30 min for preparation of composites at various KDC loading (0-60 wt %). The effect of KDC loading on the tensile strength and modulus of composites were investigated. The results demonstrated that increasing KDC loading from 0-60 wt % enhanced the tensile strength and the tensile modulus up to 34 and 107 %, respectively. The α-cellulose was initially derived from kenaf fibre (from bast) by removal of lignin and hemicellulose *via* chemical (chlorination and mercerization) processes. The absence of these components in the FTIR spectral peaks confirms their removal after been chemically treated.

Key Words: Polylactic acid, Derived cellulose, Derived cellulose, Tensile properties, Mixing torque, FTIR.

INTRODUCTION

Nowadays, biodegradable polymers or biopolymers show a large range of properties and can compete with the non-biodegradable thermoplastics for various application namely textile, biomedical and packaging[1]. Different types of biopolymer can be obtained from renewable resources such as polylactic acid (PLA), starch, cellulose ester and polyhydroxybutyrates (PHB)[2]. PLA polymers are polyesters of lactic acid that can be produced either by carbohydrate fermentation or by common chemical synthesis. The main process involve in the production of PLA polymer is ring-opening polymerization of lactide from corn starch to obtain high molecular weight polymer[3]. PLA polymers play as future commodity material due to its production

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from renewable resource and can be recycled or alternatively disposed of by incineration or land filling[4]. Currently, the development of biodegradable composite materials have been investigated using natural fibres such as flax[5], bamboo[6], sisal[7], jute[4] and kenaf[8,9]. Kenaf fibre (*Hibiscus cannabinus* L.) is mainly composed of cellulose (60 %) hemicelluloses (22 %), lignin (13 %) and extractive (3 %)[10]. The chemical structure of cellulose is illustrated in Fig. 1(a). According to Oksman *et al.*[5] the addition of kenaf fibres into the composite has enhanced the mechanical and thermal properties of PLA polymer. Natural fibres have many advantages compared to synthetic fibres (glass fibres), for example low density, renewability and relatively high specific strength and stiffness[11]. However, the major disadvantage of combining natural fibres with polymer is interfacial adhesion between the hydrophilic fibre and some hydrophobic polymer matrix. The studies have been carried out to increase the interfacial adhesion of natural fibres and polymer matrix including physical treatment such as surface oxidation activation[12], chemical treatment such as graft polymerization[13] and alkali treatment[14]. Alkali treatment or mercerization is a chemical process of subjecting natural fibres in an interaction with a relatively concentrated aqueous solution of strong base to create sufficient swelling by removing waxy materials, lignin, hemicelluloses and impurities[15]. The alkali solution causes fibre cell walls to swell and become round, thus increasing the strength, lustre and absorbency[16]. Goda *et al.*[16] also reported that the alkali treatment of ramie fibres made the fibrils more capable of rearranging themselves along the direction of tensile deformation. A major surface roughness of fibres due to alkali treatment might cause major accessibility of -OH groups on the surface of fibres and higher extension of the hydrogen bonds at fibre-matrix interface[17,18]. Delignified is often referred to holocellulose which is the combination of hemicelluloses and cellulose[19]. High amount of non-cellulosic components (lignin, hemicelluloses, pectin, waxes and impurities) in hemp fibres showed negative influence on the fiber processing and properties such as fineness, elasticity and sorption properties[20]. The following reaction takes place as a result of alkali treatment: $C_6H_9O_4$-OH + NaOH $\rightarrow$ $C_6H_9O_4$-O-Na$^+$ + H_2O[17]. To date, not much work on the production of natural fibres derived cellulose-biopolymer composites have been explored. A few studies that have been reported include some works by Khalid *et al.*[14] that reinforced empty fruit bunch derived cellulose-polypropylene (PP) composites. Therefore, the objective of this study is to investigate the extent of Kenaf derived cellulose (KDC) reinforcement in polylactic acid (PLA) polymer. The optimisation of composite processing variables such as temperature and time were also studied by monitoring both the stabilisation zone (to ensure composite is well mixed at sufficient duration) and the maximum mixing temperature (below 200 °C to avoid fibre degradation) by analysing mixing torque curves upon compounding 5 wt % KDC/PLA at range of temperature 160-180 °C for 10-30 min and then compared for their optimum tensile strength. Once the best processing variables was obtained, composite were prepared at various range of KDC loading and tensile properties such as tensile strength and tensile modulus were determined. Fourier transform infrared (FTIR) was also employed to characterise the fibre surface of kenaf fibre, cellulose, PLA polymer and composite.
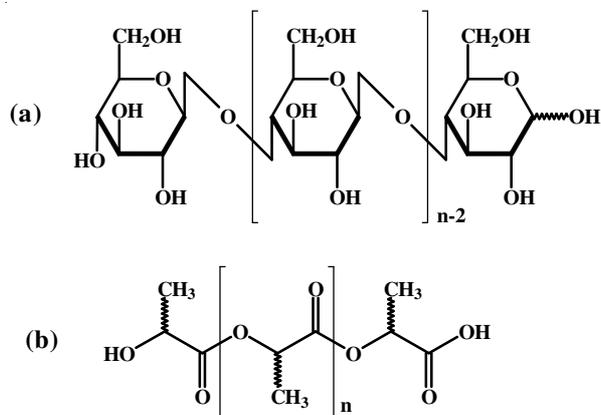
Fig. 1. Typical schematic chemical structure of (a) cellulose[36] and (b) PLA polymer[37]

EXPERIMENTAL

PLA Polymer 2002D resin was purchased from Nature Works LLC (USA) with main properties as tabulated in Table-1. Kenaf bast fibre was provided by Institute of Tropical Forestry and Forest Products (INTROP), Malaysia. This type of fibre (bast) was removed from the kenaf stem by water retting process. The length of fibre was reduced to 2 cm. Reagent grade sodium hydroxide (NaOH), acetic acid (CH₃COOH) purity and technical grade sodium chlorite (NaClO₂) of 80 % purity were purchased from Fisher Chemicals Sdn. Bhd. (Malaysia).

TABLE-1
PROPERTIES OF POLYLACTIC ACID RESIN[35]

Properties	PLA Polymer 2002D	ASTM
Melting point (°C)	150-160	–
Glass transition temperature (°C)	58	–
Specific gravity	1.24	D792
Melt index (g/10 min)	5.0-7.0	D1238
Tensile strength at break (MPa)	53	D882
Tensile modulus (GPa)	3.5	D882
Tensile elongation (%)	6	D883
Notched izod impact (J/m)	12.81	D256

Kenaf derived cellulose (KDC) preparation: Preparation of KDC involves two steps. The first step was the production of holocellulose *via* chlorination or bleaching process[14]. Initially, 2.5 g of kenaf fibers were rinsed with tap water. Then, they were soaked in a 250 mL beaker which contained 80 mL of hot distilled water. Next, the beaker was transferred into a water bath set at 70 °C. Then, both 0.5 mL acetic acid and 1 g sodium chlorite were consecutively added at every 1 h into the

beaker with a total process time of 5 h. The delignification process was indicated by the colour change of fibres from light brown to white. It was then washed and rinsed with tap water. The second step involved conversion of the holocellulose to cellulose by mercerization process[14]. The holocellulose was added with 10 mL of 17.5 % NaOH solution and was heated at 20 °C in water bath. After 5 min, 5 mL of 17.5 % NaOH was added into the mixture 3 times at every 5 min and extra 30 min at the end of the process. Then, approximately 30 mL of distilled water was added into the mixture for 1 h. Next, 100 mL of 8.3 % NaOH was added into the cellulose for 5 min *via* filtration and rinsing with tap water. Then, the alkali cellulose was subjected to acid treatment for neutralization process by adding 10 % acetic acid for 5 min. Finally, the cellulose was filtered, washed and rinsed with distilled water until the cellulose residue is free from acid as indicated by the pH meter. The cellulose was dried overnight in a vacuum oven at 100-105 °C.

Preparation of composites: Prior to mixing, KDC was dried in an oven at 105 °C for 12 h to remove the moisture content. The dried KDC was ground into smaller size using a grinder (Huang Chuan Machinery, China) and passed through a 500 μm sieve to obtain uniform size. 5 wt % of KDC was blended with PLA using an internal mixer Brabender Plastograph (Germany) in the temperature range of 160, 170 and 180 °C for 10, 20 and 30 min, respectively. These composites were prepared to investigate the optimum mixing condition at low loading of KDC (5 wt %). Composites with different percentage of KDC loading (0-60 wt %) were also prepared at 170 °C for 30 min. The test specimens were obtained by transferring each composite material into a rectangular mould with dimensions of 1.5 cm × 1.5 cm × 0.1 cm. Next, moulded composite sheets were produced at 160 °C *via* a compression machine Hot Press Machine LP-50 (Thailand). This process involved 10 min of preheating and 3 min of complete pressing at 100 kg/m^2 pressure followed by cooling for 1 min under pressure.

Tensile properties: Tensile strength (TS) and tensile modulus (TM) were measured using an Instron Universal Testing Machine Model 4301(USA) with load cell of 1 kN. Each tensile test was performed at a cross-head speed of 5 mm/min with dimension of specimens (12.7 mm × 63.0 mm × 1.0 mm) according to ASTM 1882L until the tensile failure occurred. A minimum of 7 specimens were tested for these purposes.

Fourier transform infrared (FTIR) analysis: FTIR analysis was carried out using Perkin Elmer Instrument Spectrum One (USA) with the Attenuated Total Reflectance (ATR) technique. All spectra were recorded in transmittance mode with a resolution of 4 cm^{-1} in the range of 550-4000 cm^{-1}. Ten scans were performed for each acquisition.

RESULTS AND DISCUSSION

Optimization of processing variables: Tensile strength (TS) of 5 wt % KDC/PLA composites produced at a range of temperature and time is presented in Fig. 2.
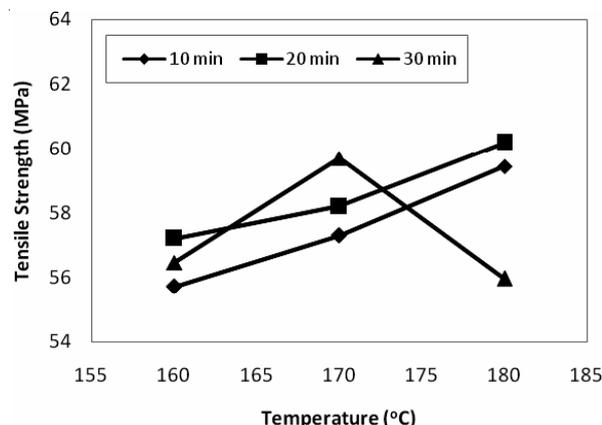
Fig. 2. Effect of temperature and time on the tensile strength of 5 wt % KDC-PLA composites

Overall, the TS increased as both the temperature and the time increased, except for extreme processing condition at 180 °C and 30 min where the strength was drastically dropped to the lowest value of 56 MPa. This is not surprising because the actual temperature during compounding records the highest value *i.e.*, 210 °C (Table-2). It has been reported by Ochi *et al.*[8] that the tensile strength of heat-treated kenaf fibres was found decreased at 200 °C. Moreover, most natural fibres have low degradation temperature (*ca.* 200 °C)[21]. From Fig. 2, it is observed that the composites prepared at conditions of 180 °C for 10 and 20 min, respectively obtain a value close to the composite that was produced at 170 °C for 30 min (59.5, 60.2 and 59.7 MPa, respectively). As seen in Table 2, the maximum temperature during processing of these composites was below 200 °C and yet obtained the highest TS. Homogeneity of composite (the distibution of fibre within the polymer matrix during mixing) was also the main concern in the current study. Sufficient processing duration is important since the stabilisation zone (commonly monitored via mixing torque curve) indicates that the KDC is well mixed and equally distributed within its matrix. Fig. 3 shows the mixing torque curves of KDC/PLA composites produced at KDC loading in the range of 0-60 wt % KDC. For neat PLA polymer, the maximum torque (highest peak) was detected at the beginning of processing followed by a rapid decrease before achieving its constant value in stabilisation zone. This peak represents the highest mechanical shear forces to flow the cold PLA resin. As the molten PLA completely melted, the torque rapidly decreased. For KDC/PLA composites, the first peak was also observed and the maximum values decreased as the loading of KDC increased due to the decrease in the amount of PLA introduced into the mixer. The second peak was recorded after 2 min and this peak is due to the addition of KDC into the mixer. This peak represents a greater torque that had been developed from the increase in mechanical shear to mix the KDC into the molten polymer. At higher loading of KDC, the torque curves show that longer time is required to gradually mix the cellulose into the mixer until achieving its constant torque value towards the end of

the processing time. Overall, increasing the cellulose loading increased the final value of stabilisation zone. Yang *et al.*[22] reported that the fibres and LDPE matrix has been completely mixed by observing the stabilisation zone. A similar mixing torque curves have also been observed by Bengtsson *et al.*[23]. For these reasons, the best processing condition for producing KDC/PLA composite at higher KDC loading was performed at 170 °C for 30 min to prevent the loss of mechanical strength due to the thermal degradation[8].

TABLE-2
MAXIMUM MIXING TEMPERATURE OF THE COMPOSITES
RECORDED DURING THE COMPOUNDING

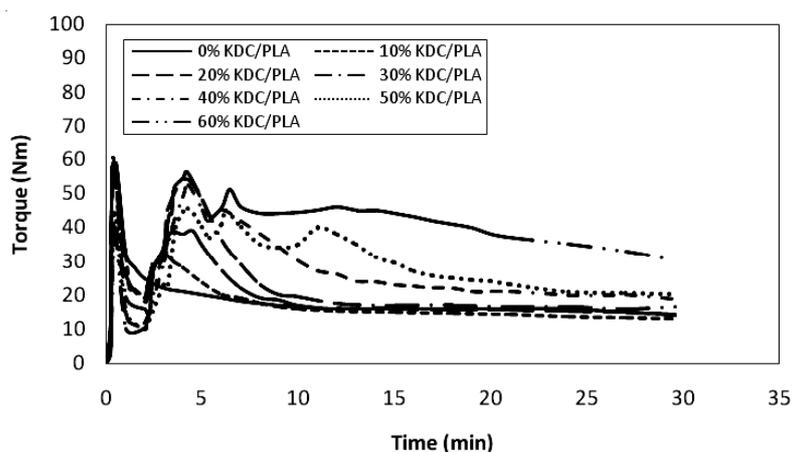| Time (min)	Set temp. (ºC)	Max temp. (ºC)	Time (min)	Set temp. (ºC)	Max temp. (ºC)	Time (min)	Set temp. (ºC)	Max temp. (ºC)
10	160	188	20	160	188	30	160	188
10	170	197	20	170	196	30	170	194
10	180	203	20	180	203	30	180	210

Fig. 3.   Mixing torque as a function of processing time for composite ranging from 0-60 wt % KDC loading

Effect of KDC loading on tensile properties: Figs. 4 and 5 depict the effect of incorporating various KDC loading in KDC/PLA composites on their TS and TM. Overall, both properties were improved with the increase of KDC loading. Fig. 4 shows that composite with 20 wt % of KDC loading or less produce no remarkable changes in their strength. Nevertheless, addition of the KDC loading from 30-60 wt % has enhanced the strength of the composite to 25 %, with 60 wt % KDC/PLA composite obtaining the highest strength to approximately 79 MPa. When comparing neat PLA polymer to the 60 wt % KDC/PLA composite, the later obtained 34 % higher in TS than the former one. It can be observed in Fig. 5 that the 60 wt % KDC/PLA composite achieved the highest TM (1639 MPa) as compared to the
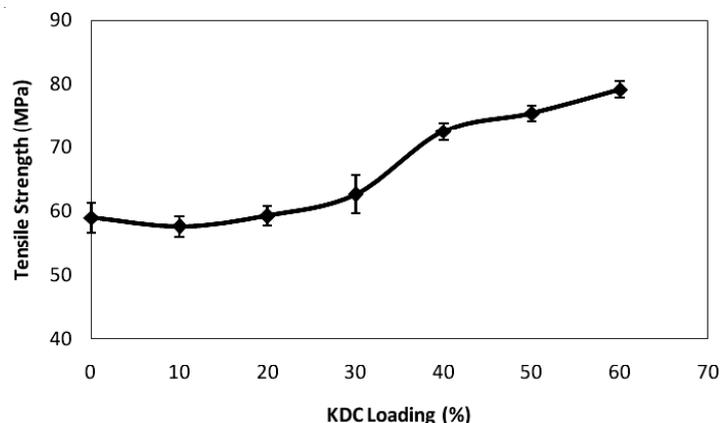
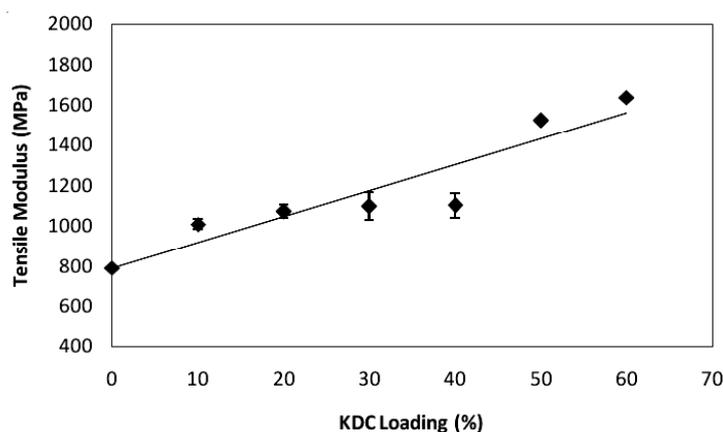Fig. 4. Effect of KDC loading on the tensile strength of KDC/PLA composites

Fig. 5. Effect of KDC loading on tensile modulus of KDC/PLA composites

neat PLA (790 MPa), with significant enhancement of approximately 107 %. This indicates that the composites become stiffer at higher KDC loading. According to Klason *et al.*[24], incorporation of cellulosic reinforcements as shown by various studies produced greater stiffness in the composites. In general, natural fibres have a higher Young's modulus thereby contributing to the higher stiffness of the composites[25]. Generally, it is known that mechanical performance of fibre-reinforced composites is mainly influenced by the fibre-matrix compatibility, fibre dispersion and fibre aspect ratio. PLA is hydrophilic in nature which has polar oxygen atoms whereas chemically treated KDC would become more hydrophilic[27]. In a study by Bax and Mussig[28], it is reported that polar oxygen atoms from PLA could form hydrogen bonds to the hydroxyl groups of the natural fibre and resulting in a good interfacial adhesion between both materials. But, they assumed that these hydrogen bonds might only have a small influence on the fibre-matrix adhesion of flax/PLA composites. A good fibre dispersion in polymer matrix may also be enhanced the mechanical

properties of the KDC/PLA composites. A similar trend in mechanicalproperties was reported for TS and TM of flax fibre reinforced starch based biopolymer (Biopar®) composites[26]. They believed that the dispersion of the flax fibre inside the polymer matrix should be good enough due to the linear dependence of the TS and TM with the Biopar® loading. In addition, even fibre distribution provides a better stress transfer from matrix to the fibre as indicated by a high TS[29]. Previous studies have also shown that the natural fibres treated with chemical had resulted in a significant improvement in tensile strength[14].

Fourier transform infrared (FTIR) analysis: Fig. 6 compares the FTIR spectra of the kenaf fibre and cellulose. The spectra show a peak in the range of *ca.* 3400-3200 cm⁻¹, which corresponds to O-H stretching vibration. It is noticed that the peak is narrower (higher intensity) for cellulose, which indicate that it contains more -OH groups. This may result from mercerization reaction between sodium hydroxide (NaOH) as the swelling agent and the fibres. In this process lignocellulosic materials would lead to swelling of the cell wall and subsenquently exposed lignocellulosic -OH group free from hydrogen bonding[30]. Meanwhile, C-H stretching refers to alkanes group at 2900 cm⁻¹ that was present in kenaf fibre and cellulose. For cellulose, the disappearence of C=O stretching frequency at 1736 cm⁻¹ can be clearly seen as compared to kenaf fibre. This peak is attributed to the carbonyl (C=O) stretching of the acetyl groups that is present in hemicelluloses for kenaf fibres as reported by Sgriccia *et al.*[21]. Alkali treatment of grass fibres exhibits the similar peak that assigned to a C=O stretching vibration of carboxylic acid or ester due to the removal of hemicellulose from the fibre surfaces[30,31]. The vibration peak at 1515 cm⁻¹ is assigned to the benzene ring of the lignin. This is due to the structure of lignin that is highly unsaturated or aromatic polymer and it has low oxygen to carbon ratio[30]. According to Sgriccia *et al.*[21] the lignin peak at 1500 cm⁻¹ is present in kenaf fibers. The absence of this peak in cellulose spectrum is due to the removal of lignin *via* alkali treatment. It is known that lignin is soluble in alkali solution[30]. From Fig. 6, the FTIR spectrum of cellulose confirmed the removal of lignin by the loss of peak at 1236 cm⁻¹ which represents C-O stretching vibration of acetyl group of lignin. Similar peak was reported[21] at 1239 cm⁻¹ for the kenaf fibres. Fig. 7 shows the FTIR spectra of 60 wt % KDC/PLA composite, neat PLA and cellulose. The composite obtained similar spectrum to neat PLA except at approximately 3340 cm⁻¹ low intensity peak was observed which corresponded to O-H stretching vibration of 60 wt % KDC/PLA. The larger amount of cellulose (KDC) in the PLA matrix may be attributed to the presence of -OH group. The C=O stretching vabriation at 1746 and 1749 cm⁻¹ were observed and this shows the presence of carbonyl (C=O) in ester group of PLA polymer. The chemical structure of PLA polymer is illustrated in Fig. 1(b). There are three bands in the range of 1300-1500 cm⁻¹ in both composite and neat PLA spectrum that may be attributed to symmetric and asymetric deformational vibration of C-H in methyl (CH₃) group[32]. This was in agreement with the study done by Pamula *et al.*[33]. Besides that the wavelength in the range of 1300 cm⁻¹ to 1000 cm⁻¹ consists of multiple peaks. The peak at 1181 cm⁻¹ represents a stretching vibration
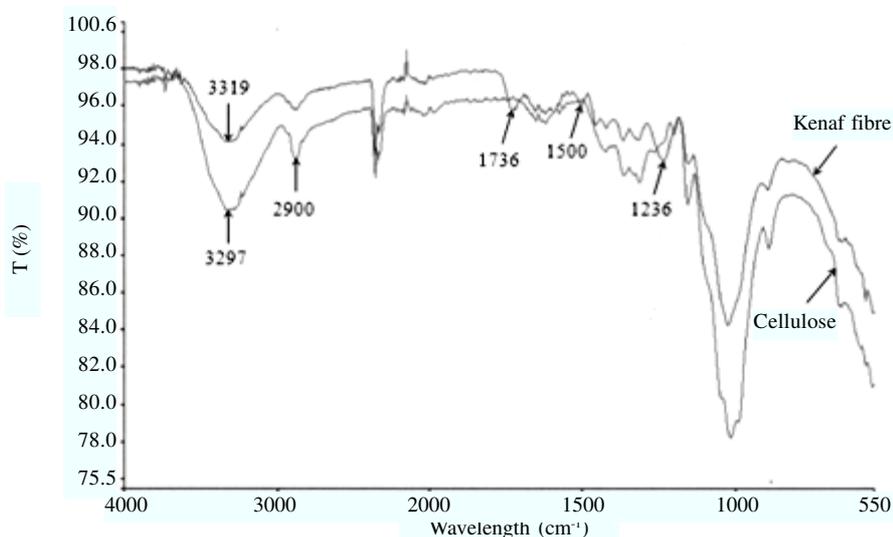
Fig. 6. FTIR spectra for the untreated and treated kenaf fibres
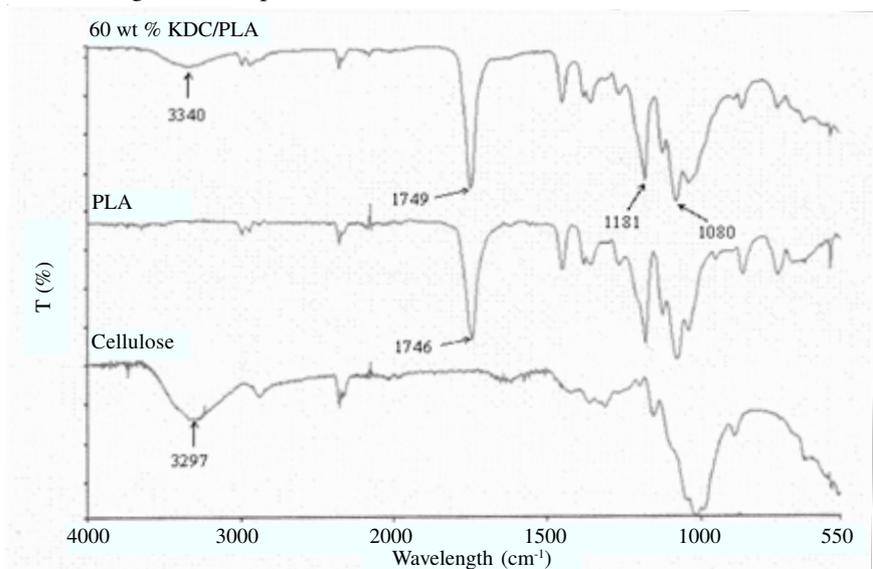

Fig. 7. FTIR spectra for the 60 wt % KDC/PLA composite, neat PLA and treated kenaf fibres

of C-O-C. This peak indicates the C-O-C of ester group which corresponded to PLA polymer[34]. Meanwhile, at wavelength of 1080 cm[-1], the C-O-C stretching vabration was observed and this was attributed to the anhydrides groups[34]. Generally, there was no new peak (Fig. 7) observed in the composite which could indicated the formation of new chemical bonding. Therefore, the composites in the current study may be enhance their strength and stiffness due to good physical interaction between the KDC and PLA matrix.

Conclusion

This study demonstrated that careful consideration of composite's mixing variables (temperature and time) coupled with the information from mixing curves (maximum mixing temperature and stabilization zone) are useful for determining the optimum mixing variables to produce composites with better tensile properties both low (5 wt %) and high loading of KDC (up to 60 wt %). The best optimum processing condition for the maximum tensile strength of low KDC loading (5 wt %) composite was found at 170 °C for 30 min. This mixing condition was employed to blend KDC/PLA composites at various KDC loading (0-60 wt %). Increasing the KDC loading up to 60 wt % has successfully enhanced both the tensile strength and the tensile modulus of composites by 34 % and 107 %, respectively.

ACKNOWLEDGEMENTS

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