Journal of Composite Materials

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Journal of Composite Materials 2013 47: 3343 originally published online 19 November 2012
DOI: 10.1177/0021998312465026

Article

JOURNAL OF COMPOSITE MATERIALS

Journal of Composite Materials
47(26) 3343–3350

DOI: 10.1177/0021998312465026
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Mechanical and thermal properties of chemical treated kenaf fibres reinforced polyester composites

HPS Abdul Khalil[1], NL Suraya[1], N Atiqah[1], M Jawaid[2] and A Hassan[3]

Abstract

Kenaf fibre reinforced polyester biocomposites fabricated by hand lay-up technique by using propionic and succinic anhydride-modified kenaf fibres. Chemical structure, mechanical, thermal and morphological properties of kenaf fibres reinforced polyester biocomposites evaluated. The Fourier transform infrared study of modified kenaf fibres carried out to look at changes in functional groups after modification. It confirmed from Fourier transform infrared spectroscopy the variation in positions of functional groups. The mechanical (tensile, flexural and impact) property results revealed that biocomposites with modified kenaf fibres exhibited better properties as compared to untreated kenaf fibres-reinforced polyester biocomposites. Morphological studies show that treated kenaf biocomposites show better fibre/matrix interaction. Thermal analysis results of modified biocomposites exhibited higher initial and final decomposition temperatures. Modified biocomposites display less char residue as compared with unmodified kenaf fibres reinforced polyester composites.

Keywords

Fibres, chemical treatments, composites, mechanical properties, thermal properties

Introduction

The environmental issues raised due to rapid industrialization have caused great threat to the environment. As humans become more aware about industrialization, the direct effect has been found on the ecosystem and these issues have become a major concern for both researchers and industries. In Malaysia, government is emphasizing and taking industries as well as academic researchers into confidence to make environment free from the negative effects of rapid growth of globalilization. The awareness effort needs conservation of natural resources for sustainable development of future. The utilization of renewable resources is one of the major solutions to overcome the environmental issues. The availability and renewability of natural fibres has made a new platform for polymer scientists, engineering researchers to develop a sustainable technology for utilization of natural fibres for sustainable existence of our future generations. Researchers over the globe are seriously looking at natural fibres as alternatives to replace synthetic fibres.[1,2] The mechanical and physical properties together with their eco-friendly nature of plant fibres has motivated researchers, particularly automotive industry, to consider these fibres as potential candidates to substitute glass fibres in environmentally safe products.[3,4]

The combination of natural fibres such as kenaf, oil palm, hemp, flax, jute, henequen, pineapple leaf, sisal, bamboo etc. with polymer matrices is often used to fabricate natural fibre reinforced polymer composites.[5] Natural fibre biocomposites such as kenaf composites offer many environmental advantages and at the same time encourage the development of industrial crops in Malaysia. Kenaf has opened up new avenues

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for exploiting these natural resources commercially. The government in Malaysia has generated a new programme known as National Kenaf Research and Development Program in order to support the development of new methods of harvesting and utilization of kenaf fibre as reinforcement in commercial products.[6] However, there are reports which indicate limitation of using these fibres due to their poor interfacial interaction with the matrix.[7] Chemical modification of fibre improve fibre/matrix interfacial bonding, and enhance natural fibres application as reinforcing agent in polymeric materials.[8] The large amount of hydroxyl group in cellulose in natural fibre gives it hydrophilic properties, when used to reinforce hydrophobic matrices it shows very poor fibre/matrix interface and poor resistance to moisture absorption.[9] The enhanced adhesion between matrix and fibres can be achieved through various methods e.g. treatment of fibres with the chemical agents. The chemical modification of plant fibres is well practiced in fibre-reinforced biocomposites technology in order to fabricate the biocomposites with better properties with environmentally friendly nature.

Present study reports the potential use of chemically modified kenaf bast fibres as reinforcement in polyester composite. This research is different from previous reported work because we try to explore the reactive chemistry of succinic and propionic anhydride towards kenaf bast fibres. We investigate the effect of modification of kenaf fibres on chemical structure, mechanical, morphological and thermal properties. We attribute that modified kenaf fibres reinforced polyester composites display better properties and enhance utilization of kenaf fibres in different applications.

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 (PA), succinic anhydride (SA), sodium formate and N, N-dimethyl formamide was purchased from Mega Makmur (Malaysia). The unsaturated polyester resin used in this research was based on commercial unsaturated polyester resin produced by Euro Chemo-Pharma Sdn. Bhd. Methyl ethyl ketone peroxide (MEKP) used as hardener was also supplied by the same supplier.

Method

Fibre treatment

The treatment of kenaf was carried out according to our previous research.[10] Kenaf bast fibre was chemically modified by using propionic anhydride and succinic anhydride. Kenaf mats were soaked in the solution of the propionic anhydride and succinic anhydride in N, N-dimethylformamide in the presence of the catalyst sodium formate (10:1). The reaction was carried out at 100°C for 180 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 dried in GOTECH Drying Oven-GT-7024, for 24 hours with a temperature of 80°C.

Composite preparation

The unsaturated polyester resin was mixed well with the hardener, MEKP, in required amounts. Then, resin was poured onto the kenaf modified mat about 10 minutes under vacuum impregnation to ensure good delivery of the resin. The wetted kenaf-modified mat in the mold of dimensions 120 mm × 150 mm × 3 mm was then placed between the platens of a pressing machine at 8 MPa pressure until it was cured at room temperature. The composite was then ready for testing.

Characterization

Fourier transform infrared

A Fourier transform infrared (FTIR) spectroscope, Nicolet Avatar 360 (USA), was used to examine the functional groups present in the fibres. A Perkin Elmer spectrum 1000 was used to obtain the spectrum of each sample; 1 mg of powdered fibre samples was mixed with 100 mg of KBr powder. The powder mixtures were then pressed into transparent thin pellets. FTIR 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 wave number.

Tensile properties

Tensile testing was carried out by using GOTECH Universal Tester-GT-A1-7000 L according to ASTM D3039. Samples with dimension 120 mm × 15 mm × 3 mm were prepared. Samples were tested at a cross-head speed of 3 mm/min and gauge length of 60 mm.

Flexural properties

Flexural testing was carried out by using GOTECH Universal Tester-GT-A1-7000 L according to ASTM D 790. Samples with dimension 120 mm × 20 mm × 3 mm were prepared. Samples were

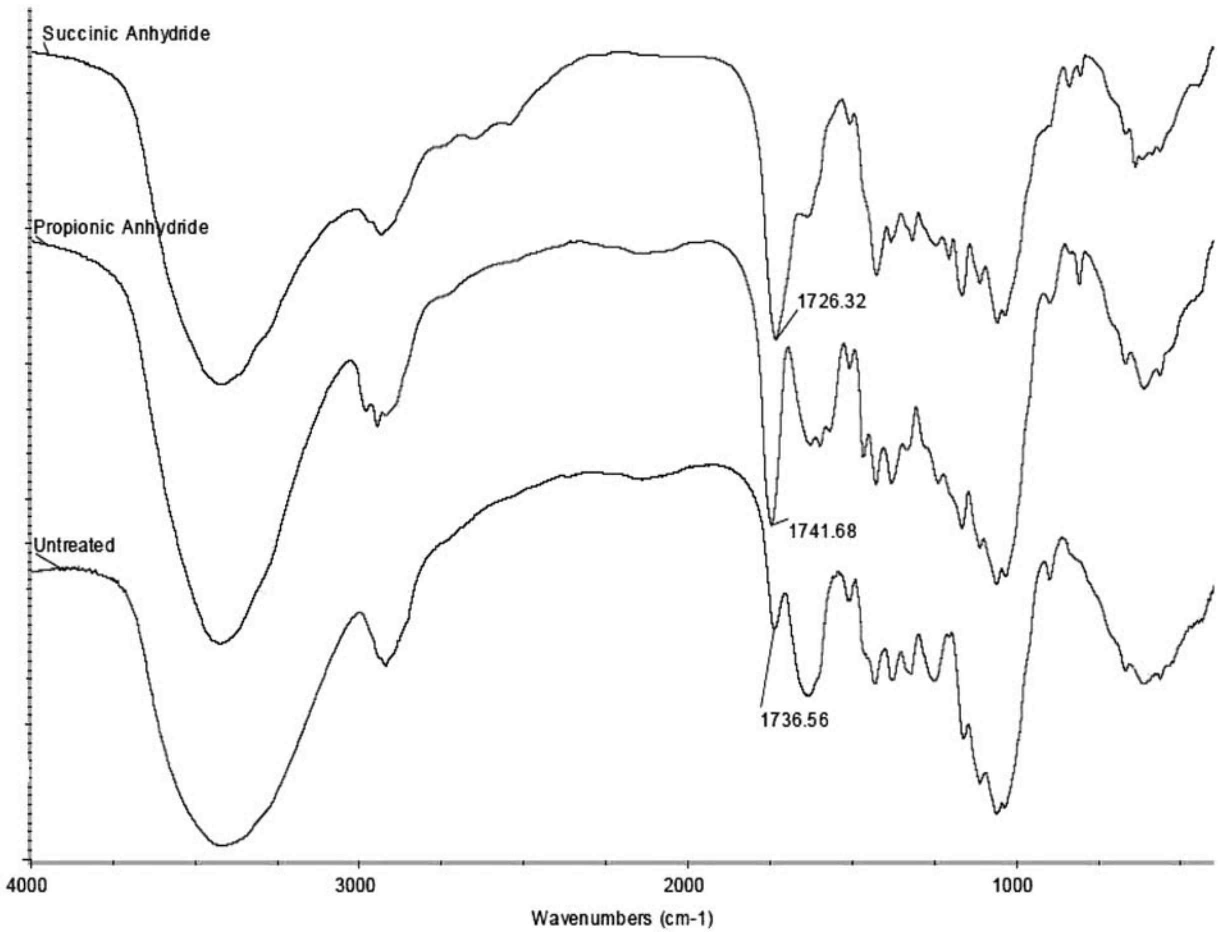

Figure 1. Fourier transform infrared (FTIR) spectra of untreated, propionylated and succinylated treated kenaf fibres.[10]

tested at a cross-head speed of 3 mm/min and gauge length of 60 mm.

Impact strength

Impact test was done according to ASTM D256. Charpy impact tests were performed using the GOTECH Impact Tester Model-GT-7045-MDL. The impact test was carried out on samples with dimensions of 65 mm × 15 mm × 3 mm. The samples were rigidly mounted in a vertical position and were struck using a pendulum with a force of 10 J at the center of the samples. The number of fracture values was calculated.

Scanning electron microscope

Morphological behaviours of untreated and treated kenaf fibres reinforced polyester composites were investigated using a scanning electron microscope (SEM) (Leo Supra, 50 VP, Carl Ziess, SMT, Germany). The samples mounted onto SEM holder using double-sided electrically conducting carbon adhesive tapes to prevent surface charge on the specimens when exposed to the electron beam. The samples were sputter with gold prior to their morphological observation. The SEM micrographs were obtained under conventional secondary electron imaging conditions with an acceleration voltage of 5 kV.

Thermogravimetric analysis

The thermal stability of biocomposites samples were characterized by using a thermogravimetric analyzer (TGA), model 2050, (TA Instruments, New Castle, DE). All specimens were scanned from 30°C to 800°C at the rate of 20°C/min under nitrogen environment.

Results and discussion

FTIR studies

Figure 1 displays FTIR spectra of untreated, treated by PA and treated by SA kenaf fibre. The FTIR studies revealed that treatment of kenaf fibres by succinic anhydride and propionic anhydride occurred and was evidenced by changes in frequencies in functional groups as per our previous article.[10] The FTIR of unmodified and modified Kenaf polyester composites revealed the shifts in respective functional groups. The composites exhibited new absorption bands between 2800 cm^{-1} to 2900 cm^{-1}. These are due to –C-H vibration indicating the presence of alkyl chains and ester groups formed during anhydride treatment.[11] The succinic anhydride kenaf fibre composite exhibited intense band at 1720 cm^{-1} and 1700 cm^{-1} due to >C=O stretching of succinic anhydride kenaf fibre composites. As compared to unmodified kenaf fibre, modified kenaf fibre composites observed shift in >C=O stretching vibration, which indicated the interaction of modified kenaf fibre with polyester matrix. In addition, the band at 1247–1270 cm^{-1} can be attributed to the –C-O stretching vibration, thereby supporting the bond formation between –OH groups of fibre and –C-O groups of anhydride. A decrease in the intensity of the hydroxyl groups (–OH) absorption band around 3500–3100 cm^{-1} indicated that the OH were reduced, which also was observed from this research.[12] Moreover, FTIR spectra of modified biocomposites revealed variation in 1800–500 cm^{-1}.[13] These results indicate a strong reaction has been occurred between modified fibres and in polymer matrix.

Mechanical properties

Tensile Properties. The tensile properties of unmodified and modified biocomposites are given in Table 1. The tensile strength results of unmodified kenaf fibre biocomposites was found to be 65.1 MPa, while tensile strength of composites modified with succinic anhydride and propionic anhydride was found to be 80.6 MPa and 79.2 MPa, respectively. The higher tensile strength obtained can be attributed to better interfacial adhesion properties between matrix and fibres, however this interfacial adhesion is greatly affected by the treatment of fibres with respective anhydrides. It has been reported that chemical treatment of fibres provides better mechanical interlocking and enhances the penetration of resin into fibres and vice versa.[14] Propionic and succinic anhydride treatment can attribute to the improvement of mechanical interlocking between fibre and matrix.[15,16] The mechanical interlocking theory of adhesion proposes that surface irregularities are the determining factors for adhesion strength between a matrix and fibre due to the ability of the matrix to key and latch the fibre.[16] Besides that, propionylated and succinylated samples give higher tensile strength due to the dipolar interactions between anhydride groups and cellulosic OH groups.[15,17] Hence, the good interfacial bond provides high resistance to fibre pullout without rupturing the fibre and greatly increases the tensile strength of composites.[18]

Tensile modulus results show similar trend like tensile strength. Tensile modulus of modified kenaf bast polyester composite gives higher value than unmodified composite. This clearly indicates that chemical treatment increase the ability of composite to resist the deformation and breaking under tensile stress.[14] Modified composite can withstand the stress exerting on it more than unmodified composite because of the good bonding between modified fibre and matrix. In polymer composites there are more to understand about its mechanical properties than merely knowing about the strength only. Strength only explains how much stress is needed to break something, but it does not explain what happens to the composite while force tries to break it. Elongation at break of unmodified, propionylated and succinylated obtained are 4.2%, 3.8% and 4.1%, respectively. The reason of the decrease in elongation at break for modified composite was due to the improvement of fibre-matrix bonding. According to Khalil et al., the improvement of fibre-matrix bonding would increase the strength, stiffness and interfacial adhesion of the composite because modified fibres were prone to split and fall apart i.e. fibre was more brittle after modification.[19] Thus, reduce the percent of elongation until it can reach the point to permanently deforming it.

Flexural Properties. The flexural properties of unmodified and modified kenaf-based polyester composites are given in Table 2. The modified composites exhibited better mechanical properties including flexural properties as compared with unmodified composite. Flexural strength of propionylated shows highest value which is

Table 1. Tensile properties of untreated, propionylated and succinylated treated kenaf fibres reinforced polyester composites

Composite	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
Untreated	65.1 ± 0.59[a]	1.6 ± 0.02	4.2 ± 0.19
Propionylated	79.2 ± 0.95	2.5 ± 0.35	3.8 ± 0.43
Succinylated	80.6 ± 0.51	2.7 ± 0.57	4.1 ± 0.26

[a]Standard error.

169 MPa followed by succinylated (166 MPa) and unmodified (128 MPa). In unmodified fibres, hemicelluloses are dispersed in the interfibrillar region and separates cellulose chains [16], these chains are in strain state. They also reported that chemical treatment removes hemicelluloses, so the internal strain disappears and microfibrils are able to rearrange in a more compact structure and treatment leads to a decrease in the spiral angle and increased molecular orientation. A cellulose chain poses a closer packaging that leads to higher fibre strength.[20] The flexural modulus of modified kenaf-based polyester composites showed higher values as compared to unmodified fibre-based composites. The propionic anhydride and succinic anhydride modified kenaf fibre composites exhibited 2.9 GPa and 3.2 GPa, respectively, as compared to unmodified samples, 1.9 GPa. The fibres in composites also become more capable of rearranging themselves along the flexural deformation and thus enhance the ability of composite to resist the applied flexural stress.[21]

Impact strength. The impact strength of unmodified, propionylated and succinylated composites is given in Table 3. Results exhibited high value of impact strength for propionylated (7.7 kJ/m^2) and succinylated (7.3 kJ/m^2) as compared with unmodified (5.4 kJ/m^2). The impact properties were directly related to the overall toughness of the materials, which were highly influenced by the interfacial bond strength, matrix and fibre properties.[22] The results apparently shows that the chemical modification of kenaf bast fibre leads to high impact strength of composite. Higher impact strength means high energy required to cause failure

Table 2. Flexural properties of untreated, propionylated and succinylated treated kenaf fibres reinforced polyester composites

Composite	Flexural strength (MPa)	Flexural modulus (GPa)
Untreated	128 ± 0.21[a]	1.9 ± 0.20
Propionylated	169 ± 0.45	2.9 ± 0.52
Succinylated	166 ± 0.21	3.2 ± 0.53

[a]Standard error.

Table 3. Impact strength of untreated, propionylated and succinylated treated kenaf fibres reinforced polyester composites

Type of composites	Impact strength (kJ/m^2)
Untreated	5.4 ± 0.21[a]
Propionylated	7.7 ± 0.23
Succinylated	7.3 ± 0.28

[a]Standard error.

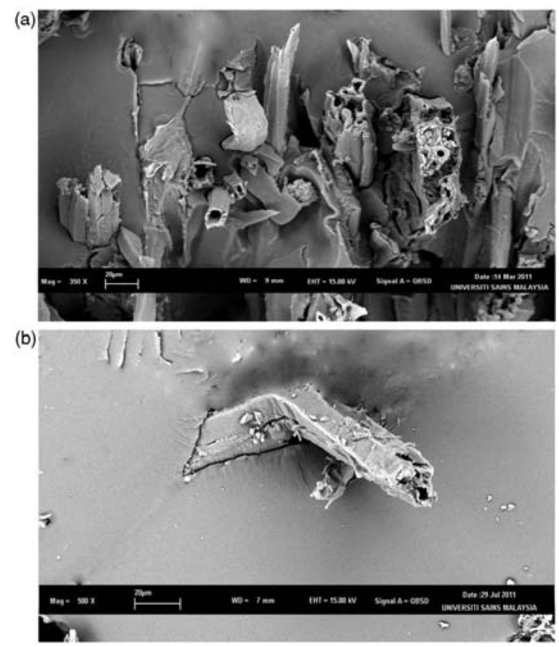

Figure 2. Scanning electron microscope (SEM) micrographs of tensile fracture of untreated kenaf fibre reinforced polyester composites.

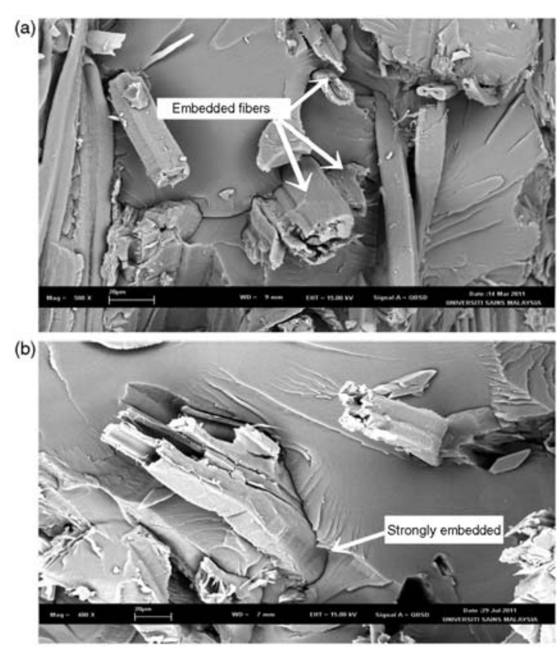

Figure 3. Scanning electron microscope (SEM) micrograph of tensile fracture of (a) succinic andyride (b) propionic anhydride treated kenaf fibre reinforced polyester composites.

towards the composite. According to Velmurugan and Manikandan, the matrix fracture, fibre matrix debonding and fibre pullout are important failure modes observed in the fibre composites due to impact loading.[23] From the results, propionylated give slightly higher impact strength than succinylated and this might be due to the different kind of chemical structure that exists in succinic and propionic anhydride. Previous research by Bullard stated that although the greatest reactivity of chemical modification of fibres was obtained with succinic anhydride, this also had drawbacks.[24]

Table 4. Thermal properties of untreated, propionylated and succinylated treated kenaf fibres reinforced polyester composites

Type of composites	Degradation temperature (°C)		Char residue (%)
	T_{IDT}	T_{FDT}	
Untreated	250	457	11.4
Propionylated	262	473	10.9
Succinylated	273	485	9.6

IDT: initial decomposition temperature; FDT: final decomposition temperature.

Morphological studies

The effect of treatment of kenaf bast fibre reinforced polyester composite is also described by using the SEM micrographs of tensile fracture samples. Figure 2 depicts the morphology of tensile fracture of unmodified kenaf composites. This observation clearly indicates that unmodified kenaf bast fibre and polyester matrix have poor adhesion and interaction between each other. Figures 3(a) and 3(b) shows the fracture surface of modified composite. SEM micrograph shows good fibre matrix interaction as the fibres are well embedded in the matrix. According to Tserki et al., fibres are covered with a layer, whose composition is probably mainly waxy substances.[25] Thus, by chemical modification, dissolution of waxy substances can expose the –OH and the –COOH groups on the fibre surface.[21] This will ultimately lead to increased polar-polar interaction with the matrix and the strong interface between fibre and matrix can prevent

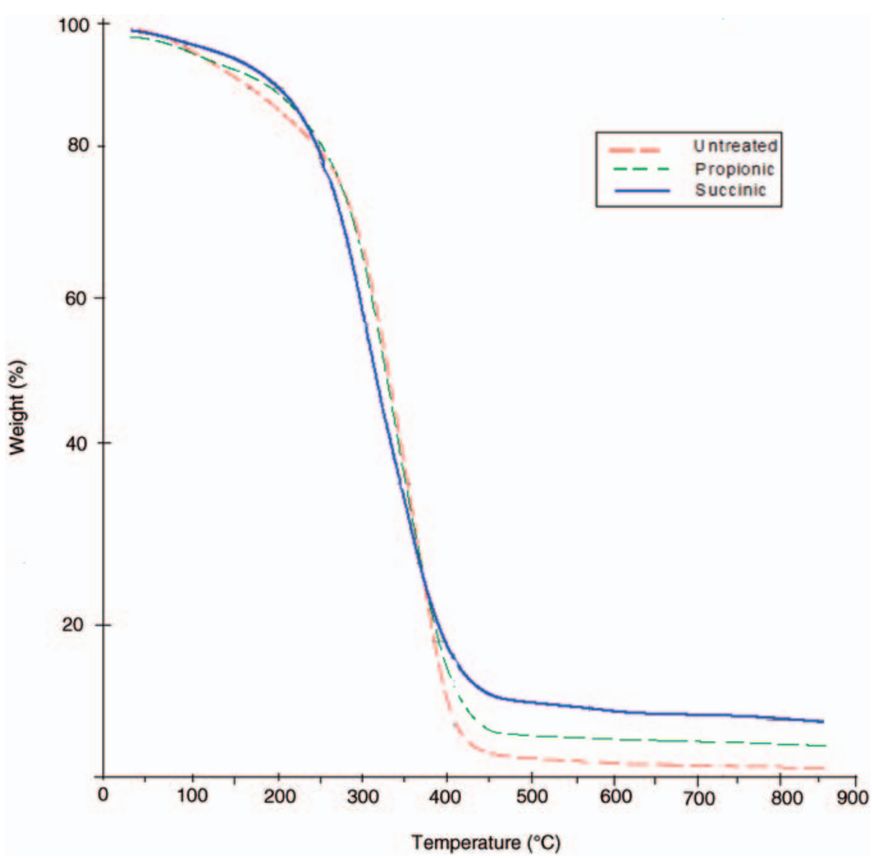

Figure 4. Thermogravimetric analysis (TGA) curves of untreated and succinic andyride, propionic anhydride treated kenaf fibre reinforced polyester composites.

interfacial slippage.[26] Figure 3 shows modified kenaf bast fibre sticking and well wetted with unsaturated polyester matrix, which proved the existence of good adhesion between them. Thus, the morphological examinations of samples by SEM can provide a strong evidence of good adhesion between the matrix and the fibre.

Thermal properties

The initial degradation temperature (T_{IDT}), final degradation temperature (T_{FDT}) and char residue content of untreated, propionylated and succinylated kenaf bast fibre reinforced composites are shown in Table 4. Generally, modified composites showed good thermal stability as compared with unmodified composite. According to Xiao et al., the first stage of weight loss was assigned to the evaporation of water from the samples.[27] TGA curves of untreated, propionic anhydride-treated and succinic anhydride-treated kenaf bast fiber reinforced polyester composites are illustrated in Figure 4. The initial degradation temperature for unmodified, propionylated and succinylated are 250°C, 262°C and 273°C, respectively. This result indicated that succinylated and propionylated composites are able to form a good bonding between the fibre and matrix. Furthermore, unsaturated polyester, which is classified as thermoset matrix, has slightly high heat resistant. This is because of the existence of its chemical cross-linking which leads to the formation of a tightly bound three-dimensional network of polymer chains. The final degradation temperature for modified composites also shifted to higher range of temperature (473–485°C) as compared with unmodified composite which is 457°C. At this point, it is clearly proved that unmodified composites degraded rapidly due to the weak bonding between fibre and matrix. Unmodified composite also left slightly high char residue, which is 11.4% as compared with propionylated and succinylated composites, which is 10.9% and 9.6%, respectively.

Conclusions

The modified kenaf fibre composites yielded better mechanical properties and were confirmed by morphological studies. The treatment by succinic anhydride and propionic anhydride led to enhanced interfacial bonding between fibre and polyester matrix. SEM investigation of modified composites revealed better interaction between fibre and matrix as compared to the unmodified biocomposites. SEM studies also revealed that increased interfacial adhesion was observed in the modified biocomposites. TGA studies showed better thermal stability as compared to unmodified composites. Therefore, this study confirms that treatment of fibre by anhydrides can be feasible to use in biocomposites technology. The biodegradable nature of kenaf fibres will make the fabricated biocomposites more environmentally friendly and will be of high economic value. Developed treated kenaf fibre polyester composites provide an opportunity for replacing the existing materials with high mechanical and thermal properties, low cost alternatives and that are environment friendly. Treated kenaf fibre reinforced polyester composites can be used for boat decking, aerospace, automobile, building and in sport components.

Funding

This research received no specific grant from any funding agency in the public, commercial or not-for-profit sectors

Acknowledgements

The researchers would like to thank the Universiti Sains Malaysia, Penang for providing facilities that has made this work possible.

Conflict of interest

None declared.

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