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Effects of reinforcing arrangement of kenaf fibres into unsaturated polyester for improved properties

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ABSTRACT

In this current study, the mechanical, structural and morphological properties of kenaf fibre based polyester composites based on reinforcing arrangement were investigated. Fibres were placed in layers into the resin to enhance reinforcement. A hand lay-up technique was used to prepare the sandwich composite. Composites were characterized by tensile, flexural and impact testing. The structural properties as well as crystalline behaviour were observed by X-ray diffraction analysis. Additionally, the surface of the fractured samples was also examined using scanning electron microscopy to understand the interaction behaviour between the fibres and matrix. Results of the analyses revealed that single layered kenaf mat enhanced the tensile strength by 80%, whereas the double layered improved by 156%, in the presence of a fixed amount of curing agent. The thermal properties of the composites based on double layered kenaf mat were also found to improve compared to the others. The double layered composites can be used as high temperature-sustained roofing materials to replace traditional products.

***Keywords*:** Polyesters; composites; kenaf fibres; mechanical properties; thermal properties.

INTRODUCTION

Natural fibres (NFs) possesses a number of benefits which add values to polymer composites in terms of strength, structural flexibility, light weight, renewability and cost reduction [1-8]. In addition, environmental sustainability and reduced demand of polymer are also considered as significant impacts of using them with polymer matrices [9-11]. There are drawbacks of using NFs, which are also found in the literature as reported by different researchers. For example, NFs are hydrophilic, which is different than natural polymer as they are more hydrophobic in nature [12, 13]. This negative characteristic enhances moisture absorption by composites, and makes them incompatible with polymer matrix, resulting in poor interfacial adhesion and weakened formulated composites. In addition, it limits the use of composites for high performance and outdoor applications. The properties of composites depend on various parameters like matrix and fibre strength, fibre aspect ratio, interfacial adhesion between fibres and matrices, etc. An effective reinforcement can produce good composite performance. The reinforcement of NFs inside polymer matrices can be done efficiently by different techniques, such as physical and chemical treatments of fibre, usage of coupling agent and processing technique of composites formulation [14-16]. Few common and efficient processing techniques

include extrusion, injection and compression moulding. For liquid resin, melt mixing and compression moulding are effective. Hot or cold pressing at high pressure and required temperature have also been found as effective reinforcing techniques.

The dispersion and placement of fibres inside polymer matrix is important. The fibre loading can be made up to 40 to 50%. The properties of the composites depend on the volume of the fibre, aspect ratio, size, orientation into the polymer, processing techniques, types and quality of the fibres, and the polymer matrix used [1, 17-19]. The alignment of fibres according to axial or bi-axial arrangement has been found influential in varying the strength of the composites. Therefore, the current study deals with fibre dispersion inside the matrices for improved performance of kenaf fibre based polyester composites. Important NFs for composite preparation are sisal, kenaf, jute, coir, oil palm empty fruit bunch, etc. Among the fibres, kenaf is long fibre and suitable to be incorporated into polymer matrix for reinforcement purposes. Kenaf fibres have been used for composite fabrication with different thermoplastic and thermosetting polymers like polypropylene, polyesters, polyethylene, etc [12, 20-23]. Studies have reported that fabrication methods use 3 to 5 mm chopped fibres [9]. The variations in properties are also claimed as a function of fibre loading and aspect ratio [24]. Polyester from among the thermosetting resins is used for composite preparation [24, 25]. They are important in hand lay-up technique for easy fabrication of the composites. The aim of this study was to determine the effects of reinforcing arrangement of kenaf mats as layers into a polymer matrix, polyesters. The reinforcing arrangement has a significant contribution on mechanical properties, which has not been reported for the case of kenaf mat and polyester composites.

MATERIALS AND METHODS

Materials

Green kenaf fibres were collected from Kuala Rompin, Pahang, Malaysia. The fibres were dried in sunlight for four days at 32^{o}C. The net structure was prepared by placing ten long (25 cm) fibres horizontally and ten vertically one-after-another like a net structure. The polymer matrix chosen was commercially available unsaturated polyester (UP) resin, whereas methyl ethyl ketone peroxide (MEKP) and cobalt naphthenate were used as accelerator and catalyst, respectively, for the curing process of the resin.

Composite Fabrication

The amount of liquid resin required to soak all of the fibers completely was considered to be as much as resin. It was estimated that 100 ml of resin is sufficient for single layered and randomly oriented fibres, whereas, 150 ml is adequate for double layered. The amount of MEKP used for 100 ml of resin was 5 ml and for 150 ml was 7 ml. The amount of cobalt naphthenate used for all samples was 0.5 mg. The fabricated composites were of two types: single layered and double layered. The process flow including the formulation of the composites and the formulated samples are illustrated in Figure 1. For comparison, 2-5 mm fibres were mixed with the liquid resins. The sample prepared without fibre is called UP, single layered fibre based UP composite (SUPC), double layered fibre based UP composite (DUPC) and randomly oriented fibre based UP composite (RUPC).

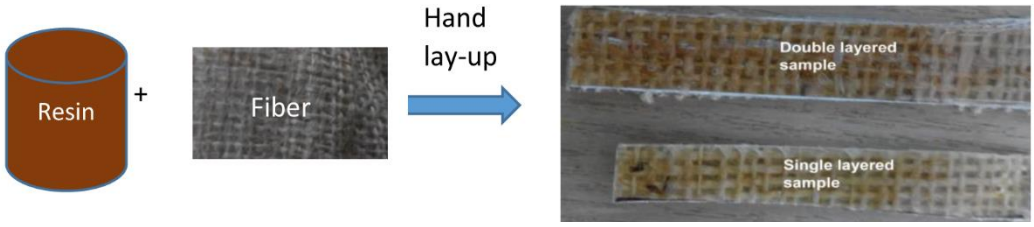

Figure 1. Samples showing double layered and single layered fibre replacement inside the polymer matrix.

Tensile Properties

The tensile test was carried out using Universal Testing Machine AG-1 (Shidmadzu, Japan), following the method described in ASTM D 638 – 01. The cross-head speed was fixed at 10 mm/min and gauge length was maintained at 65 mm. The experimental set-up for this testing is presented in Figure 2. Five specimens were tested, and the average value was taken for data analysis.

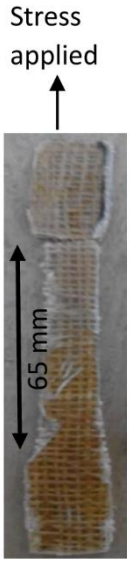

Figure 2. Tensile testing of the sample.

Flexural Properties

The flexural test was conducted according to ASTM D790-97 method by using a universal testing machine (model- SHIMADZU, AG-1) with static load cell of 1 kN. The support was set at 20 mm, and the cross-head speed was fixed at 10 mm/min. Five specimens were tested, and the average value was taken for data analysis.

Charpy Impact Test

The charpy impact tests were performed using impact pendulum tester (model-ZWICK/ROELL) at 90^{o} swing angle with a hammer of load 1J. The charpy impact tests were carried out according to ASTM D256 standard with a rectangular specimen test bar of fixed dimension (55 × 3.3 × 10mm). The Davenport notch cutting apparatus were used to notch the specimen and the notch depth was fixed at 2mm with an angle of 45^{o}. Five specimens were tested, and the average value was taken for data analysis.

X-ray Diffraction Analysis

The crystalline property of the polymer matrix and the composites were measured by X-ray diffraction (XRD) analysis. For this purpose, A Rigaku Mini Flex II, Japan, was used with tube current and operating voltage 15 mA and 30 kV, respectively. The samples were scanned step-wise from 5^o to 40^o with a scattering angle (2θ) by using Cu K_α radiation of wavelength $\lambda 1.541 Å$.

SEM

A scanning electron microscope (SEM) (model-ZESIS) was used to observe the surface images of the fractured samples. The samples were dried in an oven at 60^oC before the observation. Air dried samples were fixed to a metal-base specimen holder using double-sided sticky carbon tape, and then coated with gold using a vacuum sputter-coater to make them conductive prior to SEM observation.

Thermal Analysis

Thermogravimetric measurements were carried out using a thermogravimetric analyser (TGA) of model: TA instrument, TGA Q500. Each sample was weighed nearly 5 mg and heated at a temperature range of 40–600^oC with a heating rate of 20^oC/min. TGA analyses were conducted in a platinum crucible under nitrogen atmosphere at a flow rate of 40 ml/min to ensure inert atmosphere.

RESULTS AND DISCUSSION

Tensile Properties

The tensile strength (TS) and tensile modulus (TM) of UP and composites based on randomly-oriented short fibres as well as kenaf mat of different number of layers are illustrated in Figure 3. From the results, the tensile strength of UP was found to be 5.1 MPa, which improved by up to 7.0 MPa through the incorporation of randomly oriented short kenaf fibres. This increase could be due to the load transfer and interfacial adhesion between the fibres and matrix [12]. On the other hand, the incorporation of single layered kenaf fibres mat enhanced the TS (9.2 MPa) by 80%, which further improved by the incorporation of double layered kenaf fibre mat. DUPC showed a TS of 13.1 MPa, which was an increment by 156% from UP and 42% from SUPC. The improvement for SUPC compared to RUPC was probably due to equal load transfer by the structured kenaf mat, whereas the highest TS was observed by DUPC as a result of double layered mat, which contributes equal maximum load transfer due to structured fibres arrangement [26].

The same trend was obtained for TM. The TM of UP, RUPC, SUPC, DUPC was found to be 90, 120, 322 and 464 MPa, respectively. Significant enhancement in tensile strength and modulus was obtained with increasing number of fibre layers. The fibres mainly play an interesting role in enhancing the stiffness of the composites by supporting the applied load. Therefore, by increasing the fibre layer, the degree of obstruction increases, which consequently increases the stiffness of the composites [27].

Flexural Properties

The flexural strength (FS) and flexural modulus (FM) of the composites are illustrated in Figure 4. The FS of UP, RUPC, SUPC and DUPC was found to be 11.5, 15.7, 19.2 and 23.1 MPa, whereas the FM was found to be 235, 320, 422 and 564 MPa, respectively. FS improved due to the incorporation of randomly-oriented fibres. Normally hard PU are brittle, but due to fibres inclusion, the applied load was shared and the stress transfer was

possibly carried out through the fibres, which ultimately improved the stiffness of the composites, and as a result improved the bending strength [13]. However, the single layered kenaf mat enhanced FS from 11.5 to 19.2 MPa, which was 3.5 MPa higher than RUPC. Finally, the maximum FS (23.1 MPa) was observed with double layered mat. The same trend of results was observed for FM. The maximum FM was noted for DUPC as 564 MPa, which was 140% higher than PU. The improvement was thought to be due to higher interfacial adhesion built due to the special arrangement of the fibre mat [1].

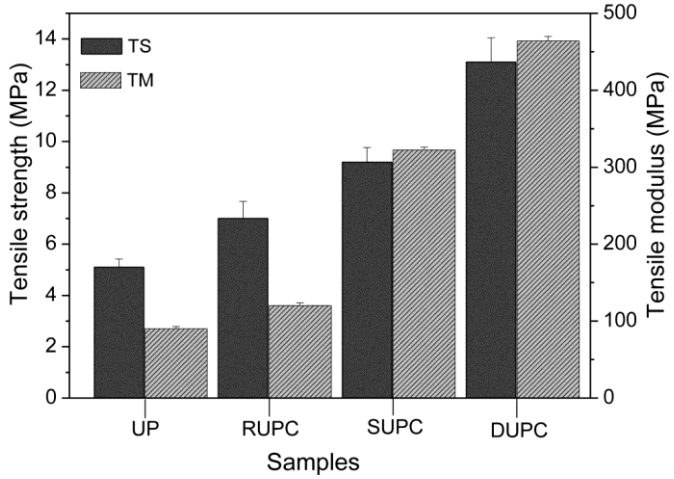

Figure 3. Tensile strength (MPa) and tensile modulus (MPa) of the composites.

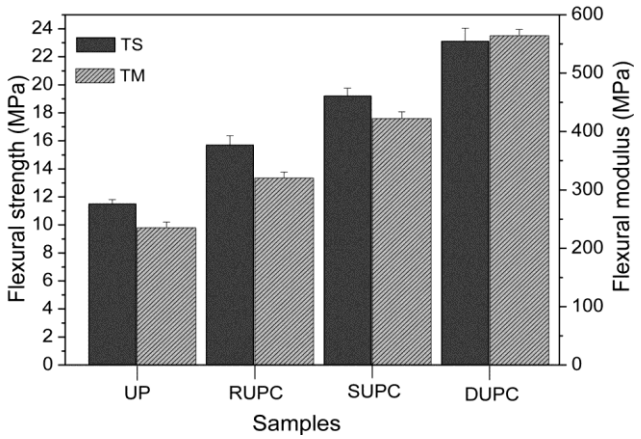

Figure 4. Flexural strength and flexural modulus of the composites.

Charpy Impact Properties

The impact strength (IS) of UP and composites are tabulated in Table 1. The IS of UP was found to be 3.82 kJ/m^2, whereas RPEC showed a higher value of 4.56 kJ/m^2. On the other hand, the incorporation of single layered kenaf mat enhanced the value to 10.83 kJ/m^2, which was found to decrease with further layer (second layer) to 9.73 kJ/m^2. The value decreased due to the probability of fibre agglomeration, which resulted in stress concentration region which requires less energy for crack propagation [28]. In addition, it was also reported that the impact strength decreased with fibre loading due to weak interfacial bonding between fibre and matrix, which causes micro crack to occur at the point of impact. Similar observation was documented elsewhere [29, 30]. It was based on the phenomenon of as fibres volume increases, the wettability decreases as an outcome

of lack of interfacial adhesion. Therefore, the formation of solid interface bond requires less energy absorption to be cracked.

Table 1. Impact strength of UP and different composites.

Samples	IS (kJ/m^2)
UP	3.82
RUPC	4.56
SUPC	10.83
DUPC	9.73

Structural Properties

The XRD diffractograms of different samples are presented in Figure 5. It can be seen that all samples including UP, RUPC, SUPC and DUPC show similar trend of curve, except for changing intensities. The curve intensity of fibre loaded UP was slightly high due to higher amount of crystallinity [31]. The fibre acted as a nucleating agent which were thought to be the reason behind this statement. Table 2 shows the crystallinity of the samples that was calculated based on the graphs generated by XRD diffractograms. RUPC showed higher intensity compared to UP. The crystallinity was found to be 44% for the case of UP, whereas RUPC showed a crystallinity of 53%. Improved interfacial adhesion between the fibre and matrix is a possible reason behind this observation. Further, the crystallinity was found to improve for the case of SUPC due to single layered kenaf mat (58%), which was found to be the lowest (50%) among the composites for the case of DUPC. The increment of crystallinity was probably due to the presence of crystalline cellulose fibre and their special arrangement into the matrix [12].

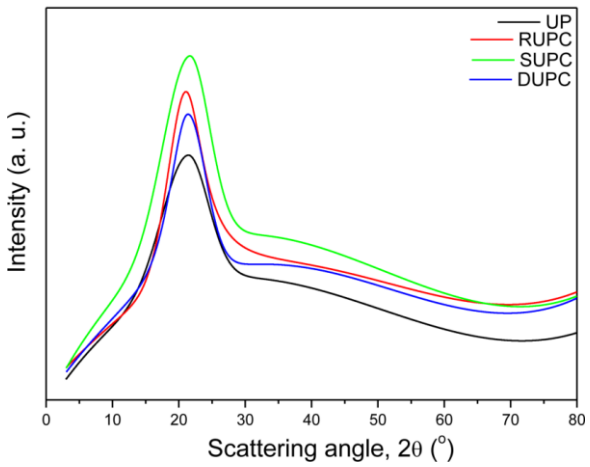

Figure 5. XRD patterns of different samples.

Surface Morphology

The surface morphology of the tensile fractured samples is represented in Figure 6. Figure 6(a) shows the surface of the UP sample. From the image, the fracture was visible on the surface due to its brittleness. After randomly loading kenaf fibres, the surface (Figure 6(b)) was found to be rigid without any visible fracture. Therefore, it can be stated that the brittleness of the sample was reduced due to the incorporation of the fibres. Likewise, the surface of SUPC and DUPC was found to be similar, where no fracture was found. From another point of view, pulled out fibre due to stress was found to be maximum with

DUPC, as a large number of fibres were visible. In comparison, the surface of RUPC and SUPC showed lesser amount of pulled out fibre which is as a consequence of better interfacial adhesion between fibres and matrix [32].

Table 2. Thermal properties of the composites.

Samples	Crystallinity (%)	T_{onset} (^oC)	T_{max} (^oC)	Residue (wt.%)
UP	44	359	410	3.0
RUPC	53	313	416	4.5
SUPC	58	358	417	5.8
DUPC	50	313	417	6.0

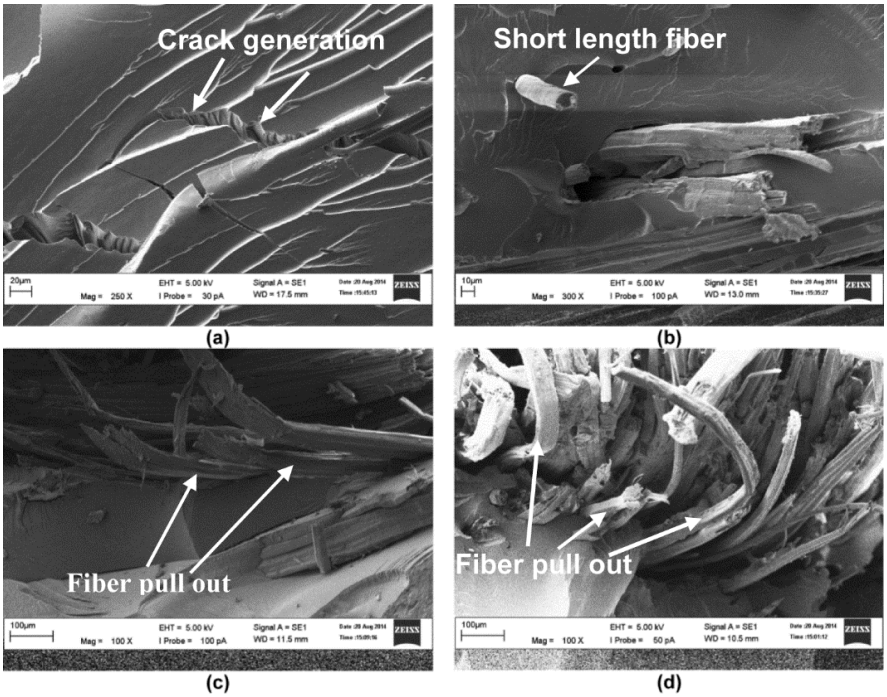

Figure 6. SEM images of the fractured samples of the composites: (a) UP, (b) RUPC, (c) SUPC and (d) DUPC.

Thermal Properties

The weight versus temperature, and the derivative weight versus temperature plots from thermogravimetric analysis are illustrated in Figures 7 and 8, respectively. Table 2 shows the onset degradation temperature (T_{onset}) of different samples. The onset of the degradation was started for PU at temperature of 359^{o}C, which was decreased due to randomly oriented short kenaf fibre incorporation and started at temperature of 313^{o}C. This is probably due to the low temperature sustainability of natural bio-based fibres [33]. On the other hand, the single layered mat reinforced composite showed an onset degradation temperature of 358^{o}C, which was close to pure UP, which was due to better reinforcing arrangement of the structured fibre mat and UP [33]. DUPC showed lower onset stability by showing degradation at 318^{o}C, which is because of fibres agglomeration, as confirmed by the SEM image. From Figure 8, the T_{max} of PU, RUPC, SUPC and DUPC was found to be 410, 416, 417 and 417 ^{o}C, respectively (Table 2). The

residue of the samples was found to be 3.0, 4.5, 5.8 and 6.0%, respectively (Figure 7). Due to the presence of natural fibres inside the polyester based composites, the degradation profile can be explained in three steps. The removal of moisture was observed after 100°C, which corresponds to about 1.0 to 2.0% weight loss. Further low molecular weight compounds were found to be degraded after 250°C and the estimated weight loss was confirmed as nearly 20%. Finally, the main degradation occurred at around 350°C due to polymer chain break down, and completed by 450°C with residues stated earlier.

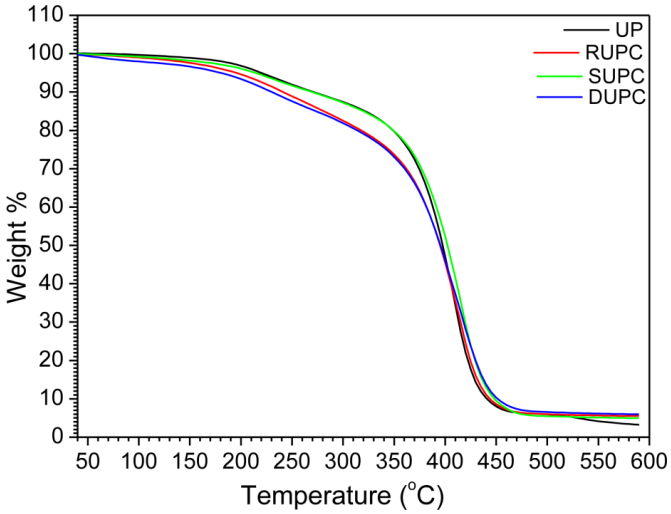

Figure 7. TGA analysis of different composites.

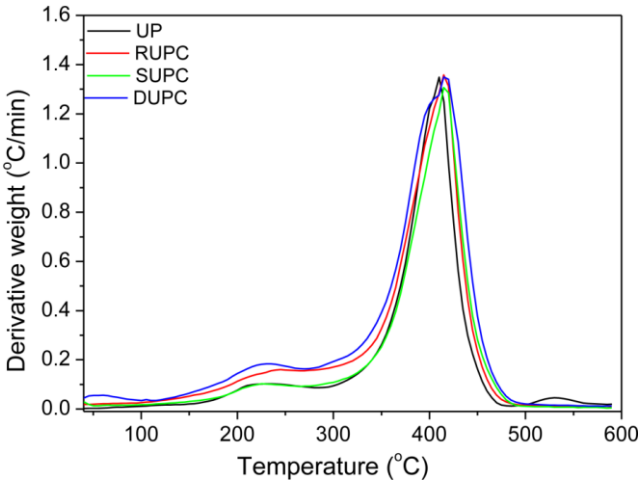

Figure 8. Derivative weight vs temperature curves of different composites.

CONCLUSIONS

The arrangement of reinforcement of long kenaf fibre was assessed for the preparation of unsaturated polyester resin based bio-composites. It was found that randomly oriented fibres based composite (RUPC) showed better mechanical strength than pure UP. On the other hand, single layer based composite (SUPC) showed improved tensile, flexural and impact properties compared to RUPC. While considering the properties of DUPC, it was found that the impact strength was lower. The surface morphology of RUPC and SUPC

was also found to be better than DUPC as confirmed by short length fibres. The crystallinity was found to be improved with single layered kenaf mat incorporation, whereas double layered mat was found to have discouraging outcome. Finally, the thermal stability of the composites was found to be close to each other and the degradation completed before reaching 450^{o}C.

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