

Jute Fabric Reinforced Engineering Thermoplastic Sandwich Composites. I. The Effect of Molding Time

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ABSTRACT: Jute fabric-reinforced sandwich composites were fabricated using engineering thermoplastics. The jute fabrics were pre-coated with thermosetting resin to improve their thermal resistance before molding of the composites. Thermal gravimetric analysis (TGA) studies revealed that the resin coated fabrics decomposed at higher temperature than the uncoated jute. The onset of degradation of the coated fibers also falls between that of jute fibers and the thermoset resins. This indicates the presence of good interfacial bonding between jute fibers and both resins. Isothermal TGA studies revealed that jute could withstand brief exposure to higher temperature at 270 and 290°C. The sandwich composites were fabricated at 270°C by compression molding for 1.5 and 3 min in each case, and then characterized by flexural, tensile and morphological studies, i.e., SEM and optical microscopy. The uncoated jute fabric yielded composites of superior mechanical properties even with 3 mins molding at 270°C which is close to the degradation temperature of uncoated jute fibers. This is an indication that it is feasible to prepare jute fiber filled engineering polymer composites provided the exposure time at high temperature during processing does not exceed 3 mins as determined by TGA isothermal studies. SEM studies revealed strong fiber/matrix interfacial bonding between jute and the thermoset resins while the inferior mechanical properties of the resin coated sandwich composites could be attributed to the poor interfacial bonding between the already cured thermoset coating and the matrix based on optical microscopy of the polished cross-sections. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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INTRODUCTION

Fiber reinforced composites, usually made of glass, aramid, or carbon fiber reinforced with polymer, are being used extensively, because of their impressive mechanical performance. However, they are nonbiodegradable and expensive materials while they do not also respond to the current environmental concerns.^{1–4}

In recent years, many researchers have shown interest in thermoplastic composites reinforced with natural fiber. Natural fibers, such as jute, sisal, and hemp can potentially serve as cheap reinforcement of polymers if compounded into polymers without decomposition. Natural fiber reinforced thermoplastic composites have been used widely, because the combination between natural fiber and thermoplastic composites can produce high performance composites and the development of natural fiber composites has been a subject of interest for the past few years. These natural fibers have low density and low cost.

Moreover, they are renewable, biodegradable, and environment friendly.^{5–10}

Textile reinforced composites enjoy technical applications in aerospace, marine, and also transportation industries, because they yield affordable and high-quality composites.^{11–13}

The main constraint of natural fiber reinforced composites has been the limitation of choice of polymer matrix. This is because natural fibers degrade at temperatures below the processing temperatures of engineering polymers. As such, the polymers currently in use as matrices for natural fiber filled composites are those with low-to-moderate processing temperature.^{14–26} These comprise of thermoplastic polymers such as polypropylene,^{22,23} polyethylene,^{18–21} polyvinyl chloride,¹⁷ and polystyrene. There is also the problem of poor compatibility between the predominantly hydrophobic thermoplastic polymers and the hydrophilic cellulosic natural fibers.²⁷ However, such incompatibility has not been reported in the case of thermoset polymers

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such as epoxy,^{18–23} polyesters,²⁸ and phenol formaldehyde.^{26,29} The processing temperatures of both thermoplastic and thermoset composites ranges below 200°C. Because of the poor interfacial interaction leading to poor bonding between the hydrophobic thermoplastics and natural fibers, polymer-fiber bonding is often improved by the use of coupling agents and fiber surface treatments.^{24,25,30,31}

To expand the scope of natural fibers in composites applications, innovative techniques are required to reduce the gap between the processing temperatures of engineering polymers and the decomposition temperatures of natural fibers. Sadhan and Alberto^{32,33} attempted this using a reactive solvent, a low molecular weight epoxy to form a miscible blend with poly(phenylene ether) (PPE). This offered much lower viscosity compared to that of PPE and processing temperature below the decomposition temperature of the natural fiber. They also noted compatibility between the polar wood flour and the epoxy component. The drawback of this system is that the resulting composite is a blend, which gets weaker with reducing PPE content.

The main objective of this study is to enhance the thermal resistance of natural fibers to facilitate their usage with high temperature engineering thermoplastics. The jute fiber-reinforced sandwich composites were fabricated using high temperature engineering thermoplastics such as poly(ethylene terephthalate) (PET) and polycarbonate (PC) as the matrix resins. The jute fabrics were precoated with thermosetting resin to improve their thermal resistance before molding of the composites. It is also expected that good compatibility would exist between jute fibers and the polar thermoset resins.³² The thermal decomposition characteristics of the jute fibers as a function of time were evaluated using thermal gravimetric analysis (TGA) while static mechanical tests were performed to evaluate the performance of the composites.

EXPERIMENTAL

Materials

The matrix materials used in this chapter were recycled polyethylene terephthalate (RPET) and PC. RPET recovered from post-consumer waste bottles, was provided by Yasuda Sangyo, PC (grade S3000) was supplied by Mitsubishi Engineering Plastic.

Two types of thermosetting resins were used as surface coating, i.e., epoxy resin and unsaturated polyester (UP) resin. Epoxy resin consists of liquid Bisphenol A 78% and butyl glycidyl ether 11% and the curing agent (grade diethyl triamine) were supplied by Refinotech, while the UP resin that consists of 50–60% of styrene (grade 150HR BQNTINW) was supplied by Highpolymer, Methyl ethyl ketone peroxide (grade PERMEK N) was used as curing agent for UP, which was supplied by NOF, Japan.

Recycled jute coffee bags with a thickness of ~0.5 mm provided by a coffee company in Japan were used as the reinforcement. The jute coffee bags were cut into 150 × 150 mm² sizes.

Sample Preparation Methods

Preparation of Matrix Resin Sheet. RPET and PC pellets were dried at 120°C for 8 h. These pellets were then used to form matrix sheets by compression molding at 290°C to form 1 mm-

thick sheets. The compound was preheated for 4 mins before applying a pressure of 20 MPa for another 3 mins. The mold was subsequently quench cooled by flowing water while the specimens were kept under constant pressure. The size of the matrix sheet was 150 mm long × 150 mm wide × 1 mm thick.

Surface coating Using Thermosetting Resin. Jute fabrics were dried at 80°C for 6 h before being immersed in epoxy or UP resin. The ratio of the resin and its curing agent was determined in weight ratio of 100 : 10 and 100 : 1 for Epoxy and UP resin, respectively. After immersion, the jute sheets were hung up and allowed to cure at room temperature for 12 h. Postcuring of the jute sheets was done in an oven set at 100°C for 2 h. The thickness of the resin coating was determined to be ~0.15 mm. The weight content of epoxy or UP coated jute was 20% of jute fabric. The coated jute sheets were 150 mm long × 150 mm wide × 0.5 mm thick. In addition, samples consisting of a layer of epoxy or UP sandwiched between two matrix sheets of either PC or RPET were prepared under similar conditions for the sake of comparison with the coated jute fabric sandwich composites.

Processing

Compression Molding of Composites. The composites consist of a layer of jute fabric sandwiched between two matrix sheets of either PC or RPET. The composites were prepared by compression molding. The molding temperature was set at 270°C. Compression molding was done at a constant pressure of 20 MPa. The molding time varied between 1.5 and 3 mins. All the composites processed had total fiber weight fraction of 10% (±1). The fiber volume fraction of composite was about 20% for every condition.

Characterization

Thermal Stability. Thermogravimetric analysis (TGA) was used to determine the thermal stability of resin coated and uncoated jute fabric under elevated temperatures using Thermogravimetric Analyzer (TGA-2950, TA Instruments). The specimen was heated from room temperature to 600°C at a heating rate of 50°C/min in air atmosphere.

Isothermal Degradation. Thermogravimetric analysis (TGA) was used to determine the thermal stability of jute fabric before and after resin coating with a thermogravimetric analyzer (TGA-2950, TA Instruments). Thermal stability resistance of jute fabric as a function of time was investigated by isothermal heating in air. The temperature was increased from room temperature at a heating rate of 100°C/min and held isothermal at 160, 270, and 290°C for 30 min in each case.

Flexural Test. Flexural test specimens were cut into strips with dimensions of 120 mm long × 10.0 mm wide × 2.8 mm thick in accordance with ASTM D790. An Instron 4206 universal testing machine was used to carry out the tests at 28°C. The flexural tests were done at a crosshead speed of 1 mm/min and span length of 40 mm. At least five specimens were tested for each sample.

Tensile Testing. Tensile testing was carried out with an Instron 4206 machine at 28°C, according to ASTM D 3039, at a cross

head speed of 1 mm/min. At least five composite specimens were test for each condition.

Morphology Studies. A scanning electron microscope (JEOL, JSM5200) was used for morphology studies of the gold sputtered fracture surfaces of the composites. The polished cross-sections of the composites were observed using a digital microscope (Keyence VH-S30). The specimens were finely polished to a mirror finish by gradually changing the roughness of the polishing medium from coarse to fine (i.e., polishing paper index from 200 to 400, 800, 1200, and 2000) with constant water flow over the specimen and polishing papers to prevent the specimens from being damaged by heat and also to flush away the debris. Further polishing using graded alumina suspensions in water was performed. The alumina practice size was gradually changed from 1.0 to 0.1 and finally 0.05 μm (3 min for each polishing stage). The polished specimens were thoroughly washed to discard any residue by immersing the specimens into an ultrasonic cleanser filled with clean water for 15 mins.

RESULTS AND DISCUSSION

The thermogravimetric analysis (TGA) thermograms for epoxy and UP coated jute fibers are compared with that of uncoated and coated jute fibers in Figure 1. It can be seen that the onset of degradation in the jute fibers commenced at a much lower temperature (274°C). In the case of the epoxy coated and UP coated jute fabrics, the onset of degradation was at 340 and 348°C, respectively. This indicates that the thermal degradation resistance of the coated jute fibers is higher than that of the uncoated jute fibers. The jute fibers initially made contact with the epoxy or UP at ambient temperature and at low resin viscosity. Wetting of the jute fibers is expected to be essentially complete before curing takes place and postcuring at higher temperature.

The onset of degradation of the coated fibers also falls between that of jute fibers and the resins in both cases. This indicates the presence of good interfacial bond between jute fibers and both resins. The jute fibers have polar surface and so are epoxy and UP. Therefore strong interfacial interaction is predictable. When these coated jute fibers are incorporated into engineering

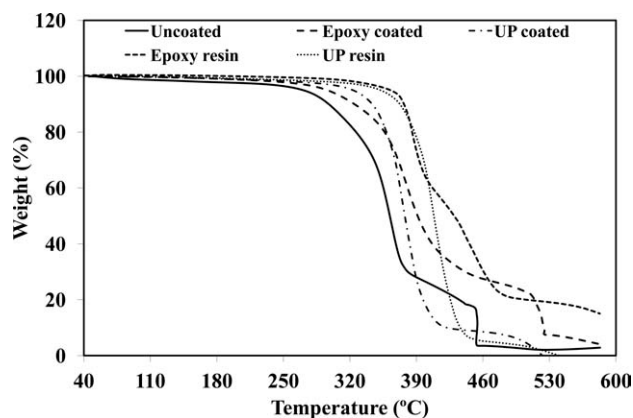


Figure 1. Thermograms showing the influence of epoxy resin and UP coating on thermal degradation resistance of jute fiber.

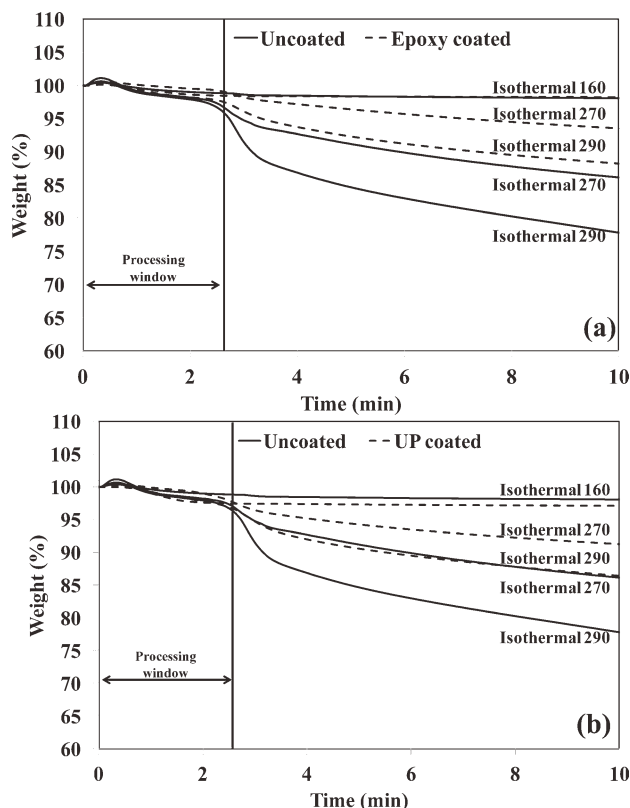


Figure 2. Thermograms of isothermal degradation of (a) epoxy-coated jute fiber and (b) UP at 160, 270, and 290°C.

thermoplastics an improvement in interfacial interaction is expected for two reasons. In the first place the coatings would serve as coupling agents between the engineering polymer and the natural fiber. Secondly, the coated jute fibers would be able to withstand the higher processing temperatures of the engineering polymers due to the improved thermal resistance indicated in Figure 1.

Isothermal Degradation

The resistance of the coated fibers to high temperatures was also investigated. The TGA thermograms obtained for the epoxy and UP coated resins under isothermal heating conditions are shown in Figure 2. The temperature was increased from ambient temperature at a ramp of 100°C/min and held at 160, 270, and 290°C for 30 min in each case. The jute fibers appeared to be thermally stable when heated at 160°C, irrespective of whether they were coated or not. At 270 and 290°C, however, the fibers would start to degrade significantly after about 3 min. Therefore, it was determined that the molding time should be kept within 3 min, as shown in Figure 2 for epoxy and UP coated jute fibers. Subsequent studies were done while bearing this in mind.

Effect of Surface Coating on Interfacial Strength and Mechanical Performance of Composites

Figure 3 shows a typical stress-strain curve during flexural loading that can be used to determine fiber-matrix interfacial strength in textile composites. The fiber-matrix interfacial strength can be estimated from the knee point at the end of the

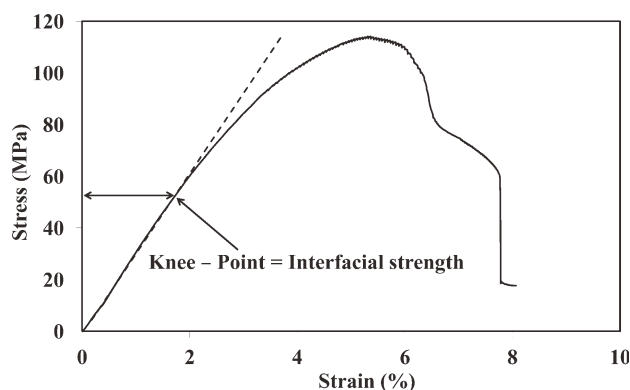


Figure 3. A typical stress-strain curve indicating knee point (interfacial strength) determination.

elastic region as indicated in the figure. The knee point is an indication of the initiation of fracture located between the plastic and elastic regions. This initiation of fracture can be used as a measure of the interfacial strength in the composites.³⁴

Figures 4 and 5 show the effect of epoxy and UP surface coating on the knee point or interfacial strength of jute fabric reinforced RPET and PC sandwich composites compared with the uncoated jute fabric composites. The composites were molded at 270°C for 1.5 and 3.0 min, respectively. It can be seen from both Figures that 1.5 mins molding time generally yielded composites with better interfacial strength than 3 mins. In the case of the RPET composites molded for 1.5 mins, the interfacial strength is 90 MPa for the uncoated jute fibers, 60 MPa for the epoxy coated and 35 MPa for the UP coated composites. In the case of composites molded for 3 mins, the uncoated one showed interfacial strength of 70 MPa, i.e., retention of 78%. The epoxy coated jute composite showed interfacial strength of 40 MPa or 67% retention while the UP showed a value of 38 MPa or 109% of the value at 1.5 mins.

In the case of the PC composites, the uncoated jute fibers yielded the highest value of 68 MPa for the 1.5 mins molding while the epoxy coated composite showed a value of 66 MPa and the polyester coated composites gave a value of 50 MPa. For the 3 mins

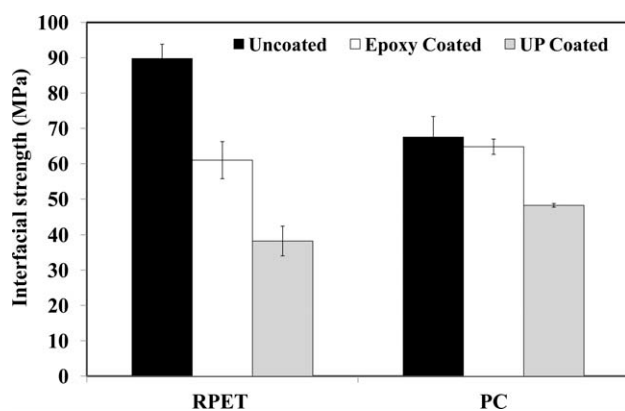


Figure 4. Interfacial strength of uncoated, epoxy and UP coated jute reinforced RPET and PC composites at the molding temperature of 270°C for 1.5 min.

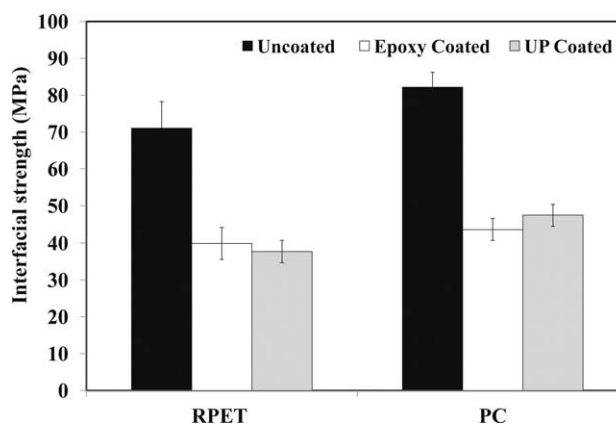


Figure 5. Interfacial strength of uncoated, epoxy and UP coated jute reinforced RPET and PC composites at the molding temperature of 270°C for 3 min.

moldings, the uncoated jute also gave the highest value of 82 MPa or 121% of the 1.5 min molding. The epoxy coated composite gave a value of 46 MPa or 70% of the 1.5 min molding while the UP coated composite gave a value of 48 MPa or 96%. It is obvious from the above that 1.5 to 3 min molding time is suitable for molding. It is again obvious that the uncoated jute fibers actually yielded composites of superior interfacial strength even with 3 mins molding. This is an indication that it is feasible to prepare jute fiber filled engineering polymer composites provided the exposure time at high temperature during processing does not exceed 3 mins. The good interfacial strength of the uncoated jute composites is easily understood as jute fibers are polar similar to the engineering plastics investigated.

It was also observed that the epoxy and UP coated jute fiber composites displayed inferior interfacial strength as compared to the uncoated jute fiber composites. This could be because the coatings are thermosets which crosslinked during the curing stage to form intractable mass with the fibers. So the coatings are not melted by subsequent heating during compression molding with the engineering polymers. Therefore there is no effective interaction between the engineering polymers and the thermoset coatings so a weak interface is formed between the thermoset coatings and the engineering polymers. Hence, the interaction between the uncoated jute fibers and the engineering plastics at the processing temperature is better than that between the cured epoxy and UP coatings. However, it can be seen that the interface was stronger between PC and the surface coating, which could be due to the availability of more amorphous chains in PC that could interdiffuse into the surface coating to form molecular entanglements. Furthermore, the fast solidification time of PC could retain more entanglements to attain better interfacial strength. RPET, however, would crystallize much easier than PC and would take more time to solidify. As such, less molecular entanglements were formed, which reduced interfacial strength.

Dependence of Flexural Properties of RPET and PC on Molding Time

The specific flexural properties of RPET and PC of epoxy and UP coated jute composites are presented in Figures 6–9. In

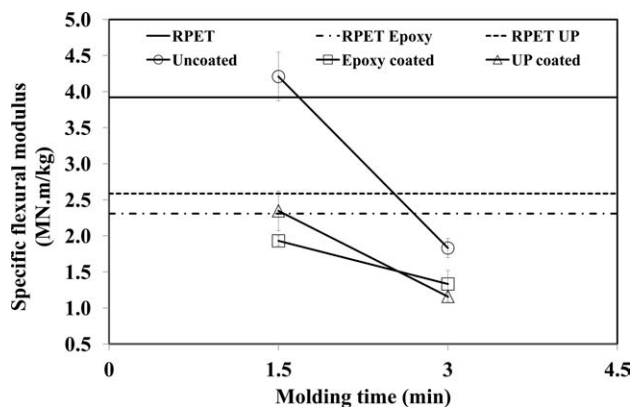


Figure 6. Specific flexural modulus of uncoated, epoxy and UP coated jute reinforced RPET at the molding temperature of 270°C for 1.5 and 3.0 min.

Figure 6, it can be seen that the specific flexural modulus of epoxy/RPET sandwich and UP/RPET sandwich were lower than RPET matrix. However, the values for epoxy/RPET sandwich and UP/RPET sandwich were higher than epoxy coated and UP coated jute fabric sandwich composites. This indicates that the presence of jute fabric in the composites further weakened them. The uncoated jute fabric showed values that were higher than those of RPET and both thermoset sandwiches at 1.5 min molding time. The uncoated jute fibers gave the highest value for RPET composites albeit it experienced a greater reduction in value at 3 min molding time, from 4.2 MN m/kg to 1.8 MN m/kg for the 1.5 and 3 min molding times, respectively, i.e., a retention of 43%. The property retention of the epoxy coated and UP coated composites is much better than the uncoated fibers. The epoxy coated composites gave values of 1.9 MN m/kg and 1.33 MN m/kg for the 1.5 and 3 min molding times, respectively indicating retention of 70% while the UP composites gave 2.3 MN m/kg and 1.2 MN m/kg for the 1.5 and 3 min molding times, respectively, indicating retention of 52%. Even though the uncoated fabric composite gave higher values at the respective molding times, it could be inferred from Figures 6 and 7 that the coated fabric composites had better retention of properties with 3 min molding. The superiority of the

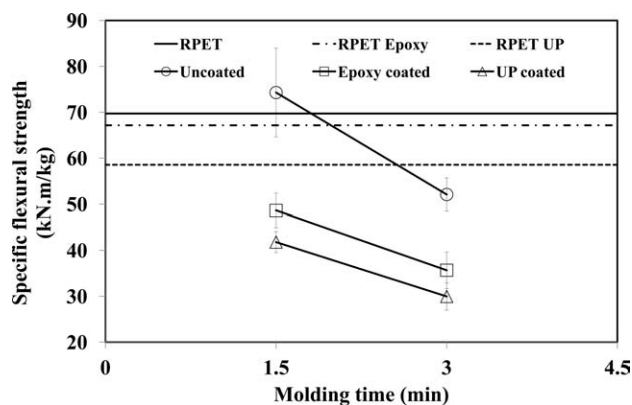


Figure 7. Specific flexural strength of uncoated, epoxy and UP coated jute reinforced RPET at the molding temperature of 270°C for 1.5 and 3.0 min.

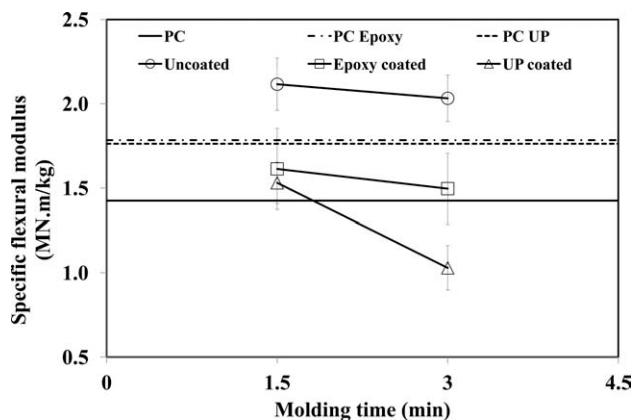


Figure 8. Specific flexural modulus of uncoated, epoxy and UP coated jute reinforced PC at the molding temperature of 270°C for 1.5 and 3.0 min.

properties at a shorter molding time could be attributed to fiber degradation as indicated in Figure 2. This degradation may not be significant but it sure has serious consequences.

The specific flexural strength of RPET composites also follow a similar trend as indicated in Figure 7. The specific flexural strength of epoxy/RPET sandwich and UP/RPET sandwich were lower than RPET matrix. It can be seen that 1.5 min molding time yielded composites with superior strength, while the 3 min molding time gave less with the resin coated composites showing better property retention. The uncoated jute fibers also gave the highest values at both molding times, followed by the epoxy coated and then the UP coated composites.

In the case of PC composites (Figure 8), the specific flexural modulus of epoxy/PC sandwich and UP/PC sandwich were higher than PC matrix. However, specific flexural modulus of the epoxy coated and UP coated were lower than epoxy/PC and UP/PC sandwiches while the uncoated jute fabric composite show the highest values. In the case of uncoated and coated jute fabric composites, the trend of specific flexural modulus is similar to the RPET composites. Here, the 1.5 min molding time gave composites of superior specific flexural modulus as

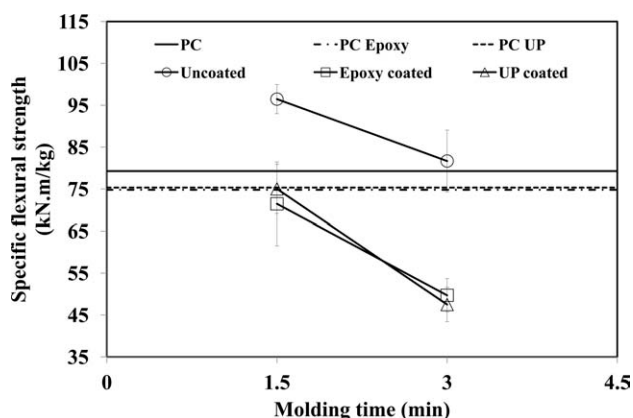


Figure 9. Specific flexural strength of uncoated, epoxy and UP coated jute reinforced PC at the molding temperature of 270°C for 1.5 and 3.0 min.

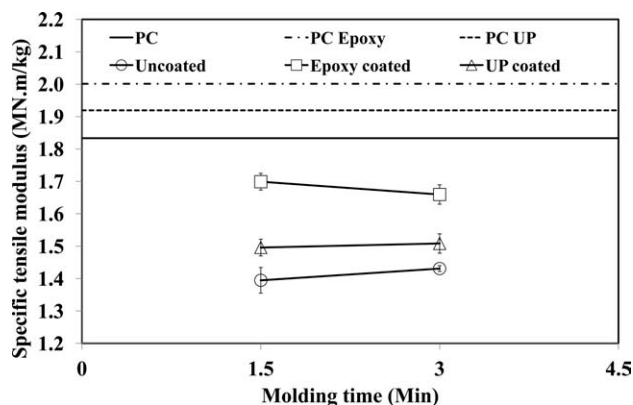


Figure 10. Specific tensile modulus of uncoated, epoxy and UP coated jute reinforced PC at the molding temperature of 270°C for 1.5 and 3.0 min.

compared to the 3 min molding time although the difference seems negligible in the cases of the uncoated and epoxy coated fabrics. Modulus is an indication of the rigidity of materials and it is possible that materials that are weak in terms of other mechanical properties could still maintain their rigidity. The uncoated fibers gave the highest values in both cases, followed by the epoxy and then the UP coated composites. The uncoated and epoxy coated fiber composites showed a slight decrease in value whereas the UP composites showed a greater decrease.

In Figure 9, The specific flexural strength of epoxy/PC sandwich and UP/PC sandwich were lower than RPET matrix. The specific flexural strength of the uncoated jute fabric and PC composites decreased considerably with increasing molding time from 1.5 to 3 min. The uncoated fabric composite also exhibited the highest value while exhibiting flexural strength that is better than that of the matrix even with 3 min molding. The epoxy and UP coated jute composites behaved in a similar manner, indicating decreases in values from 1.5 to 3 min molding time with values below that of the matrix.

Based on Figures 6–9 in both RPET and PC composites, 1.5 min molding time would not cause significant deterioration of mechanical properties. This shows that although the composites were molded at 270°C which is higher than the degradation temperature of uncoated jute fibers, the jute fibers could still withstand molding at temperature above its degradation temperature provided the exposure time is brief, i.e., kept lower than 3 min as indicated by the isothermal heating curve. However, it is obvious that 3 min molding time would cause significant deterioration in mechanical properties. This shows that although the composites were molded at 270°C, which was lower than the degradation temperature of coated jute as shown earlier in Figures 6–9, the jute fabric would still degrade with increasing molding time. Although the degree of deterioration in terms of composite modulus was quite similar in RPET and PC composites, the deterioration in strength was worse in PC composites. This indicates that longer molding times did not only caused degradation of the jute fibers but would also reduce mechanical properties.

Effect of Molding Time on Tensile Properties

Figures 10 and 11 show the effect of molding time on the specific tensile properties of uncoated and coated jute composites with PC. It can be seen from Figure 10 that the modulus of the coated and uncoated jute fiber composites remain steady with increasing molding time. The property retention is good until 3 min molding time. Moreover, the moduli of coated jute fiber reinforced composites were higher than uncoated jute fiber reinforced composites. This could be due to the improved stiffness of the jute fiber caused by the epoxy and UP coatings. This again confirms the observation based on the isothermal TGA thermograms. The specific tensile strength of the uncoated jute fibers also remain steady while those of the coated fibers showed a slight decrease with increasing molding time. It is again obvious that the tensile strength of the uncoated jute fiber composites is superior to the epoxy and UP coated ones even close to the safe limit. In all cases, it is indicated that composites fabricated with 3 min molding time could still retain their integrity.

Morphology

Figure 12(a–c) shows the scanning electron micrographs of the fracture surfaces of the uncoated, epoxy and UP coated composites respectively. It should be noted that the jute fabric was soaked in the uncured resins followed by room temperature curing and then postcuring at 100°C. So the jute fabrics are intimately bonded to the cured epoxy and UP resins. Therefore, in both cases jute is immediately surrounded by the thermoset resin. Even after the fabrication of the sandwich composite with the engineering thermoplastic matrices, the jute fabrics are still surrounded by the thermoset resins. This explains the intimate fiber/matrix bonding that is indicated in Figure 12(b, c). Intimate interfacial bonding is indicated by breakage of the fibers along the same plane as the resin. Fiber splitting plus the absence of fiber pull out indicate strong interfacial bonding. The mechanism responsible for the inferior mechanical properties of the resin coated composites can be deduced from cross sectional photographs of the composites at a lower magnification as indicated in Figure 13.

Optical photo micrographs of the cross sections of jute reinforced engineering thermoplastic composites are presented in

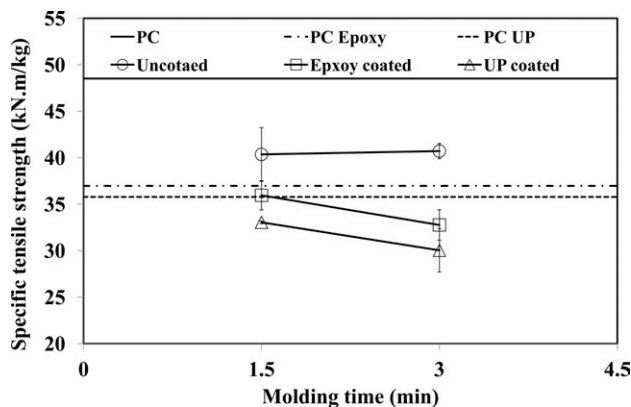


Figure 11. Specific tensile strength of uncoated, epoxy and UP coated jute reinforced PC at the molding temperature of 270°C for 1.5 and 3.0 min.

Figure 13(a–c). Figure 13a indicates very good interfacial interaction between jute and the engineering plastic thus the fibers bundles appear unperturbed. The interface between the fibers and polymer matrix undistorted. This explains why the mechanical properties of the uncoated jute fiber are superior to those of the coated fibers. Figure 13(b, c) on the other hand show the cross section of the epoxy and UP coated jute composites respectively. Insufficient interfacial interaction is indicated by the distortions and delamination at the resin/engineering polymer interface. As mentioned above, this could be due to insufficient interaction between the thermoset coatings and the thermoplastic engineering polymer due to the curing of the thermoset that has occurred before composite fabrication. The surface coated jute composites could be attributed to the fact that the thermoset resins are fully cured before the fabrication of the sandwich composite. Future investigations will be directed towards in situ curing of the thermosets during the sandwich fabrication stage.

CONCLUSIONS

TGA studies revealed that the thermoset resins coated jute fabric decomposed at higher temperature than the uncoated jute. The

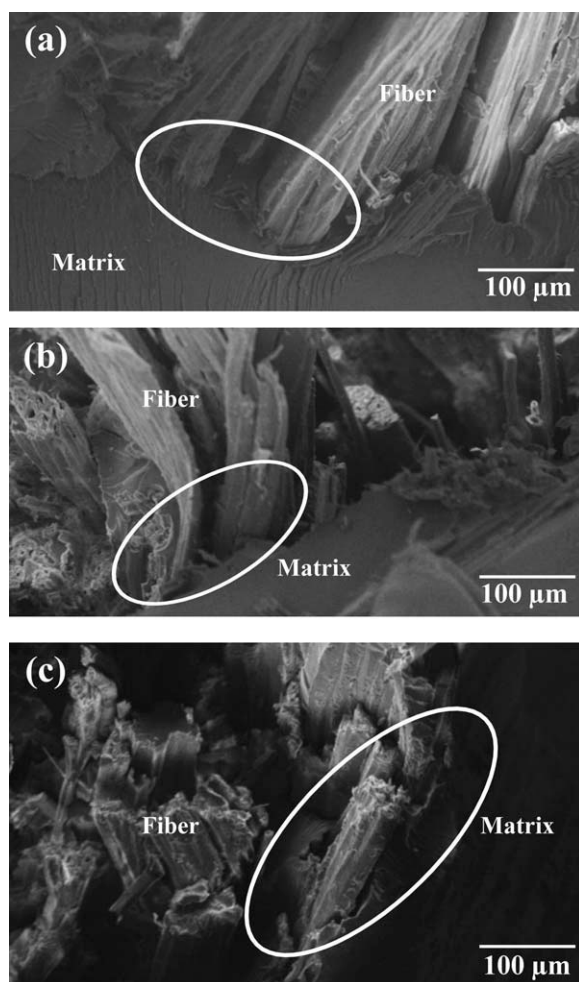


Figure 12. Scanning electron micrographs of the fracture surfaces of PC/Jute composites; (a) uncoated, (b) epoxy coated, (c) UP coated.

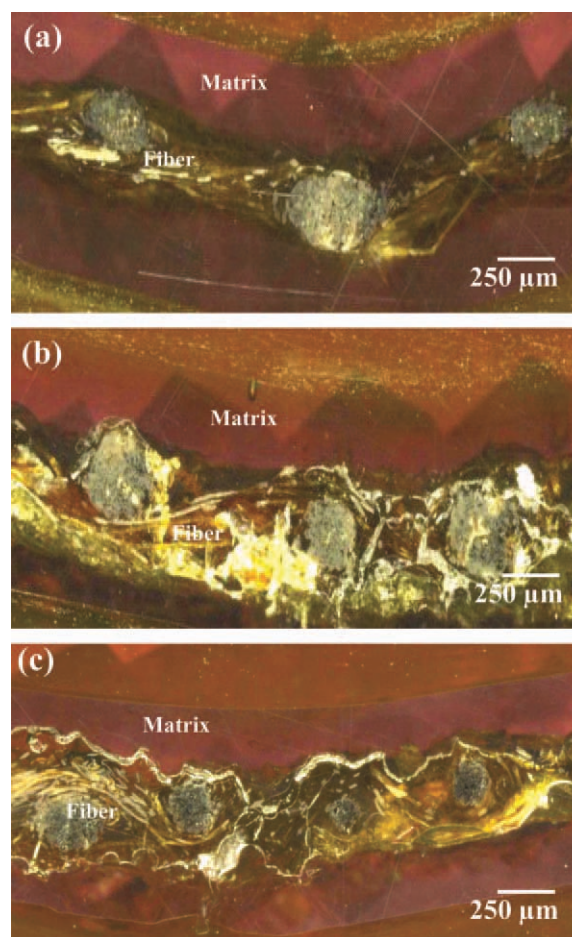


Figure 13. Photo micrographs of the cross sections of PC/Jute composites; (a) uncoated, (b) epoxy coated, (c) UP coated. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

onset of degradation of the coated fibers also falls between that of jute fibers and the thermoset resins. Indicating the presence of good interfacial bonding between jute fibers and both resins. Isothermal TGA studies revealed that jute could withstand brief exposure to higher temperature at 270 and 290°C. The uncoated jute fabric yielded sandwich composites of superior mechanical properties. This is a significant finding that it is feasible to prepare jute fiber filled engineering polymer composites provided the exposure time at high temperature is within the safe limit. SEM studies revealed strong fiber/matrix interfacial bonding between jute fabric and the engineering plastics while the inferior mechanical properties of the resin coated sandwich composites could be attributed to the poor interfacial interaction between the already cured thermoset coating and the engineering plastic.

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