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Properties of Pultruded Jute Fiber Reinforced Unsaturated Polyester Composites

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Abstract

This paper deals with an experimental study on the properties of pultruded jute fiber reinforced unsaturated polyester composite (PJFRC). The ratio of fiber to matrix is approximately 70:30 by volume. Compression and flexural testing were performed in order to study the mechanical properties while TMA and DMTA were used to study the thermal properties of the composites. Morphological aspects of the composites were also evaluated using an optical and scanning electron microscope. Compression and flexural stress–strain curves showed a linear portion at initial loading phase followed by yield and plastic deformation. For the DMTA evaluation it was found that the storage modulus is strongly dependent on temperature and degrades with increasing temperature. The TMA result showed a contraction of composite upon heating instead of expansion. The acid digestion test result confirmed that the fiber content was approximately 70% by volume. © Koninklijke Brill NV, Leiden, 2011

Keywords

Polymer composite, jute fiber, unsaturated polyester resin, pultruded jute composites

1. Introduction

The utilization of natural fiber reinforced composites has gained increased attention due to concerns of environmental preservation, renewability of raw material as well as the high specific mechanical properties of such composites. Natural fiber reinforced composites are undergoing a high technology revolution and are replacing synthetic fiber reinforced composites in high performance applications due to their significant advantages [1]. It has been reported that natural fibers like cotton, sisal, jute and coir exhibit good thermal and acoustic insulating properties, better electrical resistance and higher resistance to fracture. It has also been demonstrated that jute fiber reinforced composite has a huge potential to be used in various structural

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and non-structural applications [2]. Jute belongs to the genus *Corchorus*, family Tiliaceae, and is an example of a number of woody-stemmed herbaceous dicotyledons grown in the tropics and subtropics, from the bast of whose stems fiber can be extracted [3]. Jute has higher strength and modulus than plastic and is a good substitute for conventional fibers in many situations [4]. As far as jute fiber reinforced composite is concerned, most of the works reported have dealt with short jute fiber or jute in dust form [5]. At present, most of the pultruded composites available in the market use synthetic fiber, typically glass fiber. Unsaturated polyester resins (USP) are widely used as a matrix, thanks to a relatively lower price than other resins, ease of handling and a good balance of mechanical, electrical and chemical properties [6]. Due to the low shrinkage on curing and the fact that they are also easy to release from the die, they have become a popular choice for the pultrusion process.

Pultrusion is a process that is quite similar to filament winding and resin transfer moulding (RTM) used to obtain large fiber reinforced composite structures, using low cost facilities, tools and materials [7]. Many investigations have been done on the pultrusion process using various types of reinforcements and resin [8–11]. Pultrusion processing has shown a growth of interest because of its cost-effectiveness for high volume production of uniform cross-section parts and offers continuous production of profiles. The process involves pulling resin-impregnated fiber reinforcements through a moulding/curing station and a heated die to cure the resin. The profiles such as rods, I-beam, tubes, and various structural shapes can be produced using appropriate dies. With fiber content up to 60–70%, profiles may exhibit high strength and stiffness in the length direction. In this study, preparation of PJFRC has been described. The mechanical and thermal properties of PJFRC also have been reported. In addition, results on morphological evaluation on fractured specimen and acidic digestion were also presented.

2. Methods and Materials

2.1. Materials

Jute fibers were supplied by Alam Fiber Impex Ltd, Bangladesh in yarn form. The jute fiber yarn was produced in twisted 2-ply configuration having tex of 1500 and average diameter of 1.30 mm and the density of 1.46 g/cm³. Unsaturated polyester resin (Crystic P9901) was supplied by Revertex (Malaysia) Sdn. Bhd. with some typical properties presented in Table 1.

2.2. Preparation of Pultruded Composites

The rod profiles pultruded composites were manufactured using a thermoset pultrusion machine at MMFG Composites Sdn. Bhd. Subang Jaya, Selangor, Malaysia. The machine was equipped with a creel, guidance devices, resin bath, preforming guide, heated die, pulling device and cut-off saw, as shown in Fig. 1. A bookcase-type shelf was used to place the jute fiber which contains a roving guider to guide

Table 1.
Properties of unsaturated polyester supplied by Revertex (Malaysia)

Properties	Unit	Value	Standard
Brookfield RVT 2/10 rpm	MPa s	1200	ISO 2555
Density	g/cm ³	1.35	ISO 2811
Young's modulus	GPa	4	ISO 527
Tensile stress at break	MPa	61	ISO 527
Tensile strain at break	%	2.5	ISO 527

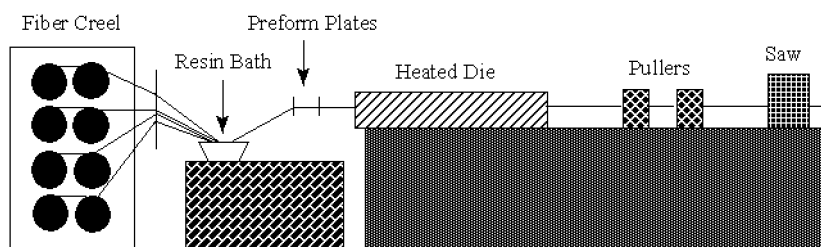


Figure 1. Schematic representation of pultrusion processing.

the strands to the resin bath. Grid plates were provided at the entrance and exit ends of the resin bath to keep the rovings in horizontal alignment as they pass through the resin bath. The composites were then cured within the electrically heated rod shape die. Two pullers were employed to pull the composites continuously. The pultruded composites were then cut by using an automatic cut-off saw. The average diameter of the composites was 12.7 mm.

2.3. Density Measurement

Density was measured using a digital density meter (pycnometer) in accordance with ASTM D792. The rod profile specimens with a diameter of 12.7 mm and length 10 mm were weighted first to the nearest 0.0001 g. Three specimens were prepared for this measurement. The average of the three measurements gave a value of 1.48 g/cm³.

2.4. Mechanical Testing

2.4.1. Compression Testing

Compression test was performed using Instron 8802 in accordance with ASTM D695-02a. The diameter and length of the specimen were 12.7 and 25.4 mm, respectively. The cross-head speed was set at 1.3 mm/min. Three specimens were prepared for the PJFRC compression testing.

2.4.2. Flexural Testing

Three-point flexural test of pultruded composites was carried out using Instron 5582 in accordance with ASTM D4476-03. Specimens were cut into two parts so that

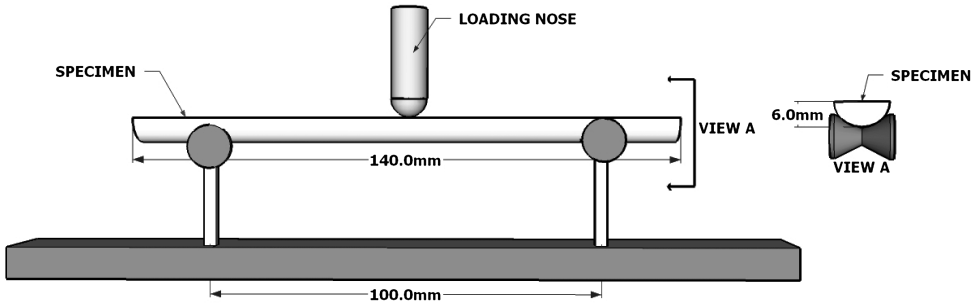


Figure 2. Schematic diagram for the three-point bending for flexural testing.

the cross-section of each part was smaller than a half-round section and the span was 100 mm, as shown in Fig. 2. The cross-head speed for flexural test was set at 3 mm/min. Three specimens were prepared for the jute fiber composites flexural testing.

2.5. Thermal Testing

2.5.1. Thermomechanical Analysis (TMA)

The thermal expansion analysis was done using the Thermal Mechanical Analyser (TMA 7) in accordance with ASTM standard E228-85 over the temperature range of 30–100°C with the heating rate of 5°C/min. The specimens were cut into cubes with dimensions of 8 × 8 × 8 mm. The coefficient of thermal expansion (CTE) α was calculated by using:

$$\alpha = \frac{L_f - L_0}{L_0 \times \Delta T} \quad (1)$$

and

$$\varepsilon = \frac{L_f - L_0}{L_0}, \quad (2)$$

where ε is strain (mm/mm); L_f is final length (mm); L_0 is initial length (mm) and ΔT is temperature change (°C).

2.5.2. Dynamic Mechanical Thermal Analysis (DMTA)

The comprehensive study on dynamic mechanical properties of PJFRC was performed using a Mettler Toledo machine (Model 861) under three-point bending configuration (ASTM D5023-7). The specimens were cut into pieces of dimensions 50 × 12 × 3 mm. The PJFRC samples were tested in a temperature range from 0°C to 200°C, with the heating rate of 5°C/min, operating frequency of 10 Hz, forced amplitude of 4.5 kN and the displacement amplitude of 10 μ m.

2.6. Morphological Assessment

The macrostructure of PJFRC and the dimensions of single jute fibers were evaluated and measured using an optical microscope (Rax Vision) and video camera

(JVC), respectively. Detailed microstructural analysis was also performed using FESEM.

2.7. Matrix Digestion Process

The rod PJFRC specimen, with 12.7 mm diameter and 100 mm length, was tarred and weighted accordingly. The specimen was then immersed in 70% nitric acid in a 100 ml beaker following ASTM D3171-06. After 24 h of immersion, the matrix was completely digested and the content was filtered. Then the content was placed in an oven at a temperature of 100°C until the sample was dried (approximately for 1 h). The content was cooled to room temperature and kept in desiccators. The specimen was then weighted again to the nearest ± 0.0001 g.

3. Results and Discussions

3.1. Mechanical Properties

3.1.1. Compressive Properties

Figure 3 shows a series of the compressive stress–strain curves for three identical PJFRC. From the figure, the curves are highly reproducible with respect to compressive strength and modulus (slope) except for slight variation in terms of the compressive failure strain recorded. The ultimate compressive strength of specimens depends upon the strength of the resin and fiber alignment with respect to the applied load. All specimens exhibit linear curves at initial loading. The compressive strength of unidirectional composites depends on the fiber–matrix adhesion and the ability of the matrix to transfer stresses from highly stressed fiber to other fibers. In general, composite specimens under compression test will undergo several major failure modes which result from matrix yield followed by fiber micro buckling, local fiber micro buckling with an elastic matrix, shear failure, and pure fiber com-

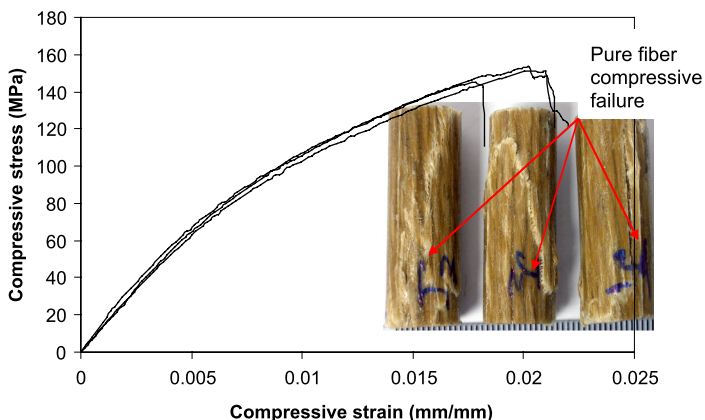


Figure 3. Compressive stress–strain curve of PJFRC. This figure is published in color in the online version.

pressive failure. Such failure modes are associated with the composites having poor, intermediate and high levels of fiber/matrix adhesion [12]. The jute fiber reinforced composites experiencing rod buckling due to the two-ply twisted jute yarn type as shown in Fig. 4 which was used in this experiment and partly aided by poor adherence between jute fiber and matrix. It was noted that a buckled rod form a kink band on the surfaces parallel to the fiber direction as depicted in small caption provided in Fig. 3. The average compression strength of the PJFRC is 149.0 MPa with the compression modulus of 7.4 GPa.

3.1.2. Flexural Properties

Figure 5 indicates a typical flexural stress–strain curve for three different specimens of PJFRC. From the figure the curves are highly reproducible with respect to flexural strength and modulus (slope) except for slight variation in terms of flexural failure strain recorded. Stress is proportionally increased with increasing applied



Figure 4. Twisted jute yarn with Tex of 1500. This figure is published in color in the online version.

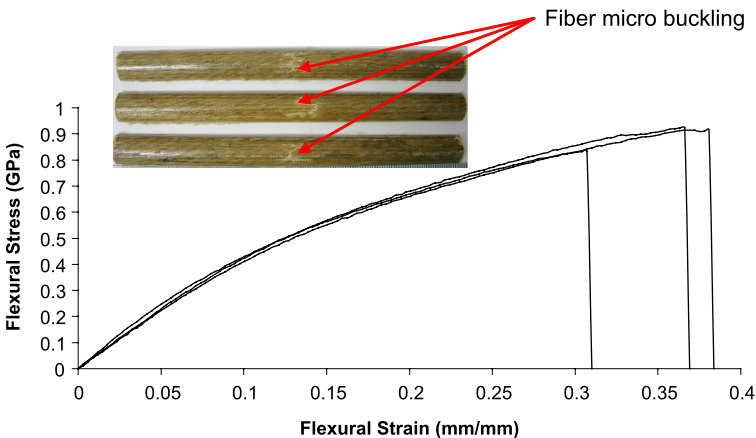


Figure 5. Flexural stress–strain curve of PJFRC. This figure is published in color in the online version.

load with highly reproducible pattern except for the values of flexural strain, which vary slightly from one sample to another. Visual inspection of the failed specimens revealed a significant degree of matrix cracking and overall buckling due to poor fiber–matrix adhesion, which resulted in brittle failure. It is not possible from stress–strain curves to locate the exact beginning of fiber failure of PJFRC under flexural load. It is believed that the crack initiates on the tension side of the beam and slowly propagates in the horizontal direction. Normally, the modulus is very sensitive to the matrix properties and matrix/fiber interfacial bonding [13]. Also, it should be noted that the brittle matrices used in this experiment possess a low tensile strain to failure. The flexural modulus obtained is 2.6 GPa whereas flexural strength is 890 MPa with the maximum failure strain of 0.3 mm/mm.

3.2. Thermal Properties

3.2.1. Thermal Expansion

Figure 6 shows a thermal expansion profile of a PJFRC specimen recorded over a temperature range of 30–100°C. From the curve, a negligible change in dimensions was recorded between the temperatures of 30–50°C. However, beyond 50°C, significant changes in dimensions were observed that could be attributed mainly to the loss of moisture in the jute fiber. A summary of the coefficient of thermal expansion (CTE) values for PJFRC at different temperatures is presented in Table 2. It has been reported previously [14] that the close packing and highly folded structure in high strength fiber could be responsible for the low CTE values. The low CTE indicated that the level of stability and ratios were changeable as a function of temperature. Another critical parameter influencing the CTE value is the size of the fibers. Fibers with smaller diameter will exhibit a big specific surface contact area between the reinforcement and matrix [15].

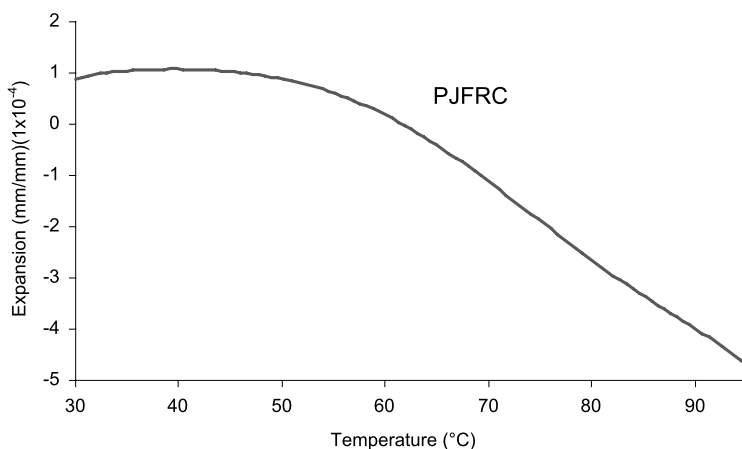
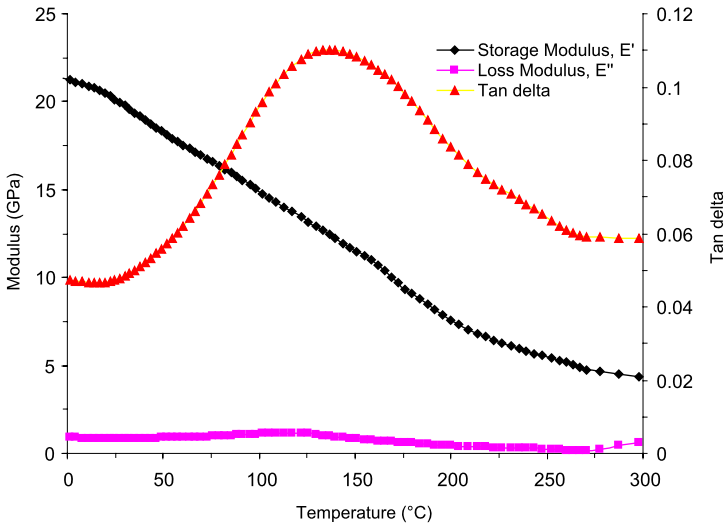


Figure 6. Thermal expansion of PJFRC at temperature range of 30–100°C.

Table 2.

Coefficient of thermal expansion (CTE) of PJFRC measured at various temperatures

Temperature range	Room temperature (°C)	30–50°C	50–70°C	70–90°C
Coeff. of thermal expansion α ($\mu/^\circ\text{C}$) of PJFRC	4.84	26.55	–8.18	–81.76

**Figure 7.** Storage modulus (E'), loss modulus (E'') and loss factor ($\tan \delta$) versus temperature for PJFRC. This figure is published in color in the online version.

3.2.2. Dynamic Mechanical Thermal Analysis (DMTA)

A series of DMTA curves for PJFRC as a function of temperature is outlined in Fig. 7. The $\tan \delta$ represents a ratio of loss modulus to the storage modulus (E''/E') of PJFRC indicated that the maximum damping parameter peak of PJFRC is inversely proportional to value of storage modulus with increasing temperature. The PJFRC specimens showed no significant change in damping peak over the temperature range of 0–25°C. Beyond 25°C, the value of the damping peak started to increase significantly up to the temperature of 130°C. This maximum value could be used as indicator for the T_g value of PJFRC. The damping of the PJFRC improves at temperatures above the T_g of the composite, but at about 150°C, the damping slowly started to drop. Because the jute fibers are very tightly constrained in the polyester and cannot deform, the composite does not show any additional damping. Incorporating reinforcing fibers restricted the mobility of the polymer molecules, raised the storage modulus values and reduced the viscoelastic lag between the stress and the strain and hence, the $\tan \delta$ values were decreased in the composites

[14, 16, 17]. It has been shown that the value of storage modulus could be used to predict the value of crosslink density in the composite [18]:

$$E' = \frac{3\phi dRT}{M_c}, \quad (3)$$

where E' is elastic modulus in the rubbery state; d is density; R is the gas constant; T is absolute temperature; M_c is molecular weight between the cross-linking and ϕ is a front factor (close to unity).

The above equation could be further simplified if the effect of dangling is neglected, then

$$M_c = \frac{d}{\alpha}, \quad (4)$$

where α is the crosslink density. Thus, equation (3) changes to equation (5):

$$E' = 3\phi\alpha RT, \quad (5)$$

where E' can be plotted from the storage modulus of DMTA; R is the gas constant; ($8.31447 \text{ cm}^{-3} \text{ MPa K}^{-1} \text{ mol}^{-1}$), T is the absolute temperature in Kelvin ($\tan \delta + 10^\circ\text{C}$); and the ϕ , the front factor, the value of which is close to unity. This equation was applied to determine the crosslink density of composites materials. From the previous workers [19], it has been reported that an increase of crosslink density is expected to increase the E' in rubbery state during thermal degradation process. Since the parameters in equation (5) remain unchanged, the increase in E' is expected to increase the crosslink density.

The relationship between T_g and crosslink density, α is given by [20]:

$$T_g = K_1 \log K_2\alpha, \quad (6)$$

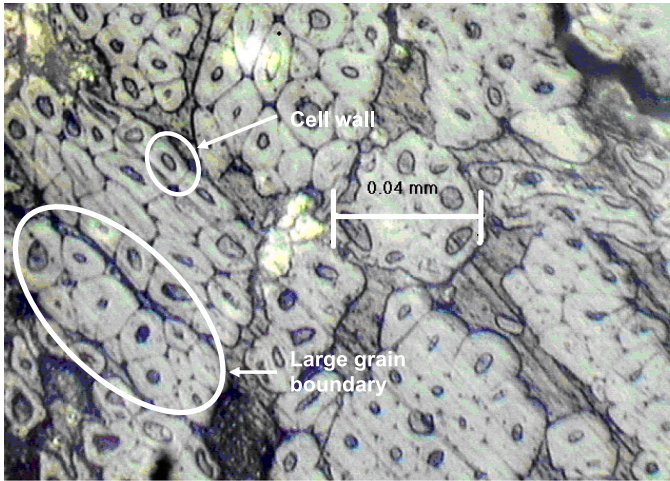
where K_1 and K_2 are constants for the same groups of resins.

From the calculation, the crosslink density obtained is $2.975 \times 10^{-3} \text{ cm}^{-3} \text{ mol}^{-3}$. Bending loads are applied perpendicular to the jute fiber, and the measured properties are sensitive to changes in the matrix to measure the storage modulus E' . Both jute fibers and unsaturated polyester matrix experiences equal strains. This is because the jute fibers do not change in structure over the measured temperature range: changes in the storage modulus reflect structural changes occurring in the matrix [21].

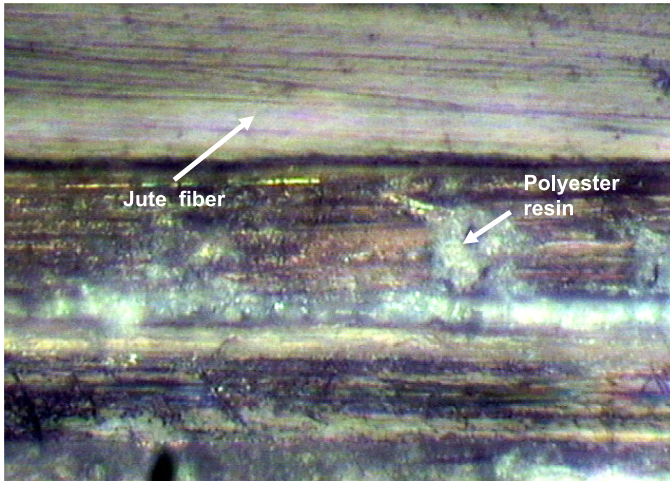
3.3. Morphological Assessment

3.3.1. Optical Microscope and Scanning Electron Microscope

Morphological assessments were done using both optical and scanning electron microscopes. Figure 8(a) shows a typical distribution of jute fiber within polyester matrix under cross-sectional view (fiber direction). Since the volume fraction of fiber is high ($\approx 70\%$ by volume), the majority of the fibers can be clearly seen with the minimum interphase region and a little resin-rich area. A close-up view of the interfacial area between fiber/matrix along the fiber direction is shown in Fig. 8(b).



(a)



(b)

Figure 8. Photomicrograph of PJFRC of (a) across fiber direction, (b) along fiber direction. This figure is published in color in the online version.

The jute fibers are shown as light grey and the epoxy matrix as shiny grey. The polyester distribution is not thoroughly mixed but the fibers seem to be attached well to the matrix with a long fiber attachment. Figure 9 shows the micrograph structure of a single jute fiber under a scanning electron microscope with the same 500 \times magnification. The impurities must be removed in order to enhance the interaction between the fiber and matrix.

3.4. Matrix Digestion

Actual fiber content was determined experimentally using matrix digestion technique. Composites with digested matrix were then used to calculate the volume

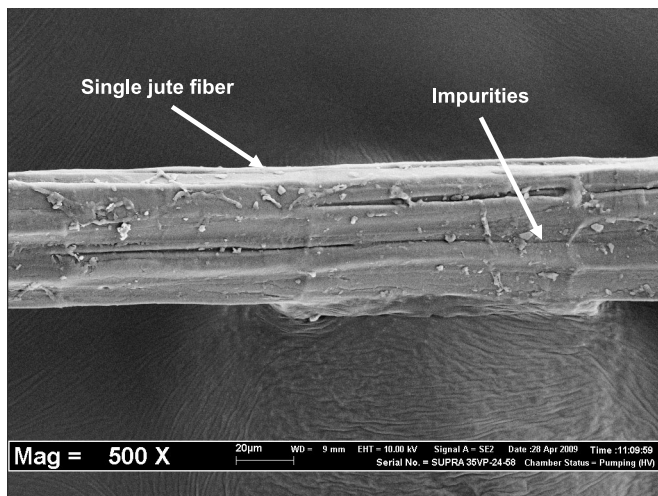


Figure 9. Micrograph of single jute fiber.

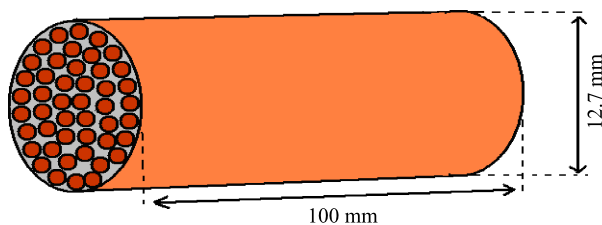


Figure 10. Schematic representation of PjFRC specimen for matrix digestion test. This figure is published in color in the online version.

percent of fiber using equation (7). Figure 10 represents the dimension of the pultruded profile for the matrix digestion testing:

$$\text{Fiber volume fraction } V_f = \frac{\rho_m w_f}{\rho_m w_f + \rho_f w_m}, \quad (7)$$

where ρ_m is the density of the matrix (g/cm^3); ρ_f is the density of the fiber (g/cm^3); w_f is the weight of fiber (g) and w_m is the weight of matrix (g).

The fiber weight fraction w_f and the matrix weight fraction w_m can be determined from this equation:

$$w_f = \frac{W_f}{W_c}, \quad (8)$$

where W_f is the weight of fiber (g) and W_c is the weight of composite (g).

The volume fraction and weight fraction of the fiber and matrix is shown in Table 3.

Table 3.Typical values of fiber weight fraction (g) and volume fraction, V_f (%) after digestion process

Tex (g/km)	Weight of composite, w_c (g)	Weight of fiber, w_f (g)	Weight of matrix, w_m (g)	Volume fraction of fiber, V_f (%)	Volume fraction of matrix, V_m (%)
1500	15.53	11.41	4.12	71.92	28.08

4. Conclusion

Pultruded jute fiber reinforced composites (PJFRC) with 70% fiber content have been successfully prepared. The properties of PJFRC have been characterized and analyzed. From the results obtained, the following conclusions could be drawn:

- (1) PJFRC specimens under compression and flexural test exhibited several major failure modes, which are matrix yield followed by fiber micro buckling, local fiber micro buckling, with an elastic matrix, shear failure, and pure fiber compressive failure.
- (2) From the thermal expansion test, a small expansion of PJFRC was observed at initial temperature range up to 50°C. Beyond 50°C, the PJFRC specimen started to contract instead of expand until the test stopped at 100°C.
- (3) From DMA curve, the T_g of PJFRC derived from the $\tan \delta$ peak is 135°C. Meanwhile, the calculated cross-linking density of the PJFRC using equation (5) is $2.975 \times 10^{-3} \text{ cm}^{-3} \text{ mol}^{-3}$.
- (4) Microscopic examination for the composite surface indicates that the interfacial adhesion strength is sufficient to transfer load until failure initiated in the jute fiber.
- (5) Matrix digestion test showed that the volume fraction, V_f and weight fraction W_f of PJFRC are 71.92 and 73.47%, respectively.

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