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Effects of Aspect Ratio and Loading on the Mechanical Properties of *Prosopis juliflora* Fibre-reinforced Phenol Formaldehyde Composites

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Abstract

In this paper, an attempt was made to use *Prosopis juliflora* fibres (PJFs) as a reinforcing agent for phenol formaldehyde (PF) composites. Mechanical properties of the composites were studied for various fibre aspect ratios (FAR) and fibre loadings (FL). A scanning electron microscope (SEM) was used to study the fractured surface of the composites. The peak range of mechanical properties was identified for composites with a FAR of 136 and fibre loading of 23.53 wt%. This study shows that the optimum FAR and fibre loading for PJFs were found to be 136 and 23.53 wt% in order to achieve good reinforcement with better mechanical properties in the PF resin matrix. Experimental results were observed to be in very good agreement with the theoretical.

Key words: *prosopis juliflora* fibres, composite, mechanical properties, scanning electron microscope, theoretical models.

Introduction

Recently the use of plant based natural fibres as a reinforcement in polymer composites has grown to be an important field of research. These composites are the best candidate for substituting synthetic fibres such as glass, carbon and aramid reinforced polymer composites [1-3]. Several researchers and material engineers have studied the various properties of natural fibres such as flax, kenaf, sisal, banana, coir, roselle, hemp, bamboo, rice husk, bagasse, wood and pineapple leaf fibre, etc. as reinforced polymer composites at various conditions [4-6]. Among the various natural fibres, PJFs have been recently introduced into cellulosic fibres as a reinforcing agent in polymer composite materials, and are getting acceptance by material researchers [7, 8]. The bark of PJ has been traditionally used to tie wet and dried stems and other large amounts of stems together into bundles for transport from one place to another. They are abundantly available in the southern region of India. However, these plants disturb other economic plants in their growing, and therefore government officials have decided to remove them

permanently from the land of Tamilnadu, India. PJFs have not been frequently utilized as a reinforcing agent in polymer composites yet, and there is even no literature available on the mechanical properties of PJF/PF composites. Some authors have studied the physico-chemical properties of these fibres with the aim of finding out their suitability as reinforcement for polymer matrix composites. The present work was undertaken to develop a PF composite using PJFs as a reinforcement. Composites were prepared with three different fibre aspect ratios (FARs) and five different fibre loadings. Based on the test results, the composites were characterized to find out the optimum FAR and fibre loading. The fracture surface of the mechanical tested composite specimens was studied by scanning electron microscope (SEM). A comparison of experimental values with predicted values was carried out using the Hirsch, Cox and modified Bowyer and Bader models.

Experimental

Materials

The PJFs used in this study were extracted manually from the bark of the

PJ plant and used in the received condition as a reinforcing agent for the resole type phenol formaldehyde resin matrix. The chemical compositions and physical properties of PJFs are presented in **Table 1**. The phenol formaldehyde resin with the cross-linking agent (divinylbenzene) and acidic catalyst (hydrochloric acid) was obtained from Pooja Chemicals, Madurai, Tamilnadu, India.

Preparations of composites

PJF/PF composite plates were prepared for various FARs of 46, 136 and to 227 mm and fibre loadings of 4.88, 10.34, 16.51, 23.53 and 31.58 wt% by the hand lay-up technique. First a mold box with a size of 150 × 150 × 3 mm was developed in our laboratory for this study. Prior to process, the releasing agent was applied on the inner side of the mold box to ensure easy removal of the cured composite plate. The fibres were evenly spread in a mold using a mechanical roller. A mixture was prepared with PF resin, a cross-linking agent and acidic catalyst using a mechanical stirrer and poured into the mold. Then the mold was closed and kept under pressure for 48 hours at room temperature.

Table 1. Chemical composition and physical properties of PJFs.

Chemical composition, %		Physical properties	
Cellulose	54.67	Thickness, mm	0.29-1.53
Hemicellulose	23.21	Length, mm	11-34
Lignin	18.19	Density, g/cm ³	0.4-0.8
Moisture content	1.80	Ultimate stress, MPa	115.5-528.2
Ash content	2.13	Strain rate, %	1.58-1.79

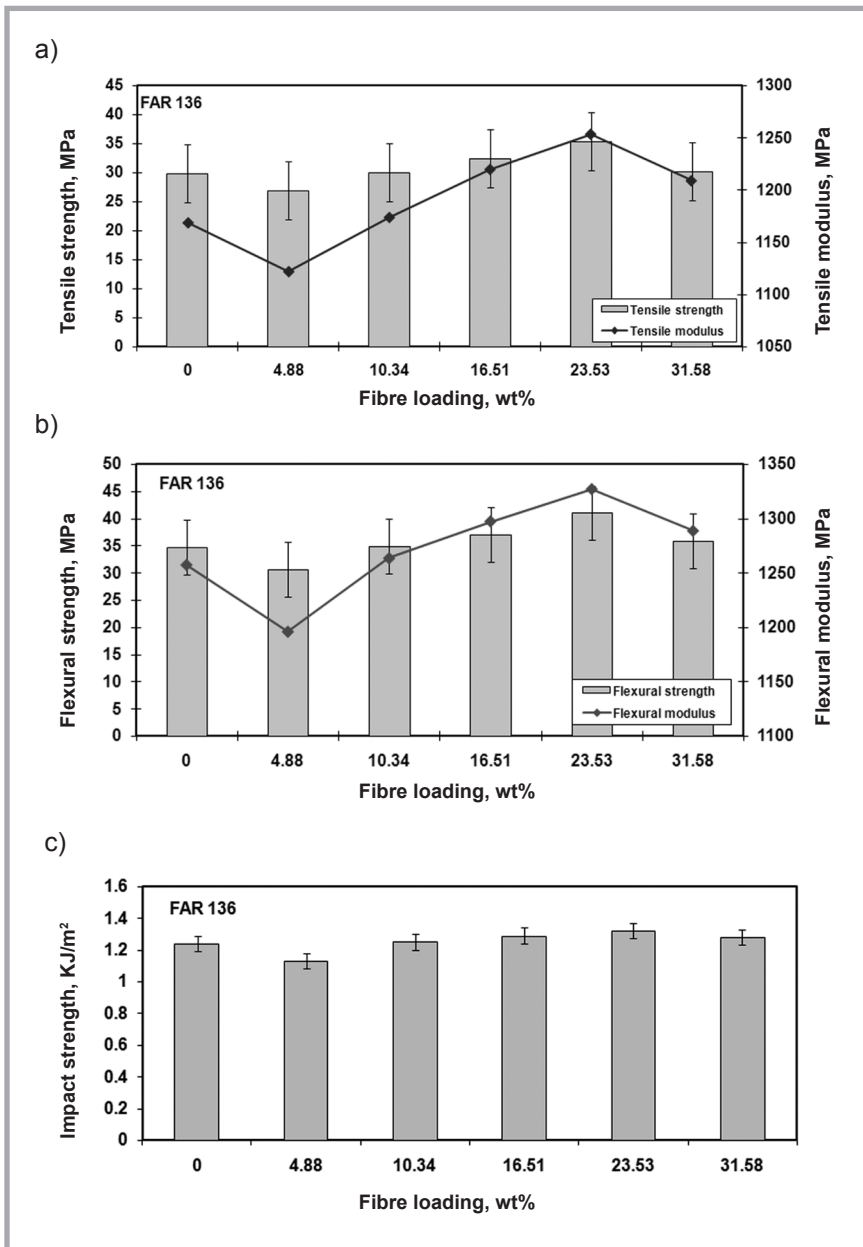


Figure 1. Variation in the mechanical properties of 136 FAR composite with fibre loadings: a) tensile strength and modulus, b) flexural strength and modulus, and c) impact strength.

Characterisation of composite specimens

Tensile tests were performed according to ASTM D 638-10 with a crosshead speed of 2 mm/min in an FIE universal testing machine at room temperature.

Flexural tests were also carried out on the same universal testing machine according to ASTM D 790-10 with a crosshead speed of 2 mm/min at room temperature. Impact tests were carried out according to ISO 180 at room temperature. A set of

Table 2. Average mechanical properties of 46-FAR composite.

Fibre loading, wt%	No. of specimens tested	Tensile strength, MPa	Tensile modulus, MPa	Flexural strength, MPa	Flexural modulus, MPa	Impact strength, KJ/m ²
0	5	29.80	1168.4	34.70	1257.4	1.24
4.88	5	25.91	1091.3	28.74	1128.6	0.97
10.34	5	27.93	1153.4	32.12	1175.8	1.18
16.51	5	30.12	1189.6	34.83	1218.3	1.26
23.53	5	32.55	1203.8	39.91	1257.9	1.29
31.58	5	27.21	1178.3	33.53	1208.7	1.25

five composite specimens of each measurement was tested for the determination of mechanical properties and the average results reported. The fracture surface of the composites was examined by an SEM machine (Hitachi S-3000N, Japan).

Results and discussion

The average values of five replications from mechanical tests of the PJF/PF composite prepared by three different FARs and five different fibre loadings are discussed here. The effect of the FAR with fibre loading was discussed separately to find out the suitable (optimum) FAR and (optimum) fibre loading, because the FAR and fibre loading play a major role in the properties of natural fibre-reinforced polymer composites.

Effect of an FAR of 46 on the mechanical properties

The variation in the mechanical properties of the 46FAR composite with varying fibre loading is presented in **Table 2**. It can be seen that with an increasing fibre loading in the PF resin matrix, mechanical properties were increased with a fibre loading of 16.51 wt% to 23.53 wt%, and then dropped. The composites reach the tensile strength and modulus of the neat resin sample at 16.51 wt%. The addition of 23.53 wt% of PJFs increases the strength of the PF composite by 9.23%. The tensile modulus of the 23.53 wt% composite was increased 3.03% more than the neat resin sample and 10.31% more than the 4.88 wt% composite.

Flexural properties of the composite are presented in **Table 2**, exhibiting a similar trend to that observed for tensile properties. Flexural strength and modulus values of the composite reached the neat resin sample values at 16.51 wt%. The flexural strength and modulus values of the composite with 31.58 wt% of fibre loading was lower than that with fibre loading of 16.51 wt%. Because the amount of resin to hold the fibres in the composites with higher fibre loading is not sufficient, the flexural properties decrease with an increasing fibre loading. It can be seen from **Table 2** that the tensile and flexure properties of the composites for a fibre loading of 4.88 wt% were less than for the neat resin sample. The logical reason is that at lower fibre loading, the stress transfer from the matrix to the fibre will not occur properly due to the poor dispersion of PJFs in the composite. Therefore the reduction in

properties was identified in the composites [9].

The trend of impact results of the composite is shown in **Table 2**. The same trend was identified in impact strength values as in tensile and flexural strength values. The composite with fibre loading of 23.53 wt% has an impact strength of 1.29 KJ/m², which is higher by 4.03% than that of the pure PF resin sample. It was observed from the results above that there is no significant variance in the mechanical properties of composites reinforced using an FAR of 46 with five different fibre loadings. At a small aspect ratio, the mechanical properties were at the lowest level due to the fact that the length might not be sufficient enough for stress transfer and also for the proper distribution of the load. Therefore failure could easily occur in the composites [10]. From the results above, it is clearly observed that the increment from one fibre loading to the other is low. The tensile strength of 16.51 wt% and 23.53 wt% composites was 24.7 MPa and 28.45 MPa, respectively, and their percentage of increment is just 8.07%. Similarly the percentage of increment between 10.34 wt% and 16.51 wt% MPa was 7.84 %. Therefore it can be observed that the increment of the weight percentage of fibre loading did not significantly affect the mechanical properties of the PF composites.

Effect of an FAR of 136 on the mechanical properties

Figures 1.a to 1.c show the effect of fibre loading on the mechanical properties of the composite with a FAR of 136. **Figure 1.a** clearly shows an increasing trend in both the tensile strength and modulus up to 23.53 wt% of the fibre loading, and then dropped. The value of the tensile strength and modulus of the 23.53 wt% composite was found to be 18.32% and 7.28% higher than for the un-reinforced neat resin sample.

The effect of fibre loading on the flexural properties of the composite shows a similar trend to that of the tensile properties results, which can be seen in **Figure 1.b**. The composites reach the flexural strength and modulus value of the neat resin sample at 10.34 wt% of fibre loading. The 23.53 wt% composite showed the highest flexural strength (41.02 MPa) and modulus (1327.2 MPa), and then decreased. The improvements were 18.21% and 5.55% compared to the neat PF composite specimen.

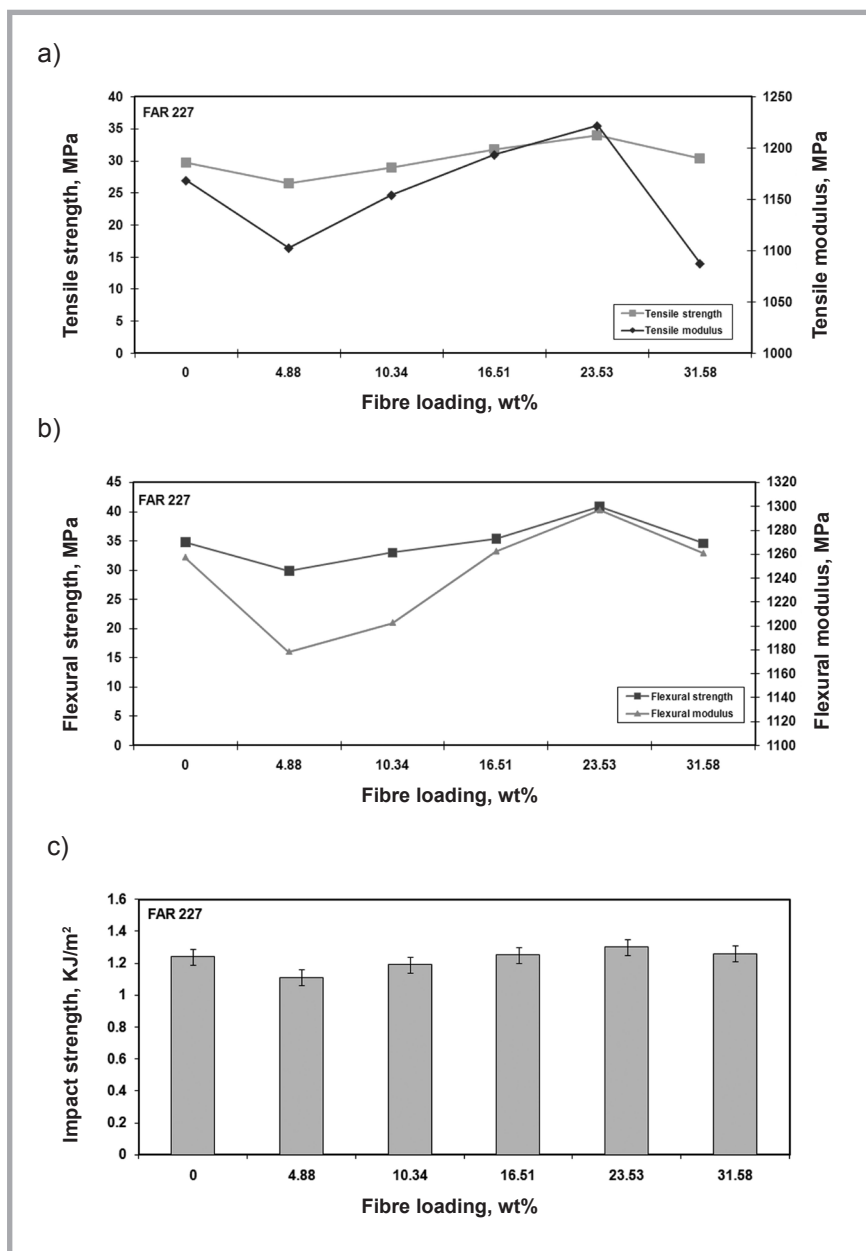


Figure 2. Variation in the mechanical properties of 227 FAR composite with fibre loadings: a) tensile strength and modulus, b) flexural strength and modulus, and c) impact strength.

The impact behaviour of the composites after impact tests is shown in **Figure 1.c**, showing that the addition of PJF loadings to the PF matrix increases the impact strength of the composite. It also exhibits a similar trend to that observed for tensile and flexural properties. The 23.53 wt% composite shows the highest impact strength compared to the other wt% composites. Generally the impact property of the natural fibre reinforced polymer composites is poor compared to the tensile and flexural properties. The probable reason is that natural fibres produce an interface of higher strength with polymer matrix due to which energy absorption decreases at these interfaces [11].

From the results above, it can be seen that the mechanical properties of the composites with a FAR of 136 are higher compared to those with a FAR of 46 at all fibre loadings. Here also it is clearly observed that the increment of the weight percentage of fibre loading has not significantly affected the mechanical properties of PF composites prepared with PJFs with a FAR of 136.

Effect of a FAR of 227 on mechanical properties

Tensile, flexural and impact properties of composites with an FAR of 227 are given in **Figures 2.a to 2.c**. Mechanical properties at all levels of fibre loading were less

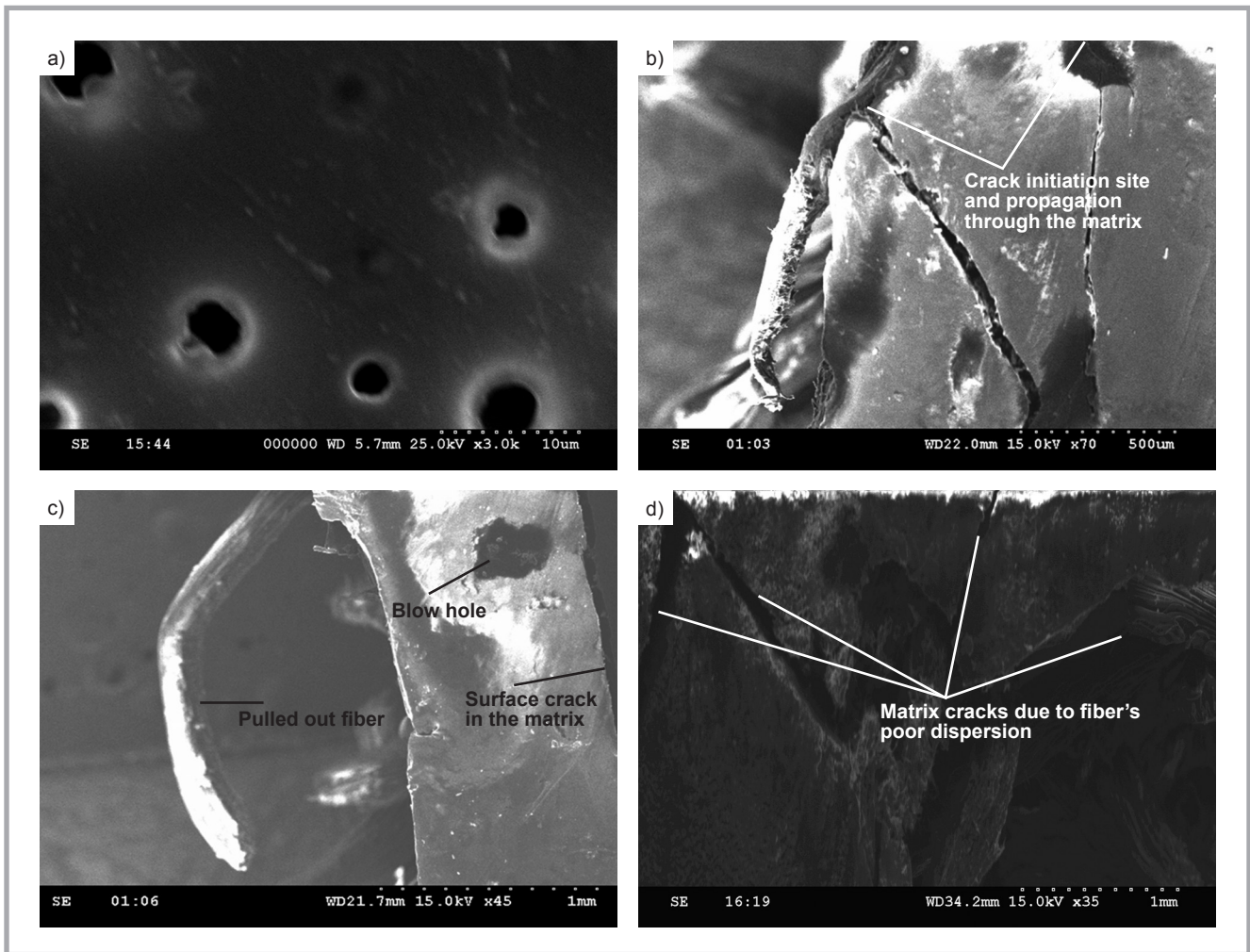


Figure 3. SEM images of a fractured surface of PJF/PF composites after mechanical tests.

than those of the composite with a FAR of 136, which may be due to the formation of fibre agglomeration and the presence of voids. The maximum level of mechanical properties was observed in the 23.53 wt% composite when compared to the other fibre loadings. On further increasing the PJF reinforcement above 23.53 wt%, the composite becomes more brittle, shows brittle behavior during testing and the overall strength of the composite decreases. Mechanical properties of the polymer composites reinforced with long natural fibre decreased compared to polymer composites reinforced with small length natural fibres. Because of the long natural fibres, fibres form agglomerates during the preparation of the composite due to fibre-to-fibre interaction i.e., the fibres may be folded. This prevents better dispersion of natural fibres into the polymer matrix, resulting in an improper bonding between the hydrophobic polymer matrix and hydrophilic natural fibres [12]. Therefore, in this investigation, PJFs with a FAR of 136 and fibre loading of 23.53 wt% were

observed as an optimised or suitable FAR and fibre loading to achieve the best combination of mechanical properties in PF composites.

Fractographic analysis

A fractured surface on the mechanically tested composite specimens (136 FAR & 23.53 wt%) was observed under an SEM. Micrographs of the fracture surface obtained by SEM are shown in **Figure 3**. All SEM images showed no particular adhesion between PJFs and the PF resin matrix. From the SEM image it is evident that there is an inhomogeneous mixture of PJFs and the PF resin matrix, leading to some voids/pores in the matrix, as shown in **Figure 3.a**. Some micro-cracks on the surface of the matrix were identified, which are responsible for the reduction in strength of the composites. A crack initiation site and propagation through the matrix were observed in **Figure 3.b**. Fibre pull-out, surface crack and blow holes were also identified in **Figure 3.c**. Composites with lower fi-

bre loading possess very poor fibre dispersion. Matrix cracks were identified during the testing of this composite, as shown in **Figure 3.d**, which was the main reason for the lower range of mechanical properties of PJF/PF composites with a fibre loading of 4.88 to 16.51 wt% at all the FAR.

Comparison of experimental results with theoretical predictions

PJF/PF composites were characterised based on the different FARs and fibre loadings. PJF/PF composites with a FAR of 136 show better or the maximum level of mechanical properties compared to other FARs. Therefore the tensile properties of PJF/PF composites with a FAR of 136 were taken for a comparative study with theoretically predicted results using various theoretical models such as the Hirsch and modified Bowyer-Bader and Cox. The Hirsch model is a combination of the rule of mixture (parallel model) and inverse rule of mixture (series model), because these are typically treated

as upper and lower limits of the tensile properties of fibre-reinforced polymer composites. According to the Hirsch model, the tensile strength and modulus are given by [13] **Equations 1** and **2** where a is a parameter in dependence on the stress transfer between the fibres and the matrix, i.e., the fibre orientation with respect to the loading direction. The following equations are used to determine the tensile strength and modulus by means of the modified MBB model [13] **Equations 3** and **4** where α_v is the overall reinforcing factor, expressing to what extent the modulus of elasticity of the fibre contributes to that of the composite. k_1 is the fibre orientation factor and k_2 the fibre length factor [13]. According to the Cox model [14], the tensile strength and modulus of the fibre-reinforced polymer can be expressed as follows **Equations 5** and **6** where r is the radius of the fibre, G_m the shear modulus of the matrix, A_f the area of the fibre, and R is the center to center distance of the fibres. For a hexagonal array of fibres in the composites, the value of R is $\left[\frac{2 \times \pi \times r^2}{3^{1/2} \times V_f} \right]^{1/2}$, and for a square arrangement of fibres, the value of R is $r \times \left[\frac{\pi}{4 \times V_f} \right]^{1/2}$.

A comparison of the variation in experimental and theoretical tensile property values of randomly oriented and intimately mixed PJF/PF composites with a weight percentage of loading of PJFs is shown in **Figure 4**. It can be observed that in all the cases the same trend was observed, i.e. the tensile strength increases with PJF loadings. Also a good correlation was identified between the experimental and theoretical tensile strength values.

Variable a in the Hirsch model (**Equations 1** and **2**) is an important parameter which determines the stress transfer between the fibre and matrix. It also plays a major role in determining the agreement between experimental and theoretical values. The value of a in **Equations 1** and **2** is 0.47 for a good agreement between experimental and theoretical values. The modified Bowyer and Bader model has one adjustable parameter, $\alpha_v = k_1 \times k_2$, which expresses to what extent the properties of the fibre contribute to those of the composite [13]. The values of k_1 and k_2 in **Equations 1** and **2** were found to be 1 and 0.4, which is a good agreement with experimental values. It

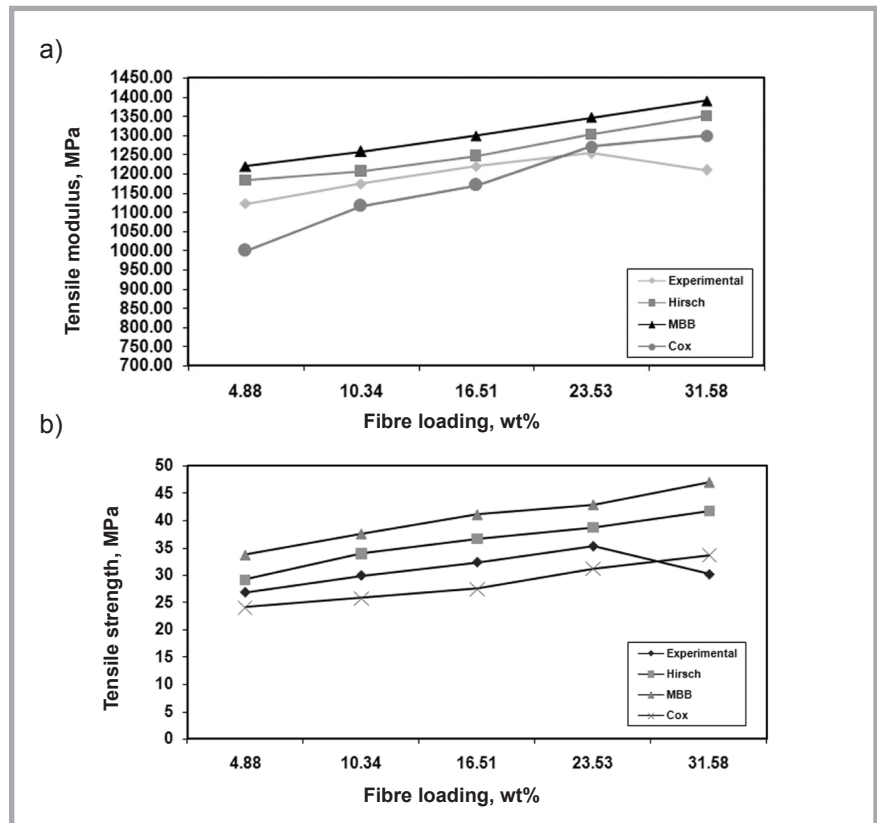


Figure 4. Variation in the experimental and theoretical a) tensile strength and b) tensile modulus of 136 FAR composites.

shows a very good agreement with experimental tensile strength values, and can be seen in **Figure 4.a**. It was followed by the Hirsch and Cox model. Furthermore the MBB model overestimated the tensile strength value at 23.53 wt%. In the case of the Cox model, it underestimated

the tensile strength for all fibre loadings, whereas the Hirsch model overestimated the tensile strength for all fibre loadings. **Figure 4.b** shows the variation in the experimental and theoretical tensile modulus of the PJF/PF composite with the fibre weight percentage. From **Fig-**

$$E_c = a(E_f \times V_f + E_m \times V_m) + (1-a) \frac{E_f \times V_m}{E_f \times V_m + E_m \times V_f} \quad (1)$$

$$\sigma_c = a(\sigma_f \times V_f + \sigma_m \times V_m) + (1-a) \frac{\sigma_f \times \sigma_m}{\sigma_f \times V_m + \sigma_m \times V_f} \quad (2)$$

$$\sigma_c = \alpha_v \times \sigma_f \times V_f + \sigma_m \times V_m \quad \text{with the same } \alpha_v \text{ value} \quad (3)$$

$$E_c = \alpha_v \times E_f \times V_f + E_m \times V_m \quad \text{with } \alpha_v = k_1 \times k_2 \quad (4)$$

$$V_f \left[1 - \frac{\tan h \left(\frac{\beta \times l}{2} \right)}{\frac{\beta \times l}{2}} \right] + E_m \times V_m \quad \text{with } \beta = \left(\frac{2 \times \pi \times G_m}{E_f \times A_f \times \ln \left(\frac{R}{r} \right)} \right)^{1/2} \quad (5)$$

$$\sigma_c = \sigma_f \times V_f \left[1 - \frac{\tan h \left(\frac{\beta \times l}{2} \right)}{\frac{\beta \times l}{2}} \right] + \sigma_m \times V_m \quad \text{with } \beta = \left(\frac{2 \times \pi \times G_m}{\sigma_f \times A_f \times \ln \left(\frac{R}{r} \right)} \right)^{1/2} \quad (6)$$

Equations (1), (2), (3), (4), (5) and (6).

ure 4.b, it can be observed that a very reasonable correlation exists between the experimental and theoretical values in the Hirsch and modified Bowyer-Bader models, better than that of the Cox model. Also a very good agreement can be seen in the case of the Hirsch model compared to the MBB model. Furthermore the Cox equation underestimated the tensile modulus values for 4.88, 10.34 and 16.51 wt%, whereas it overestimated at 23.53 wt% and 31.58 wt%. However, it can be seen that the Cox model can also be used to calculate the tensile modulus.

■ Conclusions

A preliminary study was conducted on the mechanical properties of PF composites in relation to the use of PJFs as a reinforcement agent. Mechanical properties were increased linearly with fibre loadings from 10.34 wt% to 23.53 wt%, and then started to decrease at 31.58 wt% for all cases of FARs. A 136 FAR and 23.53 wt% fibre loading were observed as optimised or suitable to achieve the best combination of mechanical properties. Only a small range of increment was identified in the strength values of the composites in all the cases. Furthermore there is also no significant effect on the modulus values of composites due to PJF reinforcement in a PF resin matrix in all cases of FARs and fibre loadings. It is proven that there was not a high level of contributions of PJFs with the PF resin matrix. Experimental tensile property results were compared with theoretical results and found to be in good agreement. In order to be a suitable reinforcement material, natural fibre must meet the following conditions: it must increase the tensile properties of the resin matrix and exceed the critical fibre weight percentage or volume fraction; and there must be optimum bonding between the natural fibres and resin matrix.



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