Polymer(Korea), Vol. 40, No. 1, pp. 1-8 (2016)
http://dx.doi.org/10.7317/pk.2016.40.1.1
ISSN 0379-153X(Print)
ISSN 2234-8077(Online)

단방향으로 배향된 Agave Sisalana Variegata 섬유 보강 Vinyl Ester 복합체의 물리적 및 기계적 성질

A. Athijayamani†, R. Ganesamoorthy*, K. T. Loganathan**, and S. Sidhardhan***

Department of Mechanical Engineering, A.C.C.E.T., *Department of Mechanical Engineering, SEC
**Department of Chemistry, A.C.C.E.T., ***Department of Civil Engineering, GCE

(2015년 3월 6일 접수, 2015년 9월 22일 수정, 2015년 9월 22일 채택)

Physical and Mechanical Properties of Unidirectional Aligned Agave Sisalana Variegata Fiber-Reinforced Vinyl Ester Composite

A. Athijayamani†, R. Ganesamoorthy*, K. T. Loganathan, and S. Sidhardhan*****

Department of Mechanical Engineering, A.C.C.E.T., Karaikudi-630 004, Tamilnadu, India
**Department of Mechanical Engineering, Sayasuriya Engineering College, Chennai-600 055, Tamilnadu, India*
***Department of Chemistry, A.C.C.E.T., Karaikudi-630 004, Tamilnadu, India*
****Department of Civil Engineering, GCE, Tirunelveli-627 007, Tamilnadu, India*

(Received March 6, 2015; Revised September 22, 2015; Accepted September 22, 2015)

Abstract: This paper presents preparation of unidirectional aligned agave sisalana variegata fiber-reinforced vinyl ester composite laminates and their mechanical properties such as tensile, shear, flexural and impact strength. Wet hand lay-up technique was used for the preparation of composites. Mechanical tests were carried out for different weight percentage of fiber by varying the number of layers. Mechanical properties were analyzed as a function of wt%. The maximum tensile, flexural, impact and shear strength was observed on a composite designated as D. But the maximum tensile and flexural modulus values were identified in a composite designated as E. Experimental results were compared with theoretical results such as the rule of mixture and Bowyer and Bader model. Bowyer and Bader model was able to predict the strength and modulus of the composites better than the rule of the mixture model. The comparison between experimental and predicted values was also done by the student *t* test.

Keywords: polymer-matrix composite, agave sisalana variegata fiber, vinyl ester, mechanical property.

Introduction

Recently the plant based natural fibers have been used in combination with polymer to prepare composite materials. These fibers are replacing synthetic fiber reinforced polymer composites in various fields (from structural to computer industry) for the benefits of the environment. Polymer composites reinforced using the plant based natural fibers provides positive environmental benefits with respect to ultimate disposability and best utilization of raw materials.[1-5] Since natural fibers like jute, sisal, coir, banana, kenaf, flax, hemp, and pineapple, etc. are light in weight, strong, abundant, non-hazardous, non-abrasive and inexpensive; they have been served as an excellent reinforcing agent for polymer matrix. Among the various natural fibers sisal fibers are one of the important natural lingocellulosic fibers compared to others fibers. Traditionally, these fibers are mainly used for the manufacture of ropes for use in agriculture, for making twines, purses, wall hangings, and mat making, etc. Many authors have been reported, their work on the use of sisal fibers as reinforcements in polymer matrices.[6-16] Agave sisalana variegata (ASV) plant is on the most important plant in Agavaceae family. They are strong and very fast-growing species. They have heavily variegated leaves. The leaves of these plants are also used for fiber production From the literature survey, it was found that there is no literature available on unidirectional aligned agave sisalana variegata fiber-reinforced vinyl ester (UAASVFRVE) composite laminates. Therefore, it was considered to take an

†To whom correspondence should be addressed.
E-mail: athimania@rediffmail.com

attempt on mechanical properties of UAASVFRVE composite laminates. Composite laminates were prepared using a wet hand lay-up technique. Mechanical properties such as tensile, flexural, impact and shear strength of composite laminates were analyzed as a function of the fiber weight fraction and the number of layers. The rule of mixtures (RoM) and Bowyer and Bader (BB) models are used to compare with experimental results. Student *t* test was also used for comparing mean values.

Experimental

Materials. ASV fibers were used as reinforcement and purchased from fiber shop, Ammapalayam, Erode, Tamilnadu, India. Fibers were used without any treatment (as received condition) in this study. Vinyl ester resin (Satyen Polymers Pvt. Ltd., Bangalore, India) was used as polymer matrices in this study. Methyl ethyl ketone peroxide (MEKP), cobalt 6% naphthenate (CoNap) and *N-N* dimethyl aniline were used as an accelerator, catalyst and promoter, respectively and they were purchased from GVR Enterprises, Madurai, Tamilnadu, India.

Specimen Preparation. For composite preparation, a mould with a size of 300×300×3 mm was prepared in our laboratory. Prior to processing, the mould is cleaned and added a releasing agent into the mould for easy removal of composite laminates. First, the aligned ASV fiber mat is prepared with interlacing of cotton fibers placed in the longitudinal direction with a gap of 10 mm to keep the ASV fibers in transverse direction. The prepared mat is placed in the mould carefully. A mixer of vinyl ester resin with a calculated amount of accelerator, catalyst and promoter is prepared using a mechanical stirrer. Then, the mixer is poured into the mould and the load is applied over the mould for the complete closure. Composite laminates were prepared for different weight percentages and different layers. Table 1 shows the designation of prepared composite laminates.

Table 1. Designation of ASV Fiber-Vinyl Ester Composite Laminates

Designation of composite	Fiber weight percentage (wt%)	Number of layer
A	12.31	Single
B	23.97	Two
C	35.08	Three
D	45.67	Four
E	55.77	Five

Density. The density of a solid composite material is a property that is measured to identify a composite material, to follow physical changes in a composite sample, to indicate degree of uniformity among different composite specimens. Densities of ASV fiber-vinyl ester composite laminates with the size of 100×20×3 mm were measured according to ASTM D792-13[17] at a temperature of 23±2 ºC. Prior to test in accordance with ASTM standard composite specimens are conditioned at 23±2 ˚C and 50±5% relative humidity for not less than 40 h. De-mineralized water at temperature of 23±2 ˚C was used as the immersion fluid and the mass was measured using a digital balance with a 0.1 mg resolution. A total of twenty five composite specimens (five composite specimens from each designation) was tested to obtain the mean value of the density of each designation.

The density of a fiber reinforced polymer composite laminate can be easily given by the simplest of the RoM. In general, the total volume (V) of an FRP composite laminate containing various constituents with different mass (M_1, $M_2 .... M_n$) has a density (ρ_c). It is given by the following eq. (6)

$$\rho_c = \frac{\sum M_i}{V} = \frac{M_1}{V} + \frac{M_2}{V} + \ldots + \frac{M_n}{V}$$

with Mass(M) = Density(ρ) × Volume(v) (1)

Therefore the eq. (1) can be rewritten as

$$\rho_c = \frac{\sum \rho_i v_i}{V} = \frac{\rho_1 v_1}{V} + \frac{\rho_2 v_2}{V} + \ldots + \frac{\rho_n v_n}{V} \quad (2)$$

But v_i / V in eq. (2) is just the volume fraction of the individual constituent V_i. For a simple composite comprising matrix and reinforcement, V_i is V_m and V_f. Then the eq. (2) is changed as

$$\rho_c = \sum \rho_i V_i \quad (3)$$

$$\rho_c = \rho_f V_f + \rho_m V_m \text{ with } V_f + V_m = 1 \quad (4)$$

$$= \rho_f V_f + \rho_m (1 - V_f) \quad (5)$$

$$= (\rho_f - \rho_m) V_f + \rho_m \quad (6)$$

where V_f and V_m are the volume fraction of fiber and matrix, and ρ_c, ρ_f and ρ_m are the densities of composite, fiber, and matrix, respectively.

Void Fractions. The information on void fractions or percentages of fiber reinforced composite laminates is essential because void contents presents in composite laminates may significantly affect some of its mechanical properties. Up to the lower percent of voids indicate a good composite laminate, but practical difficulties increase the fractions of voids in composite laminates. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering, and increased scatter in strength properties. Generally the knowledge of void content is desirable for estimation of quality of composites. The void fraction of UAASVFRVE composite laminates with the size of 25×25×3 mm was measured according to ASTM D2734-09.[18] The presence of voids in composite laminates will add to the total volume, but not to the weight of the composite. Therefore the eq. (5) may rewritten as

$$\rho_c = \rho_f V_f + \rho_m (1 - V_f - V_v) \quad (7)$$

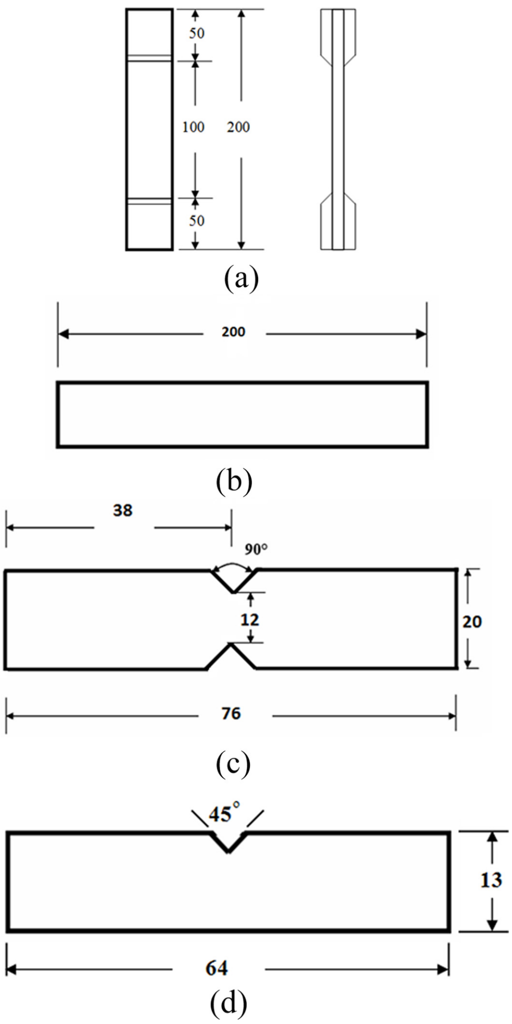

Figure 1. Composite specimens used for (a) tensile test: ASTM D3039M-08; (b) flexural test: ASTM D7264M-07; (c) impact test: ISO 180; (d) shear test: ASTM D5379-12.

where V_v is the void content. It means that the density of composites decreased with void content. If the fiber content is known and density of UAASVFRVE composite laminates can be measured with sufficient accuracy, then its void content can also be measured.

Mechanical Testing. For mechanical tests, composite specimens were cut from the prepared composite plates along the direction of the ASV fibers according to the ASTM standard. Tensile and flexural tests were performed according to ASTM D3039M-08[19] and ASTM D7264M-07[20] respectively, with a crosshead speed of 2 mm/min in an FIE (UTE, Fuel Instruments & Engineers Pvt. Ltd., Maharashtra, India) universal testing machine. Notched Izod impact and shear tests were carried out according to ISO 180[21] and ASTM D5379M-12[22] respectively. Composite specimen preparation for the characterization in this study is shown in Figure 1(a-d). At least five composite specimens of each designation are tested and reported (mean values of the five measurements) for the determination of mechanical properties. Experimental results were compared with RoM and BB results for the strength and modulus of composite laminates using regression equations and student *t* test was also performed for comparing mean values.

Results and Discussion

Density. The density of a composite is one of the most important factors determining the properties of the composites and it depends on the relative proportion of matrix and reinforcing materials. The densities of composite laminates can also be determined theoretically using RoM. The difference between theoretical and measured densities is shown in Figure 2. The difference is mainly due to the presence of voids in the prepared composite laminates. Therefore, it is required to measure the percentage of void contents in the prepared composite

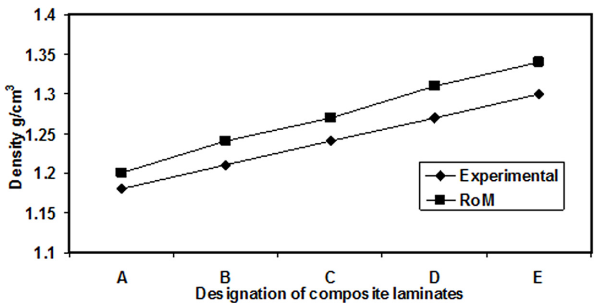

Figure 2. Variation of density with different weight percent of ASV fiber-reinforced-vinyl ester composite laminates (Fixed value: 0.02, Percentage: 5, Standard deviation (s): 1).

laminates. The density of all composite laminates designated from A to E increased with an increase in the weight fraction of the ASV fibers. It may be due to the higher density of the ASV fiber than that of the vinyl ester and thereby resulting composite density obviously increased.

Void Fraction. The knowledge about the void fraction in composite materials was desirable for estimation of the quality of the composite materials. With the addition of ASV fibers in a vinyl ester resin matrix, the void fraction is increased. The results show that void fraction varies between 0.22 to 0.69% for laminates with different weight percentages (number of layers) of ASV fibers. The void fractions were also predicted theoretically and compared with experimental values. The comparison of experimental void fractions with theoretical void fractions is also given in Figure 3. The experimental void fraction values were fitted with the predicted void fraction values by regression analysis and more than 99.07% of the variation could be predicted. If the densities of the constituents of composite materials are known, the volume fractions of fiber, resin, and void of composite materials can be calculated based on two assumptions. They are: the mass of fiber in the composite should be known; and the gas in any voids has the density of air (1.29×10^{-3} g/cm^3).

Tensile Properties. Tensile properties of UAASVFRVE composite laminates are given in Table 2. Tensile properties of composite laminate increases with increasing fiber weight percentage up to 45.67 wt% and number of layers up to five. Increase fiber wt% above a certain limit leads to the poor wetting of the fiber by resin and hence the mechanical properties of composite decrease. The maximum tensile strength was observed on composite designated as D. During the testing of composite designated as A, first, the matrix cracks take place with a loud sound and followed the failure of ASV fibers. After the failure of the matrix, the entire load is suddenly transferred to the ASV fibers, which leads to the catastrophic failure

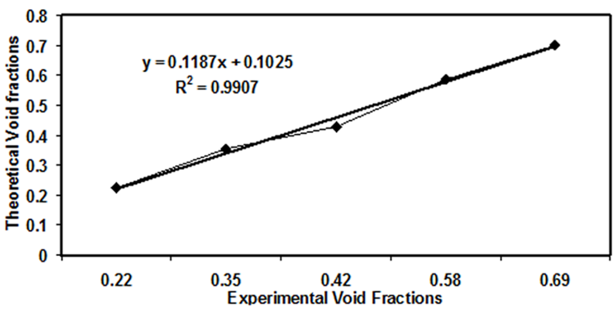

Figure 3. Comparison of experimental void fractions with theoretical void fractions.

Table 2. Tensile Properties of ASV-Vinyl Ester Composite Laminates

Designation of composite	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
A	20.4	1115.3	3.12
B	37.9	1221.7	3.89
C	52.7	1335.2	4.57
D	66.1	1447.6	4.38
E	60.5	1562.8	4.36

of the ASV fibers. The crack formed on the matrix spreads throughout the specimen along the horizontal direction. But in testing of composite designated as E, both the ASV fiber and matrix fail simultaneously and the crack extends and also spreads over a larger area. Generally, the properties of the plant based natural fibers are not constant and it depends mainly on the nature of the plant, locality in which it is grown, the age of the plant, and the extraction method used. Therefore, a few ASV fibers will break during the testing of composite designated as A. The region close to the broken end of ASV fibers is subjected to high stress concentration due to the voids created by the broken ASV fibers. Failure of other ASV fibers nearer to the broken ASV fibers takes place due to the high normal stress and stress concentration. Each broken end of ASV fibers in composite laminates creates additional stress concentration and eventually many of the ASV fibers break and also the surrounding micro cracks join to form a large crack in composite laminates. Tensile modulus of composite laminates linearly increased with increasing fiber wt% and number of layers. Composite laminate designated as E shows the maximum value of tensile modulus compared to other composite laminates. But the maximum percentage of elongation was identified in composite laminate designated as C.

Flexural, Impact and Shear Properties. The flexural, impact and shear properties are studied for different weight percentages and is given in Table 3. Both the flexural and impact strength increases with increase of fiber wt% up to 45.67 wt% and then decrease. The maximum flexural and impact strength value were identified in composite designated as D. The flexural modulus increased linearly with fiber wt%. The shear strength also increased with increasing the fiber wt% and then dropped. It was identified during the testing that deflection during bending initiates rupture of the matrix and results in fiber pull out from the matrix, fiber breakage and fiber – matrix de-bonding. It was also observed that fiber – matrix de-bonding takes place at peak load and then composite spec-

Table 3. Flexural, Impact and Shear Properties of ASV-Vinyl Ester Composite Laminates

Designation of composite	Flexural strength (MPa)	Flexural modulus (MPa)	Impact strength (KJ/m^2)	Shear strength (MPa)
A	29.4	1178.2	2.8	5.1
B	40.2	1261.8	3.5	8.4
C	56.8	1388.2	4.1	8.9
D	71.3	1514.4	4.9	9.7
E	62.1	1647.1	4.4	8.1

imens fails.

Rule of Mixtures. The strength of fiber reinforced polymer composite laminates can be predicted using a micro-mechanics approach termed the rule of mixtures. It is based on certain assumptions that the fibers are uniformly distributed throughout the matrix; a perfect bonding exists between the fibers and the matrix; the matrix is free of voids; the tensile loads applied to composite laminates are either parallel or normal to the fiber direction; there is no residual stresses; both the fiber and the matrix behave as linearly elastic.

The tensile load or force applied on composite laminates is shared by the fiber and the matrix i.e.,

$$F_c = F_f + F_m \text{ with } \text{Force}(F) = \text{Stress}(\sigma) \times \text{Area}(A) \tag{8}$$

$$\sigma_c \times A_c = \sigma_f \times A_f + \sigma_m \times A_m \tag{9}$$

$$\sigma_c = \sigma_f \times \frac{A_f}{A_c} + \sigma_m \times \frac{A_m}{A_c} \tag{10}$$

where A_c, A_f, A_m are area of cross sections of composite laminates, fiber, and matrix. We can also know that: $V_f = A_f/A_c$ and $V_m = A_m/A_c$, where V_f and V_m are volume fractions of fiber and matrix.

Invoking Hooke's Law,

$$E_c \times \varepsilon_c = E_f \times \varepsilon_f \times V_f + E_m \times \varepsilon_m \times V_m \tag{11}$$

For strain compatibility assuming that the average strains in the composite laminate, fiber, and matrix along the longitudinal direction are equal. Therefore, the longitudinal modulus is given by the following equation:

$$E_c = E_f \times V_f + E_m \times V_m \tag{12}$$

Similarly the tensile stress in a composite laminate is

$$\sigma_c = \sigma_f \times V_f + \sigma_m \times V_m \tag{13}$$

Bowyer and Bader (BB) Model. The BB model was also used for calculating the tensile modulus and tensile strength of composite laminates. The BB equations for tensile strength and modulus are

$$E_c = \alpha_v \times E_f \times V_f + E_m \times V_m \text{ with } \alpha_v = k_1 \times k_2 \tag{14}$$

$$\sigma_c = \alpha_v \times \sigma_f \times V_f + \sigma_m \times V_m \tag{15}$$

where k_1 is fiber orientation factor and k_2 is the fiber length factor, which is depending on critical fiber length, and the parameter α_v is the overall reinforcing factor. In fitting the experimental E_c *vs*. V_f data to eq. (14), one has to adjust the value of the product $k_1 \times k_2$. It is difficult to determine the value of k_1 and k_2 separately. The parameter α_v expresses to what extent the modulus of the fiber contributes to the modulus of composite laminates.

The tensile strength was calculated theoretically based on the RoM and BB is shown in Figure 4. It is seen that there is a larger deviation from the experimental values when compared with RoM. But, there was a little deviation when compared with BB. In the RoM, the theoretical strength values and the experimental strength values are very close at high fiber wt%, but at a low fiber wt% the deviation is high. Figure 5(a) and (b) shows the regression between experimental strength and predicted strength (calculated by RoM and BB). Analysis of variance (ANOVA) of the regression between experimental and predicted strength values has shown that the regression is significant at 95% confidence level. The regression equation between experimental and predicted values are also presented in Figure 5(a) and (b). The coefficient of determination (R^2) between experimental and predicted (calculated by RoM) values were determined to be 0.9894 indicating a linear correlation. R^2 value of the experimental and predicted (calculated by BB) values were 0.9999. It is also indicating a strong linear correlation. It is conformed that the predicted strength values using Bowyer and Bader model are close to the experimental

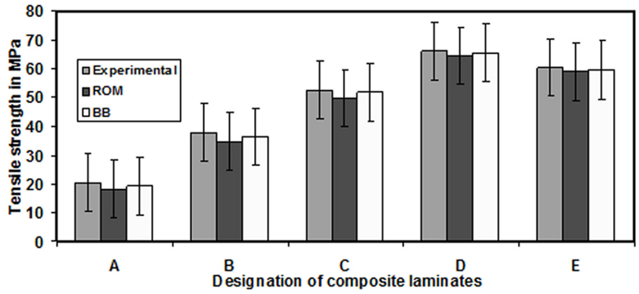

Figure 4. Comparison of experimental strength with RoM and BB models.

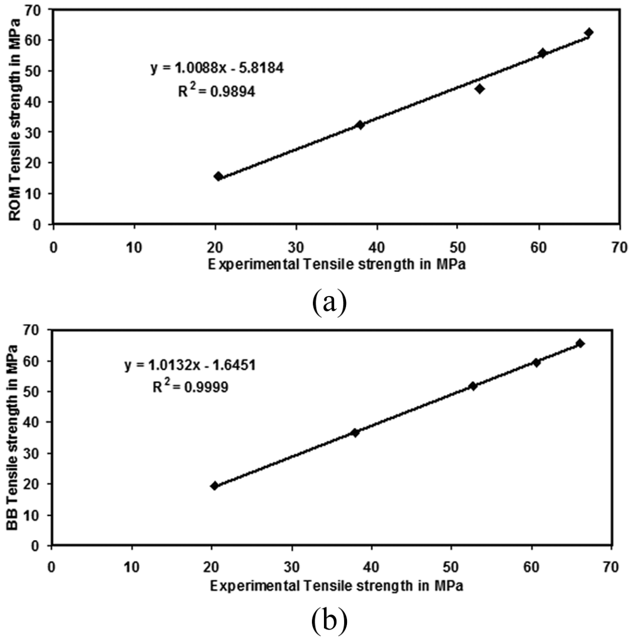

Figure 5. Comparison of (a) experimental strength with ROM strength; (b) experimental strength with BB strength.

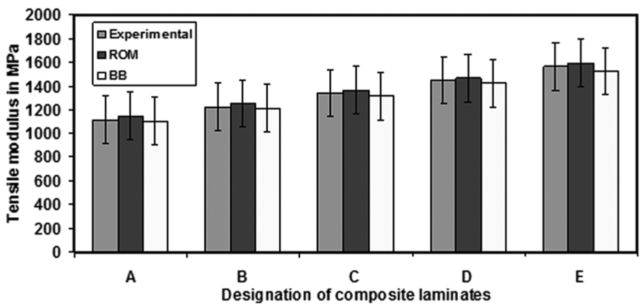

Figure 6. Comparison of experimental modulus with RoM and BB models.

strength values in all fiber wt%.

Figure 6 shows the experimental and theoretical results of tensile modulus for various fiber weight percentages. In case of the rule of the mixture model, the experimental values were less than the theoretical values. But, in case of BB model the experimental values were higher than the theoretical values. Figure 7(a) and (b) presents the regression between experimental modulus and predicted modulus using RoM and BB models. Analysis of variance of the regression between these two values has proved that the regression is significant at 95% confidence level. The regression equation between these values is also presented. The coefficient of determination (R-squared) between experimental and predicted modulus using RoM was determined to be 0.9884 which is indicating a linear

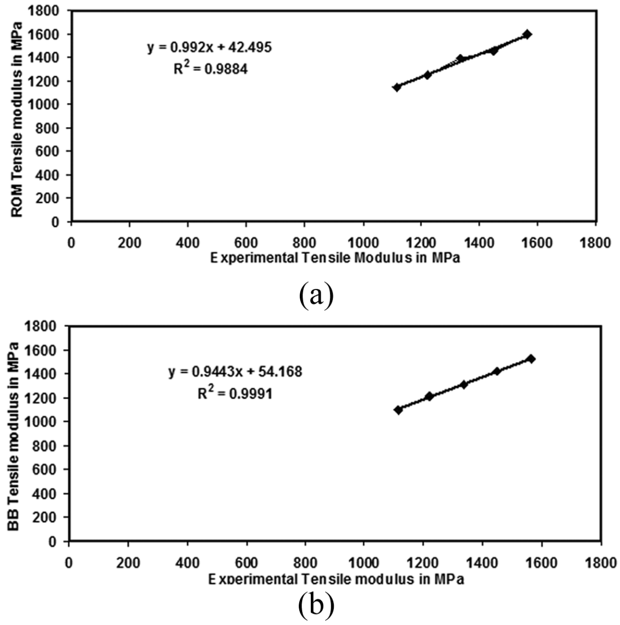

Figure 7. Comparison of (a) experimental modulus with RoM modulus; (b) experimental modulus with BB modulus.

correlation. R^2 value of the experimental modulus and predicted modulus (calculated by BB) was 0.9991. It also indicates a strong linear correlation. It was observed from the above results that the BB model is better than the RoM model to predict the tensile properties of ASV fiber - vinyl ester composite laminates.

Student *t* Test. Tables 4 and 5 show the difference of mean values between experimental and predicted (calculated by RoM and BB) tensile strength values of each composite laminate. The double symbol '*' indicates no significant difference between experimental and predicted tensile strength values for all composite laminates designated from A to E. It was observed that the percentage of difference between experimental and predicted values becomes higher in lower fiber wt% in both the case. It is also noted that the low percentage of difference is identified in composite designated as D.

After the student *t* test, the predicted tensile modulus values using RoM and BB and experimental tensile modulus values of all composite laminates designated from A-E are given in Tables 6 and 7. Again, the double symbol '*' indicates no significant difference between experimental and predicted tensile modulus values for each composite laminates. The percentage of difference between the predicted (by RoM and BB) modulus values is presented in the fourth column for each Tables 6 and 7. Moreover, the percentages of differences in the Table 7 are small compared to the Table 6. It was also observed that

Table 4. Comparison of the Predicted Tensile Strength Using RoM and Experimental Tensile Strength Means by Student *t* Test

Designation of composite	Variable	Mean strength value (MPa)	Significance p = 0.05	Difference (%)
A	Experimental Predicted	20.4 15.7	**	29.94
B	Experimental Predicted	37.9 32.3	**	17.34
C	Experimental Predicted	52.7 44.1	**	19.50
D	Experimental Predicted	66.1 62.6	**	5.59
E	Experimental Predicted	60.5 55.9	**	8.23

Table 5. Comparison of the Predicted Tensile Strength Using BB and Experimental Tensile Strength Means by Student *t* Test

Designation of composite	Variable	Mean strength value (MPa)	Significance p = 0.05	Difference (%)
A	Experimental Predicted	20.4 19.2	**	6.25
B	Experimental Predicted	37.9 36.5	**	3.84
C	Experimental Predicted	52.7 51.7	**	1.93
D	Experimental Predicted	66.1 65.5	**	0.92
E	Experimental Predicted	60.5 59.6	**	1.51

Table 6. Comparison of the Predicted Tensile Modulus Using RoM and Experimental Tensile Modulus Means by Student *t* Test

Designation of composite	Variable	Mean strength value (MPa)	Significance p = 0.05	Difference (%)
A	Experimental Predicted	1115.3 1143.2	**	2.50
B	Experimental Predicted	1221.7 1251.6	**	2.45
C	Experimental Predicted	1335.2 1394.8	**	4.46
D	Experimental Predicted	1447.6 1453.8	**	0.43
E	Experimental Predicted	1562.8 1598.1	**	2.26

Table 7. Comparison of the Predicted Tensile Modulus Using BB and Experimental Tensile Modulus Means by Student *t* Test

Designation of composite	Variable	Mean strength value (MPa)	Significance p = 0.05	Difference (%)
A	Experimental Predicted	1115.3 1103.1	**	1.11
B	Experimental Predicted	1221.7 1214.4	**	0.60
C	Experimental Predicted	1335.2 1311.5	**	1.81
D	Experimental Predicted	1447.6 1425.2	**	1.57
E	Experimental Predicted	1562.8 1527.1	**	2.34

the percentage of difference is higher in composite designated as A (Table 6). But it was identified in composite designated as E (Table 7). In both the cases, the low percentages of differences were observed in composite designated as D for Table 6 and B for Table 7.

After *t* tests, a clear result was observed from the summary of the percentages of differences from the experimental for both RoM and BB. The BB model has resulted in very small percentages of the differences between predicted and experimental values (both tensile strength and modulus) for all composite laminates. Finally, it was concluded that the BB works better than RoM in case of unidirectional aligned ASV fiber -vinyl ester composite laminates.

Conclusions

Mechanical properties of UAASVFRVE composite were investigated in the present work with special reference to the fiber weight percentage and the number of layers. Composites were characterized by following mechanical properties: tensile properties, flexural properties, impact strength and shear strength. In case of mechanical behavior, composite laminate having 45.67 wt% and four layers has been found to be more effective as compared to other composite laminates. The tensile properties of ASV fiber-vinyl ester composite laminates were compared with two different theoretical models such as the RoM and BB. The comparison between experimental and predicted values (both tensile strength and modulus) values indicated that the best prediction of values by the BB model was achieved compared to the RoM model. Furthermore, in all cases the relationships between experimental and predicted values (both tensile strength and modulus) were determined to be good by the high R^2 values obtained. This means that a strong linear relationship can be expected. The results also suggest that RoM equation can be suitably applied to the prediction of mechanical properties of unidirectional aligned ASV fiber reinforced vinyl ester composite laminates. Finally, it can be concluded that by utilizing ASV fibers in the form of laminates, we can prepare the most effective and ecofriendly composite materials possessing suitable mechanical properties and this easy available fiber from the southern Indian region especially from the South and West Tamilnadu can be a potential candidate for reinforcement in polymer composite laminates.

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