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Fabrication and characterization of Polymer laminate composites reinforced with bi-woven carbon fibers

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ABSTRACT
The present paper evaluate slaminatedcarbonbi-wove fibers Reinforced with vinyl ester composites. Vinyl ester was used as a matrix to prepare composites by in situ polymerization technique. Four planar layers were made simultaneously by keeping one over the other and each layer made sure to be weighed off by 15% which was maintained in all layers with different orientations. Pre-assumed Layer-1 is (50/50)50%,0º; Layer-2 is (35/35/30) 35% 0º, 35% +45º,30%,0;Layer-3is (25/50/25) 25% 0º, 50%+45º,25-45º; and Layer-4is (25/25/25/25) (25% 0º, 25% +45º,25% -45º,25% 90º.The composite was prepared with the help of hand layup technique. Test ready specimens were tested with the help of shearing machine in accordance with ASTM Standards .It was observed that vinyl ester made good interface with parent fiber material. Flexural strength and Tensile strength have improved up to 3rd layer and decreased afterwards whereas Flexural modulus and Tensile modulus have linearly increased up to 4th layer. Thermal stability and Glass transition temperature have also been found to be satisfactory for all the laminated layers. Chemical resistance was good for the entire chemicals except sodium hydroxide.
Keywords: laminate composites, vinyl ester, carbon fibers, flexural strength, DSC, TGA

I. INTRODUCTION

It would be hard to imagine a modern society today without plastics. Plastics have found a myriad of uses in fields as diverse as household appliances, packaging, construction, medicine, electronics, and automotive and aerospace components. The reason for its success in replacing traditional materials such as metals, wood and glass in such a diverse range of applications, is the ability to modify its properties to meet a vast array of designer's needs.

In addition to that ease with which plastics can be processed, makes them as ideal materials for the production of a variety of components. The needs of the high rise building and aerospace industry led to the development and application of composite materials. Advances in the manufacturing process and technology of laminated composites have changed the use of the composites from secondary structural components to the primary ones. Practically laminated composites are commonly used as a part of building like sandwich panel, aeronautical and aerospace industries as the main part of the structure rather than aluminum or other metallic materials. Low weight, high strength and greater rigidity were of paramount interest. A variety of structural elements such as cylinders, beams, plates and shells could be potentially used as laminated composites. The high stiffness-to-weight ratio coupled with the flexibility of the selection of

the lamination scheme that can be tailored to match the design requirement makes the laminated plate an attractive structural component for many industries. Depending upon their applications, plates can be moderately thick. To use the laminated composite plates efficiently, it is necessary to develop appropriate analysis theories to predict accurately their structural and dynamic behavior. The increased utilization of composite materials in several engineering applications has led to intensive research activities in linear and non-linear, static and dynamic analysis of laminated composite plates. The majority of the investigations on laminated plates utilize either the Classical Lamination Theory (CLT), or the First-order Shear Deformation Theory (FSDT).Advanced composite materials are widely used in aircraft and space systems due to their advantages of high stiffness and strength-to-weight ratios.

However, the analysis of multi-layered structures is a complex task compared with conventional single layer metallic structures due to the exhibition of coupling among membrane, torsion and bending strains; weak transverse shear rigidities; and discontinuity of the mechanical characteristics along the thickness of the laminates. More accurate analytical/numerical analysis based on three-dimensional models may be computationally involved and expensive. Hence, among researchers, there is a growing appreciation of the importance of developing

new kinematics for the evolution of accurate two dimensional theories for the analysis of thick laminates with high orthotropic ratio, leading to less expensive models. In this context, the applications of analytical/ numerical methods based on various higher-order theories, not only for the vibrations of thick laminates, but also for the high frequency vibrations of thin composite plates, has recently attracted the attention of several investigators. Various structural theories proposed for evaluating the characteristics of composite laminates under different loading situations have been reviewed and assessed by Noor and Burton [1,2], Tauchert [3], Kapania and Raciti [4], ReddyJN [5] and, more recently, by Mallikarjuna and Kant [6] and Varadan and Bhaskar [7]. It may be concluded from the literature that the analysis of composite plates under thermal environment is generally based on classical lamination theory and first-order shear deformation theory. Furthermore, the assumption of displacements as linear functions of the coordinate in the thickness direction has proved to be inadequate for predicting the response of thick laminates. Higher-order displacement fields yielding quadratic variations of transverse shear strains have been attempted by many researchers [8-15] for better accuracy, but the application of higher-order theory for the investigation of thick multi-layered plates under thermal load seems to be scarce in literature compared to the analysis of mechanically loaded laminates [6]. Three-dimensional elasticity analysis carried out by Bhaskar et al. [16] for thick laminates subjected to thermal loads reveals the non-linear variation of in-plane displacements through the thickness and abrupt discontinuity in slope at any interface and thickness-stretch/contraction effects in the transverse displacement. Although higher-order theories based on the discrete layer approach [17-21] account for slope discontinuity at the interfaces, the number of unknowns to be solved increases with increase in the number of layers.

Recently, Ali et al. [22] have proposed a new higher-order plate theory based on the global approximation approach for the static analysis of multilayered symmetric composite laminates under thermal/ mechanical loading, incorporating realistic through the thickness approximations of the in-plane And transverse displacements based on the work given in [16]. This formulation has proved to give very accurate results for the static analysis of symmetric cross-ply laminates, and this excellent performance of the theory for thick laminates motivated the present extension of the formulation for the dynamic analysis of thermally/ mechanically loaded general composite laminates.

In order to address the above various issues, authors focused on the carbon fiber reinforced polymer laminated composites. Composites were prepared in such a way that woven type carbon fibers cut in accordance with cast mould size of four different layers were cumulated simultaneously. Laminated composites were characterized on mechanical, thermal and chemical resistance properties.

II. MATERIALS & METHODS

The vinyl ester resin used was HPR 8711 grade, a Bakelite Hylam product. Methyl ethyl ketone peroxide (MEKP), Co-napthenate and N, N dimethylamiline were used as the catalyst, accelerator and promoter respectively. The thermal characteristics TGA, DSC measured on laminated polymer composites using SDT Q600 TGA/DSC (TA Instruments) at a rate of 10°C/min under nitrogen flow measurements were carried out at 20°C temperature, 40 % relative humidity. A JEOL JSM-6400 JAPAN scanning electron microscope at 15 kV accelerating voltage was equipped with energy dispersive spectroscopy (EDS) to ascertain the fiber/filler interfaces with the main modified matrix. Fractured specimen surfaces were gold-coated and the fractured surface was observed using a scanning electron microscope. The fractured surfaces were gold-coated with a thin film to increase the conductance. The FTIR spectra of the powders of the samples were run on an ABB-Bomem FLATA-2000 model spectrophotometer using KBr pellets. The concentration of the fabric powder was maintained at 1% in KBr. Flexural properties were measured in accordance with the procedures in ASTM D790. Flexural strength is a measure of how well, a material can resist bending. Values for flexural strength and flexural modulus are reported in MPa. Impact properties were measured in accordance with ASTM D256. For chemical resistance, test samples were cut like standard sizes (i.e.10X10X3 mm^3)on par with ASTM G 543-87.The effect of some acids, alkalis, and solvents, that is, glacial acetic acid, nitric acid, hydrochloric acid, ammonium hydroxide, aqueous sodium carbonate, aqueous sodium hydroxide, carbon tetrachloride, benzene, distilled water, and toluene were used on the matrix and the hybrid composites were studied. In each case, the samples were pre-weighed in a precision electrical balance and dipped in the respective chemical reagents for 24 h. Then they were removed immediately, washed in distilled water, and dried by pressing on both sides with a filter paper at room temperature.

III. FABRICATION OF LAMINATED COMPOSITES :

Composites were prepared using hand layup technique. A mould was prepared on par with ASTM dimensions, and it was coated with a mould releasing agent(poly-vinyl-alcohol) for easy removal of casting.

Predetermined dimensions of 300 x 300 mm^{2}carbon woven fiber were cut with appropriate scissors and made sure to ensure flat surfaces of mats. Mats were mercerized for about 2h under tensile loading to obtain the flat surfaces. Vinyl ester was stirred gently at 50^0 C to decrease its density as it facilitates easily flowing properties to the end destiny. Then the accelerator/catalyst/promoter (100:2/2/2) parts by weight was added to the modified vinyl ester mixture. The mat should be placed in the mould cavity before modified solution is to be poured, on par with the orientations mentioned in the above for different layers. Laminated mixture was poured into the mould cavity in such a way that mat should be completely drowned with laminated solution and yet it has to be in the approximately center place if it is asked to be single layer-1. Air was removed with the help of vacuum machine. Brush and roller were used to impregnate fiber. The closed mould was kept under pressure for 24 h at room temperature. To ensure complete curing, the composite samples were post-cured at 80˚C for 45 min. Now the single layered laminated polymer composites are ready. In order to get two layered laminated composites, it is made sure to keep two mats in the mould cavity one upon the other in the orientations mentioned above and then same modified solution has to be poured into the mould. Similarly three and four layered laminated composites were prepared. Specimens of required size were cut out from the post cured laminated composites for further characterizations as per ASTM standards.

IV. RESULTS AND DISCUSSIONS:

Four different laminated layers were coded as mentioned below. Layer-0 is (0/0)no fiber was inserted); Layer-1 is (50/50)50%,0º; Layer-2 is (35/35/30) 35% 0º, 35% +45º,30% 0^0;Layer-3 is (25/50/25) 25% 0º, 50%+45º,25-45º; and Layer-4is (25/25/25/25) (25% 0º, 25% +45º,25% -45º,25% 90º; (For example:(25/25/25/25) means first ply is 0º and the second ply is +45º; third ply is -45º: fourth ply is 90º). Carbon fibers were dispersed into the polymer blend in the form of four layers as mentioned above as shown in the **Fig. 1**.
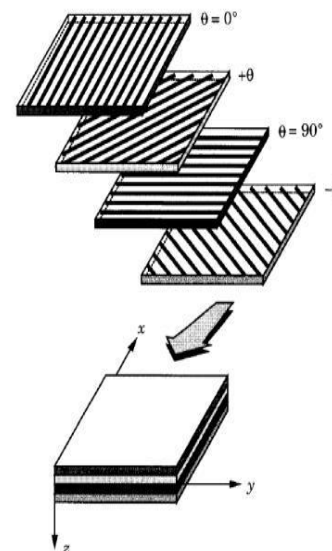

Figure: 1 A laminate made up of lamina with different fiber orientations

Improvement of flexural strength of laminated layers is shown in **Fig.2.** Flexural strength for layer-0 laminate was 41MPa approximately. Flexural strength was significantly increased from layer-1 to layer-3 linearly, on other hand after **3rd** layer laminate strength becomes decreased. In the layer-4 flexural strength was 50MPa. Reason for reduced strength for the layer-4 is poor wetting as the main reason because, then as number of layers are increased consequently resin may not go to all the places due to the entrapment of the fiber laminates, due to this reason, in the same places there will be a entrapment of air makes the crack initiation takes places. The increase in strength was due to good fiber matrix interactions, which was largely due to the orientation of fiber plane structures might have enhanced ability of the fiber resistance.
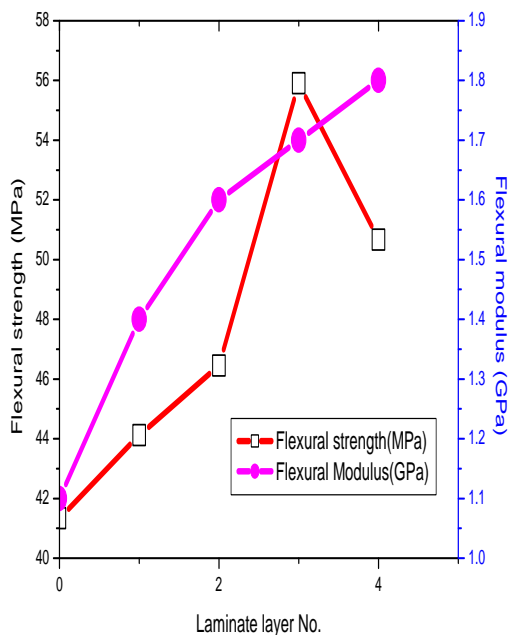
FIGURE 2: Flexural strength and modulus of vinyl ester laminated composites as a function of no of plies.

FIGURE 3: Tensile strength and modulus of vinyl ester laminated composites as a function of no of plies.

Planar type fibers have higher aspect ratio and this increases the wet ability of the fibers by the matrix, thus creating fewer micro voids between the fiber and matrix. Another reason is if layers are not perfectly bonded together as a result of this cohesion becomes poor between the matrixes and layers. Another reason is when stress is being transfers from fiber to matrix, in the process as matrix does not resist large stress, automatically crack will initiate. Another reasons are the material of each layer is linearly elastic and has three planes of material symmetry (i.e., orthotropic) (restriction),each layer is of uniform thickness (restriction), the strains and displacements are small (restriction),the transverse shear stresses on the top and bottom surfaces of the laminate are zero (restriction). Up to some extent matrix could have accommodated the resin, so much so that strength was increased up to layer-3. When fiber layers were dispersed in to the matrix with different orientations the toughening effect of the composites will raise from the occasion. This could enhance the flexural modulus of the laminates. In **fig.3,** tensile strength was increased significantly from 0-3 laminates, whereas reduces suddenly at layer-4. The decrease in tensile strength at layer-4 laminate could be attributed to poor adhesion between the layers and the modified matrix, which promotes the micro-cracks formation at the interface.
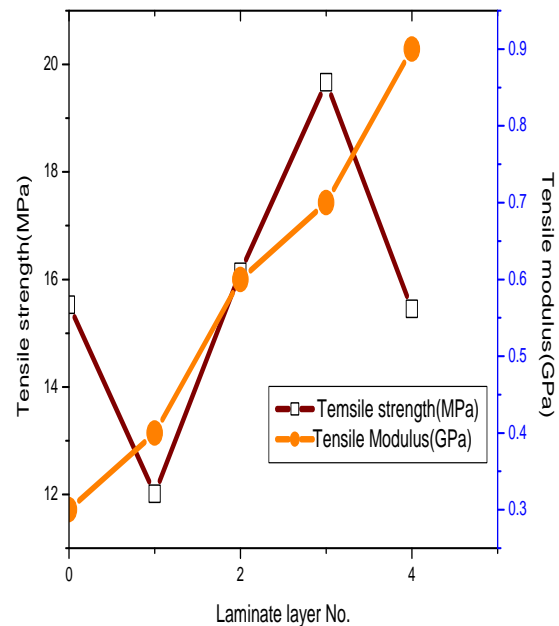
A larger layer content results in more and probably larger in more void formation during processing, which leads to micro-crack formation under loading and therefore reduces the tensile strength. [15] Study highlighted that stacking pattern of the different components in hybrid laminated composite play an important role in influencing the mechanical properties of the hybrid composites. [5, 6, 7, 12] reported that layering pattern of the fibers in hybrid composite effect tensile strength and modulus of the hybrid composites. Various researcher studied synthetic fiber/natural fiber based hybrid composite but natural fiber based hybrid composites are recent phenomena due to environmental concern. Moving on to, the variation of tensile modulus of laminate composites are tabulated table.1, in which tensile modulus significantly increased from 0-3 layers, but decreased at layer-4.

The attributed reasons are, when inclusion of filler in the matrix leads to significant increase in tensile modulus. The addition of rigid layers increases the stiffness in the matrix. This development is well explained by the percolation theory. According to this theory there is a matrix zone around each filament affected by stress concentration. If the distances between filaments are small enough, the zones join together and form a percolation network, which increase the tensile modulus [11, 13].

Thermal stability of various laminated layers as a function of temperature as shown in **fig.4(a).** Thermal stability for layer-2 was approximately 350ºC with

slight decomposition weight loss of >5%, and then for layer-3, curve shifting towards the right side shows that thermal stability was good enough for the same with negligible decomposition weight loss of >2%, and the thermal stability this was 355ºC. However for layer-4 the thermal stability got reduce, the reasons are attributed that, there might be poor miscibility of polymers or there might be insufficient resin to wet the entire resin, due to this there is possibility of formation of voids makes the matrix fast decomposition of the fiber. From the earlier researcher were observed the similar phenomenon [21, 23]. Variation of glass transition temperature with respect to the temperature are shown in the **fig.4(b)**, layers 2,3, and 4 glass transition temperature was increased cumulatively and it was observed from the figure that layer-4.
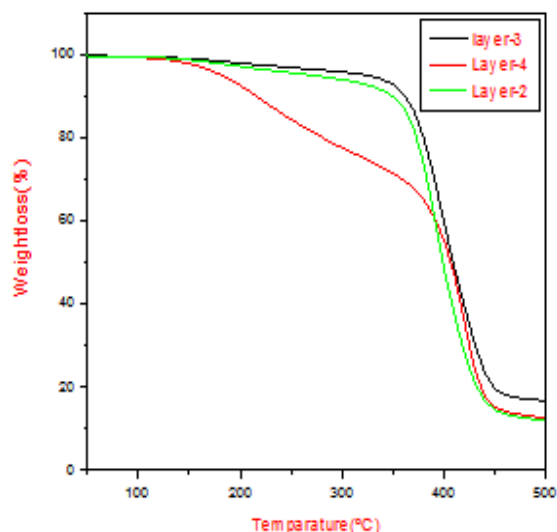

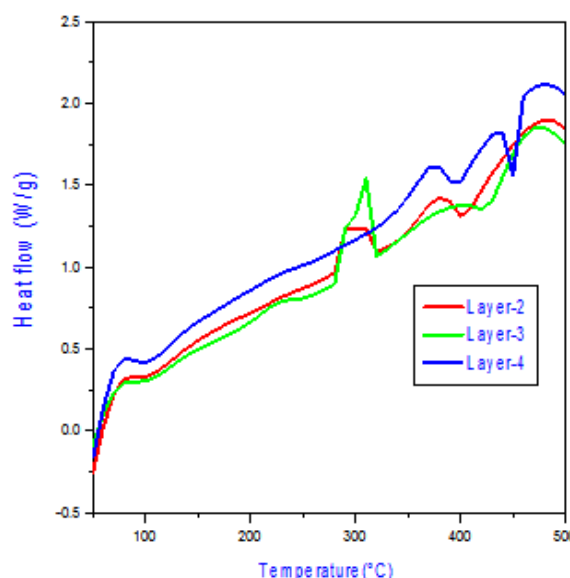**Figure: 4** (a) TGA Curves for different layers of laminated composites.

Figure: 4 (b) DSC Curves for different layers of laminated composites.

The effect of the used fiber on crystallinity of laminated composite was analyzed by DSC depicts DSC thermograms at 50^0–500ºC region of the second cooling cycle for pure laminated composite (later-2, 3 and 4) and laminated composite including. Crystallization exothermic appears as a single, distinct peak in both cases and the Tg temperature is almost unchanged (300ºC for neat laminated composite and 300ºC and 299ºC for layer-2 and 3 respectively). Thus, it seems that the fiber used in our study is active in sense of nucleation effect ,probably due to the facts that (1) the average particle size of the used fiber was relatively high (2 lm), and(2) the fiber filaments were surface treated to increase the surface energy.

Fig.5 (a) shows the variation transmittance with respect to the wave length (FTIR) analyses for (a) Layer-4 and (b) Layer-3 laminated composites. In **Fig.5(a)**, we can see that the broad peak at 2958–2858 cm^{-1} is attributed to stretching vibration of –OH of carboxyl groups. And the shoulder peak appeared at 1,468 cm^{-1} was attributed to carbonyl stretching vibration of –COOH groups. These features revealed the existence of carboxyl groups on nano clay molecular chains. In **Fig. 5(b)**, it can be found that the characteristic peaks of blend still exist at 2916$cm^{-1}$and 2848 cm^{-1} for C=O and C–O–C stretching vibration, respectively. Meanwhile, the 3431cm^{-1} for the C≡N of Nano clay particles also appeared in thelayer-4 laminated composites.

No of laminated layers	Flexural strength (MPa)	Flexural modulus(GPa)	Tensile strength (MPa)	Tensile modulus (GPa)
Layer-0	41±1.34	1.1±1.02	15±1.53	0.3±2.47
Layer-1	44±2.12	1.4±2.10	12±2.01	0.4±2.36
Layer-2	46±1.45	1.6±2.46	16±2.14	0.6±2.48
Layer-3	55±2.91	1.7±2.16	19±2.67	0.7±2.74
Layer-4	50±2.67	1.8±2.74	15±2.00	0.9±2.79

Table: 1 Variation of flexural strength and modulus and tensile strength and modulus of different layers of laminated composite
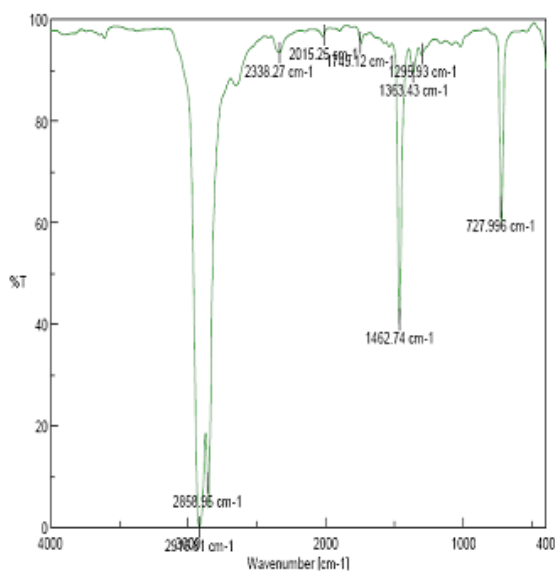
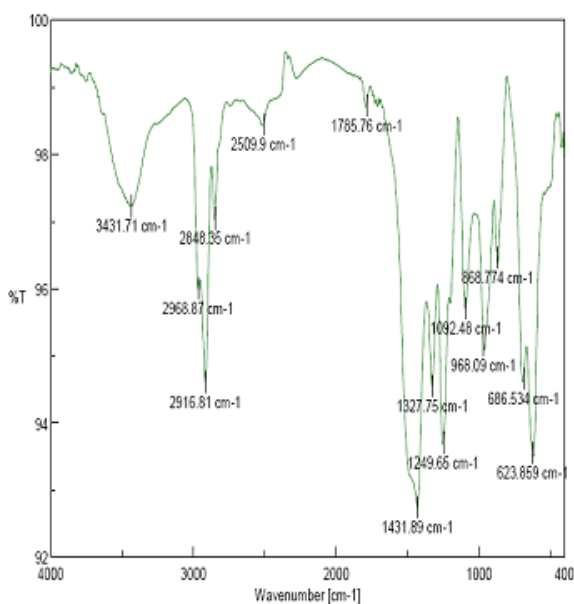**Figure: 5** (a) FTIR analyses for Layer-4 and laminated composites.

Figure: 5 (b) FTIR analyses for Layer-3 and laminated composites

Especially, the hydroxyl stretching vibration of blend shifts from 3,431 to 3,421 cm^{-1} for the composite with layer-4. **Table 2** shows the effect of some acids, alkalis, and solvents, that is, glacial acetic acid, nitric acid, hydrochloric acid, ammonium hydroxide, aqueous sodium carbonate, aqueous sodium hydroxide, carbon tetrachloride, benzene, distilled water, and toluene were used on the matrix and the hybrid composites were studied.

Chemical resistance	Layer -0	Layer -1	Layer -2	Layer -3	Layer -4
Hydrochloric acid	0.859	0.875	0.850	0.845	1.452
Acetic acid	0.235	0.236	0.230	0.214	0.485
Nitric acid	1.532	1.534	1.541	1.520	1.78
Sodium hydroxide	0.523	0.254	0.251	0.246	0.356
Sodium carbonate	-0.231	-0.230	-0.221	-0.220	-0.256
Ammonium hydroxide	0.742	0.741	0.740	0.742	0.765
Benzene	10.14	9.454	9.452	9.256	9.325
Toluene	4.85	3.994	3.951	3.489	5.687
Carbon tetrachloride	2.59	2.424	2.410	2.405	2.965
Distilled water	1.252	1.242	1.230	1.265	2.365

TABLE 2: Chemical resistance of vinyl ester laminated composites of different layers.

It was clearly evident that weight gain is observed for almost all the chemical reagents except sodium carbonate. It is also observed from the table that layer-4 gain more weight than other laminates. The reason is attack of the carbonated hydrocarbons on the cross-linked laminated system. The positive values indicate that the laminates were swollen with gel formation rather than dissolving in chemical reagents. It was further observed that laminates were also resistant to water.

V. CONCLUSIONS

The experimental investigations used for the analysis of tensile behavior of bi-woven carbon fiber reinforced polymer laminates leads to the following conclusions. The laminated specimens with lesser thickness lead to more ultimate tensile strength irrespective of fiber orientations. Young's modulus of specimens increases with increase in laminates irrespective of its orientation. Flexural strength and tensile strength were found to be improved up to layer-3 but decreased afterwards, however, flexural modulus and tensile modulus increased linearly up to layer-4. Similarly chemical resistance and FTIR results were also found to be good for the composites.

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