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Development and Assessment of Vinylester Reinforced with *Sansevieria cylindrica* / Betel Nut Fruit fibers

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Abstract

The objective of this research was to gain a better understanding of tensile properties of vinylester resin hybrid composites reinforced with *Sansevieria cylindrica* fiber (SCF)/betel nut empty fruit fiber (EFF) using rule of hybrid mixture. Tri layer hybrid composites of betel nut empty fruit (EFF) and *Sansevieria cylindrica* leaf fiber (SCF) was prepared by keeping betel nut EFF as skin material and *Sansevieria cylindrica* as the core material and vice versa. The chemical resistance, void content and tensile properties of betel nut EFF/SCF composites was investigated with reference to the relative weight of betel nut EFF/SCF, i.e. 4.5:1, the fibre loading was optimized and different layering pattern were investigated. It is found from the chemical resistance test that all the composites are resistant to various chemicals. It was observed that marked reduction in void content of hybrid composites in different layering pattern. From the different layering pattern, the tensile properties were slightly higher for the composite having SCF as skin and betel nut EFF as core material. Scanning electron microscopy (SEM) was used to study tensile fracture surfaces of different composites.

Keywords: Hybrid composites; Mechanical properties; Vinylester resin; NFF; SCF

1. Introduction

Polymer composites find their way into hundreds of new applications from golf clubs and tennis rackets to Jet Ski, aircraft, missile, spacecraft and marine applications. Other uses include transportation, chemical equipment and machinery construction, electrical and electronics equipment, fishing rods and storage tanks. A composite is a complex solid material, made by combining two or more dissimilar materials in such a way that the resulting material is endowed with some superior and improved properties. Owing to these superior properties, polymer composites find various applications in our daily life. Composites are light weight, high strength to weight ratio and stiffness properties have come a long way in replacing the conventional materials such as metals and wood (Ashok Kumar *et al* 2012a). Composites materials are attractive because they combine material properties not found in nature. Such materials often results in light weight structures having high stiffness and tailored properties for specific applications, (Ashok Kumar *et al* 2012b) thereby saving weight and reducing energy needs. In fibre reinforced composites, the fibers serves as reinforcement by giving strength stiffness to the structure while the plastic matrix serves as the adhesive to hold the fibers in place so that suitable structural component can be made (Karina *et al* 2008).

Because of the presence of hydroxyl and other polar groups in various constituents of natural fiber, the moisture uptake is high which lead to weak interfacial bonding between fibers and the relatively more hydrophobic polymer matrices, thus compromising the mechanical properties of the composites. Therefore, (Khalil *et al* 2007) data on the effects of moisture on retention of mechanical properties of natural fiber reinforced composites during long-term

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environmental exposure are crucial for them to be utilized in outdoor applications. Studies on the environmental effect on the mechanical properties of some natural fibers such as jute, sisal, and wood- flour etc., composites have been reported (John and Venkatanaidu 2004).

It is shown by previous studies that moisture causes degradation of mechanical properties of natural fiber reinforced composites to a larger extent when compared to synthetic fiber reinforced composites, as a consequence of the higher moisture sorption behavior, and the organic nature of the natural fibers (Varadarajulu and Ramadevi 2007). It is necessary to enhance the hydrophobicity of the natural fiber by chemical treatments with suitable coupling agents or by coating with appropriate resin in order to develop composites with better mechanical properties and environmental performance (Khalil *et al* 2007). Hybridization of natural fiber with stronger and more corrosion-resistant synthetic fiber, for example, glass or carbon fiber, can also improve the stiffness, strength, as well as moisture resistance of the composite. Using a hybrid composite that contains two or more types of different fibers, the advantages of one type of fiber could complement what are lacking in the other. As a consequence, a balance in performance and cost could be achieved through proper (Bledzki and Gassan 1999; Noorunnisa Khanam *et al* 2007; Idicula *et al* 2005) material design. However, only a few studies on the mechanical properties of natural and synthetic fiber reinforced polymer matrix hybrid composites are available to date and in most cases durability issues are not addressed (Abu Bakar *et al* 2005; Padma Priya *et al* 2007; Padma Priya *et al* 2005; Venkata Reddy *et al* 2007). Although there have been numerous studies of fatigue and environmental fatigue of composite materials (Jawaid *et al* 2011; Khalil *et al* 2009), only a few references are available on fatigue behavior of natural fiber and their composites. The physical and mechanical properties of SCF and betel nut EFF fibre are shown in Table 1 (Idicula *et al* 2010; Idicula *et al* 2005). (Padma Priya *et al* 2005) studied the tensile, flexural, and chemical resistance properties of waste silk fabric-reinforced epoxy laminates and also studied the impact, compression, density, void content, and weight reduction were studied on waste silk fabric/epoxy composites. (Venkata Reddy *et al* 2007) studied the chemical resistance of kapok/glass and kapok/sisal fabrics reinforced unsaturated polyester hybrid composites and they also studied the compressive, chemical resistance, and thermal properties on kapok/ sisal fabrics polyester composites. Various researcher studied chemical resistance, physical and mechanical properties of natural fibre reinforced polymer composites (John and Venkata Naidu 2007; Sreenivasulu *et al* 2006; Ramakrishna *et al* 2005). (Ashok kumar *et al* 2012a) were studied on characterization of light weight epoxy composites reinforced with short sansevieria cylindrica fibers and he was shown that the removal of the amorphous hemi- cellulose on alkali treatment was played an instrumental in improving properties.

Table 1 Physical and mechanical properties of SCF and EFF fibers.

Fibre	Density (g/cm ³)	Tensile strength (MPa)	Tensile Modulus (MPa)	Elongation at Break (%)
SCF	2.403	586-890	30-37	1.8-2.09
EFF	0.67-1.43	259	1-10	9-20

In the present study, chemical resistance, void content and tensile properties of vinylester based chopped tri layer EFF/SCF/EFF and SCF/EFF/SCF fibre reinforced hybrid composites has been made with reference to the relative weight fraction of two fibers. Relative weight ratios of two fibers (i.e. betel nut EFF/SCF) were kept constant as 4.5:1 and also studied the different layering pattern. Chemical resistance, void content and tensile properties of composites were analysed. The interaction between the reinforcement fibre and the matrix was studied by observing fracture surface of composites by using scanning electron microscope.

2. Materials and methods

2.1 Materials

Betel nut empty fruit (EFF) fibre mat was extracted from betel trees of wet fields at Enumuladoddi at Kalyanadurgam, Anantapur-Dist, Andhra Pradesh, India and fiber was extracted from natural retting process (John and Venkata Naidu 2007). SCF mat was procured from Enumuladoddi, forest, Anantapur, Andhra Pradesh, India. In the present work, a commercially available vinylester, methyl ethyl ketone peroxide (catalyst), Cobalt naphthenate (accelerator) were purchased from the V.G.R. Enterprises, Madurai, Tamilnadu, India. Vinyl-ester monomers with two reactive vinyl end groups enable the cross-linking for network formation. The liquid resin has a density of 1.045 g/cm³ and a viscosity of 350 centipoises (cps) at room temperature. Toluene, benzene, carbon tetra chloride, hydrochloric acid, nitric acid, NaOH, Na₂CO₃ and NH₄OH were also supplied by Aldrich Company.

2.2 Fabrication of composite

The betel nut empty fruit fiber (EFF) and *Sansevieria cylindrica* (SCF) fibers were used for preparing hybrid composites. Duo fibers were chopped in order to get short fibers. A glass mould of dimensions 300 mm X 300 mm X 3 mm was used. The mould cavity was coated with a thin layer of polyvinyl alcohol (PVA) solution, which acts as a releasing agent. The vinylester (VE) composites were made first adding the methyl ketone peroxide (1.2mL) in to the VE resin (100g) and catalyst (2mL). Cobalt naphthenate (0.7mL), accelerator (2mL) (Raghu *et al* 2010) was added with resin. EFF fibre mat and SCF fibre mat were stacked together with the layer of SCF fibre mat sandwiched in between the layer of EFF fibre mat and vice versa in mould. It was then impregnated with vinylester resin in mould. Different layering pattern of fibers were maintained (Padma Priya *et al* 2005). A neat vinylester matrix (unfilled) sample was prepared and vinylester resin with pure EFF and SCF fibre composites were also prepared. The test specimens for chemical resistance void content and tensile properties were cut from the composites according to ASTM standards.

2.3 Characterization of hybrid composites

2.3.1. Chemical resistance properties

The chemical resistance of the VE resin based EFF/SCF/EFF and SCF/EFF/SCF fibre reinforced hybrid composites were studied using ASTM D 543-87 (Noorunnisa Khanam *et al* 2007) method. The effect of some solvents i.e. toluene, benzene, carbon tetra chloride, the effect of some acids i.e. nitric acid, hydrochloric acid

and the effect of some alkalis i.e., NaOH, Na₂CO₃, NH₄OH were studied on the matrix and hybrid composites. In each case five pre weighed samples were dipped in the respective chemical reagents for 24 h. They were then removed and immediately washed in distilled water and dried by pressing them on both sides with a filter paper at room temperature. The samples were then weighed and the percentage weight loss/gain was determined. The percentage of weight loss/gain was determined using the literature (Varadarajulu and Ramadevi 2007).

2.3.2. Void content of composites

Void content of composite was determined by ASTM D 2734-94 (Idicula *et al* 2010; Idicula *et al* 2005). The void content was determined from the theoretical and experimental density of the composites as per the literature (Abu Bakar *et al* 2005).

2.3.3. Tensile properties

In the present work, the tensile strength and modulus of the plain matrix as well as EFF/SCF/EFF and SCF/EFF/SCF fibre reinforced composites were measured by using an INSTRON 3399 Universal testing machine. This test was conducted as per the ASTM D 3039 (Venkata Reddy *et al* 2007) specifications. In each case, five specimens were tested and the average value is tabulated. A scanning electron microscope (JEOL JSM-6400 JAPAN) was used to analyze the morphological images of the hybrid composites. A thin section of the sample was mounted on an aluminum stub using a conductive silver paint and was sputter-coated with gold prior to morphological examination. The SEM micrographs were obtained under conventional secondary electron imaging conditions with an acceleration voltage of 5 kV.

3. Results and discussion

3.1 Chemical Resistance Tests

Chemical resistance tests are used to find the ability of a composite to withstand exposure to acids, alkalis, solvents and other chemicals. The chemical resistance tests of these hybrid composites were performed in order to find out whether these composites can be used for making articles that are resistant to chemicals. The weight loss/gain for the VE, pure EFF, pure jute, EFF/SCF/EFF and SCF/EFF/SCF reinforced hybrid composites with different chemicals were shown in Table 2. Chemical resistance properties of pure EFF and SCF reinforced composites were also presented in the same table. From this table it is clearly seen that in all cases weight gain is observed. This clearly indicates that the composites did not lose weight, and therefore it does not seem as if any erosion occurred. The weight increase of the composites was larger for aqueous solutions, and this was to be expected as a result of the hydrophilicity of the fibre (Bledzki and Gassan 1999). The weight gain (%) was more when the EFF as skin and SCF as core material in the composites, although there are enough exceptions not to make this a general rule. In these cases, betel nut EFF of OH groups in the cellulose probably were more exposed, and this increased the hydrophilicity of the system. The performance of tri layered hybrid composites against almost all chemicals used for chemical testing slightly enhanced when compared to that of pure systems, thus emphasizing the fact hybridization with SCF significantly increase the chemical

resistance of the material. Totally it was observed that matrix and all the composites showed better chemical resistant to all chemicals.

Table 2 Chemical resistance properties of different fibre reinforced composites

Chemicals	VE	Pure SCF	Pure EFF	SCF/EF F/SCF	EFF/SCF/ EFF
Benzene	0.431	7.425	0.425	3.254	3.254
Toluene	0.238	4.125	0.423	3.452	3.124
CCl ₄	1.080	4.521	0.326	3.568	1.833
H ₂ O	1.322	19.420	3.001	9.568	2.412
HCl	0.236	14.895	1.568	9.745	6.895
HNO ₃ (40%)	0.225	12.964	2.103	15.639	12.365
CH ⁺ COOH (5%)	0.364	19.563	3.256	10.245	11.265
NaOH (10%)	0.091	14.002	5.324	18.254	13.256
Na ₂ CO ₃ (20%)	0.213	9.256	0.893	4.698	3.852
NH ₄ OH (10%)	0.312	20.147	4.025	12.365	3.568

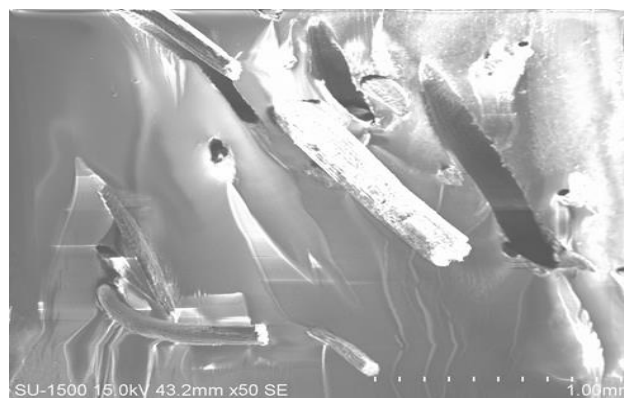


Fig. 1a. SEM micrograph of pure SCF fibre reinforced composites.



Fig. 1b. SEM micrograph of EFF/SCF/EFF hybrid composites.

3.2. Void content

The presence of void content in the composites significantly reduces the mechanical and physical properties of the composites. During impregnation of fibres into the matrix or during manufacturing of fibre reinforced composites, the trapped air or other volatiles exist in the composites (John and Venkatanaidu 2004).

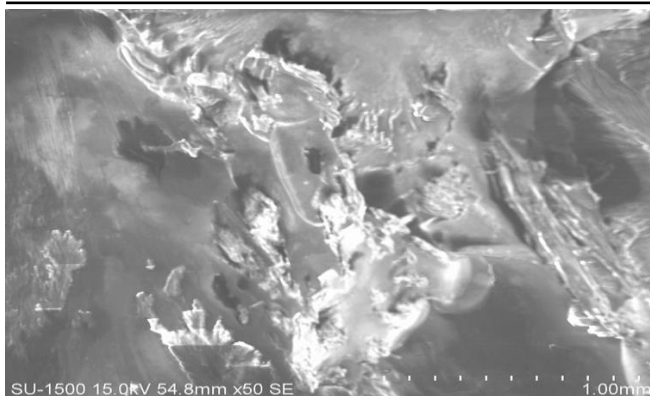


Fig. 1c. SEM micrograph of 3 layered SCF/EFF/SCF hybrid composites.

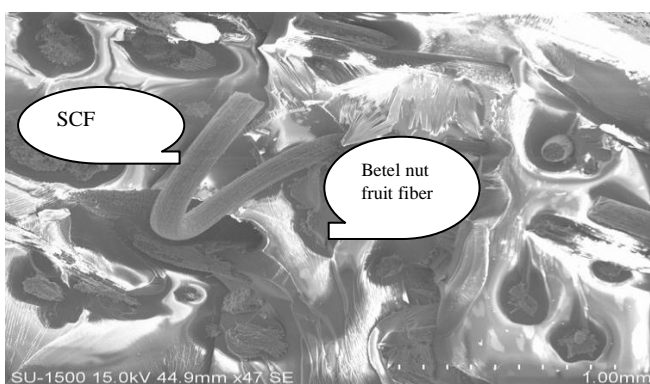


Fig. 1d. SEM Micrograph of SCF reinforced composites.

The most common cause of voids is the incapability of the matrix to displace all the air which is entrained within the woven or chopped fibres as it passes through the matrix impregnation. The void content (%) of composite samples with different layering pattern is presented in Table 3. The betel nut EFF composites exhibited a higher amount of voids (8.6%) compared to the SCF composites (2.6%), it's due to incompatibility between the VE resin and betel nut EFF. According to (Ashok Kumar *et al* 2012b) and (John and Venkata Naidu 2004), incomplete wetting out of the fibres by the matrix would lead to the formation of voids. Betel nut EFF are porous and the mat is loosely packed hence a large amount of resin would be squeezed out of the mat during pressing molding. In the SCF/ EFF/SCF hybrid composites, EFF layer is sandwiched between two SCF skins resulting in a reduced exposure of betel nut fibres compared to EFF/SCF/EFF hybrid composites. SCF mats are tightly packed and also more compatible towards the VE resin. As a result, slightly fewer amounts of voids are present in SCF/ EFF/SCF hybrid composites compared to the EFF/SCF/EFF hybrid composites.

Table 3 Void contents of SCF/EFF fibre reinforced vinylester hybrid composite.

Type of composite	Void content (%)
Pure SCF	6.532
Pure EFF	2.365
SCF/EFF/SCF	4.253
EFF/SCF/EFF	3.456

Tensile properties (Ashok Kumar *et al* 2012a) 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. (Idicula *et al* 2010; Idicula *et al* 2005; Guduri *et al* 2007; Raghu *et al* 2010) were reported that layering pattern of the fibres in hybrid composite effect tensile strength and modulus of the hybrid composites. Various researcher studied synthetic fibre/natural fibre based hybrid composite but natural fibre based hybrid composites are recent phenomena due to environmental concern. Study of tensile properties of sisal/silk, kapok/sisal, and sisal/banana hybrid composites were reported that hybridization of two natural fibres enhanced mechanical performance of hybrid composites compared to individual fibre composites (John and Venkata Naidu 2007; Sreenivasulu *et al* 2006; Ramakrishna *et al* 2005). While using a hybrid composite that contain two or more types of fibre, the advantages of one type of fibre could complement with what are lacking in the other. As a consequence, a balance in cost and performance could be achieved through proper material design (Khalil *et al* 2007).

Table 4 Tensile strength and tensile modulus of SCF/EFF/SCF and EFF/SCF/EFF fibre reinforced hybrid composites

S.No.	Samples	Tensile strength(MPa)	Tensile modulus(MPa)
1	Pure VE	18.63	1.75
2	Pure SCF	48.35	2.42
3	Pure EFF	25.42	2.36
4	SCF/EFF/SCF	35.42	2.45
5	EFF/SCF/EFF	30.42	2.59

The results of the tensile properties such as tensile strength and tensile modulus of the EFF/SCF/EFF and SCF/EFF/SCF fibres reinforced hybrid composites are shown in Table 4. It observed that tensile strength to be higher when SCF was used as skin and betel nut EFF as the core material. The tensile strength will be higher when the high strength material is used as skin, which is the main load bearing component in tensile measurements. In EFF/SCF/EFF, the value of tensile strength is slightly lower because the low strength EFF is used as the skin material. (Idicula *et al* 2005) was done similar study on sisal and banana hybrid composites and reported that sisal/banana/sisal show slightly lower tensile strength compared to banana/sisal/banana hybrid composites because low strength sisal fibre is used as a skin material. It is observed from Table 4, that the tensile strength of pure EFF composite (25.42MPa) is much lower than pure SCF (48.35MPa). This indicated that the high porosity or the presence of voids on the surface of pure EFF composite. With the presence of voids on the surface of composite, which enables air on the surface or inside to be trapped could contribute to the high porosity in the betel nut EFF composites (Jawaid *et al* 2011). The pure SCF composite showed highest value of tensile strength due to good bonding between SCF fibre and VE matrix. The fibre-matrix interface plays an important role in determining the mechanical properties of composite materials.

The scanning electron micrograph of betel nut EFF composite show the poor adhesion between the betel nut EFF fibre and VE matrix, which is evident by the pull out of the betel nut EFF as shown in Fig. 1a, leads to a weak interfacial bond, resulting in an

inefficient stress transfer between the VE matrix and the betel EFF. Due to this behavior the betel nut EFF composites fails at a lower load compared to the SCF fibre reinforced composites. As clear from tensile modulus of the EFF/SCF/EFF trilayer fibre reinforced hybrid composites as shown in Table 4. It is found from the table that the tensile modulus of EFF composites have lower than SCF composites. It is seen that pure SCF composites have higher tensile modulus than other composites. When the EFF were placed at the skin and the SCF were at the core (EFF/SCF/EFF), the hybrid composite exhibited a low tensile modulus compared to the SCF/EFF/SCF hybrid composites and pure SCF reinforced composites. (Idicula *et al* 2005) were studied reported that layering pattern did not affect tensile modulus. In Figs. 1b and 1c show the scanning electron micrograph of tensile fracture surface of EFF/SCF/EFF and SCF/EFF/SCF hybrid composite, respectively. Fibre–matrix debonding, matrix crack and fibre pull out is more evident in hybrid composite (EFF/SCF/EFF), compared to SCF/EFF/SCF hybrid composites. The scanning electron micrograph of EFF/SCF/EFF reveals the fibre pull out from the matrix, indication poor adhesion between the reinforcement and the matrix. The tensile failures of a hybrid composite are mainly dependence on the breaking strain and modulus of the individual reinforcing fibres (Khalil *et al* 2009; Idicula *et al* 2010; Idicula *et al* 2005). The high tensile strength SCF at the outer layer are able to withstand the tensile stress while the betel nut EFF core absorbs the stresses and distributes them evenly in the composites. This is also explained that SCF composite has strong composite showed higher tensile strength and modulus. The increase in the tensile strength and modulus of the hybrid composites is also due to the higher tensile strength of SCF than the EFF fibre. Extensive fibre pullout was observed not only in the pure EFF composite but also in the EFF layer of the hybrid composite (Fig. 1b). The SEM micrograph of SCF composites is shown in Fig. 1d. This micrograph shows that fibre pullout is less than pure EFF composites. It can be clearly seen that apart from some fibre pulled out from the matrix, some places matrix skin formation on the fibres can be observed. Further, at some places it observed that some of the fibres broken instead of a pull out. Hence overall bonding between the SCF and the matrix was found to be good.

4. Conclusion

In this study the chemical resistance of vinylester based trilayer EFF/SCF/EFF and SCF/EFF/SCF fibre reinforced hybrid composites clearly indicates that these composites are strongly resistant to all chemicals. The void content of composites decreases with hybridization of betel nut empty EFF composite with SCF fibers. SCF/ EFF/SCF trilayer composite show less void content compare to pure EFF and EFF/SCF/EFF composites because SCF fibre mats are tightly packed and also more compatible towards the vinylester resin. The high strength SCF fibers at the outer layer are able to withstand the tensile stress while the EFF fibre core absorbs the stresses and distributes them evenly in the composites. This is showed the upper layer of SCF fibre was capable of withstanding stronger loads as compared to EFF fibers. This is also explained that hybrid composite has higher tensile strength and modulus compare to pure EFF composite. Pure EFF fibre reinforced composites showed low tensile strength and

modulus than all other composites. The low tensile properties of the pure EFF fibre composites were enhanced with the addition of SCF fibers. From the SEM micrograph of tensile fracture surface samples, it is concluded that hybrid composite show better adhesion to matrix than pure EFF fibre composite.

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