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Effect of fiber treatments on mechanical properties of Grewia serrulata bast fiber reinforced polyester composites ☆

Mahesha G T[1*], Satish Shenoy B[2], Vijaya Kini M[3], Padmaraja N H[4]

[1, 2,4] *Department of Aeronautical & Automobile Engineering, Manipal Institute of Technology, Manipal University, Manipal,576104-India*

[3]*Department of Mechanical & Manufacturing Engineering, Manipal Institute of Technology, Manipal University,Manipal, 576104-India*

- Corresponding author- E mail: mahesh.gt@manipal.edu
  Contact no:+91-9480572702

Abstract

Natural fiber reinforced polymer composites were prepared using unsaturated polyester resin reinforced with bast fibers extracted from Grewia Serrulata trees. The extracted fibers were subjected to sodium hydroxide pretreatment followed by exclusive chemical treatments such as acetylation, permanganate treatment and Silane coupling treatment to modify the fiber surface. Hand lay-up technique was used for the fabrication of uni-directionally oriented untreated and treated fiber reinforced laminates. Mechanical characterization was carried out as per the ASTM- 3039 and ASTM D 790 standards for the tensile and flexural properties respectively. It was found that the tensile modulus of Silane treated Grewia Serrulata fiber reinforced composites was 27% higher and tensile strength of acetylated fiber reinforced composites was 53% higher than untreated fiber reinforced composites. The flexural modulus and strength of treated fiber reinforced composites were considerably higher than the untreated fiber reinforced composites. The fractographs of the samples revealed the improvement in bonding between the chemically treated fibers and the matrix.

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1. Introduction

Traditional materials such as metals and alloys are being increasingly replaced with bio-degradable and ecofriendly materials like natural fiber reinforced composites. This will facilitate easy disposal after their service life which eases the problems associated with land fillings. Alternative materials such as composites that have both environmental as well as economic benefits are being developed for applications in the automotive, building, furniture, and packing industries [1]. Natural fibers are economical, easy to work with as well as biodegradable which makes it a better choice than its synthetic counterpart. The morphology of natural cellulose fibers can be easily modified than the inert carbon, aramid and glass fibers [2, 3], which improves the compatibility the fiber and the matrix.

However, a major drawback of natural fiber-polymer composites is the lack of compatibility between the hydrophilic natural fibers and the hydrophobic polymeric matrices. This degrades the properties of the composites prepared using such natural fibers. It is therefore necessary to modify the fiber surface/structure by employing physical/chemical modifications to improve the adhesion between fiber and matrix [4]. Some of the methods adapted by the researchers for fiber surface modification are discussed below.

1.1 Mercerization

This is an alkali treatment in which the raw fibers are soaked in an aqueous solution of Sodium Hydroxide for a stipulated period of time. This results in higher degree of access to resin systems. Alkali sensitive hydrogen bonds existing across the fibers break down during the process and new reactive hydrogen bonds form between cellulose molecular chains. Due to this, hydrophilic hydroxyl groups are partially removed and moisture resistance property is improved [5, 6]. Typical reaction taking place during the alkali treatment of natural fibers is

Fiber-OH+NaOH----------→ Fiber-O-Na+ H_20

1.2 Acetylation

Acetylation is the principle of replacing reactive hydroxyl groups on cellulose fibers coupling agents may reduce the number of cellulose hydroxyl groups in the fiber matrix interface which leads to enhancement with acetyl groups. This results in hydrophobic fibers and hence having greater resistance to biodegradation. The obtained hydrophobic nature of the fibers is more compatible with non-polar polymers used as matrix [4]. The reaction taking place during the treatment can be expressed as

Cellulose–OH + CH_3 – CO – O – CO – CH_3→ Cellulose– O – CO – CH_3 + CH_3COOH

1.3 Silane treatment

Silanes are used as coupling agents to let natural fibers adhere to the polymer matrix, thereby stabilizing the composite material. Natural fibers exhibit micropores on their surfaces and silane coupling agent act as a surface coating which penetrates into the pores and develop mechanically interlocked coating on their surfaces [7]. Silane coupling agents may reduce the number of cellulose hydroxyl groups in the fiber matrix interface which leads to enhancement in the mechanical properties of the composites.

2. Materials and methodology

In the present work, fibers extracted from the Grewia Serrulata tree, belonging to Tiliaceae family is taken as the source plant. Grewia Serrulata plants are about 15-20 feet tall, ever green shrubs, commonly found in the tropical western ghats of Indian peninsular region.

The fibers from the well grown branches were used to ensure the consistency. The fibers were extracted from the stem branches manually after retting in water for three days. The following treatments were carried out to modify the surface morphology of the fibers.

2.1 Alkali pre- treatment

The fibers were subjected to 5% NaOH pre-treatment for 2 hours. It was followed by flushing with distilled water several times to ensure that surface impurities were cleansed. The pH measurements were carried out to ensure that no residues of NaOH were present in the dried fiber samples.

2.2 Acetylation

The alkali pretreated fibers were soaked in demineralized water for an hour, filtered and placed in a round bottom flask, containing acetylating solution. Acetylating solution was prepared using 250 ml toluene, 125 ml acetic anhydride [5]. The acetylation process was conducted for a duration of 1.5 hours at a temperature of 60°C.

After the modification, the fibers were washed periodically with distilled water and pH value was evaluated. The acidic residues were removed by treating with 5% sodium Bicarbonate solution until the fibers were acid free. The modified fibers were dried using hair dryer for about 15 minutes.

2.3 Silane treatment

Silane coupling agents were used to improve the compatibility at the fiber- matrix interface. 3-methacryloxy-propyl-trimethoxy-silane (Sigma Aldrich) was used to modify the fiber surface [6] in the current work. Grewia Serrulata fibers were soaked in a solution of acetone/water (50/50 by volume) with a 1% Silane concentration for 2 hours. After the Silane treatment, the fibers were dried using hair dryer for about 15 minutes.

2.4 Potassium permanganate treatment

Alkali Pretreated Fibers were washed with 2% detergent solution and then soaked in 0.2% potassium permanganate ($KMnO_4$) solution for 1 hour. Fibers were rinsed in distilled water and dried after the permanganate treatment.

2.5 The Resin

Unsaturated polyester resin (Ash Polymers, Bangalore, density 1.089 gm/cc) was employed for the composite

preparation. Matrix material was prepared by blending polyester with 2% by volume of cobalt solution as accelerator and 2% by volume of MEKP (Methyl Ethyl Ketone Peroxide) as catalyst.

2.6 Composite preparation

Hand lay-up technique was used to prepare unidirectional fiber reinforced laminates. Composites were prepared with 15% by weight fraction of the fiber reinforcement. The unidirectional fiber mats were prepared by tying them on to rectangular metal frames. Such woven mats were wrapped with adhesive tapes on both sides all around the frame and were separated from the frame by carefully cutting with a scissor. Photographs of the hand layup technique employed for the composite preparation and the prepared laminate are shown below in figure 1a and 1b respectively.

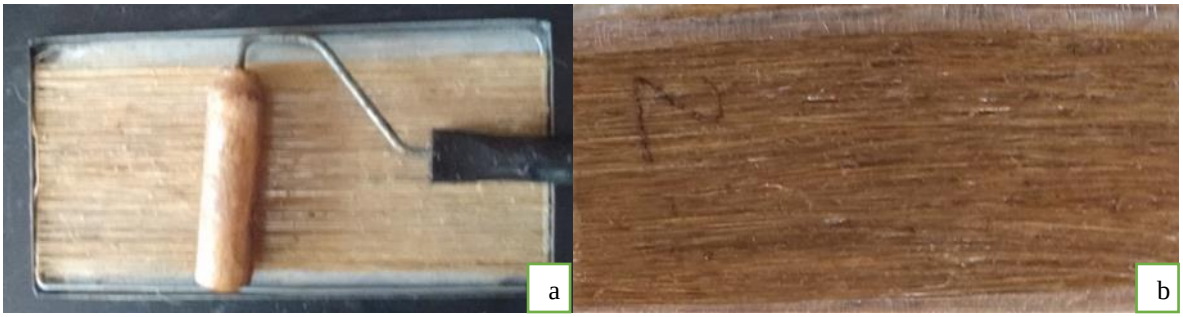

Fig. 1. (a). Hand Layup technique; (b) Prepared laminate

3. Experimental characterization

The prepared composite specimens were subjected to Mechanical characterization based on ASTM standards.

3.1 Tensile test

Tensile test samples were prepared according to ASTM D3039 Standards. The tests were carried out on a Biss universal testing machine with a strain rate of 1mm/min. Rectangular specimens of dimensions 200mm x 12.5mm x 4mm were used for the test with a gauge length of 140 mm. The tensile strength and elastic modulus were calculated by analysing the load versus extension graphs. Five specimens of each type were tested and average values of the results are used for analysis.

3.2 Flexural test

The flexural modulus of a material indicates the stiffness as well as the extent of deformation of a material when it is subjected to bending stress. It gives a measure of the ductility of the material. For determining bending strength and flexural modulus, tests were performed on an INSTRON 3366 universal testing machine. Flexural test samples were prepared and test protocols were followed according to ASTM D790-03 standard. The load applied and corresponding deflection were obtained by the computerized data acquisition system. The flexural strength and modulus were calculated as shown below:

$$FS = \frac{3P\max L}{2bd^2} \quad \text{------(1)} \qquad ; \qquad E_f = \frac{L^3}{4bd^3}\left(\frac{dp}{d\delta}\right) \quad \text{-------(2)}$$

Where P_{max}- maximum load, *L*- span between supports(77 mm), *b*-width of specimen(12.7mm), *d*- depth of specimen(4mm) , $dp/d\delta$ is obtained from the elastic region of load-deflection plot.

4. **Results and discussions**

The following tests were conducted to characterize the samples. Five specimens of each class were tested following the respective ASTM standards. The results obtained are discussed below to analyse the performance of the samples.

4.1 Tensile test

The elastic modulus and tensile strength of 15% (weight) Grewia serrulata, unidirectionally oriented long fiber reinforced composites is shown in table1.

The specimens tested are designated as shown below.

UT GFRP -Untreated Grewia Serrulata Fiber reinforced Polyester ; ATGFRP -Acetylated Grewia Serrulata Fiber reinforced Polyester; PTGFRP -Permanganate treated Grewia Serrulata Fiber reinforced Polyester; STGFRP – Silane treated Grewia Serrulata Fiber reinforced Polyester.

The mean values of the results obtained from the tension test are reported in the table below.

Table 1. Tensile and flexural test results.

Specimen	Tensile strength(MPa)	Young's Modulus(MPa)	Flexural strength(MPa)	Flexural modulus(MPa)
Neat resin	31.27	1072	34.1	440
15%UTGFRP	32.98	2871	57.62	2250
15%ATGFRP	49.18	3111	50.18	2555
15%PTGFRP	36	2488	71.89	3152
15%STGFRP	36.4	3657	99.7	3489

The silane treated fiber reinforced composites have higher value of Young's modulus of about 3657 MPa which is 30% higher than 15% untreated fiber reinforced composites (2871MPa).The acetylated fiber reinforced composites shows an improvement of 8.3% in modulus and an increase of 49% in tensile strength when compared to the untreated fiber reinforced composites. Improved tensile strength for acetylated fiber-based composites could be because of better interfacial bonding between acetylated fiber which has been rendered hydrophobic[6] and the polymer matrix which is by nature hydrophobic.

Improved performance of silane treated composites is attributed to the better interaction between the treated fibers and the matrix as a result of silane initiated crosslinking/grafting. This restricts the chain movement during deformation which results in stiffening of the composites [7-8].

4.2 Micrograph analysis

The micragraph studies of the fractured samples were carried out to analyse the type of failures. The study was done

using ZEISS EVO 50 analytical scanning electron microscope. The fractured surface images of untreated fiber reinforced and Silane treated fiber reinforced composites are shown in figure 2a and 2b respectively.

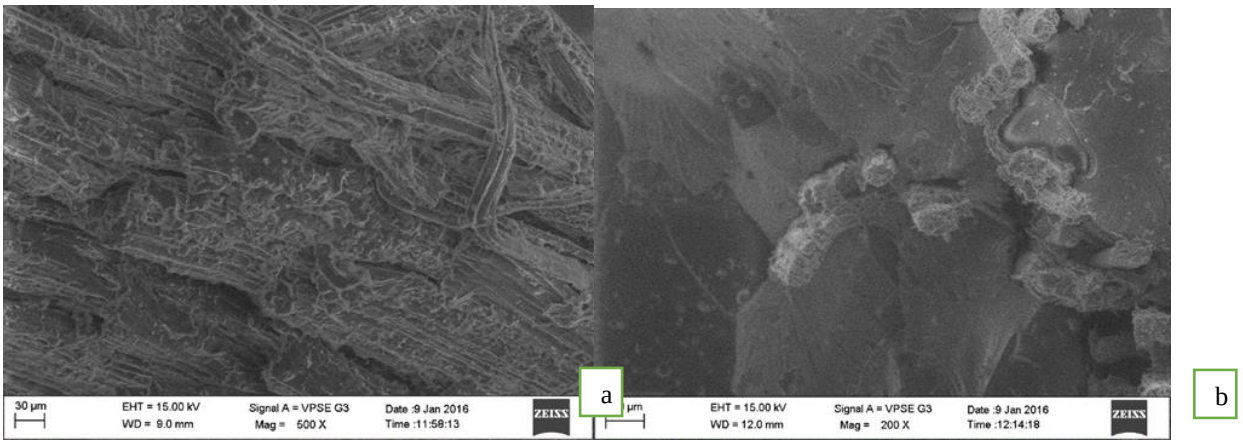

Fig.2.(a) SEM images of untreated; (b) Silane treated fiber reinforced composite fractured surfaces.

In the fractured samples, the composite with untreated fibers showed poor interfacial bonding between the fiber and the matrix, which resulted in a relatively clean surface of the pulled out fibers due to a greater extent of delamination. It can be seen that the untreated fibers were detached from the matrix surface, due to the poor interfacial bonding. Such observation illustrates that there is a poor interaction between fibers and matrix. This could be due to the waxy substances present on the surface of the untreated fibers that results in ineffective fiber-matrix bonding and poor wetting.

In the Silane treated fiber reinforced composite, the SEM micrograph shows that the coupling agent facilitates the direct contact between the cellulosic fibers and the polyester matrix. This indicates the existence of strong bonding between the reinforcement fabrics and matrix. Thus, treatment and usage of predominantly polar coupling agent enhanced the interfacial bonding between Grewia serrulata fibers and polyester in the composites and hence contributed for improved mechanical properties.

4.3 Flexural test

The flexural modulus and flexural strength of the samples were found out using the data obtained from the three point bending tests. It was found that all the three treatments lead to improved flexural properties. The Silane treated fibers enhanced the flexural modulus of the composite specimens by 55%, while the Permanganate treated ones enhanced the modulus by 40%. A maximum flexural strength of 99.7MPa was obtained with Silane treated fiber reinforced composites. The flexural strength of permanganate treated fiber reinforced composites was 24% higher than the untreated composites. Both permanganate and Silane treatments resulted in improved graft co-polymerization where strong cross linked network structure was formed between the fibers and the matrix. The

resulting increase in interlaminar shear strength improves the load bearing capability of the composites.

Conclusions

The following conclusions are drawn from the current research work.

1. The adhesion between the Grewia serrulata fibers and polyester resin can be improved by Silane coupling treatment, permanganate treatment and acetylation of fibers.
2. The tensile modulus of alkali pre-treated and Silane treated fiber reinforced polyester composites is 27% higher and acetylated fiber reinforced composites is higher by 8% than the untreated fiber reinforced composites.
3. The tensile strength of acetylated fiber reinforced composites is 53% higher and Silane treated fiber reinforced composites is 10% higher than untreated fiber reinforced composites.
4. The flexural strength of alkali pretreated and Silane treated fiber reinforced composites is 73% higher and permanganate treated fiber reinforced composites is 24% higher than untreated fiber reinforced composites.
5. The flexural modulus of Silane treated fiber reinforced composites is 55% higher and permanganate treated composites is 40% higher than untreated fiber reinforced composites.

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References

[1]A.S.Singha,B.S.Kaith,A.J.Khanna,,Bio Resources. 6(2)(2010) 2101-2117.

[2]R.Akter, R.Sultana, M. Z. Alam,M.R. Qadir, M.H..A.Begum , M.A. Gafur, Int j Engg & Tech. 13(02)(2013) 122-128.

[3]A. C. Milanese, M.O.H.Cioffi , H.J.C.Voorwald, Procd Engg.10(2011) 2022–2027.

[4] A. Valadez-Gonzaleza, J.M. Cervantes-Uca, R. Olayob , P.J. Herrera-Franco, Composites: Part B.30(1999) 309–320.

[5] A. K. Bledzki, S. Reihmane , J. Gassan**,** J. App Poly sci.59(8)(1996)1329-1336.

[6]J. Jayaramudu, G. Siva Mohan Reddy, K. Varaprasad, E. R. Sadiku, S. S. Ray , A. Varada Rajulu, Fibs. and Polymers.15(7)(2014)1462-1468.

[7]Ana Espert**,** M S thesis. Royal Institute of Technology,Stockholm, Sweden.(2003)

[8] P. Threepopnatkul, N. Kaerkitcha ,N. Athipongarporn, Composites: Part B.40(2009) 628–632.

[9] A.V. Ratna Prasad , K. Mohana Rao, Matls and Desi. 32(8)(2011) 4658-4663.

[10] T.H.Mokhothu, , M S thesis, University of the free state.(2010).

[11]A.Gopinatha, M.B.Senthil Kumar , A.Elayaperumal , Procd Engg. 97(2014) 2052 – 2063.

[12]I.O. Oladele, j of the Asson of Proff Engrs of Trinidad and Tobago.42(1)(2014) 12-15.