

Preparation and characterization of polymer matrix composite using natural fiber lantana-camara

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ABSTRACT: Environmental awareness today motivates the researchers, worldwide on the studies of natural fiber reinforced polymer composite and cost effective option to synthetic fiber reinforced composites. The availability of natural fibers and ease of manufacturing have tempted researchers to try locally available inexpensive fibers and to study their feasibility of reinforcement purposes and to what extent they satisfy the required specifications of good reinforced polymer composite for different applications. With low cost and high specific mechanical properties, natural fiber represents a good renewable and biodegradable alternative to the most common synthetic reinforcement, i.e. glass fiber. Despite the interest and environmental appeal of natural fibers, their use is limited to non-bearing applications, due to their lower strength compared with synthetic fiber reinforced polymer composite. The stiffness and strength shortcomings of biocomposites can be overcome by structural configurations and better arrangement in a sense of placing the fibers in specific locations for highest strength performance. Accordingly extensive studies on preparation and properties of polymer matrix composite (PMC) replacing the synthetic fiber with natural fiber like Jute, Sisal, Pineapple, Bamboo, Kenai and Biogases were carried out. These plant fibers have many advantages over glass fiber or carbon fiber like renewable, environmental friendly, low cost, lightweight and high specific mechanical performance

I. INTRODUCTION

A typical composite material is a system of materials composing of two or more materials (mixed and bonded) on a macroscopic scale. As defined by Jartiz, [1] Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form. Kelly [2] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength or resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

1.1 Characteristics of the Composites

Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the 'reinforcement' or 'reinforcing material', whereas the continuous phase is termed as the 'matrix'. Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties.

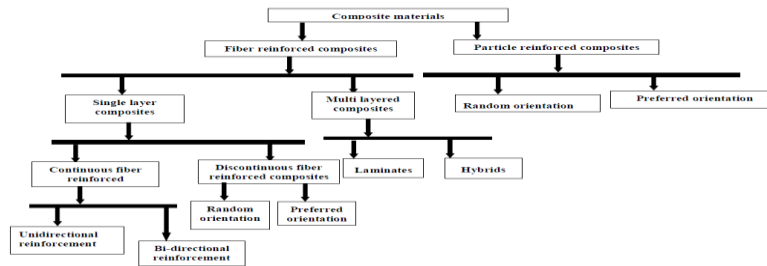
1.2 Classification of Composites

Composite materials can be classified in different ways [3]. Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high performance of the composites. A typical classification is presented in . The two

broad classes of composites are:

Fibrous composites

Particulate composites



1.3 Components of a composite material

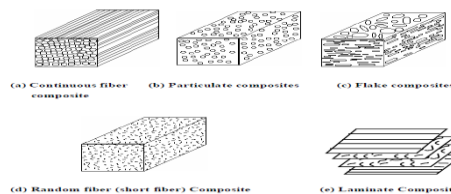
- 1. Role of matrix in a composite
- 2. Materials used as matrices in composites
- 3. Reinforcement
- 4. Interface
- 5. Interface

3. Bulk-Phases

- (a) Metal Matrices
- (b) Polymer Matrices
- (c) Ceramic Matrices

1.4 Types of composite materials

- (a) Fiber-reinforced composites
- (b) Continuous or long fiber composite
- (c) Discontinuous or short fiber composite
- (d) Laminate Composites



1.5 Natural fibers: source and classification

Growing environmental awareness has triggered the researchers world wide to develop and utilize materials that are compatible with the environment. In the process natural fibers have become suitable alternatives to traditional synthetic or manmade fibers and have the potential to be used in cheaper, more sustainable and more environmentally friendly composite materials. Natural organic fibers can be derived from either animal or plant sources.

1.6 Structure of plant fiber

Natural plant fibers are constituents of cellulose fibers, consisting of helically wound cellulose micro fibrils, bound together by an amorphous lignin matrix. Lignin keeps the water in fibers; acts as a protection against biological attack and as a stiffener to give stem its resistance against gravity forces and wind. Hemicellulose found in the natural fibers is believed to be a compatibilizer between cellulose and lignin. The cell wall in a fiber

is not a homogenous membrane [4]. Each fiber has a complex, layered structure consisting of a thin primary wall which is the first layer deposited during cell growth encircling a secondary wall. The secondary wall is made up of three layers and the thick middle layer determines the mechanical properties of the fiber. The middle layer consists of a series of helically wound cellular micro-fibrils formed from long chain cellulose molecules. The angle between the fiber axis and the micro-fibrils is called the microfibrillar angle.

1.7 Chemical composition of natural fibers

The constituent of any natural fiber vary with origin, area of production, variety and maturation of plant. The major constituent of a fully developed natural fiber cell walls are cellulose, hemicellulose, lignin and pectin. These hydroxyl-containing polymers are distributed throughout the fiber wall [5].

1.8 MATRIX MATERIAL

- 1. Thermosets

2. Bio-derived Thermoplastic Matrices

1.9 Mechanical characterization of lantana-camara fiber epoxy composite

In general natural fibers are hygroscopic in nature and they absorb or release moisture depending on environmental conditions. Amorphous cellulose and hemicelluloses that are present in the natural fiber are mostly responsible for the high moisture absorption, since they contain numerous easily accessible hydroxyl groups which give a high level of hydrophilic character to fiber. The high moisture absorption of the fiber occurs due to hydrogen bonding of water molecules to the hydroxyl groups within the fiber cell wall. This leads to a moisture build-up in the fiber cell wall (fiber swelling) and also in the fiber-matrix interface. This in turn becomes responsible for changes in the dimensions of cellulose-based composites, particularly in the thickness and the linear expansion due to reversible and irreversible swelling of the composites [6]. Another problem associated with fiber swelling is a reduction in the adhesion between the fiber and the matrix, leading to deterioration in the mechanical properties of the composite [7]. A good fiber-matrix bonding can decrease the rate and amount of moisture absorbed by the composite as well as improving the mechanical properties [8]. However in order to overcome this problem, chemical treatment has been considered as a good technique to reduce the hydroxyl group in the fibers. Different chemical treatments such as mercerization or alkali treatment, isocyanate treatment, acrylation, benzoilation, permanganate treatment, acetone treatment, acetylation, silane treatment etc. are reported by several researchers [9, 10-12].

1.10 Chemical modification of fiber

Processing of plastic composites using natural fibers as reinforcement has increased dramatically in recent years. A better understanding of the chemical composition and surface adhesive bonding of natural fiber is necessary for developing natural fiber reinforced composites. The interfacial bonding between the reinforcing fibers and the resin matrix is an important element for improving the mechanical properties of the composites. Realizing this, several authors [13-15] have focused their studies on the treatment of fibers to improve the bonding with resin matrix. The mechanical properties of the composites are controlled by the properties and quantities of the individual component and by the character of the interfacial region between matrix and reinforcement. Lack of good interfacial adhesion makes the use of cellular fiber composites less attractive. Often the interfacial properties between the fiber and polymer matrix is low, because of hydrophilic nature of natural fiber which reduces its potential of being used as reinforcing agents. Hence chemical modifications are considered to optimize the interface of fibers. Chemicals may activate hydroxyl groups or introduce new moieties that can effectively interlock with the matrix. There are various chemical treatments available for the fiber surface modification. Chemical treatment including alkali, silane, acetylation, benzoilation, acrylation, isocyanates, maleated coupling agents, permanganate treatment are discussed in details in [96]. The chemical treatment of fiber aimed at improving the adhesion between the fiber surface and the polymer matrix by modifying the fiber surface and the fiber strength. It also reduces the water absorption capacity of the fiber and helps in improving the mechanical properties.

1. Alkaline Treatment 2. Acetone Treatment 3. Benzoilation Treatment 4. FTIR Spectroscopy 5. X-ray Diffraction

1.11 Single fiber pull-out test

To find out the critical fiber length of Lantana-Camara to be used for preparation of composite, single fiber pull-out test was carried out. shows the schematic diagram of the sample to be used for test. Single fibers were taken and partially embedded in the mixture of epoxy resin and hardener (ratio 10:1) inside a per-pex sheet mould to prepare the samples. The embedded lengths and diameter of the fiber were measured by electron microscope. The embedded lengths were found to be 1.25 to 15.14mm with fiber free length of 30 mm. After curing, the specimens were taken out from the mould. Pull-out test was then conducted on an Instron-4204 tensile testing machine at crosshead speed of 1 mm/min and using 5KN-load cell. Five specimens were prepared for each embedded length and average value was taken

Composite fabrication

Lantana-Camara fiber

Preparation of Lantana-Camara Fiber

Fresh Lantana-Camara stems were collected locally. They were cut to sizes between two nodes. The upper skin was removed by scrapping without damaging the fiber surface. Then they were cut to sizes of 100mm lengthwise. Long fibers were washed with pressurized water to remove unwanted organic materials present on the surface. These fibers were then spread over a water proof sheet and stored in an enclosed shed to reduce the

moisture content. After two weeks the long fibers were cut to lengths of 10mm (optimum fiber length found from single fiber pull-out test) and of width 1mm with a pair of scissor. Due to low moisture content of the fibers, no fungus grew during storage. The Lantana-Camara fibers after cutting were again washed with

pressurized water to remove the fine particle and other organic material that grew and adhered to the surface of fiber during storage and cutting. The fibers were then dried with compressed air at a pressure of approximately 145 kPa at 108°C. The required drying time was determined by weighing a trial sample every 10 min. until the measured mass became constant. A drying time of 40 min. was established to provide subsequent drying of the fiber.

1 Epoxy Resin

The type of epoxy resin used in the present investigation is Araldite LY-556 which chemically belongs to epoxide family. Its common name is Bisphenol-A-Diglycidyl-Ether. The hardener with IUPAC name NNO-bis (2-aminoethylethane-1,2-diamin) has been used with the epoxy designated as HY 951. Both the epoxy and hardener were supplied by Ciba-Geigy of India Ltd.

2 Composite preparation

A Per-pex sheet mold of dimension 130×100×6 mm was used for casting the composite sheet. The first group of samples were manufactured with 10, 20, 30 and 40% volume fraction by weight of fiber. The usual hand lay-up technique was used for preparation of the samples. For different volume fraction of fibers, a calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with gentle stirring to minimize air entrapment. For quick and easy removal of composite sheets, a mould release sheet was put over glass plate and mould release agent was applied at the inner surface of the mould. After keeping the mould on glass sheet a thin layer (≈2mm thickness) of mixture was poured. The required amount of fiber was then distributed on the mixture. The remaining mixture was then poured into the mould. Care was taken to avoid formation of air bubbles. Pressure was then applied from the top and the mould was allowed to cure at room temperature for 72 hrs. During application of pressure a small amount of mixture of epoxy and hardener was squeezed out. Care has been taken to consider this loss during manufacturing of composite sheets. After 72 hrs the samples were taken out from the mould, cut in to different sizes and kept in an air tight container for further experimentation.

STUDY OF ENVIRONMENTAL EFFECT

Moisture absorption test

$$\%M_t = \frac{(W_t - W_0) \times 100}{W_0}$$

where 'W₀' and 'W_t' denote the oven-dry weight and weight after time 't', respectively. Equilibrium Moisture Content (EMC) of the sample is the moisture content 45 when the periodic weight change of the sample was less than 0.1% and thus the equilibrium state was assumed to be reached. The thickness swelling (TS) was determined by using the following equation: where 'H_t' and 'H₀' are

the composite thickness after and before the water immersion respectively.

$$TS(t) = \frac{H_t - H_0}{H_0} \times 100$$

III. Figures and Tables

Table-3.1 crystallinity index of untreated and treated lantana-Camara fiber

Embedded Fiber Length (mm)	Pullout Load (Kg)
1.25	1.54
1.78	2.05
2.41	3.28
2.84	3.69
3.58	4.56
4.02	3.58
4.51	4.26
5.8	4.97
6.45	6.92
7.28	7.74
8.75	8.55
9.22	8.68
10.42	8.83*
11.25	8.32*
12.05	8.98*
13.34	8.37*
14.05	9.08*
15.14	9.24*

Note: * * did not pullout / ruptured

Table- 3.2 pullout testing results

Types of Fiber	I _{nm} (2θ=15°)	I ₀₀₂ (2θ=23°)	Crystallinity Index
Untreated (UT)	715	5108	86.00
Acetone treated	707	5910	88.03
Alkali treated	782	6372	87.73
Benzoyl-chloride treated	798	6768	88.21

Table-3.1 crystallinity index of untreated and treated lantana-

Fiber Content (wt%)	Tensile strength (Mpa)	Yong's Modulus (Mpa)	Elongation of Break (%)	Flexural strength (MPa)	Flexural Modulus (MPa)	Impact strength (KJ/m2)	Vickers Hardness (HV)
0%	18.031	521	3.4	45.519	632	25.78	17.894
10%	17.680	821	4.83	39.346	1284	31.73	17.375
20%	18.020	965	4.46	48.082	1356	36.36	18.145
30%	19.080	1132	5.22	55.491	1425	34.69	19.455
40%	18.440	1172	4.12	46.597	1373	30.38	17.315

Table-3.3 mechanical properties of untreated lantana-camar

camara fiber

Fiber Content (%)	Type of fiber	Tensile strength (Mpa)	Yong's Modulus (Mpa)	Elongation of Break (%)	Flexural strength (Mpa)	Flexural Modulus (Mpa)	Impact strength (KJ/m2)
30	Untreated	19.080	1132	5.22	55.491	1425	34.69
30	Acetone treated	20.078	1435	4.98	58.351	1489	36.24
30	Alkali treated	23.451	1542	5.29	69.527	1658	42.36
30	Benzoylated	25.621	1631	5.36	72.047	1785	45.42

table-3.4 mechanical properties of treated lantana- camara fiber epoxy composite

II. CONCLUSION

Based on experimental results, this study has led to the following conclusions:

1. The Lantana-Camara fiber can successfully be used as reinforcing agent to fabricate composite by suitably bonding with epoxy resin.
2. The effective fiber length for fabrication of Lantana-Camara epoxy composite as found out from the single fiber pull-out test is approximately 9.11 mm or longer.
3. On increasing the fiber content the strength, modulus and work of fracture increases and the best combination is found with 30 vol% of fiber.
4. The fiber surface modification by chemical treatments significantly improves the fiber matrix adhesion, which in turn improves the mechanical properties of composite. Benzoyl-chloride treatment shows the highest improvement in comparison to alkali and acetone treatment. These results are confirmed through SEM, FTIR and XRD analysis.
5. The moisture uptake and thickness swelling values increases with increase in fiber loading. Both values are found to be higher in steam environment than in saline water and sub-zero temperature environments. However these values are considerably reduced with chemical treatments of the fiber.
6. Under all environment conditions, the moisture diffusion process of both treated and untreated Lantana-Camara fiber composites are found to follow the Fick's law.
7. Fiber breakages are found to be the predominant mode of failure as ascertained from the morphology of the treated fiber composites.

REFERENCES

Books:

- [1] Jartiz, A.E., 1965, "Design," pp. 18.
- [2] Kelly, A., 1967, *Sci. American*, 217, (B), pp. 161.
- [3] Agarwal, B.D. and Broutman, L.J., 1980, "Analysis and performance of fiber composites," John Wiley & Sons, New York, pp.3-12.
- [4] Rong, M.Z., Zhang, M.Q., Liu, Y., Yang, G.C. and Zeng, H.M., 2001, "The effect of fiber treatment on the mechanical properties of unidirectional sisalreinforced epoxy composites," *Compos. Sci. Technol.*, 61; pp. 1437-1447.
- [5] Haigler, C. H., 1985, "The Functions and Biogenesis of Native Cellulose," *Cellulose Chemistry and Its Applications*. T. P. Nevell and S. H. Zeronian. West Sussex, Ellis Horwood Limited: pp.30-83
- [6] Rowell, R.M., 1997, "Chemical modification of agro-resources for property enhancement", *Paper and Composites from Agro-based Resources*, CRC Press. p. 351-375.
- [7] Espert, A., Vilaplana, F. and Karlsson, S., 2004, "Comparison of water absorption in natural cellulosic fibres from wood and one-year crops in polypropylene composites and its influence on their mechanical properties", *Compos Part A* ; 35: pp.1267-76.
- [8] Sanadi, A.R., Caulfield, D.F. and Jacobson, R.E., 1997, "Agro-Fibre Thermoplastic Composites", *Paper and composites from agro-based resources*, Boca Raton: CRC Press: Lewis Publishers, Chapter 12, pp. 377-401.
- [9] Valadez-Gonzales, A., Cetvantes-Uc, J.M., Olayo, R. and Herrera Franco, P.J., 1999, "Effect of fibre surface treatment on the fibre-matrix bond strength of natural fibre reinforced composites", *Composites, Part B* 30 (3): pp.309-320.
- [10] Maya Jacob John, Anandjiwala Rajesh D., 2008, "Recent Developments in Chemical Modification and Characterization of Natural Fiber-Reinforced Composites", *Polymer composites*, pp.187-207.
- [11] Joseph, K., Thomas, S. and Pavithran, C., 1996, "Effect of chemical treatment on the tensile properties of short sisal fibre-reinforced polyethylene composites", *Polymer* 37: pp. 5139-49.
- [12] Joseph, P.V., Joseph, K. and Thomas, S., 1999, "Effect of processing variables on the mechanical properties of sisal-fiber-reinforced polypropylene composites", *Compos Sci Technol.*, 59: pp.1625-40.
- [13] Gassan J, Bledzki AK, 1999, "Alkali treatment of jute fibers: relationship between structure and mechanical properties." *Journal of Applied Polymer Science*, (71): p. 623-629.

[14] Zadorecki P., Flodin P., 1985, "Surface modification of cellulose fibers. I. Spectroscopic characterization of surface-modified cellulose fibers and their copolymerization with styrene." *J Appl Polym Sci*, (30): pp. 2419–2429.

[15] Zadorecki, P. and Flodin, P., 1985, "Surface modification of cellulose fibres. II. The effect of cellulose fibre treatment on the performance of cellulose– polyester composites." *J Appl Polym Sci*, (3): p. 3971–3983.