STUDY OF WATER ABSORPTION BEHAVIOUR OF NATURAL FIBRE REINFORCED COMPOSITES

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Abstract: Environmental perception today encourages empiricism worldwide on the learning of plant or natural fibre reinforced polymer composite and cost efficient alternative to synthetic fibre reinforced composites. The accessibility to natural fibers and simplicity in manufacturing have persuaded researchers to aim for locally existing low cost fibers and to investigate their possibility of reinforcement intensions and up to what extent they can satisfy the essential detailing of superior reinforced polymer composite intended for different application program. Natural fibre represents a superior biodegradable and renewable alternative to the most popular synthetic reinforcement, i.e. glass fibre possessing high mechanical properties and low cost. Regardless the curiosity and environmental request of natural fibers, there usage is restricted to non-bearing uses, because of its lower strength than that of synthetic fibre reinforced polymer composite. The stiffness and strength limitations of bio composites can be chased by operational arrangement by placing the fibers at particular locations to have higher strength performance. Research regarding preparation and properties of polymer matrix composite (PMC) replacing the synthetic fibre with natural fibre like Jute, Sisal, Jute, Bamboo, Pineapple, Bagasse and Kenaf were carried out. Renewable, environmental friendly, low cost, lightweight and high specific mechanical performances are the advantages of these plant fibres over the glass fibre or carbon fibre. Composites are exciting materials which are finding increasing application in transportation, aerospace, defence, communication, sporting, electronics and number of other commercial and consumer products. Composite materials have become one of the fastest growing research and development areas of Material Science because of their high potential. In current years there is swift growth in the arena of fibers, matrix, materials, processing, boundary structure, bonding and their characteristics on the final properties of composites. The technological developments in composite materials help in meeting the global industrial demand for materials with improved performance capabilities.

Keeping this in view the present work has been undertaken to develop a polymer matrix composite (epoxy resin) using Luffa Cylindrica fibre and to study its moisture absorption behavior and mechanical properties. The composite are prepared with different volume fraction (number of layers) of Luffa Cylindrica fibre.

Keywords: Natural fibre, plant fibres, plant fibres, Luffa Cylindrica fibre, reinforced polymer composite.

1. INTRODUCTION

It is a platitude that technological advances depends on fosters in the sector of materials. If sufficient materials to bear the service loads and conditions are not available then one does not have to be a skilful to realize the most advanced turbine or air-craft pattern. Whatever the field may be, the ultimate restriction on progression is to be governed by materials. Composite materials in this regard signify a big step in the constant accumulation of optimization in materials. Composites are mixture of two or more materials such as reinforced plastics, metals, or ceramics. The reinforcements may be in the form of fibers, particles, whiskers or lamellae and are embedded in a suitable matrix, thereby providing a material that contains the most useful properties of the constituents. High structural strength, glass fibre reinforced plastics were developed in the early 1940’s and the application of reinforced plastics composites, the glass fibre provides strength and stiffness while the plastic matrix provides the temperature capabilities of the composite. Initially the glass fibres were incorporated in a polyester matrix which could withstand temperature up to 200°C. They were applied in car bodies, appliances, boats etc., because of their light weight and mitigate of production. Intricate composites parts can be made by injection moulding. Polymer matrices are usually thermosets such as epoxies. Later, resins which can withstand high temperatures, of the order of 300°C were developed such as polyamides. Other thermo setting resins include benzyoclylobutene – bismaleimides. Advanced composites are manufactured by using the above polymers with reinforcements of stronger fibres such as aramid and carbon. As a result advanced composites are finding increasing applications in aircraft, automotive industry, etc. In order to reduce the manufacturing time, thermoplastics polymers such as polyether – ether ketone (PEEK) have been developed. The plastic requires only a short revelation to heat to soften the plastics, thereby allowing faster processing of the composite.

1.2. COMPONENTS OF A COMPOSITE MATERIAL

In its most basic form a composite is the element which comprises of two or more elements (at least two elements) that are bound together to produce a different material which produces enhanced properties that are better and different to the properties of the individual elements. Usually composites comprises of reinforcement and a matrix, incorporated mainly to raise the stiffness and the strength of the matrix.

1.2.1. Role of matrix in a composite
Numerous materials when are in a fibrous form display very high strength but in order to attain these properties the fibers must be joined by an acceptable matrix. The role of matrix is to separate the fibers from each other in order to avert abrasion and development of fresh surface flaws and to stick the fibers in place by acting as a bridge. A quality matrix must possess the capability to transfer the load onto the fibers, deform easily under applied load.

In case of laminates [6] the nature of bonding forces indicates that upon initial loading there is a proneness for the adhesive bond between the matrix and reinforcement to be failed. The frictional forces between them portrayal for the high strength properties of the laminates.

1.2.2. Material used as matrices in composites

1.2.2.1 Bulk-Phases

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

1.2.2.2. Reinforcement

In a composite material the purpose of the reinforcement is basically to increase the mechanical properties of the neat resin structure. The properties of the composite are affected differently by different fibers used in it as these fibers have different properties. For various applications, the fibres are required to be organised into the structure of sheet, known as a fabric, in order to make handling possible.

1.2.2.3. Interface

The important feature of interface is that its properties are not rendered by any component in the system on isolation. It is a bounding zone at which disturbances occurs, it can be mechanical, chemical, physical etc. It is required that fibre must be wetted by the matrix material and in order to improve this wettability coupling agents are used frequently. Increase in the interface surface area is only possible when the fibre is well “wetted”. The applied load must be effectually transferred to the fibers from the matrix through the interface to get the advisable properties and this concludes that the interface should be large and the fibers and matrix must be joined strongly.

1.3. TYPES OF COMPOSITE MATERIALS

The composite materials are classified into the following categories as shown in Figure-1.1 (a - e).

1.3.1. Fibre-reinforced composites

Because of intrinsic high specific strength and stiffness these composites are universally used in numerous industrial applications. These composites are acquiring high potential in tribological applications also as they possess brilliant structural presentation. Fiber reinforced composites materials comprises of fiber of high strength in or bonded to a matrix with discrete interfaces between them [4, 5]. In this form physical and chemical identities are retained by both fibres and matrix. Yet they produce an amalgamation of properties which is difficult to achieve with either of the composite constituents individually. In general, the role of fibers is to carry load, whereas the role of matrix is to keeps them in the crave position and alignment [5, 6].Fibrous composite can be further classified into two groups: continuous (long) fiber composite and discontinuous (short) fiber composite.

1.3.2. Laminate Composites

Laminate Composites consists layers of material held together by matrix. Mainly, these layers are organized alternatively in order to provide a better bonding between the reinforcement and the matrix. According to the end use of the composite these laminates can have uni- directional or bi-directional orientation of the fiber reinforcement. The different types of composite laminates are angle-ply, unidirectional, symmetric laminates and cross- ply. A blended laminate can also be fabricated by the use of alternate constituent materials or of the same material with alternate reinforcing design. In most of the applications of laminate composites, man-made fibers are used because of their good amalgamation of physico-mechanical and thermal behaviour.
Natural Fiber Composites: Initiative in Product Development

1.4. NATURAL FIBER COMPOSITES: Initiative in Product Development

The cost effective option to synthetic fibre reinforced composites and the interesting studies of plant or natural fibre inspires the researchers to make advances in the field of composites. Ease in access and built-up simplicity of natural fibre have convinced these researchers to try natural fibres which are available locally and to study their practicability of reinforcement motives. These are also studied to have the information that up to what limit they can fulfill the desired specifications and properties for various uses. Natural fibre appears as a good renewable and biodegradable substitute to most of the synthetic fibre such as glass fibre.

Vegetables, animal, mineral fibers etc. fall under the area of natural fibre. Generally it is referred as wood and agro based fibre, leaf, stem and seed fibers in the composite engineering. A natural fibre frequently contributes to the structural presentation of plant and they can deliver substantial reinforcement, when used in the production of plastics composites.

Is curtailed to non-bearing applications because of their bring down strength compared to synthetic fibre reinforced polymer complex in malice of the interest and environmental appeal of natural fibers. By the sense of positioning the fibers in particular locations for maximum strength presentation, the limitations in stiffness and strength of bio composites can be succeeded.

Accordingly vast studies on construction and properties of polymer matrix composite (PMC) substituting the synthetic fibre with natural fibre like Jute, Pineapple, Sisal, Kenaf, Bamboo, luffa cylindrica, ipomea carnea and Bagasse were executed [7-12]. Above natural fibers have numerous advantages over the glass fibre or carbon fibre such as renewable, low cost, lightweight, high specific mechanical performance.

1.5. Applications

- Automobile industry:
- Packaging industry:
- Consumer products:
- Building and construction industry:
1.5.1. *Luffa cylindrica* as a natural fiber

Numbers of potential natural resources are there, which India has in abundance. Most of which comes from the forest and agriculture.

Luffa cylindrica, locally called as ‘Sponge-gourds’ is that natural resource whose capability as fiber reinforcement in polymer composite has not been explored to date. The fibrous cords are liable in a multidirectional array resulting in a natural mat in ligneous netting system possess by "Sponge gourds". It comprises 62% cellulose, 20% hemicellulose and 11.2% lignin [1]. The sponge-gourd (*Luffa Cylindrica*) plant with fruit which belongs to the curcubitaceae family is shown in Fig. 1.1(a).

The main objective of this project is to prepare a PMC using luffa fiber as reinforcement and epoxy as matrix material and to study its moisture absorption characteristics under different environmental conditions and then to find its mechanical properties i.e.; tensile and flexural strength. Out of the available manufacturing techniques, we have chosen hand-lay-up method to construct the composite. Then the composites were manufactured by varying the no. of layers of fiber i.e.; single, double and triple layers composite using these techniques. The surface of fracture and worn out samples have been studied using Scanning Electron Microscope (SEM) to have an idea about the fracture behaviour of the composite.

2. LITERATURE SURVEY

2.1. NATURAL FIBERS: Source and Classification

Growing environmental awareness has activated the researchers worldwide to enhance and utilize materials that are companionable with the environment. In the procedure natural fibers have become suitable options to traditional synthetic or manmade fibers and have the prospective to be used in cheaper, more sustainable and more environment friendly composite materials. Natural organic fibers can be obtained from either animal or plant sources. Most of the useful natural textile fibers are obtained from plant, with the anomaly of wool and silk. All plant fibers comprises of cellulose, whereas protein act as a chief content of fibers of animal origin. Hence, the natural fibers are categorized on the basis of their origin, whereas the plant fibers can be further classified on the basis of plant parts from which the parts are originated. An overview of natural fibers.

The chief driver for switching natural fibers for glass is that they can be grown with lesser cost than glass. The price of glass fiber is around Rs. 300.0/- per kg and has a density of 2.5 gm/cc. On the other hand, natural fiber costs Rs. 15.0/- to 25.0/- per kg and has a density of 1.2-1.5 gm/cc.
On the other hand, when the specific modulus of natural fibers is measured, the natural fibers show values that are similar to or even better than glass fibers. Material cost savings, suitable to the use of natural fibers and high fiber filling levels, coupled with the benefit of being non-abrasive to the mixing and moulding tools make natural fibers a thrilling outlook. These reimbursement mean natural fibers could be used in many applications, including building, automotive, household appliances, and several other applications.

3. MECHANICAL CHARACTERIZATION OF LUFFA CYLINDRICA FIBRE EPOXY COMPOSITE

In common natural fibers are absorptive in nature and they take up or liberate moisture relying on to the environmental conditions. For high moisture absorption rate amorphous cellulose and hemicellulose present in the fibre are the main reasons, as they possess innumerable easily available -OH groups which provide an increased level of hydrophilic property to the fibre. The high moisture absorption of the fiber happens due to hydrogen bonding of water molecules to the -OH groups in the fiber cell wall. This guides to a moisture growth in the fibre cell wall (fibre swelling) and also in the fibre-matrix border. This in turn becomes liable for variations in the dimensions of cellulose-based composites, mainly in the thickness and the linear expansion because of reversible and irreversible swelling of the composites. In order to solve this problem, chemical treatment has been taken into account as a good method to reduce the -OH group in the fibers. Different chemical treatments such as alkali treatment, acrylation, benzoylation, isocyanate treatment, acetone treatment, acetylation, silane treatment, permanganate treatment etc. are reported by many researchers [22, 23].

The moisture uptake of composites comprising natural fibers has some unfavourable causes on their properties and hence disturbs their long-term presentation. In view of the sternness of moisture absorption and its consequences on composite properties, a number of efforts have already been made by several researchers to address this issue.

Chemically and the chemically treated fibres showed a decrease in water absorption because of the enhanced interfacial bonding. As the temperature stimulates the diffusion procedure, it was found that the water absorption of the composite increases with the temperature. Plasticization effect of water was the reason to decrease the tensile properties of the composites.

3.1. CHEMICAL MODIFICATION OF FIBER

Utilizing natural fibers as reinforcement in the organization of plastic composites has improved intensely in recent years. For a well-developed composite using natural fibre as reinforcement the understanding of surface adhesive bonding of fibre and the chemical composition is essential. Interfacial bonding between fibers and the resin needs to be good as it plays a significant role in enhancing the mechanical properties of the composites. Understanding the importance of chemically modified fibers, numerous authors have started studying treatment of fibers in order to develop the bonding with resin matrix. The quantity of individual material in a composite and the nature of interfacial region between matrix and reinforcement decides the mechanical properties of the composites.

Absence of good interfacial adhesion makes the usage of cellular fiber composites less attractive. Frequently, due to water absorbing nature of natural fibre the interfacial properties between the fiber and polymer matrix tends to be low, which decreases its potential of being used as reinforcing agents. Hence chemical alterations are taken into account to enhance the interface of fibers. There are several chemical treatments that exist for the fibre surface modification. Chemical treatment comprising alkali, silane, acetylation, benzoylation, acrylation, isocyanates, maleated coupling agents, permanganate treatment are discussed in details in.

The main reason for performing chemical alterations on natural fibres is to improve the adhesion between fibre surface and the polymer matrix by altering the fibre surface and the fibre strength. It also helps in enhancing the mechanical properties by decreasing the water absorption capability of the fibre. Out of the available treatments, for the present case to have a decent bonding between the fibre and the matrix Luffa Cylindrical fibre have been treated with alkali. The subsequent section will elaborate separately the treatment of the fibre surface by alkali methods, study of mechanical properties of both treated and untreated fibre reinforced polymer composite followed by studying environmental effects on mechanical performance of the composite along with moisture absorption characteristics.

3.1.1. Method of Chemical Modification

3.1.1.1. Alkaline Treatment

When it comes to reinforce thermoplastics and thermosets, alkaline treatment is one of the mostly used treatments. In the modification done by the alkaline treatment the disruption of hydrogen bonding in the network structure takes place resulting in increased surface roughness. By using this treatment, certain amount of lignin, wax and oils covering the outer surface wall of the fibre was removed, depolymerizes cellulose and depicts the short length crystallites [36]. Addition of aqueous sodium hydroxide (NaOH) to natural fibre stimulates the ionization of the -OH group to the alkoxide.

\[
\text{Fiber} -\text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O}
\]

Alkaline treatment has two effects on the fibre:

- It increases surface roughness by the disruption of hydrogen bonding resulting in better mechanical linking.

Fiber + NaOH → Fiber – O – Na + H₂O
It increases the number of possible reactions sites by increasing the amount of cellulose exposed on the fibre surface.

Subsequently, this treatment has a lasting effect on the mechanical behaviour of flax fibre, especially on the strength and stiffness of the fibre.

For performing this treatment, firstly the Luffa Cylindrica fibre were kept in a solution containing 5% NaOH at room temperature maintaining a liquor ration of 15:1 for 4hrs. Secondly, the fibers were washed many times with water in order to remove the NaOH sticking to the fibre surface followed by neutralizing with dilute acetic acid and washed with distilled water, so that pH of 7 was maintained. Lastly, the fibers were dried at room temperature for 48hrs followed by oven drying for 6hrs at 100°C. The alkali reaction between Luffa Cylindrica fibre and NaOH is as follows:

$$(\text{Luffa Cylindrica}) - \text{OH} + \text{NaOH} \leftrightarrow (\text{Luffa Cylindrica}) - \text{O}^\text{-Na^+} + \text{H}_2\text{O}$$

3.2. COMPOSITE FABRICATION

For preparation of composite the following materials have been used;

(1) Luffa Cylindrica fiber

(2) Epoxy

(3) Hardener

(1) Preparation of Luffa Cylindrica Fiber Mats

Dried Luffa Cylindrica was collected locally. These fibres were then treated with water for 24 hrs in order to remove wax, lignin and oil from the external surface of luffa fibre and then dried at room temperature. After these the fibres were cut with appropriate dimensions (150×140 mm) and then these fibres were kept between two wooden boards followed by pressing it into the bench vice to straighten the fibres.

(2) Epoxy Resin

The epoxy resin used in this examination 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 (2aminoethyl)amine-1,2diamin) has been used with the epoxy designated as HY 951.

(3) Composite preparation

Initially, wooden moulds with dimensions of 140 x 120 x 10 mm³ were prepared for the fabrication. For different number a layer of fibre, epoxy resin and hardener (ratio of 10:1 by weight) with a calculated amount was mixed thoroughly in a glass jar. Figure 3.1(a) illustrates the mould used to construct the composite. Mould release sheet was put over the glass plate and a mould release spray was sprayed over the inner surface of the mould for quick and easy removal of composite. After keeping the mould on a ply board a thin layer of the mixture was poured. Then the fiber lamina was distributed on the mixture. Then again resin was applied over the fiber laminate and the procedure was repeated to get the desired thickness. The remaining mixture was then poured into the mould. Precaution was taken to prevent the air bubbles formation. Then from the top pressure was applied and the mould was kept at room temperature for 72 hrs. During application of pressure some amount of mixture of epoxy and hardener squeezes out.
Care has been taken to consider this loss during manufacturing of composite sheets. After 72 hrs the samples were taken out of the mould. Figure 3.2 (a, b) shows the photograph of the composite specimen cut for further experimentation.

Figure 3.1 Mould used for fabrication of the composite

Figure-3.2 (a) Flexural test samples

Figure-3.2 (b) Tensile test samples

3.4. STUDY OF ENVIRONMENTAL EFFECT

To study the effect of environmental conditions on performance of Luffa Cylindrica fiber epoxy composite, the composite sample with both untreated and chemically treated fibers were subjected to various environments such as:

(1) Saline treatment
(2) Distil treatment
3.4.1. Moisture absorption test

Moisture absorption test and thickness swelling tests were conducted in accordance with ASTM D570-98. Four specimens for different layers (Single, Double and Triple layers) were cut with dimensions of 140 x 15mm (length x width) and the experiment was performed using test samples. The specimens prior to testing were dried in an oven at 80°C and then were allowed to cool to room temperature and kept in a desiccator. The weight of the samples were taken before subjected to steam, saline water and distil water environments. After exposure for 12 hr, the specimens were taken out from the moist environment and all surface moisture was removed with a clean dry cloth or tissue paper. The specimens were reweighed to the nearest 0.001 mg within 1 min. of removing them from the environment chamber. The specimens were weighed regularly from 12-156 hrs with a gap of 12hrs of exposure. The moisture absorption was calculated by the weight difference. The percentage weight gain of the samples was measured at different time intervals by using the following equation:

\[
\% \Delta M_t = \frac{(W_t - W_0) \times 100}{W_0} \tag{3.1}
\]

Where ‘\(W_0\)’ 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 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:

\[
TS(t) = \frac{H_t - H_0}{H_0} \times 100 \tag{3.2}
\]

Where, ‘\(H_t\)’ and ‘\(H_0\)’ are the composite thickness after and before the water immersion respectively.

3.4.2. Mechanical testing of sample

3.4.2.1. Tensile test

The tensile test is generally performed on flat specimens. The most commonly used specimen geometries are dog-bone and the straight side type with end tabs. The specimen used in present case is shown in fig 3.3 (a). The tensile tests were conducted according to ASTM D3039-76 standard on a computerized Universal Testing Machine INSTRON H10KS. The span length of the specimen was 42 mm; the tests were performed with constant strain rate of 2 mm/min.

Figure-3.4 (a) UTM machine sample unloaded for tensile testing
3.4.2.2. Flexural test

Three point bend test was carried out in an UTM machine in accordance with ASTM D790-03 to measure the flexural strength of the composites. The loading arrangement for the specimen and the photograph of the machine used are shown in figure 3.5. All the specimens (composites) were of rectangular shape having length varied from 100-125 mm, breadth of 100-110 mm and thickness of 4-8 mm. A span of 70 mm was employed maintaining a cross head speed of 0.5mm/min.

The flexural strength of composites was found out using the following equation

\[ \tau = \frac{3fl}{2bt^2} \]

Where \( \tau \) is the flexural strength, \( f \) is the load, \( l \) is the gauge length, \( b \) is the width and \( t \) is the thickness of the specimen under test.
3.4.3. Results and discussion

3.4.3.1. Moisture absorption behaviour

The results of both untreated and treated fibre composite samples exposed to different environments are shown in Table 3.1 to 3.24.

3.4.3.2. Measurement of Diffusivity

The water sorption kinetics in LCF-reinforced epoxy composite has been studied through the diffusion constants k and n. The behaviour of moisture sorption in the composite was studied by the shape of the curve represented by the equation (3.3) [37, 38]:

\[
\frac{M_t}{M_m} = k t^n
\]  

(3.3)

Where, \( M_t \) is the moisture content at specific time ‘t’, \( M_m \) is the equilibrium moisture content (EMC), and \( k \) and \( n \) are constants.

The diffusion coefficient or diffusivity (\( D_x \)) of moisture absorption was calculated using the following equation:

\[
D_x = \pi \left[ \frac{h}{4M_m} \right]^2 \left( \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2
\]  

(3.4)

where ‘\( M_m \)’ is the maximum percentage of moisture content, ‘\( h \)’ is the sample thickness, ‘\( t_1 \)’ and ‘\( t_2 \)’ are the selected points in the initial linear portion of the plot of moisture absorption (\( M_t \) versus \( \sqrt{t} \)) (Figure 3.21) and ‘\( M_1 \)’ and ‘\( M_2 \)’ are the respective moisture content.

From the plot of \( M_t \) versus square root of time (t) the value of \( D_x \) has been evaluated and summarized.
3.4. CONCLUSIONS

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

- The Luffa Cylindrica fibre can successfully be used as reinforcing agent to fabricate composite by suitably bonding with epoxy resin.
- On increasing the fibre content the strength, modulus and work of fracture increases and the best combination is found with Double Layered composite.
- The fibre surface modification by chemical treatments significantly improves the fibre matrix adhesion, which in turn improves the mechanical properties of composite.
- The moisture uptake and thickness swelling values increases with increase in fiber loading. Both values are found to be higher in saline environment than in distil water environments. However these values are considerably reduced with chemical treatments of the fibre.
- Under all environment conditions, the moisture diffusion process of both treated and untreated Luffa Cylindrica fibre composites are found to follow the Fick’s law.
- Fibre breakages are found to be the predominant mode of failure as ascertained from the morphology of the treated fibre composites.

REFERENCES