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Chemical Properties of Natural Fiber Composites and Mechanisms of Chemical Modifications

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In recent years, natural fibers reinforced composites have received a strong attention because of their light weight, nonabrasive, combustible, nontoxic, low cost and biodegradable properties. However, the lack of good interfacial adhesion, low melting point and poor resistance towards moisture make the use of natural fiber reinforced composites less attractive in a number of applications. Chemical treatment of the natural fibers can clean the fiber surface, chemically modify the surface, stop the moisture absorption process and increase the surface roughness. A number of specific chemicals are used for chemical modifications of these composites. Such chemically modified are being used increasingly for engineering applications (particularly under hard working conditions). Over the life-span of the composites, the environmental conditions also affect the behaviour of composite to a large extent. Thus, it becomes necessary to take care of the chemical properties of composites to ensure their long term stable behaviour in the practical applications. This paper outlines the chemical properties of natural fibers composites and discusses the latest trends in chemical modification mechanisms. A number of commonly used natural composites have been cited as examples in different mechanisms.

Key Words: Natural fiber, Composites, Chemical treatment.

INTRODUCTION

Composite materials, plastics and ceramics have been the dominant emerging materials in the last thirty years. This has a high relevance as the source of metallic materials is limited and is getting depleted everyday. Composite materials are one of the best replacements to a number of metallic, concrete and plastics with enhanced properties at low cost. A composite material consists of two or more different kind of constituent materials, these constituents are insoluble in each other and maintain their physical phases. The constituents can be physically and (or) chemically separated by a clear-cut interface or inter-phases. The composition gives rise to a new material, which exhibits a combination of properties of both the constituent phases. Thus, the 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. Composite materials can be classified¹ into three different groups on the basis of matrix being used: a) Metal matrix composites (MMC); b) Ceramic matrix composites (CMC) and c) Polymer matrix composites (PMC).

Metal matrix composites: Metal matrix composites have many advantages over monolithic metals like higher specific

modulus, higher specific strength, better properties at elevated temperatures and lower coefficient of thermal expansion. Because of these attributes metal matrix composites are under consideration for wide range of applications *viz.* combustion chamber nozzle (in rocket, space shuttle), housings, tubing, cables, heat exchangers, structural members *etc.*

Ceramic matrix composites: One of the main objectives in producing ceramic matrix composites is to increase the toughness. It is often found that there is a concomitant improvement in strength and stiffness of ceramic matrix composites.

Polymer matrix composites: Polymers are the most commonly used matrix materials. The reason for this is two fold; firstly the mechanical properties of polymers are inadequate for many structural applications as their strength and stiffness are low as compared to metals and ceramics. This problem is overcome by reinforcing the other materials with polymer matrix. Secondly, the processing of polymer matrix composites need not require very-high pressure and temperature conditions. Also, the equipments required for manufacturing the polymer matrix composites are simpler than the metallic or ceramic composites. Because of these reasons, polymer matrix composites developed rapidly and got popularity for the structural applications. Polymers can be reinforced with the fibers or the particles to fabricate the fibre reinforced or particle reinforced composites.

TABLE-1
COMPOSITION OF VEGETABLE FIBERS

Fiber	Cellulose (Wt %)	Hemicelluloses (Wt %)	Lignin (Wt %)	Pectin (Wt %)	Moisture content (Wt %)	Waxes	Microfibrillar angle (°)
Flax	71	18.6-20.6	2.2	2.3	8-12	1.7	5-10
Hemp	70-74	17.9-22.4	3.7-5.7	0.9	6.2-12	0.8	2.62
Jute	61.1-71.5	13.6-20.4	12-13	0.2	12.5-13.7	0.5	8
Kenaf	45-47	21.5	8-13	3-5			
Ramie	68.6-76.2	13.1-16.7	0.6-0.7	1.9	7.5-17	0.3	7.5
Sisal	66-78	10-14	10-14	10	10-22	2	10-22
Henequen	77.6	4-8	13.1				
Banana	63-64	10	5		10-12		
Cotton	85-90	5.7		0-1	7.85-8.5	0.6	
Coir	32-43	0.15-0.25	40-45	3-4	8		30-49

Particle reinforced polymer: Metal particles such as aluminium and carbon black, ceramics glass particles are used as reinforcements in these composites. These particles also help to increase the modulus of the matrix, decrease the ductility of the matrix and to reduce the cost of the composites. Ceramics and glasses also offer high melting temperature, low density, high strength, stiffness, wear resistance and corrosion resistance properties. Automobile tyre is a good example of particle reinforced composite, it consists of carbon black particles in a matrix of poly-isobutylene elastomeric polymer.

Fiber reinforced polymer: Fiber reinforced composites are composed of fibers and a matrix. Fibers are the reinforcement and the main source of strength, while matrix glues all the fibers together in shape and transfers the stress between the reinforcing fibers. The fibers can further be natural (*e.g.* bamboo, pine *etc.*) or synthetic (*e.g.* glass, carbon *etc.*). Natural fibers are being predominantly used in polymer composites because of their versatile applications and availability of fundamental research². They have the specific advantage of being renewable, inexpensive, recyclable (completely or partially) and are biodegradable in nature^{3,4}. Plants, such as flax, cotton, hemp, jute, sisal, kenaf, pineapple, ramie, bamboo, banana, *etc.*, as well as wood are being used as a source of natural fibers. These fibers are commonly used as reinforcements in the composites. Easy availability and low price, along with the excellent mechanical properties make them an attractive ecological alternative to glass, carbon and man-made fiber. Natural fiber-composites are more environment friendly and offer plenty of applications in transportation (automobiles, railway coaches, aerospace), military applications, building and construction industries (ceiling paneling, partition boards), packaging and consumer goods⁵.

Natural fiber composites: Use of natural fiber as reinforcement in the composites has received increasing attention both by the academia and industry. Natural fibers play an important role in developing high performing fully biodegradable 'green' composites, which will be a key material to solve the environmental problems. Natural fibers offer many attractive technical and environmental qualities, when used as reinforcements in polymer composites. Natural fiber exists in the form of vegetable fiber, animal fiber or mineral fiber. Vegetable fiber finds extensive applications due to its easy availability and inexpensive nature. Cellulose, hemicellulose, lignin and pectins⁶ form a major component in the vegetable fibers. Cellulose is a linear macromolecule consisting of D-anhydroglucose

repeating units joined by β -1,4 glycosidic linkages. It is the main component, which provides strength, stiffness and structural stability. Hemicelluloses are the branched polymers containing five- and six-carbon sugars of varied chemical structure, Lignin is an amorphous, cross-linked polymer network consisting of an irregular array of variously bonded hydroxy- and methoxy-substituted phenylpropane units⁷. Lignin is less polar than the cellulose and acts as a chemical adhesive within and between the fibers. Pectins are complex polysaccharides, the main chains of which consist of a modified polymer of glucuronic acid and residues of rhamnose. Their side chains are rich in rhamnose, galactose and arabinose sugars. The chains are often cross-linked by calcium ions, improving structural integrity in pectin-rich areas. Lignin, hemicellulose and pectin collectively function as matrix and adhesive to hold together the cellulosic framework structure of the natural fiber composite. Composition of major vegetable fiber is shown in Table-1⁸.

Mechanisms of chemical modifications: One of the major problems associated with the use of natural fibers in composites is their high moisture sensitivity, adversely affecting the mechanical properties and lamination. The reduction in mechanical properties may be due to poor interfacial bonding between resin matrices and fibers. It is therefore necessary to modify the fiber surface to render it more hydrophobic and also more compatible with resin matrices. Natural fibers are amenable to chemical modification due to the presence of hydroxyl groups. Surface characteristics, such as wetting, adhesion, surface tension, or porosity of fibers, can be improved upon chemical modification⁹. In this paper, we have focused on the chemical treatments adopted for natural fibers and common modification techniques are explained below:

Alkali treatment: Alkali treatment leads to the increase in the amount of amorphous cellulose at the expense of crystalline cellulose. The important modification occurring here is the removal of hydrogen bonding in the network structure. The following reaction takes place as a result of alkali treatment.



The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. The type of alkali (KOH, LiOH, NaOH) and its concentration will influence the degree of swelling and hence the degree of lattice transformation into cellulose-II¹⁰. Na⁺ has got a favourable diameter, able to widen the smallest pores in

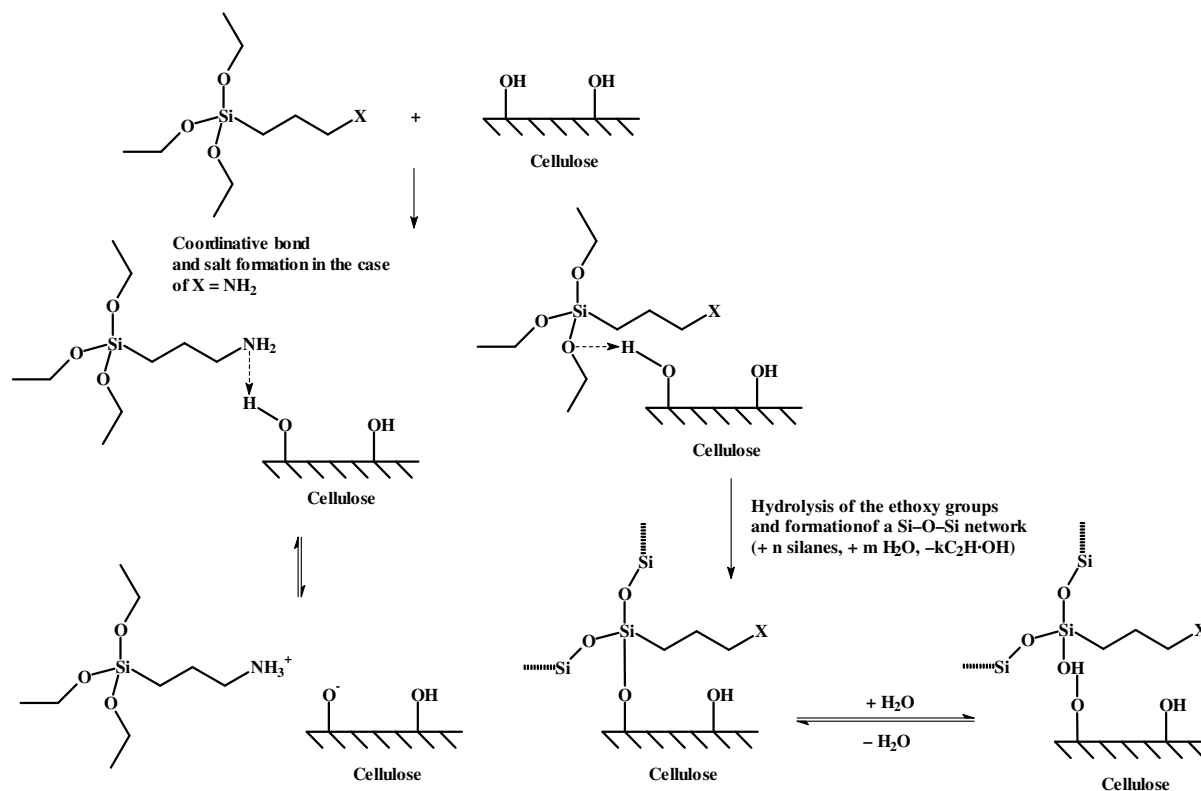
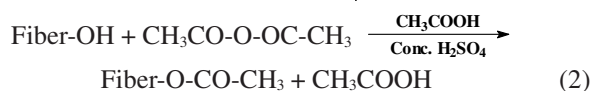


Fig. 1. Interaction of silanes with cellulosic fibers

between the lattice planes and penetrate into them. Consequently, sodium hydroxide treatment results in a higher amount of swelling. This leads to the formation of new Na-cellulose-I lattice, a lattice with relatively large distances between the cellulose molecules and these spaces are filled with water molecules. In this structure, the OH-groups of the cellulose are converted into ONa-groups, expanding the dimensions of molecules. Subsequent rinsing with water will remove the linked Na-ions and convert the cellulose to a new crystalline structure, *i.e.* cellulose-II, which is thermodynamically more stable than cellulose-I. Sodium hydroxide can cause a complete lattice transformation from cellulose-I to cellulose-II, in contrast to other alkalis that produce only partial lattice transformation. The alkali solution influences not only the cellulosic components inside the plant fiber but also the non-cellulosic components (hemicellulose, lignin and pectin)¹¹. The effect of chemical treatment of natural fibres with sodium alginate and sodium hydroxide has also been reported for coir, banana and sisal fibres by Mani *et al.*¹². This modification results in an increase in adhesive bonding and thus improves ultimate tensile strength up to 30 %. Samal and Ray¹³ have studied the chemical modification of pineapple leaf fibers using alkali treatment, diazo coupling with aniline and cross-linking with formaldehyde. These chemical treatments result in significant improvements in mechanical properties and chemical resistance and reduced the moisture regain. Joseph *et al.*¹⁴ have investigated the influence of chemical treatment with sodium hydroxide, isocyanate and peroxide on the properties of sisal/polyethylene composites. Furthermore, Razi *et al.*¹⁵ found that the treatment of wood with sodium hydroxide followed by drying with vinyltrimethoxysilane is superior, for obtaining maximum bonding strength at the wood/polymer interface that yields improved mechanical properties.

Acetylation: Acetylation is an attractive method of modifying the surface of natural fibers and making it more hydrophobic. The main principle is the reaction of the hydroxyl groups (OH) of the fiber with acetyl groups (CH₃CO), therefore rendering the fiber surface more hydrophobic. The hydroxyl groups that react are those of the minor constituents of the fiber, *i.e.* lignin and hemicelluloses and those of amorphous cellulose¹⁶. The hydroxyl groups in the crystalline regions of the fiber are closely packed with strong interchain bonding and are inaccessible to chemical reagents. The acetylation of the OH group in cellulose is represented in eqn. (2).



Acetylation has been shown to be beneficial in reducing moisture absorption of natural fibers. Reduction of about 50 % of moisture uptake for acetylated jute fibers and of up to 65 % for acetylated pine fibers has been reported in the literature¹⁷. Acetylation has also been found to enhance the interface in flax/polypropylene composites¹⁸.

Silane treatment: Silanes are the hydrophilic compounds with different groups appended to silicon such that one end will interact with matrix and the other end can react with hydrophilic fiber, which act as a bridge between them. The uptake of silane is very much dependent on a number of factors including hydrolysis time, organo-functionality of silane, temperature and pH. Alkoxy silanes are able to form bonds with hydroxyl groups. Silanes undergo hydrolysis, condensation and the bond formation stage. Silanols can form polysiloxane structures by reaction with hydroxyl group of the fibers¹⁹. The chemical reaction²⁰ is illustrated in Fig. 1.

Herrera-Franco and coworkers²¹⁻²³ have studied the influence of different silane coupling agents on the properties of

henequen fiber-reinforced polymer composites. FTIR and XPS spectroscopy have been used by the research group to show that the reaction between silanes and cellulose takes place only at temperatures above 708 °C.

Chemical properties: During the last decades, there has been a tremendous growth in the use of composite materials in various applications, ranging from sport goods to structural components for the automotive and aerospace industries. Long term properties of the materials are of primary importance for such applications. High performance polymer composite materials are being used increasingly for engineering applications (particularly under hard working conditions). The materials must provide unique mechanical and tribological properties combined with a low specific weight and heat resistance to degradation in order to ensure safety and economic efficiency. The effect of environmental condition on composites has been a subject of much debate and many studies are ongoing to study the behaviour of composites under various conditions. The commercial feasibility of composites depends highly on the chemical properties of the material. The chemical properties, which play a significant role in applications and long life behaviour of the material, are: (i) Chemical resistance; (ii) Water absorption; (iii) Swelling; (iv) Moisture absorption; (v) Void content and density.

Chemical resistance: In order to probe, whether a composite can find applications in chemical and storage tanks, study of its chemical resistance is necessary. The chemical resistance test is normally carried out for the prominent acids, alkalis and the solvents. In the category of acids, the most commonly used chemicals include: concentrated hydrochloric acid (10 %), concentrated nitric acid (40 %) and glacial acetic acid (8 %), the resistance against the alkalis is normally checked for the aqueous solutions of sodium hydroxide (10 %), ammonium hydroxide (10 %) and sodium carbonate (20 %) ²⁴. However, the prominent solvents like benzene, carbon tetrachloride, toluene and water are used to study the chemical resistance against the chemical solvents. In the normal experimental process the samples are weighed and then immersed in the chemical reagents for 24 h. The experiments are normally carried out at room the temperature. After the time period of 24 h, the samples are removed, washed with distilled water and dried by pressing them between filter papers. The weight is again reported to find the change in weight. The chemical resistance is normally reported in terms of the percentage chemical resistance as shown in eqn. (4).

$$\text{Chemical resistance (\%)} = (T_i - W_{\text{aci}}) / T_i \quad (4)$$

where, T_i = initial weight; W_{aci} = weight after certain interval

Rajulu *et al.* ²⁵ studied the chemical resistance and tensile properties of epoxy and unsaturated polyester (UP) blend coated bamboo fibres. Both the uncoated and coated fibers showed good chemical resistance. The fibers coated with unsaturated polyester showed weight loss, when they were treated with the solvents: benzene and toluene. But fibers coated with a blend of epoxy/unsaturated polyester showed weight gain. This indicates the formation of the gel instead of dissolution in the second case. However, the blend coated fibers showed lower water absorption. These better properties of chemical resistance and lower water absorption may be attributed to the hydrogen bonding between unsaturated polyester

and epoxy which promotes close packing at molecular level. Hence, making them more suitable for the high moisture locations. Rajulu *et al.* ²⁶ also studied the chemical resistance and the tensile properties of short bamboo fiber reinforced epoxy composites and reported that 30 mm is the critical length for the fibers for maximum tensile strength of the composites. Kaushik *et al.* ²⁷ studied the chemical resistance of short glass fibre reinforced epoxy composites. Chemical absorption increased with increase in fiber fraction and it was maximum for DMF. The samples were completely destroyed in concentrated sulfuric acid, pyridine and nitric acid. Singha *et al.* ²⁸ studied the chemical resistance of bio fibre based polymer composites. Chemical resistance of *Hibiscus sabdariffa* reinforced polymer composites decreases with the increase in fiber dimension. This may be due to increase in fiber content in the composite, which is vulnerable to the chemical attack resulting in decreased resistance towards chemicals. Srinivasulu *et al.* ²⁹ studied the chemical resistance and tensile properties of short bamboo fibre reinforced epoxy/poly carbonate composites

A number of research groups have studied the chemical resistance of hybrid composites. These composites find a variety of applications because of their high strength-to-weight ratio and ease of fabrication. Raghu *et al.* ³⁰ have studied the chemical resistance properties of sisal/silk hybrid composites, whereas Guduri *et al.* ³¹ have studied the chemical resistance, void contents and morphological properties of Hildegardia fabric/polycarbonate toughened epoxy composites. It has been shown by these research groups that Alkali treatment in the presence of the silane coupling agent gives good bonding between the reinforcement and the matrix. Hildegardia/polycarbonate-toughened epoxy composites have shown reasonable chemical and water resistance. The liquid absorption increased when the fabric was treated with an alkali, when the coupling agent was used and in the presence of water and aqueous solutions. Khanam *et al.* ³² have also studied the chemical resistance of randomly oriented unsaturated polyester based sisal/carbon fiber reinforced hybrid composites with different fibre weight ratios.

Water absorption: New applications and end uses of composites for decking, flooring and outdoor facilities with strong exposure to atmosphere or contact with aqueous media have made it necessary to evaluate the water uptake characteristics of natural fiber composites. Because of the hygroscopic nature of natural fibers, water uptake of composites containing these fibers as fillers and/or reinforcement can be a limiting parameter for a number of applications of the composites. Water absorption can adversely affect a number of mechanical properties and can also buildup the moisture content in the fiber cell wall and in the fiber-matrix interphase region. Moisture buildup in the cell wall could result in fiber swelling and affect the dimensional stability of the product. If necessary, the moisture absorbed in the fiber cell wall can be reduced through the acetylation of some of the hydroxyl groups present in the fiber ³³. Good wetting of the fiber by the matrix and adequate fiber-matrix bonding can decrease the rate and amount of water absorbed in the interphase region of the composite ³⁴. For Water absorption test, composite samples are weighed and then weighed samples are immersed in the

distilled water at room temperature. The samples are taken out periodically and weighed immediately after wiping out the water on the surface of the sample, using a precise four digit balance to find out the content of water absorbed. All the samples are dried until constant weight with four digit balance, previous to immersing in water. Percentage of water uptake is calculated by the following equation.

$$\text{Water absorption (\%)} = [(W_t - W_0)/W_0] \times 100 \quad (5)$$

where, W_t = weight of the sample at time t ; W_0 = initial weight of the sample at $t = 0$.

Water absorption behaviour of natural fiber thermo-plastic composites have been studied by a number of researchers and the effectiveness in reducing the amount and rate of water absorption has been well-documented in the literature³⁵⁻³⁷. Tajvidi *et al.*³⁸ have investigated the long-term water absorption behaviour of various natural fiber (wood flour, kenaf fiber, rice hulls, newsprint *etc.*) polypropylene composites and have also studied the effect of natural fiber type and fiber content. Authors have found that the chemical composition of the natural fibers is responsible for the different water uptake behaviour. It appears that the RH/PP has the lowest water absorption. This behaviour can be attributed to the higher amount of extract and ash and lower amount of cellulose and pentose in rice hulls³⁹. Kenaf fibers are rich in cellulose and hemicelluloses and they possess low lignin content. Hence, their higher water absorption can be explained by the higher amount of cellulose and pentosan and lower amount of lignin. Rashdi *et al.*⁴⁰ have studied the effects of water absorption on the mechanical properties of Kenaf fibre reinforced unsaturated polyester composites. A decrease in the tensile properties of the composites was observed of the water-saturated samples compared to the dry samples. The percentage of moisture uptake increased as the percentage of the fibre weight increased due to the high cellulose content. The water absorption pattern of these composites was found to follow the Fickian behaviour. Rajulu *et al.*⁴¹ has observed that the uncoated bamboo fibres absorbed a higher quantity of water than epoxy coated fibres. The polycarbonate/epoxy blend coated fibres showed intermediate water absorption. Suhara Panthpulkal *et al.*⁴² studied the water absorption of injection-molded short hemp fibre/glass fibre reinforced poly propylene hybrid composites. Hybridization with glass fiber enhanced the resistance to water absorption properties of the hemp fiber composites.

Swelling behaviour: Swelling behaviour of the composite samples is usually evaluated by studying the swelling in different solvents such as methanol, isobutanol, carbon tetrachloride and water. For the swelling test, the specimens are normally dried in an oven for a specified time at a particular temperature and then cooled in a dessicator. Known weight (W_i) of the initial samples is immersed in 100 mL of different solvents at room temperature for 15 days. The samples are then filtered and the excess solvent was removed with the help of filter paper, patted dry with a lint-free cloth and then final weight (W_f) is noted. The percent swelling is calculated from the increase in initial weight in following manner:

$$\text{Swelling (\%)} = [(W_f - W_i)/W_i] \times 100 \quad (6)$$

where, W_f = final weight of the sample, W_i = initial weight of the sample.

Singha *et al.*²⁸ have studied the swelling behaviour of biofiber based polymer composites in various solvents. The swelling behaviour of *Hibiscus sabdariffa* fiber reinforced polymer composites increases with increase in fiber-dimension due to greater affinity of water for OH groups present in the fiber reinforced polymer composites. It has been shown by the authors the the swelling behaviour of composites in different solvents follow the trend $H_2O > CH_3OH > C_4H_9OH > CCl_4$.

Moisture absorbance: All natural fiber reinforced polymer composites absorb moisture in humid atmosphere. The effect of moisture absorption leads to the degradation of fibre-matrix interface region, which creates poor stress transfer efficiencies resulting in a reduction of mechanical properties. Moisture absorbance is usually measured by placing the known weight (W_i) of dry samples in a humidity chamber (Swastika make), which was set at a particular humidity level for about 12 h and then the final weight (W_f) of the samples exposed at a particular relative humidity (RH) is taken. The per cent moisture absorbance is then calculated as:

$$\text{Moisture absorbance (\%)} = (W_f - W_i)/W_i \times 100 \quad (4)$$

where, W_f = final weight of the sample; W_i = initial weight of the sample.

Rashidi *et al.*⁴⁰ have studied the kenaf unsaturated polyester composites and observed that their moisture absorption indicated that polyester composites gave the most superior bonding and adhesion, apart from higher storage modulus. It has been shown by the authors that the moisture uptake increases with the increase in the percentage of the fibre content. Singha *et al.*²⁸ have studied moisture absorbance on *Hibiscus sabdariffa* fiber reinforced phenol-formaldehyde resin matrix based polymer composites at various humidity levels (varying from 20 to 100 %). Moisture absorbance increases with increase in humidity level ranging from 20 to 100 %.

Void content and density: Void content is measured for the composites, in which the matrix, reinforcement and any added filler burn off completely. Generally, a lower void content indicates improved bonding between the fabric and matrix. This is possible only when there is a strong interaction between the components. The density of resin, reinforcement and composites are measured separately. The theoretical composite density (T_d) is calculated using the following formula:

$$T_d = 100/(R/D + r/d) \quad (5)$$

where, R is the weight % of the resin in the composite; D is the density of the resin matrix; r is the weight % of the reinforcement in the composite; d is the density of the reinforcement.

This is compared with measured density. The void content is calculated with the following equation:

$$\text{Void content} = ((T_d - M_d)/T_d) \times 100 \quad (6)$$

where, T_d = theoretical density; M_d = measured density

Guduri *et al.*³¹ have made Hildegardia fabric/polycarbonate-toughened epoxy composites and studied variation of void contents with various fiber contents (15, 20, 25, 30, 35 & 40 %) and fiber orientations (0°, 15°, 30°, 45°, 60°, 75°, 90°). Authors have reported that the void content was minimum at

40 % fiber content. However, the void content must be less than 3 % for good quality composites. Rajulu *et al.*⁴³ has fabricated short bamboo fiber reinforced epoxy composites with varying fiber content. They have determined void content, density and weight reduction of these composites. Authors have reported that the void content of these composites decreased with increase in fiber content because of good bonding between bamboo fiber and epoxy matrix. The bamboo fibers and the epoxy resin matrix constitute lighter composites.

Conclusion

Chemical modification of natural fibers is necessary for increased adhesion between the hydrophilic fibers and hydrophobic matrix. The most promising approach is the one, in which covalent bonds are formed between the fiber and matrix. This can be concluded from the studies that the most common and efficient methods of chemical modification is alkali treatment of fibers and it has been used to treat almost all types of natural fibers with successful results. Most of the chemical treatments decreased the strength properties because of the breakage of the bond structure and also because of the disintegration of the non-cellulosic materials but silane treatment led to strong covalent bond formation and thereby increasing the strength marginally. This is in accordance with the common use of silane treatment by most of the researchers. Thus, the chemical treatment remains an important aspect in delivering industry specific applications of composites. However, a lot more research has to be conducted to develop novel methods of modification and investigate the exact mechanism of reactions occurring at the fiber-matrix interface.

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