Synthesis, Fabrication, Mechanical, Electrical, and Moisture Absorption Study of Epoxy Polyurethane–Jute and Epoxy Polyurethane–Jute–Rice/Wheat Husk Composites

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ABSTRACT: Bisphenol-C-epoxy-toluene diisocyanate polyurethane (PEBCT) has been synthesized and used for the fabrication of jute, jute–rice husk (JRH), and jute–wheat husk (JWH) composites. The composites have been fabricated by hand lay-up technique under a hydraulic pressure of 30.4 MPa at 135°C for 2.5 h. PEBCT-J, PEBCT-JRH, and PEBCT-JWH possess respectively, tensile strength of 37.4, 9.5, and 14.7 MPa, and flexural strength of 39.6, 12.9, and 21.3 MPa, electric strength of 1.3, 1.8, and 1.9 kV/mm and volume resistivity of 1.40×10^{13}, 1.84×10^{13}, and 1.91×10^{13} ohm cm. Tensile strength and flexural strength have decreased, while electric strength and volume resistivity are improved upon hybridization. PEBCT-JWH has better interfacial bond strength and stiffness as compared to PEBCT-JRH. Moisture uptake behavior of PEBCT-J in water, 10% HCl and 10% NaCl at room temperature is quite different. Equilibrium moisture content of PEBCT-J in 10% NaCl (5.5%) is almost half of those in water (11.3%) and 10% HCl (13.6%) environments. Equilibrium time for saline environment is also comparatively low. Equilibrium moisture uptake in boiling water has increased 1.84 times, while equilibrium time has decreased 15.3 times. The composites may be useful for low load bearing in construction industries and for marine applications. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 1228–1233, 2007

Key words: epoxy resins; polyurethane; hybrid composites; mechanical and electrical properties; moisture uptake

INTRODUCTION

Recent research and development in natural fiber reinforced composites as potential structural material has attracted the attention of material scientists allover the world because of their low cost, easy availability, light weight, renewable and biodegradable in nature, low density, and high specific strength make them the most suitable candidates for low load bearing applications.[1–9] Various items have been made from jute fiber based composites. Natural fiber reinforced composites offer many advantages over the traditional construction materials namely steel and aluminum in respect of low density, low thermal conductivity, excellent corrosion and chemical resistance, high strength to weight ratio, better design flexibility, excellent fatigue and impact properties, improved acoustical performance, and low maintenance.

Hybrid composites are materials made by combining two or more different types of fibers in a common matrix. They offer a range of properties that cannot be obtained with a single kind of reinforcement. By careful selection of reinforcing fibers, the material cost can be reduced substantially.[10] The mechanical performance of composites is mainly dependent upon the properties of the matrix, reinforcement, and the interactions between matrix and reinforcing agent. Natural fibers have already been recognized for their role in composites and can be advantageously utilized for the development of environment friendly composite materials with good physical properties.[11]

Low cost hybrid composites are produced by the use of biomass or agro waste in combination with jute. In India, rice husk (RH) and wheat husk (WH) are the biomass materials, which are byproducts from the crops. Considerable work has been carried out on utilization of biomass for particle board, medium density board, pulp,[12] and composites.[13–17] The incentives of utilizing agro waste in the fabrication of composites are their low density, less abrasiveness to equipment, low cost, etc. The main drawback of natural fibers in producing good composites is their compatibility with fillers and resins.

Epoxy resins are the most suitable resins for engineering applications because of their strength and stiffness, good dielectric behavior, resistance to chemicals, corrosion, and microbial attack, low shrinkage

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during cure and good thermal properties.[18–25] They are also suitable for land, marine and surface transportation, automobile and electrical components, adhesives, and natural or synthetic fiber reinforced composites.[26–28]

Polyurethanes are well known for their adhesion, flexibility, high cohesive strength, good resistance to abrasion, amenable curing speed, effective wetting property, and H- bond forming tendency with various substrates such as textile fibers, metals, plastics, wood, glass, etc.[17,29–33] With a view to improve the physicochemical properties of the composites an attempt has been made to use epoxy polyol and toluene diisocyanate to synthesize novel polyurethane containing a cyclohexyl cardo group, which enhances many improved physicochemical properties.

To our knowledge, no work has been reported on polyurethane of epoxy resin of bisphenol-C and toluene diisocyanate based natural fiber-reinforced composites. With a view to investigate new composites, present work deals with synthesis, fabrication, mechanical, electrical, and moisture absorption study of epoxy polyurethane–jute and epoxy polyurethane- rice/wheat husk composites.

EXPERIMENTAL

Materials

Solvents and chemicals used in the present study were of laboratory grade and purified by appropriate methods prior to their use.[34] Epoxy resin of bisphenol-C (EBC) was synthesized and purified according to our recent work.[35] Woven jute fibers (Brown jute, *Corchous capsularis*) used in the present study was obtained from Indian Jute Industries' Research Association (IJIRA), Kolkata, India. The agro wastes RH and WH were obtained from the local market. The toluene diisocyanate (TDI) (2,4-TDI : 2,6-TDI, 80 : 20) was supplied by Narmada Chematur Petrochemicals Ltd. (NCPL), Bharuch, India.

Synthesis and fabrication of epoxy-TDI polyurethane–jute composites

To a 250-mL beaker, 0.018/0.005 mol (22 g/6.5 g) EBC (epoxy equivalent 605.1) was dissolved in 70/15 mL methyl ethyl ketone (MEK) at room temperature. To this solution 0.035/0.010 mol (6.26 g/1.85 g) TDI in 30/15 mL MEK was added portion wise over a period of 10 min and stirred manually for about 25–30 min. The resultant polyurethane (Fig. 1) solution (45 wt % of jute fibers) was applied to 150 × 150 mm^2/50 × 50 mm^2 (62.3 g/19 g) woven jute fibers with a smooth brush. The prepregs were allowed to dry in sunlight for about 15 min. Ten such prepregs were stacked one over the other and pressed between two preheated stainless steel plates under hydraulic pressure of 30.4 MPa at 135°C for 2.5 h. Silicone spray was used as a mold releasing agent. The edges of 50 × 50 mm^2 composites were sealed with the same polyurethane solution for moisture absorption study. Here after composite is designated as PEBCT-J. An attempt has been made to cast EBCT film on a leveled glass

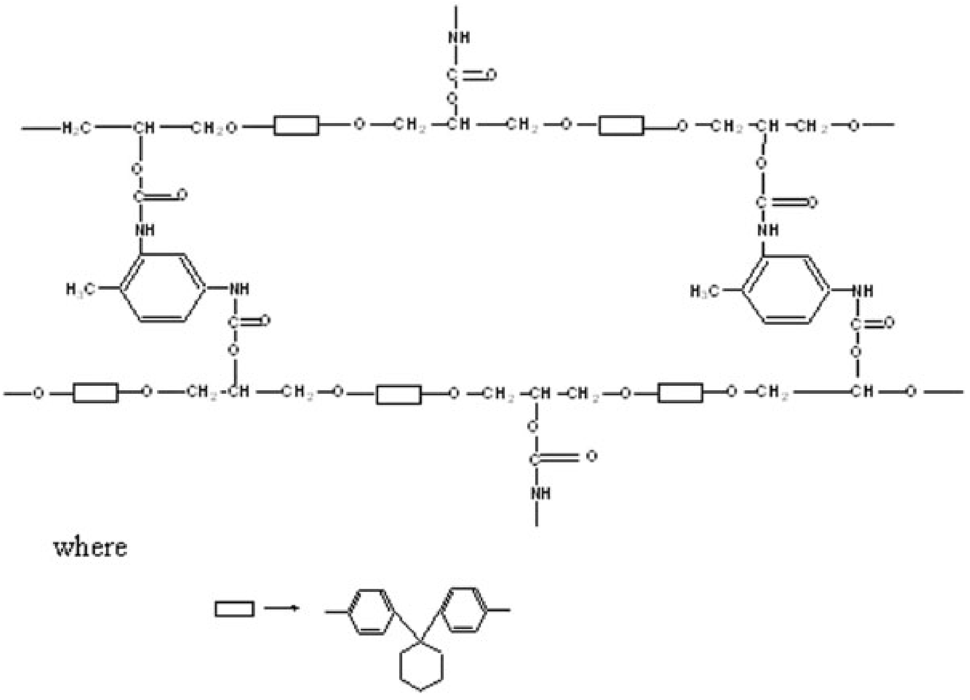

Figure 1 The structure of PEBCT.

mold but EBCT film was found hard and brittle and therefore its mechanical and electrical properties are not determined.

Fabrication of epoxy-TDI polyurethane–jute–RH/WH hybrid composites

An ordinary sieve was used to separate the different sizes of rice husk/wheat husk fillers. The average filler size used in the present study was 2–3 mm RH and 6–8 mm WH. The hybrid composites are made by one shot method. Epoxy-TDI polyurethane resin used for the preparation of hybrid composites was prepared according to above-mentioned method. Polyurethane resin was applied to two 150×150 mm^2 (12.3 g) woven jute fiber sheets with the help of a smooth brush and remaining resin was mixed with 40 g RH/WH at room temperature. Resin coated jute sheets and RH/WH filler were allowed to dry at room temperature for about 15 min. Resin coated RH/WH filler was uniformly sandwiched between two resin coated jute sheets and placed between two preheated stainless steel plates and pressed under hydraulic pressure of 30.4 MPa at 135°C for 2.5 h by using silicone spray as a mold releasing agent. Here after composites are designated as PEBCT-JRH and PEBCT-JWH.

Measurements

The tensile strength (IS : 11,298-Pt-2-87) and flexural strength (ASTM D-790-92), electric strength (IEC-243-Pt-1-88) and volume resistivity (ASTM D-257-92) measurements were made on a Universal Tensile Testing Machine, Model No. 1185 at a speed of 50 mm/min, a high voltage tester (Automatic Electric-Mumbai) in air at 27°C by using 25/75 mm brass electrodes and a Hewlett–Packard high resistance meter at 500 V DC after charging for 60 s, respectively. Moisture absorption study of PEBCT-J composite was determined by a change in mass method. For this preweighed samples were immersed in distilled water, 10% HCl, and 10% NaCl solutions at room temperature. Samples were periodically taken out from the solutions, wiped with tissue papers on both the sides, reweighed and reimmersed in the solutions. The process was carried out till equilibrium was established. Moisture absorption study of PEBCT-J composite was also carried out in boiling water. For this a preweighed PEBCT-J sample was immersed in boiling water and change in mass was determined at the interval of 1 h till the equilibrium was reached.

RESULTS AND DISCUSSION

Mechanical properties

The performance and durability of composites rely on strength and stiffness of the fibers, the strength and stability of matrix, and interfacial bond strength between fiber and matrix, fillers, compatibilizers and modifiers, fiber content, temperature, voids, dimensions, matrix material composition, fiber orientation,[36–38] etc. For engineering design, tensile properties are most widely used for quality characteristics. Bending strength and stiffness of the materials are useful for quality control and classification of materials. The test specimens were prepared by punching technique as per specified standard tests. The tensile strength (σ) of the composites was calculated by measuring the load value (W) at the point of breaking and cross-sectional area (A). A comparative tensile strength of PEBCT-J, PEBCT-JRH, and PEBCT-JWH is reported in Table I from which it is evident that tensile strength has decreased substantially upon hybridization mainly due to random orientation of RH and WH, which may not transfer stress in the direction of application. Low mechanical strength is probably also due to brittle and hard nature of the PU resin. When load is applied to the composite, flexible epoxy moiety throws it away to the rigid urethane part and consequently stress is concentrated at that point resulting in rupture at low load. PEBCT-JWH has 1.5 times more tensile strength than PEBCT-JRH composite indicating high Jute-PEBCT-JWH interfacial bond strength as compared to Jute-PEBCT-JRH interactions.

The flexural strength of the composites was determined at 50 mm span length. A comparative flexural strength of PEBCT-J, PEBCT-JRH, and PEBCT-JWH is reported in Table I from which it is clear that the flexural strength has decreased upon hybridization. PEBCT-JWH has 1.65 times more flexural strength than PEBCT-JRH indicating better stiffness of PEBCT-JWH. In hybrid composites biomass fibers are not lined up in any direction and they are just tangled mass. The composites can be made stronger by lining up all the fibers in the same direction as in woven jute fabric. Oriented fibers are strong when pulled in fiber direction but they are weak at right angle to the fiber

TABLE I
Mechanical and Electrical Properties of Composites

Composite	Tensile strength (MPa)	Flexural strength (MPa)	Electric strength (kV/mm)	Volume resistivity (10^{13}) (ohm cm)
PEBCT-J	37.4	39.6	1.3	1.40
PEBCT-JRH	9.5	12.9	1.8	1.84
PEBCT-JWH	14.7	21.3	1.9	1.91

direction. The woven fibers give a composite a good strength in many directions and under tension, the strength of the composite is entirely due to reinforcement. There are two types of interactions namely covalent and H bonding. Hydroxyl groups of lignocelluloses serve as reaction sites with various functional groups present in the resin. Thus, RH and WH both can be used as fillers as well as reactive components (polyols). Under tension, the strength of the composite is entirely due to reinforcement. The tensile strength and flexural strength of PEBCT-J are almost same but they decreased upon hybridization due to random orientation of filler fibers, which are responsible for discontinuous stress transfer and decrease in strength and stiffness of the polyurethane. The interfacial bond strength can be judged better by SEM analysis of the fractured surfaces of the specimen. Unfortunately because of lack of facilities, SEM analysis of the composites could not be performed. The lone pairs of electrons of urethane linkages form H-bonds with hydroxyl groups of jute and RH/WH fibers and hence resulted in improved interfacial adhesion. Urethane segments are hard and brittle in nature and therefore they cause lowering in mechanical strength. Thus, composites possess moderate tensile strength and some what better flexural strength indicating their usefulness for low load bearing and other applications.

Electrical properties

Electrical properties of materials are useful to designers in predicting the relative insulation quality characteristic of material selection for specific properties in combination, to access the effects of material composition and environment. Electrical properties of the composites rely on area and geometry of the electrodes, sample thickness, temperature, humidity, time of voltage application, extent of ageing, frequency of current, impurities, degree of resin cure, nature of reinforcing agents, etc. Electric strength and volume resistivity of the composites were calculated by measuring puncture voltage and volume resistance, respectively. A comparative electric strength and volume resistivity data of PEBCT-J, PEBCT-JRH, and PEBCT-JWH are reported in Table I from which it is clear that both electric strength and volume resistivity are improved 1.3–1.5 times upon hybridization indicating their usefulness as good insulating materials for various applications. H-bond formation between urethane linkages and unreacted hydroxyl groups of jute and RH/WH causes neutralization of partial charges and hence improvement in 39–46% electric strength and 31–36% volume resistivity. The improvement in electrical properties is mainly due to the nature of the matrix and reinforcing materials, fiber orientation and interfacial bond strength as well as degree of resin cure. The H bond formation and covalent bond formation between reactive functional groups of matrix and reinforcing materials caused improvement in electrical properties.

Moisture absorption

All natural fibers have a tendency to absorb moisture and possess low wettability by hydrophobic resins due to presence of hydrophilic hydroxyl groups of cellulose, hemi cellulose, and lignin.[39–42] Hemicellulose is mainly responsible for the moisture absorption in composites. Non crystalline cellulose and lignin also play an important role in moisture uptake process. Swelling of fibers continues till the cell walls are saturated with water. After the saturation point, moisture exists as free water in the void structure leading to composite defects such as delamination and void formation.[40,41] Absorbed moisture weakens the interfacial bonding, accelerates the delamination, and hence decreases the tensile properties especially tensile strength.[42] Absorbed moisture in composites causes hydrolytic degradation of resin, fibers or at an interface during service.[43,44]

The percentage moisture uptake by PEBCT-J in pure water, 10% HCl and 10% NaCl as a function of time at room temperature is shown in Figure 2 from which it is evident that the moisture uptake behavior is quite different in water, acidic and saline environments supporting different diffusion processes. The % equilibrium moisture uptake and equilibrium time are reported in Table II from which it is evident that not much difference in equilibrium moisture uptake is observed in the pure water (11.3%) and acidic (13.6%) environments, but in saline (5.5%) atmosphere it is almost half of water and acidic environments. The equilibrium time is considerably longer in water and acidic environments than that in saline

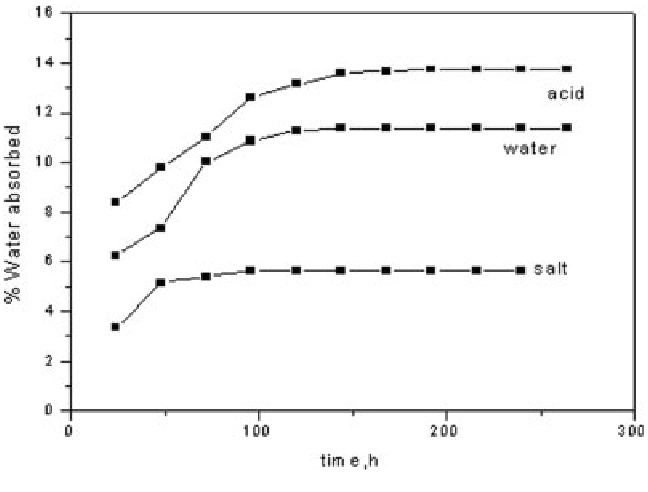

Figure 2 The plots of percentage of water absorbed against time for PEBCT-Jute composites in water, 10% salt, and 10% acid at room temperature.

atmosphere. The observed trend in moisture uptake is acid > water > salt. Thus, substantially low moisture absorption tendency of PEBCT-J in 10% NaCl supports its utility in marine applications. The moisture absorption study of hybrid composites was not done due to delamination in the above media.

Absorbed water may induce irreversible changes like chemical degradation and debonding. Cracking and blistering lead to high moisture uptake, while leaching of small molecules leads to decrease in moisture uptake.[45] Assuming one dimensional moisture uptake in semi-infinite plate exposed on both sides to the same environment,[45] the total amount of moisture uptake was determined by determining the weights of the moist and dried sample as a function of time. The amount of moisture uptake is related to diffusivity and sample thickness:

$$M = \frac{4M_m}{h}\sqrt{\frac{t}{\pi}}\sqrt{D_x} \quad (1)$$

where M_m = equilibrium moisture content, D_x = diffusivity and h = sample thickness.

In present case, the diffusivities in pure water, 10% HCl and 10% NaCl solutions were determined by determining the initial slope of percentage M versus $\sqrt{t}$ plots:

$$D_x = \pi\left(\frac{h}{4M_m}\right)^2(\text{slope})^2 \quad (2)$$

The diffusivity of PEBCT-J in different environments at room temperature is reported in Table II from which it is clear that the diffusivity in saline environment is about 3.94 times more than that in water environment and almost half in acidic environment. The different magnitudes of diffusivity in different environments indicate ionic effect on the diffusion rate. High diffusivity in saline atmosphere is probably due to electrostatic interactions between hydrated ions and polar groups present in composites. A decrease in D_x in 10% HCl is due to apparent increase in hydrated ionic size, which restricts the diffusion in cell walls. Saline solutions affect the water structure and from the present observations, it is concluded that NaCl causes breaking of water structure and hence increases the diffusivity. The moisture uptake by PEBCT-J is mainly due to presence of hydroxyl groups in jute fibers and to some extent due to solvation phenomena. The π- electrons of benzene ring form weak H-bonds in aqueous environment. The lone pairs of electrons of ether oxygen and urethane linkages also form H-bonds in water and ionic media. Hydrated ions play an important role in moisture uptake behavior.

TABLE II
Moisture Uptake and Diffusivity Data of PEBCT-J Composites at Room Temperature

Property	Water	10% HCl	10% NaCl
Equilibrium time (h)	120	144	92
Equilibrium moisture content (%)	11.3	13.6	5.5
Diffusivity, D_x, 10^{-12} (m^2/s)	2.36	1.33	9.31

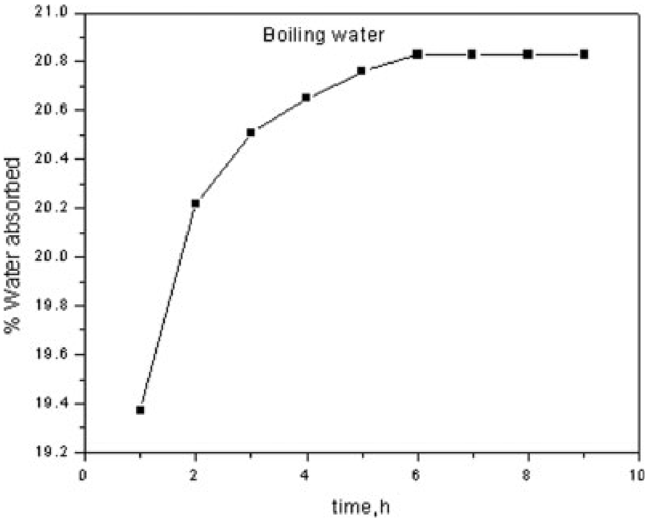

Figure 3 The plot of percentage of water absorbed against time for PEBCT-Jute composite in boiling water.

Moisture uptake in boiling water

The effect of boiling water on moisture uptake with time is shown in Figure 3 from which it is clear that the equilibrium (6 h) moisture uptake is 20.8% and beyond 6 h it remained almost constant. From Table II, it is interesting to note that the equilibrium time has reduced 15.3 times and % equilibrium moisture content has increased 1.8 times in boiling water as compared to room temperature process supporting powerful interfacial bonding. Thus, increase of temperature caused increase in moisture uptake but the equilibrium time is reduced drastically.

CONCLUSIONS

1. The tensile strength and flexural strength of PEBCT-JRH and PEBCT-JWH have decreased substantially as compared to PEBCT-J, due to random orientation of RH and WH fillers.
2. PEBCT-JWH possesses higher tensile strength (1.5 times) and flexural strength (1.65 times) as compared to PEBCT-JRH supporting high interfacial bond strength and stiffness of PEBCT-JWH.
3. Both electric strength and volume resistivity are improved upon hybridization.
4. Moisture uptake behavior of PEBCT-J is quite different in pure water, 10% HCl, and 10% NaCl

solutions. The equilibrium moisture uptake and equilibrium time in saline environment are much lower than those in water and acidic environments.

5. Equilibrium moisture uptake in boiling water has increased 1.84 times, while equilibrium time has decreased 15.3 times.

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