Jute-Fiber-Reinforced Polyurethane Green Composites Based on *Mesua ferrea* L. Seed Oil

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ABSTRACT: Two types of environmentally friendly jute-fiber-reinforced green composites based on *Mesua ferrea* L. were prepared with poly(urethane ester) and poly(urethane amide) resin blends with commercially available partially butylated melamine–formaldehyde and epoxy resins through solution impregnation and hot-curing methods. The composites were cured at a temperature of about 130–140°C under a pressure of 35 ± 5 kg/cm^2 for about 2 h. The mechanical properties, such as tensile strength, flexural strength, elongation at break, hardness, and density, of all of the composites were measured and compared. The mode of interaction between the filler and the matrix were studied by Fourier transform infrared spectroscopy and scanning electron microscopy of the fractured composite samples. The water uptake in different chemical media was observed, and we found that all of the composites possessed excellent hydrolytic stability against almost all of the media except the alkali. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were used to analyze the thermal behavior of the composites. TGA of the composites showed degradation much above that of the virgin blends, which indicated their high thermostability. The glass-transition temperatures, as shown by DSC analysis, were found to be much higher. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 115: 843–850, 2010

Key words: biofibers; blends; composites

INTRODUCTION

The replacement of traditional tough materials by natural-fiber-reinforced composite materials is gaining vast popularity because of growing environmental awareness throughout the world. These materials are emerging as realistic alternatives to the existing composites reinforced with traditional reinforcing materials, such as glass fibers, talc, and mica.[1] The main incentives for the production of natural-fiber-reinforced green composites are their easy availability and processability, light weight, high specific strength, low toxicity, low cost, and most importantly, their biofriendly nature. A lot of research works are being done in this field, particularly on jute-fiber (JF)-reinforced polyurethane composites. This is because of the structural versatility of polyurethane resins and their good compatibility with renewable lignocellulosic fibers such as jute. However, such compatibility is obtained in an efficient manner only after the modification of the surface chemistry of JFs by alkali treatment.[2,3] This results in an improvement in the interfacial adhesion by the provision of additional sites for mechanical interlocking, which promotes more fiber matrix interpenetration at the interface.[4,5] These composite materials are now in great demand because of their light weight, durability, and cost effectiveness, especially in the automotive market. These can also be successfully used to make buildings, low-cost housing units, door panels, seat frames, roofing, wood substitutes, pipes, and more.[6–9]

Polyurethane, a versatile resin, exhibits the advantage of being produced either from nonrenewable petroleum oil or from renewable vegetable oils. Recently, emphasis has been given to the development of polyurethane resins from vegetable oils because of several environmental concerns and also because of the rising cost of petroleum-based feed stocks.[10–14] One such renewable vegetable oil available in the north eastern region of India is *Mesua ferrea* L. seed oil, which contains about 70% nonedible oil.[15] Polyester,[16] poly(ester amide),[17] and polyurethane resins[18,19] have been successfully synthesized from this vegetable oil. Further, the performance characteristics of polyurethane resins based on *M. ferrea* L. seed oil can be enhanced to a considerable extent by blending with commercially available

resins such as melamine–formaldehyde (MF) and epoxy resins.[20,21] These blends could be fabricated into composites by reinforcement with naturally renewable vegetable fibers because of the possible reaction of various functional groups present in the polymer backbone of the polyurethane blends with the hydroxyl groups of the fibers.[22] Such reactions considerably improve a number of properties, such as hardness, mechanical strength, impact strength, and dimensional stability.[23]

The main objective of this study was to develop a new kind of green composite material consisting of MF and epoxy-modified polyurethane blends based on *M. ferrea* L. seed oil as the matrix reinforced with renewable JFs. Attempts were also made to characterize the developed composites by various techniques and to compare the performance characteristics of the composites by the evaluation of the physical, mechanical, chemical, and thermal properties.

EXPERIMENTAL

Materials

M. ferrea L. seeds were collected (Jorhat, Assam, India) and were used to extract the oil by the solvent extraction method. Metallic sodium, *n*-hexane, sodium hydroxide, diethyl ether, diethanolamine, anhydrous sodium sulfate, xylene, poly(ethylene glycol) (number-average molecular weight = 200 g/mol), glycerol, *para*-toluene sulfonic acid, and lead monooxide were used (Merck, Bombay, India) as received. MF resin was used as obtained as a gift sample from Asian Paints India, Ltd. (Mumbai, India), and the technical specifications of this resin were reported earlier.[20] Bisphenol A based epoxy resin and poly(amido amine) hardener (Ciba Geigy, Bombay, India) were used as supplied, and the technical specifications were reported earlier.[21] Toluene diisocyanate and dibutyl tin dilaurate (Merck, Schuchardt, Germany) were used without further purification. The JFs used in this study as reinforcing agents were collected from the local market (Napaam, Tezpur, India).

Methods

Step 1: Preparation of the polyurethane [poly(urethane ester) (PUE)/poly(urethane amide) (PUA)] and MF resin blends

Both the polyurethane resins (PUA and PUE) with NCO/OH ratios of 0.5 were prepared as reported earlier[18,19] with toluene diisocyanate (0.75 mol) and fatty acid diethanolamide (1 mol) or monoglyceride (1 mol) of *M. ferrea* L. seed oil along with poly(ethylene glycol) (0.5 mol) as the chain extender, dibutyl tin dilaurate (0.05 wt % with respect to total diol) as

TABLE I
Specification for the Composites

Sample code	Weight percentage (g)		
	Polyurethane	Epoxy	MF
EM50/JF50[a]	50 (PUE)	50	—
EA50/JF50[a]	50 (PUA)	50	—
MM30/JF50[a]	70 (PUE)	—	30
MA30/JF50[a]	70 (PUA)	—	30

[a] Indicates 50% JF loading with respect to the total resin content.

the catalyst, and xylene as the solvent. The blend solutions were prepared by the mixture of PUE and PUA resins (40% solid content in xylene) with MF resin (60% solid content in xylene/butanol = 90/10 by volume) separately in the presence of *para*-toluene sulfonic acid as an acid catalyst (0.5 wt % with respect to total resin) in the desired proportions, as shown in Table I. The mixing was carried out with constant vigorous stirring for about 0.5 h at 30 ± 1°C.

Preparation of the polyurethane (PUE/PUA) and epoxy resin blends. The resin blend solutions were prepared by the mixture of PUE- and PUA-based resins (30 ± 5 wt % solid content in xylene) with epoxy resin (100% solid content) separately and the poly(amido amine) hardener (50 wt % with respect to epoxy) in the desired proportions, as shown in Table I. The mixing was carried out with constant vigorous stirring for about 0.5 h at 30 ± 1°C.

Step 2: Surface modification of the JFs by the alkali treatment method

JFs with a density of about 1.5×10^3 kg/m³ as obtained from the local market were first chopped uniformly into small pieces about 1 cm in length. The well-chopped fibers were scoured with a 2% hot detergent solution and washed thoroughly with distilled water; they were then dried in a vacuum oven at 70°C for a sufficient duration of time. The dried fibers were dispersed in 2% NaOH solution for about 2 h with frequent stirring and then washed with distilled water several times to leach out the absorbed alkali. The fibers were immersed in distilled water overnight and again washed repeatedly to remove any trace amounts of alkali. Finally, the alkali-treated fibers were made free of water by drying under the sun and in a vacuum oven at about 60–70°C for 24 h and then stored at ambient temperature in a desiccator.

Step 3: Composite fabrication

The well-dried alkali-treated JFs in desired proportions were introduced into the dilute solutions of the

MF and epoxy-modified *M. ferrea* L. seed oil based polyurethane (both PUE and PUA) blends by a hand layout technique in a big tray. The compositions of the four resulting composites are given in Table I, where EM and EA represent epoxy-modified PUE and PUA, respectively, and MM and MA represent MF-modified PUE and PUA, respectively. After the removal of a sufficient amount of solvent under atmospheric conditions, the polymer-impregnated fibers were placed into a mold $26 \times 28 \times 0.3$ cm^3 in size. The molds were then subjected to hot curing by compression in a hydraulic press at about 130–140°C under a pressure of 35 ± 5 kg/cm^2. The molded articles were taken out after a minimum of 4 h, and thick plates of composites with various compositions were obtained. All of the composite samples were stored for 48 h at room temperature before testing.

Water absorption and chemical resistance tests

Water absorption and chemical resistance tests were done as per the ASTM D 570 method. The samples were immersed in different media for 10 days, and weight change readings were taken every 48 h. The percentage weight gain was calculated with the following mathematical equation:

$$\text{Weight gain}(\%) = (W_f - W_i)/W_i \times 100\%$$

where W_i is the weight of the composite specimen before immersion and W_f is the weight of the composite specimen after immersion.

Instruments and testing methods

We fabricated the composites into thick sheets by pressing in a steel mold with a volume of $26 \times 28 \times 0.3$ cm^3 in a hydraulic hot press (ISO 9001:2000 certified) from Peeco Hydraulic Pvt., Ltd. (Kolkata, India). Fourier transform infrared (FTIR) spectra of the postcured composite samples were recorded in a Nicolet FTIR Impact 410 spectrophotometer (Madison, WI, USA). Small quantities of the fine-powder composite samples were dispersed in KBr and further ground into a fine mixture in a mortar before they were pressed to form transparent KBr pellets for analysis. Thermogravimetric analysis (TGA) of the cured composites was done with a Shimadzu TG 50 thermal analyzer (Tokyo, Japan) under a nitrogen flow rate of 30 mL/min at a heating rate of 10°C/min from 50 to 600°C. Differential scanning calorimetry (DSC) analyses were performed in a Perkin-Elmer Pyris differential scanning calorimeter (Boston, MA, USA) at a heating rate of 10°C/min from 25 to 400°C under a nitrogen flow rate of 20 mL/min. The tensile strength, flexural strength, and elongation at break of the composite specimens were measured with a Zwick Z010 universal testing machine (Germany) with samples with dimensions of $10 \times 1.0 \times 0.3$ cm^3. These mechanical properties, along with the thickness, were measured on five specimens from each sample of composite to avoid irregularity. The surface morphology of the fractured tensile test samples were studied with a Jeol scanning electron microscope (Peabody, MA, USA) (model JSM-6390LV SEM) after the sample surfaces were platinum-coated. The specific gravity, water absorption, impact resistance, hardness, and chemical resistance of the composite samples were determined according to standard methods.[24–26]

RESULTS AND DISCUSSION

FTIR study

The completion of curing of the resins was confirmed for all of the composites by the disappearance of the oxirane ring (band at 827 cm^{-1}) in the structure of the epoxy-based composites and an increase in the intensity of the ether linkage (band at 1225–1232 cm^{-1}) in both the cases in the FTIR spectra (Fig. 1).[27,28] As also shown in the figure, there was no considerable difference between the spectra of the virgin blends[20,21] and those of the jute-reinforced composites, which confirmed that the curing mechanism remained almost the same. However, the absorption peak at about 1730–1740 cm^{-1} due to carbonyl stretching broadened a little in the composites as compared to the virgin blends. This indicated that more carbonyl groups of the composites were involved in H-bond formation. Furthermore, the broadness of the band at about 3410–3430 cm^{-1} remained almost the same because of two contradictory effects. The reaction of the hydroxyl groups of the polyurethane/epoxy/MF with the hydroxyl groups of the fibers caused sharpening of the bands, whereas H-bonding caused broadening of the same bands.

Morphological study

Figure 2 shows the scanning electron micrographs of the fractured surfaces of the jute-reinforced polyurethane composites at two different magnifications. Despite the more rough surface morphology observed in the scanning electron microscopy (SEM) micrographs of the alkali-treated JFs compared to untreated one, alkali treatment improved the interfacial bonding by providing additional sites for mechanical interlocking, which resulted in better wetting of the fibers by the matrix.[4,5,29] The voids, as observed in the SEM micrographs (Fig. 2), were created by the pullout of the fiber during fracture. More fiber was pulled out in the MF-modified
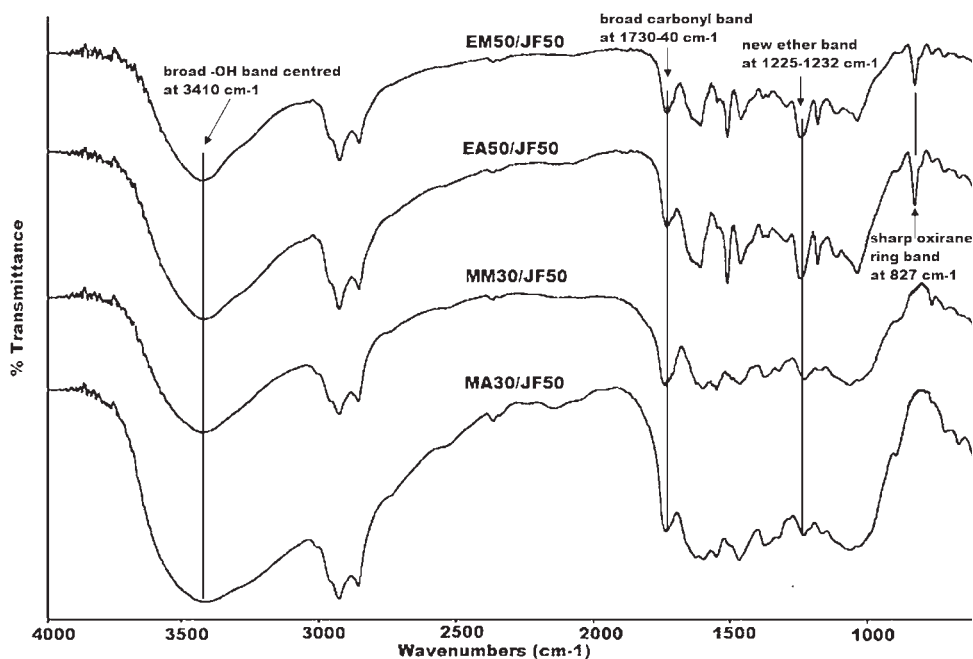
Figure 1 FTIR spectra of the composites.

polyurethane-based composites, MM30/JF50 and MA30/JF50 [Fig. 2(c,d)], because of the lower level of polymer–filler adhesion, than in the case of the epoxy-modified polyurethane-based composites, EM50/JF50 and EA50/JF50 [Fig. 2(a,b)]. This was also reflected in the low tensile and flexural strength values in the former cases. A reduction in the amount of fiber pulled out in the latter case was due to the continuous stronger interfacial interactions between the polymer and fiber [Fig. 2(a,b)], as evident from the cracks at the broken fiber ends. It was obvious, as the interactions between the cellulosic fiber and epoxy-modified polyurethane were significant because of the improved flexibility of the matrix. These results indicate the active role of the JF as a reinforcing agent and modified *M. ferrea* L. seed oil based polyurethane resin as matrix for the green composites.[30]

Mechanical and physical properties

Composites with satisfactory mechanical properties were obtained only when there was good interfacial interaction between the polymer matrix and the fibers. As the alkali treatment of JFs causes delignification that results in significant roughness of the surface and that encourages strong fiber–matrix adhesion, only alkali-treated JFs were used as the reinforcing fiber in this investigation. The reinforcing effect of the JFs could be understood from the improved interfacial interactions between the hydroxy of the cellulosic JFs and the epoxy/hydroxy

of the matrices through H bonding, polar–polar interaction, and so forth.[2,3] The tensile and flexural strengths were better for the epoxy-modified polyurethane-based composites, EM50/JF50 and EA50/JF50, than for the MF-modified polyurethane-based composites, MM30/JF50 and MA30/JF50. This was because of enhanced interfacial adhesion (also supported by the SEM study as discussed earlier), which resulted in a better transfer of stress from the matrix to the fibers in the former case as compared to the latter. Interfacial adhesions are dependent on partial chemisorption (covalent and H-bonding interactions) of the matrix on the surface of the fibers. Thus, there was discontinuous stress transfer from the matrix to the fiber in the MF-modified polyurethane-based composites, MM30/JF50 and MA30/JF50, which resulted in low tensile and flexural strengths, particularly in the latter case, because of a lesser extent of compatibility between the fiber and the rigidity of the matrix, which caused random orientation with of the sandwiched JFs.[31,32]

The hardness values (Shore A) found for all of the composites are given in Table II. The EA50/JF50 composite exhibited the highest hardness ($\sim$ 95), and the MA30/JF50 composite showed the lowest ($\sim$ 55) hardness. The results of hardness were explained by the fiber–matrix interactions of the composites and their compatibility. The higher hardness of the EM50/JF50 and EA50/JF50 composites as compared to the MF-based composites was due to better compatibility between the matrix and fiber, which arose from the presence of a flexible epoxy moiety. The increase in uniform crosslink density
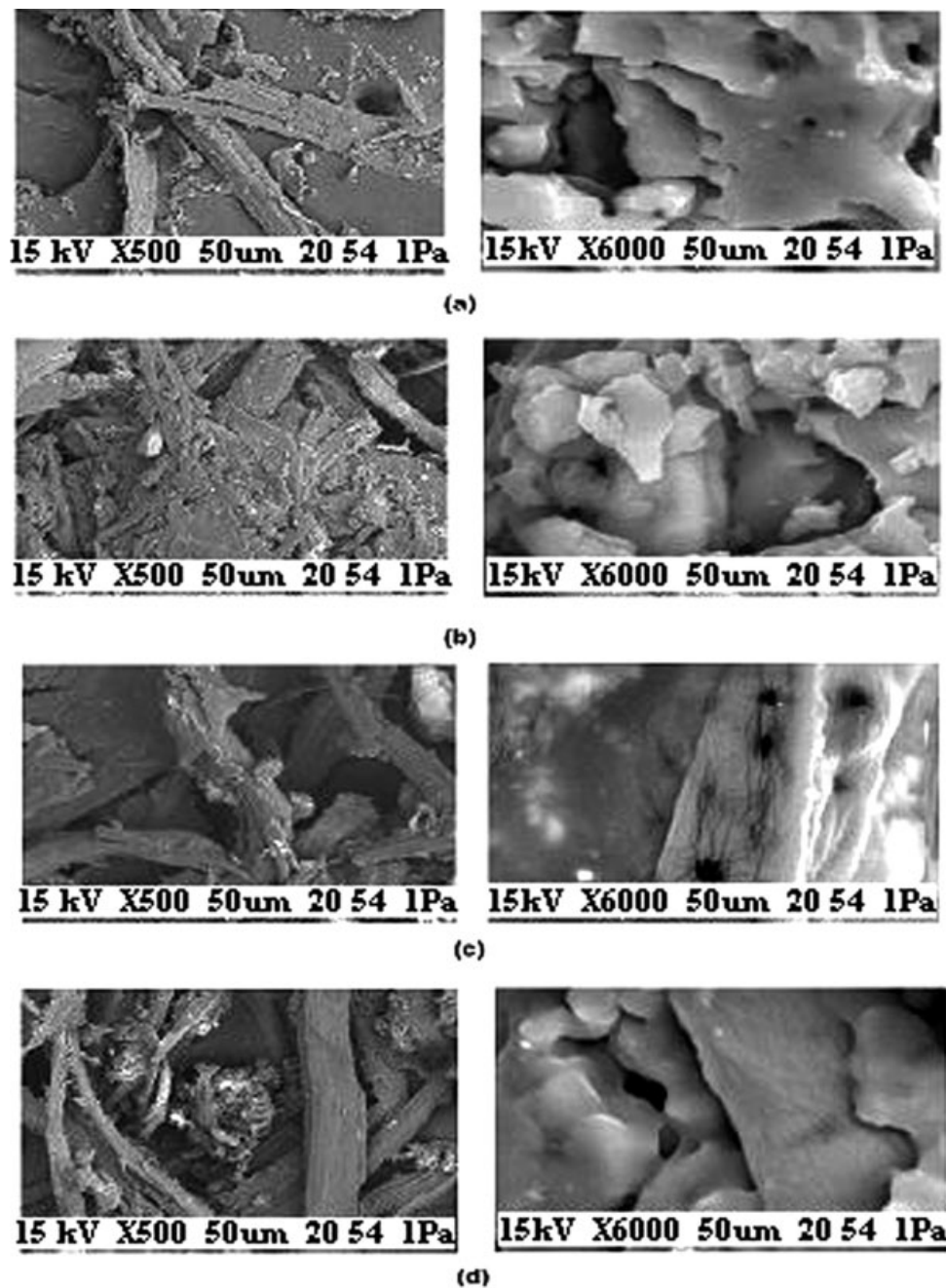
Figure 2 SEM micrographs of the composites at resolutions of 500 and 6000×: (a) EM50/JF50, (b) EA50/JF50, (c) MM30/JF50, and (d) MA30/JF50.

also increased the hardness in the epoxy-based composites compared to the MF-based composites. However, between the two MF-modified polyurethane-based composites, MM50/JF50 and MA50/JF50, the hardness value was found to be considerably higher for MA50/JF50. This was because of improved polymer–filler adhesion and distribution of polymer in the fibers.

Because of the lower density in the alkali-treated JFs, both epoxy- and MF-modified *M. ferrea* L. seed oil based polyurethane composites were found to possess low densities (Table II). Thus, the synthe-

sized composites could play a crucial role as lightweight commercial composite materials.

Thermal properties

As shown in the DSC thermograms (Fig. 3), the composites possessed high glass-transition temperatures (T_g's; marked by arrows). The lower T_g values (184–188°C) obtained for the MF-modified polyurethane-based composites compared to those of the epoxy-modified polyurethane-based composites

TABLE II
Mechanical and Physical Properties of the Composites

Property	EM50/ JF50	EA50/ JF50	MM30/ JF50	MA30/ JF50
Tensile strength (MPa)	32.4	36.0	15.6	4.0
Flexural strength (MPa)	42.6	50.3	29.2	9.75
Break load (MPa)	39.9	45.1	29.8	14.5
Elongation at break (%)	12.0	16.1	6.6	0.9
Hardness (Shore A)	93	96	85	55
Specific gravity (g/cm^3)	1.16	1.10	0.88	1.02

Figure 4 TGA thermograms of the composites.

(T_g = 195–206°C) could partly be attributed to the plasticizing effect of the dangling polymeric chains, that is, entanglement, which were not included in the network during crosslinking and left dangling.[33] This may also have been due to the phase separation of the hard and soft domains due to the incompatibility of the MF resin blend with JF, which was greater in the MA30/JF50 composite (Fig. 2). High T_g's and stiffness values of the composites hinted at good fiber dispersion, efficient wetting, and good fiber matrix adhesion.[34] All of the composites were stable up to temperatures greater than about 200°C, as evaluated by TGA (Fig. 4). Furthermore, degradation up to about 600°C occurred in a three-step process similar to that of the virgin blend. It was evident from the thermograms (Fig. 4) that the incorporation of JFs into both the epoxy- and MF-modified polyurethane blend matrix enhanced the thermal stability to a considerable extent compared to the virgin
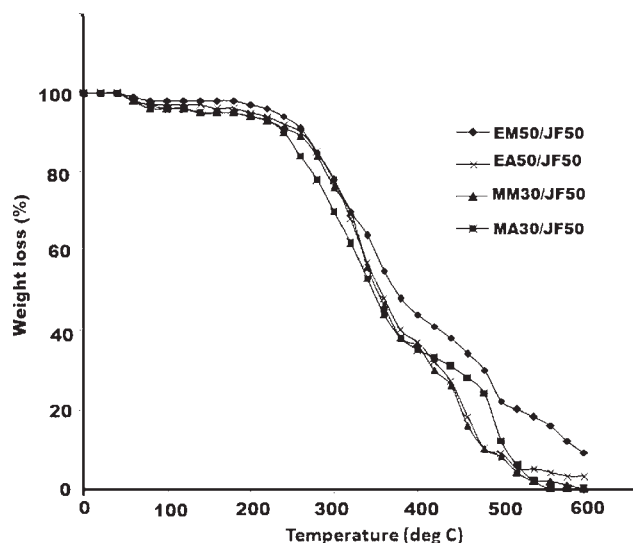
blends.[20,21] This may have been due to the improved fiber–matrix interfacial adhesion.

Water absorption and chemical resistance studies

The water absorption results are represented pictorially in Figure 5. All of the composites absorbed water to a measurable extent, which may have been due to the hydrophilic nature of the JFs. The cell walls of JFs contain hydroxyl and other oxygen containing groups, which attract water molecules through H bonding.[35,36]

From Figure 5, the percentage water absorption was found to be in the following order: EA50/JF50 < EM50/JF50 < MM30/JF50 < MA30/JF50. The lowest water absorption in EA50/JF50 again con-
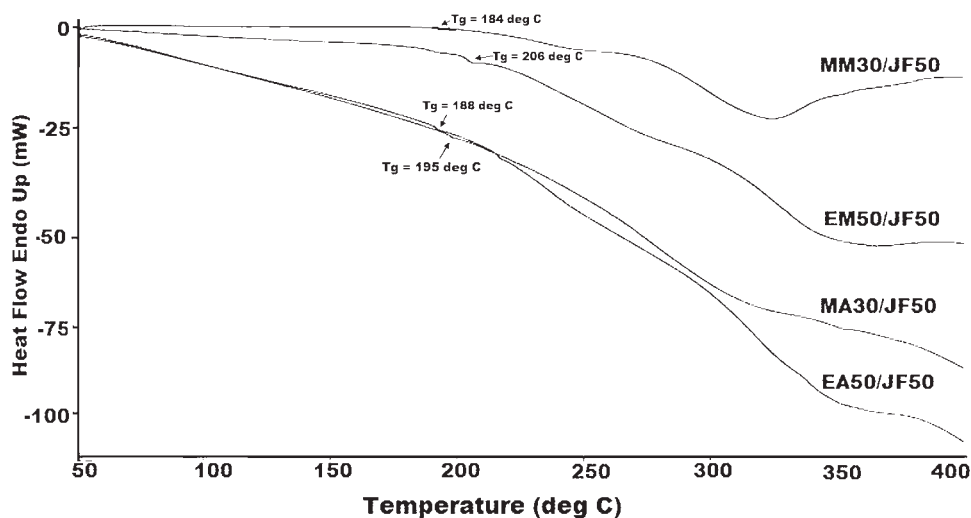

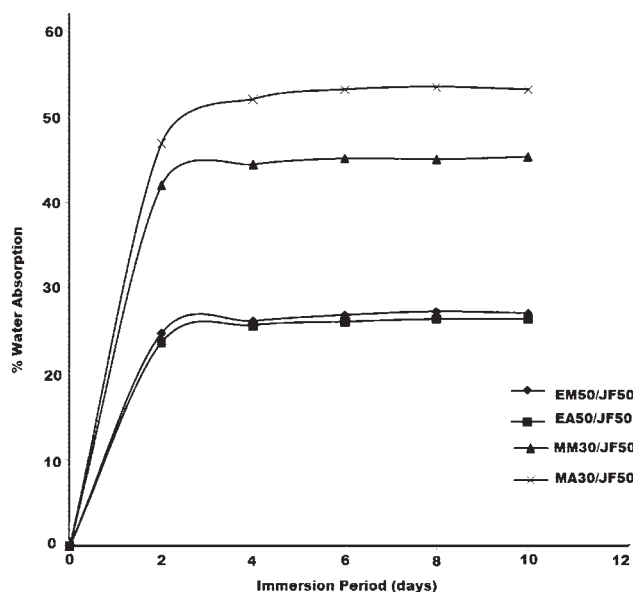**Figure 3** DSC thermograms of the composites.

Figure 5 Variation of the water absorption of the composites with time.

firmed its better fiber–matrix adhesion. Because of the greater compatibility of the matrix with the fibers in the epoxy-modified polyurethane-based composites EM50/JF50 and EA50/JF50, the fibers got fully masked with the matrix, which caused stronger adhesion and greater hydrophobicity and, hence, showed lesser water absorption. The main factors responsible for the poor water resistivity of the MF-modified polyurethane-based composites MM30/JF50 and MA30/JF50 were weak fiber matrix adhesion, agglomeration of the JFs, and incomplete encapsulation of the matrix by the fibers. Figure 5 also depicts the water absorption of the samples as a function of time. The percentage water absorption after 2 days was quite high because of the porous

structure of the JFs, which allowed transportation of water through their capillaries into the gaps and flaws at the interfaces between the fibers and matrix. After day 2, the rate of water absorption became slow. Immersion in water caused debonding between the matrix and the fibers as the time progressed. The absorption of water reached a maximum value after 6 days and then remained almost unchanged as observed for up to 10 days.

The results of the chemical resistance of the composites in aqueous acidic (10% HCl), basic (3% NaOH), and salt (10% NaCl) environments are shown in Table III. The observed trend in weight gain for all of the composites was NaOH > HCl > NaCl. The results of weight gain were due to the absorption of water by the composites as a whole and cellulosic fiber in particular. The loss in weight as observed after 4 days for the composites in NaOH solution and after 6 days in HCl solution may have been due to the presence of acid/alkali hydrolysable ester groups in all of the composites. Also, with such irreversible reactions (chemical degradation by ester hydrolysis), the other factors that have caused this type of observation were cracking and leaching. The fiber ends and the fiber matrix interfaces provided easy routes for the cracks to grow. Such cracking caused more absorption of water, whereas leaching caused a decrease in weight.[37]

CONCLUSIONS

The results for the JF-reinforced *M. ferrea* L. seed oil based green composites proved that the primary requirements for composite materials with sound mechanical properties were the good dispersion and wetting of fibers in the polymer matrix. The

TABLE III
Chemical Resistance of the Composites

Medium	Duration (days)	Weight gain (%)			
		EM50/JF50	EA50/JF50	MM30/JF50	MA30/JF50
3% NaOH	2	34.5	39.9	46.0	55.6
	4	36.2	40.7	47.1	55.7
	6	34.4	40.1	47.0	53.6
	8	32.1	36.2	44.4	52.1
	10	30.4	34.1	44.3	51.7
10% HCl	2	23.5	25.0	30.7	47.2
	4	23.6	25.6	31.6	48.9
	6	23.8	25.5	31.9	49.0
	8	23.6	24.7	32.0	46.1
	10	23.4	24.6	31.1	46.2
10% NaCl	2	20.6	20.9	32.0	37.1
	4	21.4	22.0	32.9	39.9
	6	21.7	22.6	33.6	40.4
	8	22.3	22.8	33.7	40.6
	10	22.3	22.7	33.6	40.6

properties were shown to be dependent on the nature of the matrix. The physical, mechanical, and chemical properties were better for the epoxy-modified polyurethane composites than for the MF-modified ones. They also possessed excellent chemical resistance and hydrolytic stability in water, acid, and salt solutions, which will make them useful for low-load-bearing applications. Thus, from this study, we concluded that cost-effective and environmentally friendly value-added composite materials can be obtained from epoxy- and MF-modified *M. ferrea* L. seed oil based polyurethane and locally available JFs.

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