

Novel Low-Cost Jute–Polyester Composites. III. Weathering and Thermal Behavior

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ABSTRACT: Jute–polyester composites were fabricated with untreated (control) and bleached slivers with 60% loading of fiber by weight and were designated as JPH(C) and JPH(B), respectively. Both types of composite specimens were subjected to water absorption and outdoor weathering tests to assess their relative performance under environmental conditions. While both composites showed low water absorption, JPH(B) showed lesser water absorption (8.48%) than did JPH(C) (12.25%). The mechanical properties like tensile and flexural strengths were measured for both the weathered and unweathered specimens and compared. The tensile strength of JPH(C) and JPH(B) decreased while the tensile modulus increased after weathering. The flexural strength, moduli, and ILSS of the weathered specimens were less than those of the unweathered ones. The nature of the fiber–matrix adhesion could be established from these results. The cause of every observation is explained. Thermal analyses (TG/DTG and DSC) of the composite specimens were also done. The overall thermal stability of JPH(C) was found to be better than that of JPH(B). © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 78: 1671–1679, 2000

Key words: jute–polyester composites; outdoor weathering; water absorption; thermal characterization

INTRODUCTION

Jute fiber has received considerable attention for its diversified use as a potential reinforcement in polymer composites both in academic and industrial research.^{1–6} However, these composites encounter dimensional changes during consumer and industrial use, especially in humid and hot

environments, because jute fiber is highly hydrophilic in nature and is not highly compatible with the hydrophobic organic matrices like polyester and epoxy. They age rapidly at moderately high temperature conditions, experienced particularly in most tropical and equatorial regions. The poor wettability of jute with organic matrix resins^{7–14} is responsible for the poor environmental performance of jute composites. The presence of hydroxyl and polar groups in various constituents of jute accounts for its high moisture regain value,⁴ leading to poor adhesion with the resin. Swelling

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of fibers occurs due to moisture absorption and this continues until the cell wall is saturated with water. Beyond the saturation point, moisture exists as free water in the void structure, leading to composite defects like delamination and void formation. Important properties of jute composites like delamination resistance and dimensional tolerance are, hence, affected by these environmental factors.

Therefore, to develop jute composites with better environmental performance under different temperature conditions, it is necessary to decrease the hydrophilicity of the fibers by chemical modifications like mercerization, bleaching, or grafting with various organic polymeric materials and surface coating with various interfacial agents. Such chemical modifications of jute not only are expected to decrease water absorption but also to increase wettability of the fiber with resin and improve interfacial bond strength, both of which are critical factors for the enhancement of the mechanical, weathering, and thermal performance of the composites. Bleaching of jute fiber with sodium chlorite has been carried out^{15,16} to decrease its hydrophilicity. Philip^{17,18} and the Fabric Research Laboratory¹⁹ reported the weathering of jute–polyester laminate exposed to water. They found that there is a loss of mechanical strength upon immersion in water/sea water. They suggested that this loss could be minimized by use of a glass fiber surface layer, gel coat, and edge protection. In other words, such manipulations could restrict water penetration into the composites. The work on natural fiber–glass hybrid composites with polymer matrices has gained importance in recent years with a view to decrease the water uptake of the composites and, hence, reduce the extent of environmental degradation and aging. Studies have been carried out on the moisture-absorption characteristics of jute composites based on polyester and epoxy resin systems by Rao et al.,²⁰ who reported that the rate of moisture absorption increased with increase in the fiber volume fraction. Accelerated weathering was carried out by Shah and Lakkad²¹ for the hybrid composites of jute/glass with epoxy and polyester matrices. Reduction in mechanical properties was also observed in this case. For better fiber–matrix interaction, multifunctional resins like polyesteramidepolyol (PEAP) have been used as suitable interfacial agents having compatibility with the fiber and the resin matrix. Mukherjea et al.⁹ used PEAP as an interfacial agent to study both the natural and

accelerated weathering performance of epoxy and polyester composites using jute fibers with nearly unidirectional as well as random orientation. The improvement of the flexural strength and water-uptake behavior in the PEAP-treated composites was justified on the basis of hydrogen bonding between the hydroxyl group of cellulose and PEAP.

Surface modification of jute fiber has also been achieved with phenol–formaldehyde resin and this modified jute has been used for preparing composites with polyester resin by Ghosh and Ganguly.²² Such composites were found to exhibit greater weathering resistance as they could retain better mechanical properties after exposure to water and moisture for different periods. Besides the use of interfacial agents, grafting was also found to be an effective method for the improvement of weathering properties^{22,23} of jute–polyester composites. Although a large number of reports on the water-uptake and moisture-absorption properties of jute–polyester and jute–epoxy composites are available, very little information could be found in the literature on the outdoor weathering behavior of composites. Information on the long-term behavior of the composite material is more essential to the manufacturer than are the routine laboratory tests of the composites after fabrication. In this article, we report the long-term outdoor performance of jute–polyester composites prepared in a very typical method developed by us. Besides this, we also report the thermal properties of the composites.

Very little work has been reported on the thermal stability of jute composites in the literature. Rana et al.⁴ investigated the thermal analysis of untreated (control) and chemically modified jute fibers, while thermal characterization of jute composites was studied by Mitra et al.²⁴

An attempt was made in this report to study the outdoor weathering behavior, water-uptake capacity, and thermal stability of a polyester composite reinforced with untreated (control) and bleached jute fibers designated as JPH(C) and JPH(B), respectively. The extent of water absorption, outdoor weathering behavior, and thermal properties (TGA/DTG and DSC) of both types of composites were analyzed and compared.

EXPERIMENTAL

Chemical Modification: Bleaching

Jute fibers in the form of slivers were obtained from the Indian Jute Industries Research Associ-

ation (IJIRA) (Calcutta, India). Jute fibers are changed into sliver form through an operation called carding, which opens up the mesh structure of jute reeds and makes the fibers orient almost unidirectional in a very compact form. These slivers, available with an approximate breadth and thickness of 15 and 1 cm, respectively, and of continuous length, can be cut to a desired length and can directly be used for fabricating laminates after impregnating with the plastic resin.

Chemical modification of jute sliver was done by bleaching or delignification with sodium chlorite following the method of Guha Roy et al.¹⁵ The extent of delignification achieved through the method followed was limited to 75% of the total lignin content of the fiber.

Composite Preparation

Composites were prepared separately with untreated (control) and bleached slivers as follows. General-purpose unsaturated polyester resin (GP fiber bond 333) having a viscosity of 0.5–0.6 Pa s at 30°C and specific gravity of 1.12–1.13, obtained from Ruia Chemicals (Calcutta, India) was used as the matrix resin. Ultrapure-grade benzoyl peroxide (BPO) obtained from Merck was used as the curing agent.

Jute slivers (both control and bleached) were oven-dried at 100°C for 1 h to remove moisture. Sliver pieces, cut into an approximate length of 60 cm, were impregnated with a dilute solution of polyester resin in acetone with 2% BPO in it. Four such dried prepregs were pressed into the laminate at 160°C and 4.35 MPa pressure for 5 min in a semiautomatic hydraulic press so as to obtain a laminate having 60% loading of fiber by weight and thickness of 3 ± 0.2 mm. The two varieties of composites prepared from the control and the bleached slivers were designated as JPH(C) and JPH(B), respectively. The letters J, P, and H stand for Jute Polyester Hot-cured while (C) and (B) represent Control and Bleached sliver, respectively. Further details on the preparation and the optimization of fiber loading for achieving the highest mechanical properties can be found in our published work.¹⁶ Since this is beyond the scope of this communication, we present here the preparative method very briefly. It may be recalled here that the best mechanical properties of both composites were obtained at the fiber loading of 60% by weight. So, we have taken this composition of the composites for our study on outdoor weathering, water uptake, and thermal behavior.

Water Absorption

The water-absorption test of the composite specimens was carried out as per the ASTM D-570 method. Rectangular bars of length 76.2 mm and width 25.4 mm were cut from the laminates longitudinally along the direction of the fiber axis. A protective gel coat (araldite) was applied on the cut sides to prevent penetration of water from the cut sides. The samples were conditioned for 24 h at $50 \pm 3^\circ\text{C}$ and then cooled in a desiccator. The weight of the samples (w_1) after conditioning were then recorded. The samples were immersed in distilled water for 24 h at 23°C. The samples were then removed from the water, dried by pressing in a cotton cloth, and weighed. This gave the wet weight of the sample (w_2). The samples were reconditioned again for 24 h at $50 \pm 3^\circ\text{C}$ in a dried condition and reimmersed in distilled water again. The samples were taken out of the water after the test period, dried, and reweighed (w_3). This gave the final weight of the sample after the removal of soluble matter, if any, in the composite. The following relations were used:

$$\text{Percent increase in weight due to water absorption} = (w_2 - w_1)/w_1 \times 100$$

$$\text{Percent of soluble matter lost} = (w_1 - w_3)/w_1 \times 100$$

$$\text{Percent of water absorption} = [(w_2 - w_1) + (w_1 - w_3)]/w_3 \times 100 = (w_2 - w_3)/w_3 \times 100.$$

Although the percent increase in weight after first immersion is commonly taken as the percent of water absorption by the specimen, ASTM-D-570 defines water absorption as the sum of the increase in weight after the first immersion and of the weight of the water-soluble matter.

Outdoor Weathering

The composite specimens were subjected to natural outdoor weathering for 45 days to assess their relative environmental performance as per the ASTM-D-1435 standard. The weathering study was carried out in the month of June with an average temperature and relative humidity of 38°C and 90%, respectively. The samples also experienced substantial rainfall during the test period. The test specimens were mounted on the plate holders and positioned at a 45° angle facing the equator. The specimens were removed from the racks after 45 days and subjected to tensile

Table I Water-Absorption Test

Sample Specimen	Immersion Time (h)	W_1 (g)	W_2 (g)	W_3 (g)	Increase in Weight (%)	Soluble Matter Lost (%)	Water Absorption (%)
JPH(C)	24	4.654	5.152	4.582	10.70	1.547	12.247
JPH(B)	24	4.650	5.011	4.617	7.77	0.710	8.480

and flexural tests. These results were compared with the corresponding values of the unweathered specimens, which were kept carefully inside a desiccator at room temperature to prevent the samples from outdoor weathering effects.

Thermal Analysis

TG/DTG and DSC analyses of the composite specimens were carried out using a Mettler TG 50 and DSC 25 module attached to a Mettler TC 11 4000 thermal analyzer. All the measurements were made under a nitrogen flow (150 mL per min), keeping a constant heating rate of 10°C per min and using an alumina crucible with a pinhole.

RESULTS AND DISCUSSION

Water Absorption

Table I gives the percentage of water absorption by JPH(C) and JPH(B) specimens in which the data given for each test are the average of five specimens. Both the percentage increase in weight after the first immersion and percentage of water uptake after the second immersion were found to be low, of the order of 10%. Mitra et al.²⁵ reported water absorption as high as 40% for normal jute–polyester composites using a hand layup technique. In the face of such a high value of water uptake reported in the literature, our results look very encouraging. From these results, it seems that interfacial adhesion in our case is much higher than in the earlier reports. This high

interfacial adhesion which restricts the percent of water uptake in our case is likely due to the following reasons: We used the compression-molding technique involving a high-pressure and high-temperature curing method with an adequate degassing procedure prior to molding to prepare the composites. This must have minimized the extent of void formation during processing although complete elimination of voids and their quantitative estimation in the composite specimens is practically impossible. Mitra et al.²⁵ used the hand layup technique for the preparation of composite specimens and it is very likely that a large number of voids must have been introduced during processing. These voids might have been the potential cause of debonding, which results in a very high water uptake in their case. JPH(B) was found to absorb a little less water (8.48%) than did JPH(C) (12.25%) in our case. This may be due to greater interfacial bonding in JPH(B) than in JPH(C).

Although bleaching removes a large quantity of lignin from jute and decreases the polarity of the bleached fiber, the surface roughness and microporosity²⁶ of the fiber are greatly increased. This results in good mechanical anchorage and interlocking of the matrix, with the bleached fiber imparting greater adhesion. This is corroborated by the fact that while the untreated and the bleached fibers have nearly equal water absorption (70%), which was confirmed by separate blank experiments, the composite from the bleached fiber, that is, JPH(B), was found to have less water absorption than did JPH(C). The in-

Table II Tensile Properties of JPH(C)

Sample Specimen	Nature of the Sample	Ultimate Stress (MPa)	Ultimate Strain (%)	Toughness (MPa)	TEA (N/mm)	Modulus (GPa)
JPH(C)	Weathered	88.00	5.417	2.718	8.775	3.639
JPH(C)	Control (unweathered)	132.40	5.834	3.608	9.767	2.956

Table III Tensile Properties of JPH(B)

Specimen	Nature of the Sample	Ultimate Stress (MPa)	Ultimate Strain (%)	Toughness (MPa)	TEA (N/mm)	Modulus (GPa)
JPH(B)	Weathered	78.15	4.460	1.898	6.075	3.902
JPH(B)	Control (unweathered)	117.00	6.677	3.764	10.6	2.106

creased interfacial adhesion is partly due to the chemical reaction (chemisorption) taking place between the OH groups of fiber (cellulose) and COOH end groups of the unsaturated polyester resin in both the composites.¹⁶ Since JPH(B) possesses more condensable OH groups than does JPH(C), the extent of chemical bonding is expected to be higher in the former. This contributes to lesser water uptake in JPH(B). The percentage of soluble matter lost is more in the case of JPH(C) than of JPH(B). This is most likely because the untreated (control) fibers from which the JPH(C) was prepared contain a large amount of noncellulosic and nonligneous materials, which are, absent in bleached fibers from which JPH(B) was made. It is owing to this reason that JPH(C) loses more soluble matter after the water absorption test than does JPH(B).

Weathering Studies

Tables II and III give comparative data of the tensile properties of JPH(C) and JPH(B) both before and after weathering. The data given are the average of five samples in each case. As very much expected, the tensile strength, percent strain, toughness, and tensile energy absorption (TEA) of the weathered samples in both cases are less than those of the respective unweathered ones. Tensile strength decreases from 132.4 to 88 MPa for JPH(C) and from 117 to 78.15 MPa for JPH(B). The moisture absorbed by the composites weakens the interface due to void formation induced by water in the presence of heat and light

during the exposure. The moisture acts like a plasticizer for accelerating delamination and, consequently, decreases the strength of the composites. Another interesting but surprising observation was recorded in the tensile modulus values of the specimens. The moduli for the weathered samples were found to be greater than their unweathered counterparts (Table II). This might be due to increase in the stiffness of the material resulting from the chain scission brought about by environmental weathering. Similar types of observations were reported by Raj et al.,²⁷ that at higher fiber loading like ours the elongation at break decreases by the effect of aging.

Tables IV and V show the comparative flexural property of JPH(C) and JPH(B) in respect to the weathered and unweathered samples, respectively, which are the average of five samples in each case. Like the tensile strength, the flexural strength of the weathered samples was found to be lesser than that of the corresponding unweathered specimens. For JPH(C), the flexural strength decreased from 140.4 to 100.7 MPa, while for JPH(B), it decreased from 171.8 to 105.7 MPa. But unlike the tensile modulus, the flexural modulus was found to be lesser for the weathered specimens than for their unweathered counterparts. This can be attributed to the following reason: Although the samples become stiffer after exposure, the interface becomes weakened as explained before. In the flexural experiment, the stress is applied perpendicular to the direction of the fiber axis and the fibers are brittle in this

Table IV Flexural Properties of JPH(C)

Sample Specimen	Nature of the Sample	Yield Stress (MPa)	Flexural Modulus (GPa)	Energy at Break (J)	Toughness (MPa)	ILSS (MPa)
JPH(C)	Weathered	100.7	8.94	0.4190	0.1017	3.192
JPH(C)	Control (unweathered)	140.4	13.85	0.4008	0.1157	3.865

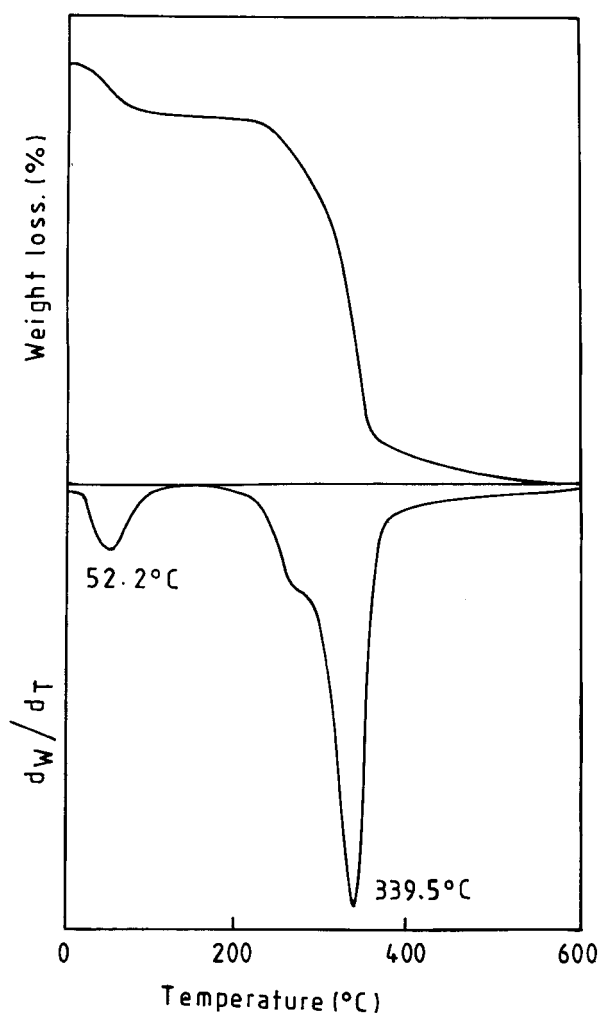
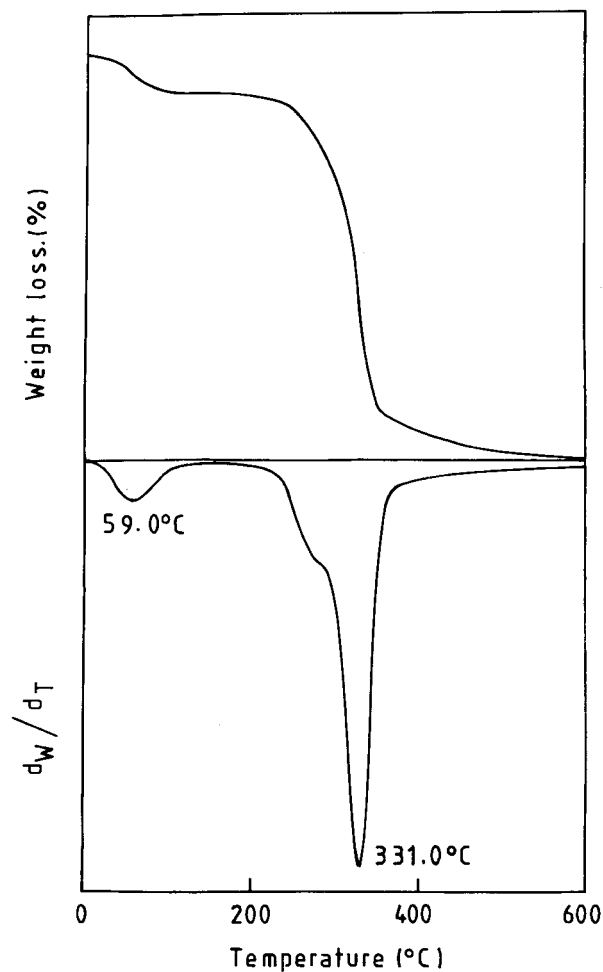
Table V Flexural Properties of JPH(B)

Sample Specimen	Nature of the Sample	Yield Stress R (MPa)	Flexural Modulus (MPa)	Energy at Break (J)	Toughness (MPa)	ILSS (GPa)
JPH(B)	Weathered	105.76	7.333	0.7560	0.1870	3.30
JPH(B)	Control (unweathered)	171.8	18.440	0.3828	0.1263	4.032

direction. Although there is more crosslinking due to postcuring in the weathered specimens, the brittleness along with the increased tendency for debonding by water, heat, and light are responsible for the lowering of flexural strength, moduli, and interlaminar shear strength (ILSS).

Thermal Analysis

Figures 1 and 2 show the TG/DTG curves of sliver(C) and sliver(B), respectively, while Figures 3 and 4 give the TG/DTG curves of JPH(C) and JPH(B), respectively. The TG/DTG results are given in Table VI, which reveal that sliver(C) and JPH(C) are thermally more stable than are sliver(B) and JPH(B), respectively. It is interest-

**Figure 1** TG/DTG curve of sliver(C).**Figure 2** TG/DTG curve of sliver(B).

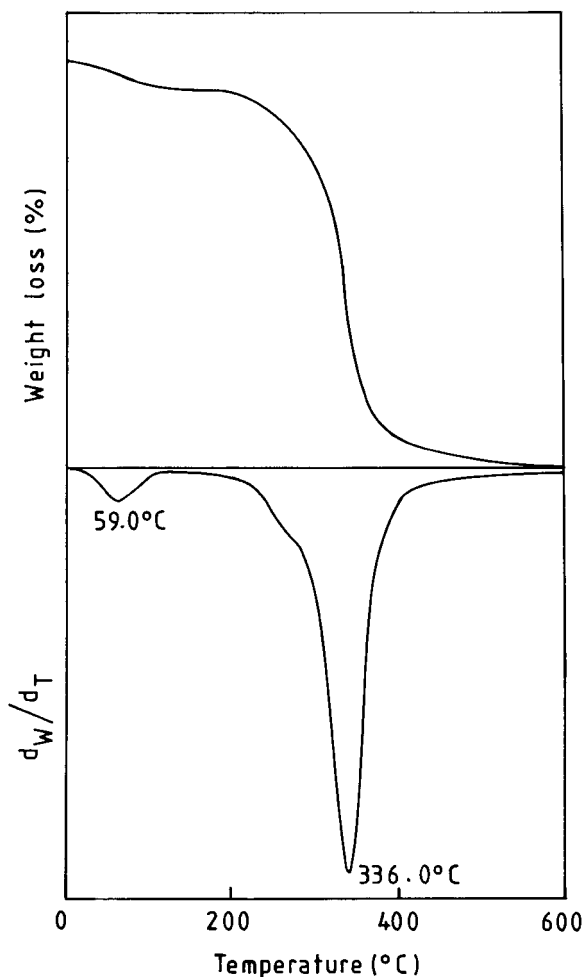


Figure 3 TG/DTG curve of JPH(C).

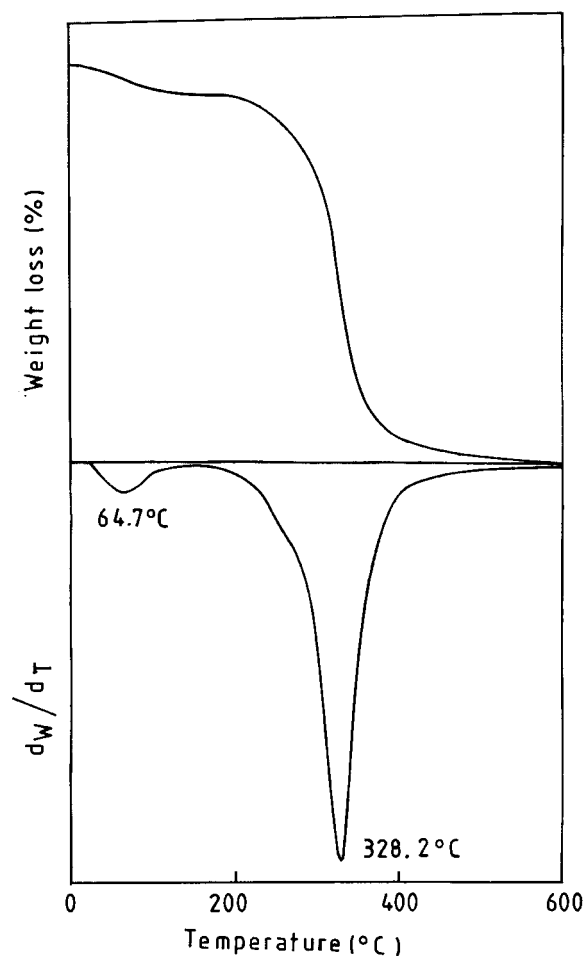


Figure 4 TG/DTG curve of JPH(B).

ing to observe that the decomposition pattern for the slivers and the composites are very similar because of the presence of a high weight percent of sliver in the composites that makes it behave predominantly like the sliver and not like the plastic matrix. The major decompositions as revealed from the DTG inflections that occur at 339.5 and 336.7°C in the case of sliver(C) and JPH(C) (Figs. 1 and 4), while it is 331.2 and 328.2°C for sliver(B) and JPH(B) (Figs. 2 and 3), respectively. This indicates that the sliver(C)–JPH(C) pair is thermally more stable than is the sliver(B)–JPH(B) pair, although the difference is not remarkable. The percentage weight loss due to loss of moisture in the case of sliver(C) and JPH(C) are observed at temperatures of 56.2 and 59°C, respectively, while the corresponding values are 59 and 64.7°C for sliver(B) and JPH(B), respectively. The percentage weight loss during major decomposition in the case of sliver(C) and

JPH(C) are less than that of sliver(B) and JPH(B), respectively. A comparison of the TG/DTG data of sliver(C) and sliver(B) (Table VI) reveals that the percentage loss of moisture in the case of sliver(C) is more than that of sliver(B),

Table VI TG/DTG Analysis

Sample Specimen	DTG Peak Specimen	Peak Temperature (°C)	Weight Loss (%)
Sliver(C)	1	56.2	8.88
	2	339.5	73.7
Silver(B)	1	59	6.96
	2	331	72.4
JPH(C)	1	59	5.3
	2	336.7	82.6
JPH(B)	1	64.7	4.75
	2	328.2	81.5

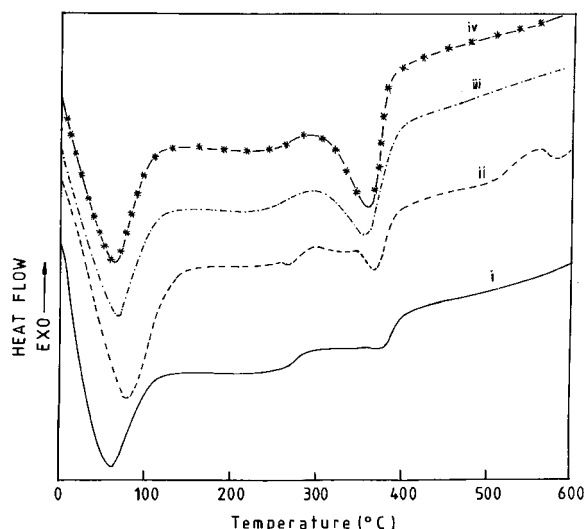


Figure 5 DSC curves of (i) sliver(C), (ii) sliver(B), (iii) JPH(B), and (iv) JPH(C).

which indicates that sliver(B) is less hydrophilic than is sliver(C). This may be due to the decrease in hydrophilicity effected by bleaching due to delignification in sliver(B). All these results indicate that sliver(C) is thermally more stable than is sliver(B).

Comparison of the TG/DTG results of JPH(C) and JPH(B) presented in Table VI reveals that the percentage weight loss due to loss of moisture in the case of JPH(C) is higher than in JPH(B). This may be due to increase in the hydrophobicity in the case of JPH(B). These results are in agreement with the water-absorption results discussed before. From the main decomposition temperatures, it is clear that the overall thermal stability of JPH(C) is greater than that of JPH(B). This may be due to the presence of lignin in JPH(C),

which is acting as a thermal stabilizer in the composite.

DSC Analysis

The DSC thermograms of sliver(C), sliver(B), JPH(B), and JPH(C) are shown in Figure 5. The corresponding results are presented in Table VII.

Figure 5(i) shows two endothermic peaks centering at 65.4 and 380.8°C in the case of sliver(C). The first peak is due mainly to loss of moisture and the second peak is the major decomposition peak. The enthalpy change for the peak at 65.4°C is found to be greater than expected for only moisture loss (Table VII). Therefore, it may be suggested that this peak is also associated with the glass transition temperature of hemicellulose and alpha cellulose present in jute fiber. Similar trends are also observed for sliver(B), JPH(B), and JPH(C) in Figure 5(ii), (iii), and (iv), respectively.

CONCLUSIONS

Jute-polyester composites exhibit excellent mechanical properties (tensile and flexural strengths) due to high fiber loading (60%) and high interfacial adhesion. Jute composites reinforced with chemically modified (bleached) fiber have a lower water-uptake value than that of the unmodified one, because chemical modification increases hydrophobicity and interfacial adhesion in the composites. This property also makes JPH(B) more resistant to weathering and other environmental conditions. However the thermal stability of JPH(C) is slightly better than that of JPH(B). Since untreated jute fiber-reinforced

Table VII DSC Analysis

Sample Specimen	Peak Specimen (°C)	Peak Temperature	Enthalpy Change (mJ)	ΔH (J/gm)	Nature of the Peaks
Sliver(C)	1	65.4	1173.3	223.61	Endothermic
	2	380.8	120.45	22.96	Endothermic
Sliver(B)	1	77.2	1847.6	255.69	Endothermic
	2	368.7	285.25	39.475	Endothermic
JPH-60(C)	1	69.2	1511.1	177.53	Endothermic
	2	368.7	1299.7	152.69	Endothermic
JPH-60(B)	1	67.9	1556.1	180.83	Endothermic
	2	362.8	994.97	115.63	Endothermic

composites are more susceptible to weathering, JPH(B) can be accepted as a better-performing composite than is JPH(C). The very low value of water absorption in the case of these composites is expected to make them more resistant to weathering on a long-term basis. Future experiments on these can throw better light on their potentiality.

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