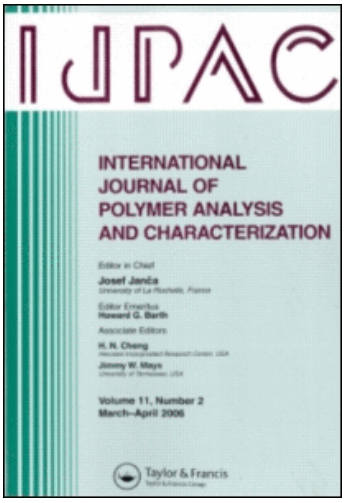

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Characterization, Biodegradation, and Water Absorbency of Chemically Modified Tossa Variety Jute Fiber via Pulping and Grafting with Acrylamide

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Abstract: Jute fiber (tossa variety) was chemically modified by the cooking alkaline sulfite process to unbleached and bleached pulps that were further modified via graft copolymerization with acrylamide monomer, using a complex initiating system: $CuSO_4$/glycine/$KHSO_5$. The samples so prepared were characterized and morphologically analyzed by IR, TGA, DSC, SEM, and XRD. The biodegradability and water absorbency of the samples were also evaluated for their novel commercial applications. It was found that the grafted samples were more resistant to biodegradation and showed higher water absorbency than their ungrafted counterparts.

Keywords: Pulping; Grafting; DSC; SEM; XRD; Biodegradation; Water absorbency

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INTRODUCTION

Jute is a low-cost multicellular and lignocellulosic fiber[1,2] produced in large quantities every year in India and neighboring Bangladesh. Chemical treatment of jute, such as reduction of lignin by pulping and bleaching and grafting, is expected to open up a new demand for jute by making it superabsorbent. In addition to its application in diapers and sanitary napkins, agricultural uses, and superabsorbents, jute can also be used as a geotextile to stabilize sandy soils in desert areas for food growth or forestation. The structural morphology, biodegradation, and superabsorbent applications of the pulp and grafted pulp materials cannot be confirmed without proper analysis and characterization.

IR spectroscopy of jute fiber and changes in spectra due to alkali treatment have been discussed.[3] IR spectroscopy has been shown to be a useful tool for investigating the fine structural characterization of cellulose and its chemical modification.[4–6] There has been little reported in the literature, on analysis and characterization of tossa jute fiber, pulp, and grafted pulps.

The present work deals with the IR, TGA, DSC, SEM, and XRD analyses of tossa variety jute fiber, chemically modified jute pulp, and grafted pulp samples. The structural and physical properties of this fiber, its modified pulp, and grafted pulp will be helpful in determining the technological performance of the fiber in terms of its applicability in paper, textiles, superabsorbent and biodegradable plastics, as compared to starch bioplastics.[7]

EXPERIMENTAL

Materials

Raw jute samples were taken from *Corchorus olitorius* (tossa variety). Acrylamide (AM) monomer (E. Merk, Germany) was purified by recrystallization from acetone and dried at room temperature under vacuum. Potassium monopersulfate ($KHSO_5$) was a gift sample from DuPont (USA) and was used without purification. All other reagents were from British Drug House (BDH) and were used after purification by standard methods.

Methods

Preparation of Jute Pulp

The pulp was prepared from the above variety of jute by the alkaline sulfite process.[8] The material was digested in a 10 L capacity electrically

heated rotary digester. The pulping parameters were described as total chemicals (as Na_2O), 8.73%, and as fiber to liquor ratio, 1:4. The cooking cycle employed was; time from room temperature to 120°C 45 m, time from 120°C to 160°C 1 h, and time for cooking at 160°C 3 h.

After cooking, the pulp was washed thoroughly to remove alkali. The pulp was then bleached by using a process consisting of alkali chlorination followed by hypochlorite treatment, where the total chlorine consumed for bleaching was 7.26%.[9]

Grafting onto Jute Pulp

Graft copolymerization reaction was carried out in a constant-temperature bath with an accuracy of 0.1°C, under an oxygen-free nitrogen atmosphere. Both the bleached and unbleached pulps were first soaked with appropriate quantities of monomer (acrylamide) and other reagents in a reaction vessel and deaerated using oxygen-free nitrogen. A known quantity of $KHSO_5$ was injected through a rubber tube after proper thermal equilibrium. After a specific time interval, each reaction was arrested by quenching the vessel in ice-cold water, followed by the addition of ammonium ferrous sulfate (AFS) solution, which spontaneously consumed unreacted $KHSO_5$. The homopolymer PAM (polyacrylamide) was removed by repeated washing with warm water, until the extract gave no precipitation with methanol. The grafted jute pulp was washed repeatedly in triply distilled water and dried to constant weight. The percentage of grafting was determined by using the equation:

$$\% \text{ grafting} = \left[\frac{(\text{wt of grafted sample} - \text{wt of sample})}{(\text{wt of sample})}\right] \times 100$$

Characterization

The IR spectra of the raw jute, jute pulps, and acrylamide-grafted pulps in the form of KBr pellets were recorded using a Perkin Elmer model Paragon-500 FTIR.

Thermogravimetric analysis (TGA) was conducted using a Shimadzu DTA -500 system (Shimadzu Corp.). TGA was carried out in air from room temperature and was gradually raised to 800°C at a heating rate of 10°C/min.

Differential scanning calorimetry (DSC) was done on a DuPont 910 DSC Calorimeter under nitrogen atmosphere at heating rate of 10°C/min using a sample weight of 1.5 mg.

The surface morphology of the samples was observed by using a model 5200 scanning electron microscope (SEM) (JEOL Ltd., Japan) at different magnifications after coating it with gold.

The X-ray diffraction (XRD) of the fiber and the modified fiber samples was recorded and scanned by a Philips Analytical BV diffractrometer.

The biodegradation and superabsorbency of the modified pulp and grafted pulps were studied according to the techniques described in the discussion.

RESULTS AND DISCUSSION

Graft copolymerization of the jute fiber increases with decreasing lignin content in the pulp.[10] Graft copolymerization of acrylamide onto chemically modified bleached and unbleached jute pulp was done to increase the graft percentage. It was found that graft percentage in bleached pulp is higher than in the unbleached pulp, which is greater than it is in raw jute.[11]

Infrared Spectroscopy

Infrared spectra of jute pulp have sharper and more clearly defined bands than the spectra of raw jute fiber. Modified jute pulp contains mostly cellulosic units whereas the raw jute fiber contains cellulose, lignin, and hemicellulose. The spectra of raw jute fiber show additional bands near $1735\,cm^{-1}$, $1595\,cm^{-1}$, and $1640\,cm^{-1}$ and a broad band in the region $1200–1300\,cm^{-1}$ having a maxima near $1245\,cm^{-1}$. There is a strong broad band near $3300–3400\,cm^{-1}$ in the spectra of jute fiber due to hydrogen bonded $-OH$ vibration.[12]

The presence of an absorption band near $1730\,cm^{-1}$ in the IR spectra is due to $>C=O$ stretching at the carboxyl groups. The sharp bands at 1595 and $1505\,cm^{-1}$ show the presence of aromatic rings in jute fiber. The spectra of lignin show sharp bands in these regions, due to the stretching modes of the benzene ring.[13] The bands near 1250 and $1235\,cm^{-1}$ are possibly due to $-C-O-C-$ bond in the cellulose chain[14] and $-OH$ deformation,[15,16] respectively.

Infrared spectroscopy is a valuable tool for identifying graft copolymerization,[17,18] helping to ensure complete removal of ungrafted products, and is used to determine the functional groups of the grafted samples as in Figure 1. It is observed that the IR spectra of ungrafted unbleached and bleached pulps show the characteristic broad absorption band at $3400\,cm^{-1}$ for free $-OH$ band of $O-H$ stretching vibration and $1040\,cm^{-1}$ $C-O$ stretching vibration. In addition to these peaks, asymmetric and symmetric $N-H$ stretching vibrations at 3520 and $3400\,cm^{-1}$, respectively, were observed in unbleached and bleached

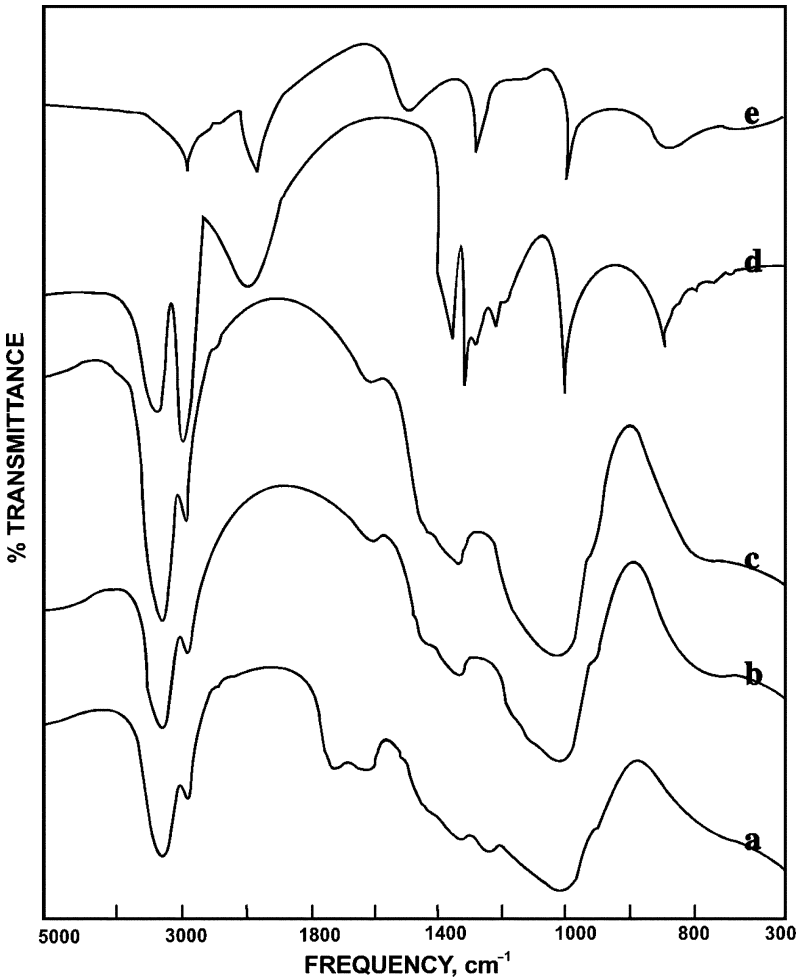

Figure 1. Infrared spectra curves of (a) raw jute fiber, (b) unbleached pulp, (c) bleached pulp, (d) unbleached pulp-*g*-PAM, (e) bleached pulp-*g*-PAM.

samples. A band at $1690\,cm^{-1}$ was observed due to $>C{=}O$ amide stretching vibration and $705\,cm^{-1}$ for C–N stretching of a primary amide, indicating the occurrence of grafting of acrylamide onto both bleached and unbleached jute pulps.

Thermogravimetric Analysis

Thermograms of raw jute fiber, bleached jute pulp, unbleached jute pulp, jute-*g*-PAM (40.2% grafting), bleached pulp-*g*-PAM (76.3% grafting),

and unbleached pulp-*g*-PAM (63.4% grafting) are shown in the Figure 2. The temperature of decomposition (T_D) is very much influenced by the grafting of the sample. As compared to the unbleached pulp with T_D of 386°C, the T_D values increased significantly to 428°C in the case of grafted pulps.

This implies that the grafted samples are moderately thermally stable as compared to the ungrafted samples. Increase in thermal stability in grafted fiber may be attributed to the increase in crystallinity resulting from the decrease in the amorphous content of the jute pulp due to AM grafting. This finding is similar to that of Verma et al.[17] on the effect of chemical treatment on density and crystallinity of the jute fibers.

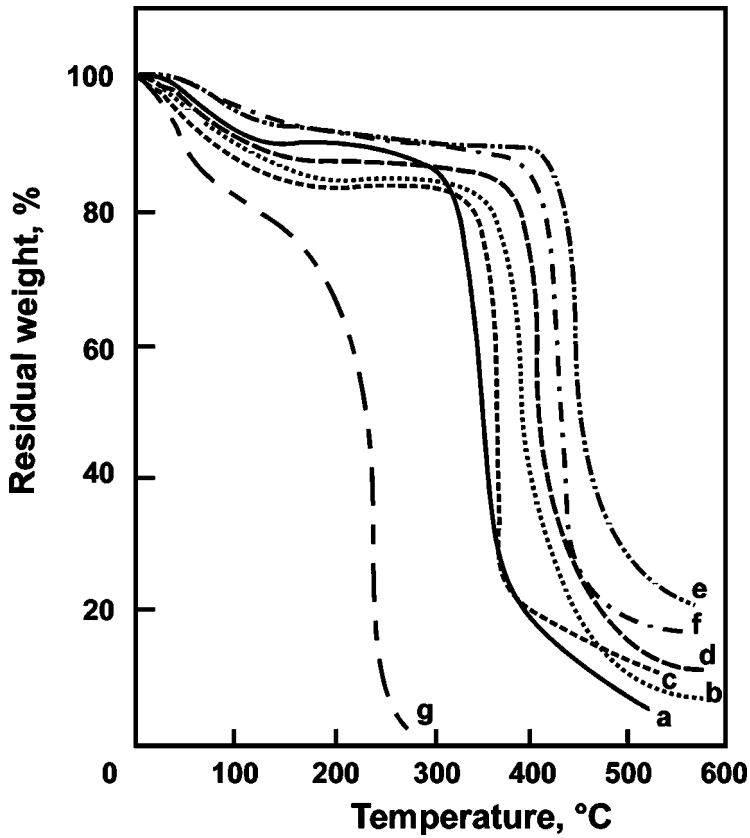

Figure 2. TGA curves of (a) raw jute fiber, (b) unbleached pulp, (c) bleached pulp, (d) raw jute-*g*-PAM (40.2%), (e) unbleached pulp-*g*-PAM (63.4%), (f) bleached pulp-*g*-PAM (76.3%), (g) PAM.

Differential Scanning Calorimetry

DSC curves of raw jute, unbleached pulp, bleached pulp, and unbleached and bleached grafted samples with AM are shown in Figure 3. The DSC

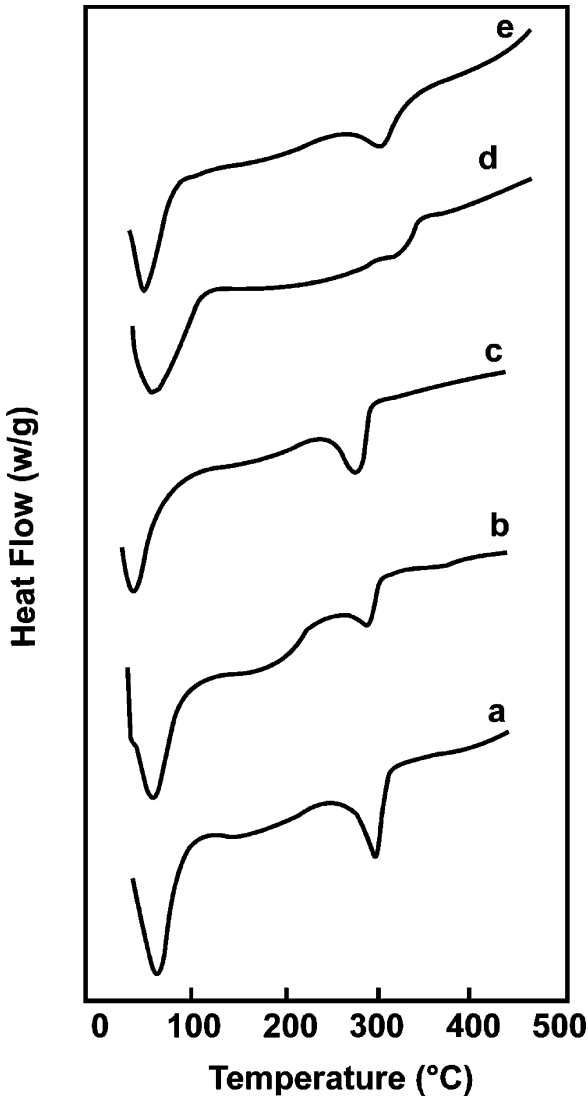

Figure 3. Differential scanning calorimetry curves of (a) raw jute fiber, (b) unbleached pulp, (c) bleached pulp, (d) unbleached pulp-*g*-PAM, (e) bleached pulp-*g*-PAM.

Table I. Results of DSC of tossa variety jute fiber, pulp (bleached and unbleached), and grafted pulps in a nitrogen atmosphere

Sample	Peak temperature (°C)	Moisture content(%)	Nature of peak
Jute fiber	78	10.4	Endo
	307		Exo
	366		Endo
Unbleached pulp	76	11.2	Endo
	329		Exo
	358		Endo
Bleached pulp	66	12.9	Endo
	307		Exo
	353		Endo
Unbleached pulp-g-PAM	72	14.0	Endo
	320		Endo
	337		Endo
Bleached pulp-g-PAM	63	15.2	Endo
	311		Endo
	330		Endo

curve of all samples show an endothermic peak below 100°C due to the evaporation of moisture. The exothermic peaks of the hemicellulose decomposition are at 307°C (raw jute), 329°C (unbleached pulp), 307°C (bleached pulp), 337°C (unbleached pulp-*g*-PAM), and 330°C (bleached pulp-*g*-PAM), and the endothermic peaks of cellulose decomposition[18–21] are at 366°C (raw jute), 358°C (unbleached pulp), 353°C (bleached pulp), 330°C (bleached pulp-*g*-PAM), and 337°C (unbleached-*g*-PAM), respectively. An exothermic peak at 225°C in unbleached jute pulp is due to lignin content. These data are consistent with data already reported by Schneider and Zoepel[22] regarding the thermal degradation of bleached sulphite pulp (225°C) and kraft pulp (240°C) and the fast endothermal degradation of bleached sulfite pulp (330°C).

In the case of modified and grafted pulps, the moisture content gradually increases, which is shown in Table I. This may be due to the increase in the porous nature of pulp and in grafted samples by the introduction of the hydrophilic polymer PAM onto the backbone of the pulp. This has been verified from the water retention capacity of these samples already reported by us.[11]

Scanning Electron Microscopy

Chemically modified jute pulps (unbleached and bleached) and their PAM-grafted counterparts were characterized in terms of surface

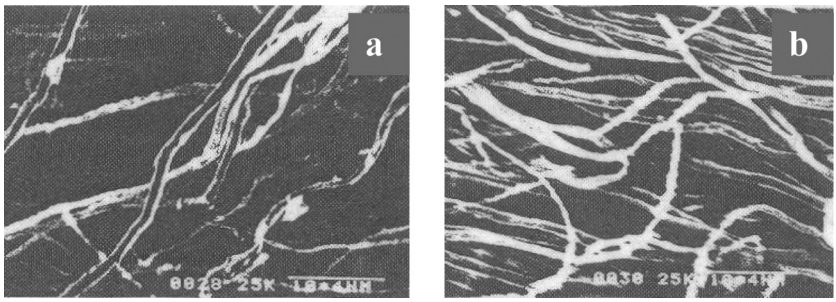

Figure 4. Scanning electron micrographs of bleached pulp without (a) and with (b) acrylamide grafting, magnification $\times 300$.

morphology by SEM. Figure 4 represents the surface morphology of the bleached pulp and PAM-grafted bleached pulp. Grafting of acrylamide polymer onto jute pulp is clearly shown as a white deposit of PAM via bonding by hydrogen atom abstraction from the jute pulp backbone. This fact has already been verified from the increase in tensile strength of the grafted sample.[11]

X-ray Diffraction

The XRD pattern shown in Figure 5 reveals the degree of crystallinity in the unmodified and modified pulps and grafted pulps. All the samples show two major peaks. The first peaks in all cases occur at approximately the same (angle of orientation) value, while the second peaks occur at slightly different values, with a small difference in their diffraction patterns resulting in different degrees of crystallinity. The degree of crystallinity was determined from the instrument by comparing the sharp diffraction of peaks with the general scattering of the polymers[23] determined to be 58.8%, 65.8%, 52.2%, 65.2%, and 56.5% for raw jute fiber, unbleached pulp, unbleached pulp-*g*-PAM, bleached pulp, and bleached pulp-*g*-PAM respectively. The degree of crystallinity of raw jute (*C. olitorius*)[24] and that of its pulp[25] were reported to be within the range of 58–66% and 62–70% respectively. It has been observed that the degree of crystallinity decreases in the case of grafting with PAM onto both bleached and unbleached pulp, suggesting that the amorphous region increases due to grafting. This is supported by the fact that the water retention capacities of grafted samples are greater, which has already been reported by us.[11] Further XRD curve intensities reveal that higher intensity of the peak will result in higher thickness,[26] as shown in case of raw jute fiber, whereas low intensity results in lower thickness, as

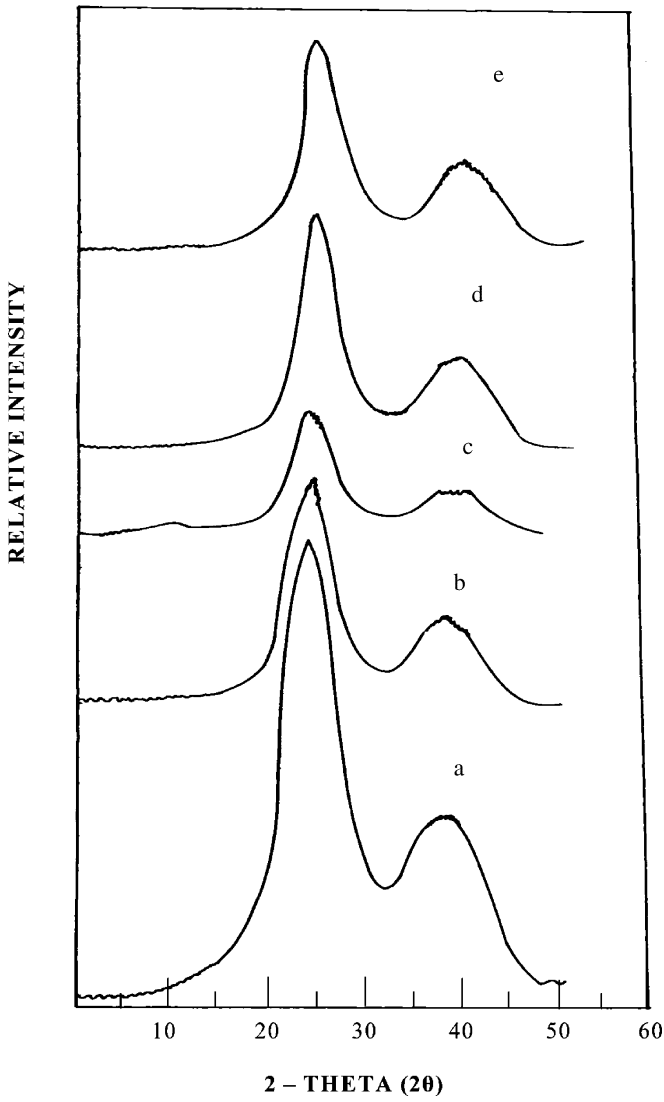

Figure 5. X-ray diffraction curves of (a) raw jute fiber, (b) unbleached pulp, (c) bleached pulp, (d) unbleached pulp-*g*-PAM, (e) bleached pulp-*g*-PAM.

shown in case of bleached pulp. This effect may be due to the removal of lignin and other binding materials. In the case of grafted bleached and unbleached pulp, the thickness is in between that of the raw jute and bleached pulp as evidenced from their curve intensities (Figure 5), further confirming grafting of PAM onto the pulp.

Biodegradation

Biodegradation studies of jute fiber, pulp, and their grafted samples in sewage water showed some interesting results (Figure 6). Supernatant water was collected in PET bottles from a nearby open dump yard where all solid as well as liquid wastes from different departments of the university are collected. The experiment was conducted under aerobic conditions by incubating the grafted and the ungrafted samples and the collected water in glass test tubes of 1:100 (w/v) at $28 \pm 2^{\circ}C$. There was 14–16% weight loss in raw jute fiber and unbleached pulp after seven days of inoculation, and the rate of weight loss increased with increased days of incubation. However, the percentage of weight loss was significantly less in bleached pulp during the same period. About 25% weight loss was noticed in bleached pulp in comparison to 65% and 62% weight loss in unbleached pulp and raw jute, respectively within 28 days of incubation (at room temperature). This may be due to lower lignin content (which is used as a source of nutrients for the degrading agent) in bleached pulp compared to unbleached pulp or raw jute. Interestingly, there was very little percentage of weight loss in the samples of polyacrylamide grafted pulps (both bleached and unbleached) after 28 days of incubation, showing negligible biodegradation. This may be ascribed to unavailability of $-OH$ group for hydrolysis in the polymer backbone

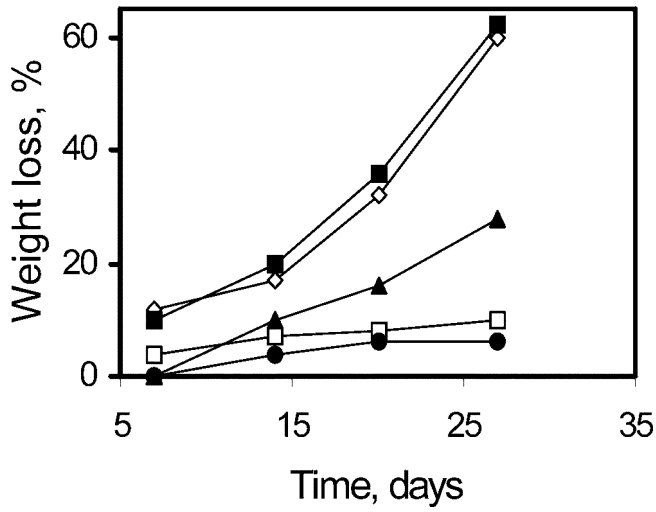

Figure 6. Plots of weight loss vs. time in sewage water of (◇) raw jute fiber, (■) unbleached pulp, (▲) bleached pulp, (□) unbleached pulp-*g*-PAM (73.8%), (●) bleached pulp-*g*-PAM (98.0%).

of grafted samples. It has been reported that an increase in derivatized −OH group decreases biodegradation.[7]

However, in the present case, the adjacent −OH group forms only H−bonding with the N-atom of acrylamide, which is confirmed from the IR peak at 3100 cm^{-1}. The biodegrading agent was found to be a fungus rather than bacteria because there was clearly visible growth of fungal mycelium in the degraded samples. According to early reports, fungi are more efficient in degrading polymer than bacteria.[27] The minor difference in biodegradation of raw jute and unbleached pulp may be due to the easier growth of fungal hyphae in loosened pulp than in tightly packed fibers of raw jute.

Water Absorbency

The water absorbency (Q_{H_2O}) of jute pulps and grafted copolymers was determined by immersing 1 g of each sample in distilled water (1000 mL) for at least 10 h at ambient temperature to reach swelling equilibrium. The residual water was removed by hanging samples to drain for 30 min. The water absorbency (Q_{H_2O}) of the samples was determined by weighing the swelled samples after centrifuging for 1 h and using:

$$Q_{H_2O} = \frac{(\text{wt of swelled sample} - \text{wt of dried sample})}{\text{wt of dried sample}}$$

Figure 7 shows the water absorbency of samples as a function of time. The water absorbency increases with time for each sample in the

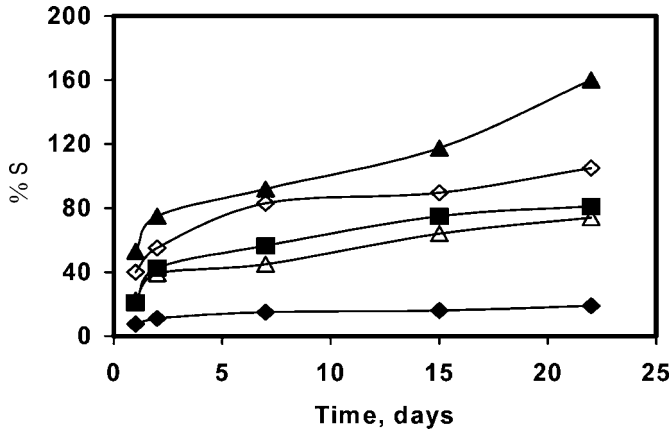

Figure 7. Variation of time with water absorbency for (◆) raw jute fiber, (△) unbleached pulp, (■) bleached pulp, (◇) unbleached pulp-*g*-PAM (73.8%), (▲) bleached pulp-*g*-PAM (98.0%).

order of jute fiber < unbleached pulp < bleached pulp < unbleached pulp-*g*-PAM (73% grafting) < bleached pulp-*g*-PAM (98% grafting). Superabsorbent properties are shown by graft copolymerization of the delignified or partially delignified jute via pulping and bleaching. Absorptive polymers containing suitable absorptive groups and appropriate cross-links could hopefully be polymerized and attached to the surface of jute fibers. In the present case, superabsorbency was shown to be maximum in the case of the grafted samples (98% grafting).

CONCLUSION

Chemical modification of tossa variety jute fibers via pulping and grafting showed different IR spectra, indicating a difference between unbleached and bleached pulps. The amide >C=O stretching vibration and C−N stretching of primary amide indicated grafting of AM onto both unbleached and bleached pulps, which is further confirmed from SEM surface morphology. Thermal analysis by TGA and DSC exhibited increased thermal stability in grafted pulps due to the increase in crystallinity and hence a decrease in amorphous content of jute pulp due to AM grafting. DSC studies showed an increased moisture content in modified samples due to the increase in the porous nature of pulps and grafts. These facts are in agreement with the increased superabsorbency nature of the grafted samples. Finally, the biodegradation studies of the bleached pulp and PAM grafted pulps showed a significant decrease in biodegradation due to a lower lignin content, a nutrient for degrading agents assumed to be fungi. These initial findings show potential application of this superabsorbent in diapers and sanitary napkins and in agricultural use, with the major use being hydromulching.[28]

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