

Effect of Silane Monomer on the Improvement of Mechanical and Degradable Properties of Photografted Jute Yarn with Acrylamide

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ABSTRACT: UV radiation was used to graft acrylamide (AA) to jute yarn in order to improve the mechanical properties. The physicochemical properties of the grafted jute yarn were investigated. A series of solutions of different AA concentrations in methanol along with photoinitiator (Irgacure-907, 2%) were prepared. The jute yarns were soaked in the solution for 30 min before radiation. The effect of irradiation time, concentration of monomer on polymer loading, and tensile properties of the jute yarn were studied. The highest polymer loading (22%) and tensile strength (95%) of the yarn were observed when the yarn was treated in 30% AA in methanol with 60 min of UV radiation. The surfaces of both treated and untreated jute were characterized by X-ray photospectrometry, infrared spectroscopy, and envi-

ronmental scanning electron microscopy, and it was observed that the AA reacted or deposited on the jute surface. A minute amount (1%) of silane monomer [3-(trimethoxysilyl)-propyl methacrylate] was used as an additive in the AA solution to further improve the mechanical properties of jute yarn. Better improvement was achieved by using 1% silane monomer. Water uptake, simulating weathering, and soil degradation tests of untreated and treated yarns were also performed. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 89: 3530–3538, 2003

Key words: jute; photografting; UV radiation; mechanical properties; biodegradable

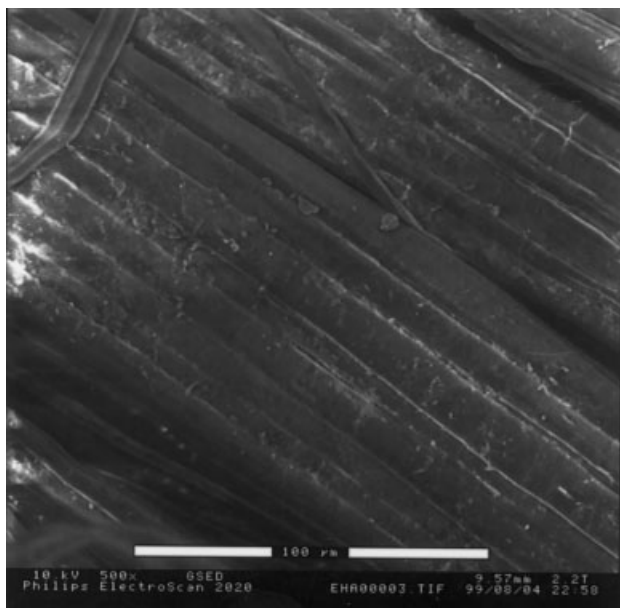
INTRODUCTION

Naturally occurring polymers are both abundant and biodegradable. Such natural polymers are obtained from fibers such as jute, cotton, and rayon. These natural fibers are lignocellulosics and contain all classes of cellulose as their main components along with hemicellulose and lignin. Lignin is a phenolic component generally resistant to microbial degradation. Cellulose is a hydrophilic glucan polymer comprising a linear chain of 1,4- β -bonded anhydroglucose units pendant with OH groups. These hydroxyl groups in the cellulose structure account for its hydrophilic character, which is a drawback of this natural fiber.

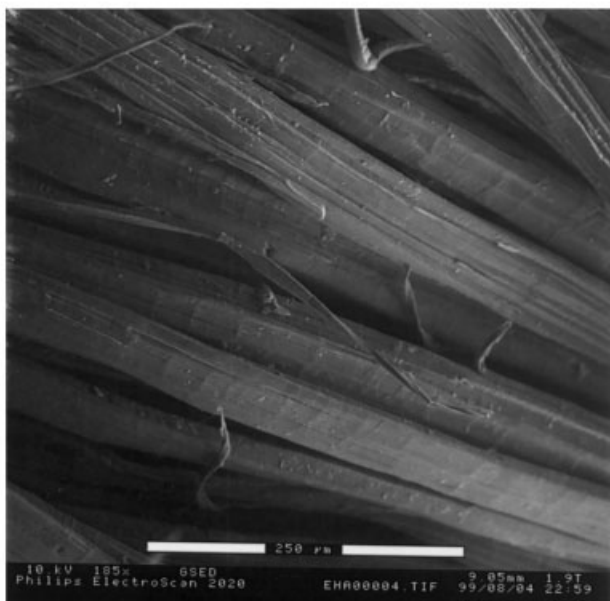
Jute is the cheapest of all natural fibers in Bangladesh and has long been used for inexpensive products such as ropes sacks and Hessian clothes. Jute contains a relatively hard cellulose fiber and has high tensile modulus and low elongation at break.¹ In contrast, synthetic polymers tend to have

higher tensile strength and durability compared to natural polymers. It is important, therefore, to improve the physicochemical properties of natural polymers with different materials in different modes. Photoinitiated graft polymerization is one of the fastest and most efficient methods for modifying the durability of the surface properties of natural polymers.^{2–3} Using UV radiation technique to graft vinyl monomer onto cellulosic materials offers several advantages, including: (a) reaction occurring at ambient temperatures, (b) reduction in reaction time, (c) continuous operation, (d) improved monomer stability, (e) less atmospheric pollution, and (f) increased design flexibility through process control.⁴ Some developments in enhancing mechanical properties, such as maintaining their inherent degradation, have been achieved on jute plastic composites using different impregnation solutions under radiation^{5–6} or UV radiation.^{7–10} The ultimate aim of this research was to improve the tensile strength and elongation at break of jute yarns, which showed reduced water absorption and could be suitable for diverse industrial applications such as wall coverings, curtains, jute blanket, carrier bags, carpets, and jute plastic products.

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(a)



(b)

Figure 1 (a) ESEM photographs of untreated jute yarn; (b) ESEM photographs of acrylamide-grafted jute yarn.

EXPERIMENTAL

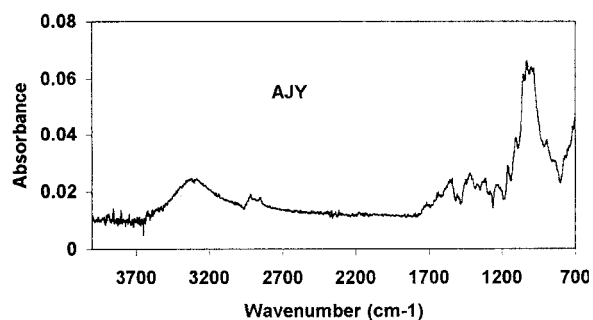
Materials

Jute yarns were collected from Islam Jute Mills, Bangladesh. The monomer acrylamide (AA) was procured from Aldrich (USA). Methanol (MeOH) was used as a solvent received from Merck (Germany). The additive 3-(trimethoxysilyl)-propyl methacrylate was procured from Fluka Chemical, Switzerland. Irgacure 907, [2-methyl-1-(4-methylthio phenyl)-2-morpholinopro-

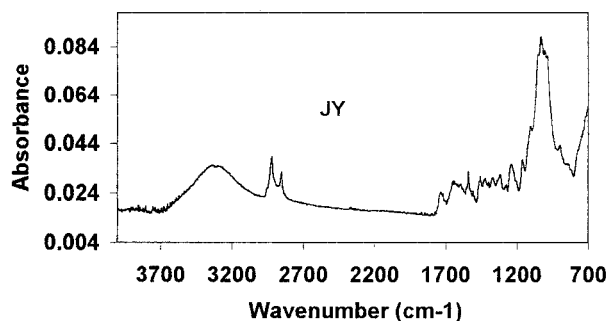
panone-1], (Ciba-Geigy, Switzerland) was used as the photoinitiator.

Methods

A series of solutions were prepared at different acrylamide (AA) concentrations (5%–50%) in methanol along with 2% (w/v) photoinitiator in different Pyrex test tubes, each tube containing a total of 20 mL of solution. Jute yarns (20 cm long) were dried at 105°C for 3 h to remove moisture and then stored in a desiccator. The dried yarns were immersed for 30 min in test tubes containing monomer and methanol solutions with different proportions of monomer. These tubes were placed in a rotary rack that moved around a central UV lamp of 100W (300–400 nm). During irradiation the test tubes were spun about their own axes while rotating around the lamp, which was hung in the middle. This arrangement ensured equal radiation to all the test tubes containing the different samples. The irradiation period varied from 15 to 300 min. After the desired length of irradiation, the jute yarns were withdrawn from the test tubes and washed in acetone for 30 min; then the samples were dried at 105°C for 3 h. Polymer loading was determined on the basis of weight gained by the jute sample after the entire treatment process. The change in tensile prop-



— Fig. 2b



— Fig. 2a

Figure 2 (a) FTIR spectra from 700 to 4000 cm^{-1} of untreated jute yarn (JY); (b) FTIR spectra from 700 to 4000 cm^{-1} of acrylamide grafted jute yarn (AJY).

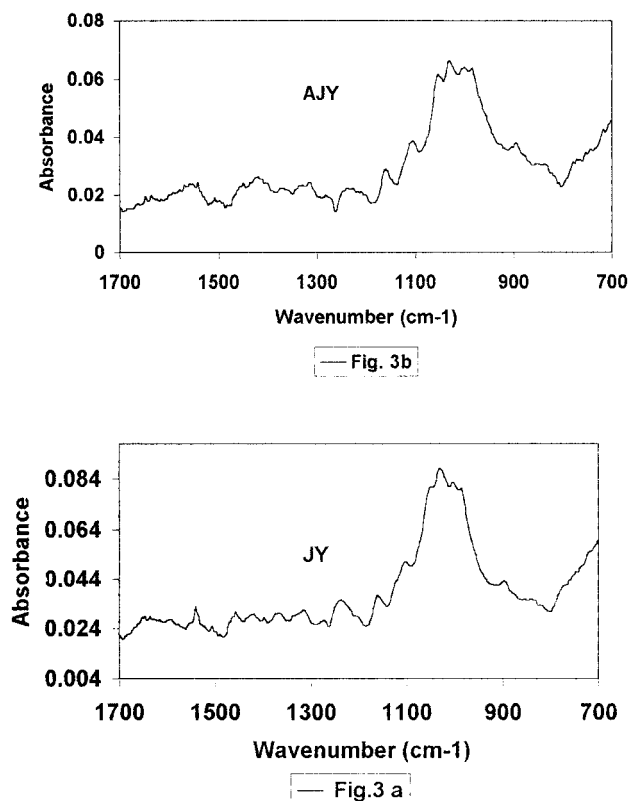


Figure 3 (a) FTIR spectra from 700 to 1700 cm^{-1} of untreated jute yarn (JY); (b) FTIR spectra from 700 to 1700 cm^{-1} of acrylamide grafted jute yarn (AJY).

erties of the jute yarns as a result of the above treatment with monomers is expressed by factors such as tensile strength factor (Tf) and elongation at break factor (Ef). Tensile properties of the jute yarn samples, such as strength (TS) and elongation at break (Eb), were directly measured with a universal testing machine (Instron model 1011) at a crosshead speed of 2 mm/min and a gauge length of 30 mm. Water uptake of the treated (irradiated) and untreated samples into a static water bath at 25°C was sufficient so that they were completely immersed. Weights of the samples were recorded after different contact times. Samples were wiped carefully to remove surface water before weighing. Finally, the weight gain for the samples for water treatment was calculated.

Environmental scanning electron microscopy

Both treated and untreated jute yarns were examined with an environmental scanning electron microscope (ESEM; model 2020). The ESEM was equipped with an La B6 filament and was operated at an accelerating voltage of 20 kV, with water vapor pressure ranging from 2 to 3 Torr.

Fourier transform infrared spectroscopy

Grafted and virgin jute yarns were analyzed by Fourier transform infrared spectroscopy (FTIR; Spectra

model 2000, Perkin-Elmer) using the attenuated total reflectance (ATR) technique.

Differential scanning calorimetry

Thermal properties of the untreated and grafted samples were investigated using differential scanning calorimetry (DSC) with a DSC-50 (Shimadzu, Japan). The samples were investigated in the sample pan and sealed. Thereafter they were introduced into the heating cell of the DSC. Heat treatment was performed at a programmed rate of 10°C/min, and then the data were collected and analyzed by a computer.

X-ray photoelectron spectroscopy

The X-ray photoelectron spectroscopy (XPS) spectra of the untreated and grafted samples were recorded with a physical electronic instrument (PHI 5400 ESCA). All spectral data were collected using a Mg anode operated at a power of 300W with an analyzer pass energy of 33 eV. The optimum spot size for the experimental conditions was the aperture 1.00 mm in diameter. Chemical information indicating changes in the surface-treated samples was elucidated by curve fitting the carbon 1s (C_{1s}) and the oxygen 1s (O_{1s}). The spectra were fitted with a Lorentzian-Gaussian mix Voigt profile function, using a nonlinear least-squares curve-fitting program. The model used for the C_{1s} spectra was based on the assumption of approximately 1.5 eV chemical shift per bond to oxygen.

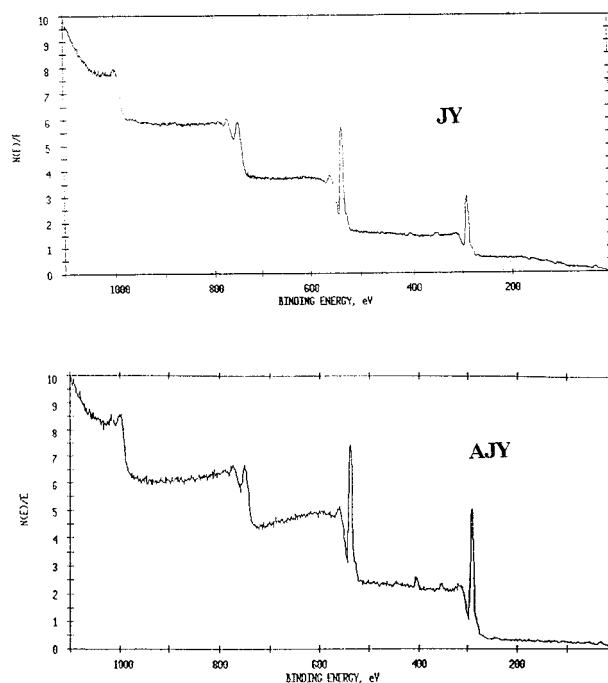


Figure 4 (a) XPS spectra of untreated jute yarn (JY); (b) XPS spectra of acrylamide grafted jute yarn (AJY).

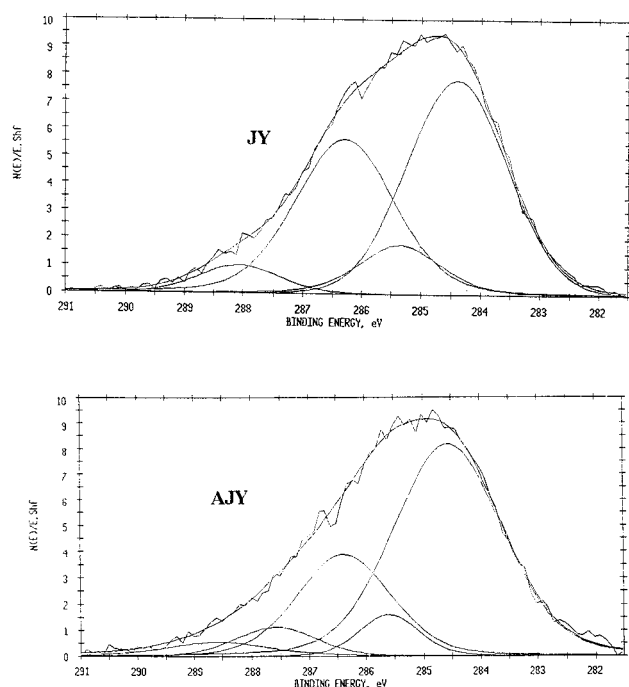


Figure 5 (a) XPS spectra of carbon (C_{1s}) peaks of untreated jute yarn (JY); (b) XPS spectra of carbon (C_{1s}) peaks of acrylamide-grafted jute yarn (AJY).

Simulated weathering test

The grafted and virgin samples were treated using a simulated weathering tester from Q-Panel Co. (model QUV, USA). The weathering test was performed in alternating cycles of sunshine over 4 h ($65^{\circ}\text{C} \pm 2^{\circ}\text{C}$) followed by dews and condensation over 2 h ($45^{\circ}\text{C} \pm 2^{\circ}\text{C}$). This treatment was carried out for about 600 h.

Soil degradation study

Cellulose has a tendency to degrade when buried in soil whose moisture level is at least 25%. For this purpose, the grafted sample and untreated samples were weighed individually and buried in garden soil for 72 days. After that the samples were carefully withdrawn, washed with distilled water, dried at a temperature of 105°C for 20 min, and then kept at room temperature for 24 h, after which their weights were recorded. Finally, the weight loss of the various degraded samples was calculated.

RESULTS AND DISCUSSION

Characterization of jute fiber surface topography

To analyze the surface topography and morphology of both virgin and AA-grafted jute fibers, an investigation was done with an environmental scanning electron microscope (ESEM). The ESEM pictures of both the treated and untreated surfaces are shown in Figure

1. The surface of the untreated fiber [Fig. 1(a)] is smooth and appears multicellular in nature. The fibrillar structure as well as the porosity of the individual fibers is revealed on the fiber surface. In Figure 1(b), which shows fibers treated with AA, a rough surface topography and fragments on the surface can be observed. The significant change in morphology and topography of the fiber surface is a result of various etchings out of the structure by acrylamide (AA).

Characterization of jute by FTIR

To study the chemical reaction between acrylamide and the cellulose of the fiber in untreated jute (JY), AA-treated jute (AJY) was characterized by FTIR using the attenuated total reflectance (ATR) technique. The IR spectra of JY are shown in Figures 2(a), for $4000\text{--}700\text{ cm}^{-1}$, and 3(a), for $1700\text{--}700\text{ cm}^{-1}$, and those of AJY are shown in Figures 2(b), for $4000\text{--}700\text{ cm}^{-1}$, and 3(b), for $1700\text{--}700\text{ cm}^{-1}$. The characteristic features of the jute spectrum are a result of its lignin and hemicellulose components. The characteristic absorption band of the carbonyl group ($>\text{C}=\text{O}$) of lignocellulosic fiber, at 1730 cm^{-1} , was observed in both JY and AJY. The intensity of the OH groups in the treated jute (AJY) was lower than that of the untreated jute (JY). A higher intensity of the carbonyl group was observed in AJY compared to that of JY. This may be attributed to the jute backbone, composed of AA grafted with cellulose, which occurred through a graft copolymerization reaction. A new sharp band was observed at 1539 cm^{-1} , which may be attributed to the $\text{C}=\text{C}$ stretching of AA.¹¹ Another new peak was observed at 1396 cm^{-1} , corresponding to the CH_3 deformation of AA.¹² The absorption bands of the above peaks are indicative of the existence of AA deposition on jute yarn, and AA might have reacted with the cellulose backbone of the jute through a graft copolymerization reaction.

Characterization of jute surface by XPS

To study the chemical environment of jute fiber surfaces, they were examined by X-ray photoelectron spectroscopy (XPS) before and after the photografting of AA onto the jute fiber. The XPS spectra of electron intensity above the bonding energy for jute (JY) and

TABLE I
XPS Analysis of Surface Composition of Treated and Untreated Jute

Samples	Elements				Ratio	
	C_{1s}	O_{1s}	N_{1s}	Other	C/O	C/N
Untreated JY	48.79	38.79	2.17	10.25	1.26	22.48
Treated JY	36.82	50.68	8.02	4.48	0.72	4.59

TABLE II
Curve Fit Summary from XPS for Carbon and Oxygen Atoms

A: Carbon					
Band No.	Peak position		Location of carbon atom in symbol (⁶)	Total area (%)	
	Untreated JY	Treated JY		Untreated JY	Treated JY
1	284.38	284.54	—C—C*—/—C*—H	47.94	56.74
2	285.35	285.59	—C*—O—C—	9.99	6.33
3	286.28	286.37	—C*—OH	36.91	24.37
4		287.57	O—C*—O		6.06
5	288.07	288.59	—C*=O	5.16	4.47

B: Oxygen					
Band No.	Peak Position		Location of oxygen atom in symbol (⁶)	Total Area (%)	
	Untreated JY	Treated JY		Untreated JY	Treated JY
1	532.57	532.81	—C—O*H	85.17	79.44
2	531.38	531.38	—C—O*—C—/—C=O*	14.83	20.56

AA-treated jute (AJY) are shown in Figure 4(a,b), respectively. The main components of jute are cellulose and lignin; two distinct peaks of C_{1s} and O_{1s} were observed, at 285 and 533 eV, respectively, in both the treated and untreated jute fibers.^{13–14} The carbon peaks of untreated jute (JY) and treated jute (AJY) are presented in Figure 5(a,b). The presence of four carbon peaks in JY indicates the presence of different types of carbon linkage in the jute surface (JY). A new peak was observed in AJY [Fig. 5(b)] at 287.43, which may be attributed to the —CO—O ester group; this free ester group indicates the presence of AA. The peak positions and locations of the carbon and oxygen atoms and the total area of the curve are shown in Table II (A for carbon, B for oxygen).^{13,15} The changes in total area of different peaks of carbon and oxygen might be because of the addition of various groups of AA to the cellulose of the jute fiber. The atomic concentrations of carbon, oxygen, nitrogen and other elements of both the treated and untreated fibers are tabulated in Table I; the carbon atom concentration of JY was higher and the oxygen concentration lower than those of the treated jute. The carbon:oxygen ratios of the treated and untreated samples were 0.72 and 1.26, respectively. The peak positions and locations of the carbon and oxygen atoms and the total area of the curve are shown in Table II (A for carbon, B for oxygen).^{13,15} A relatively sharp N_{1s} peak was observed in the treated jute compare to that of the untreated jute [Fig. 4(a,b)]. The nitrogen atom, N_{1s} , concentration in the treated jute surface also was higher compared to that of the untreated jute (JY). The N_{1s} peak of AJY was shifted toward a higher bond energy because of the formation of tertiary nitrogen from a secondary moiety [Fig. 6(a,b)]. From the above findings it can be ascertained that AA deposited on the jute surface or that a chemical reaction took place with the cellulose backbone of the jute fiber.¹⁶

Thermal analysis by DSC

DSC thermograms obtained from the untreated AA and AA+silane grafted samples exhibited both endothermic and exothermic transitions, as can be seen in Figure 7, which shows peak temperatures different than those of the sample conditions. The exothermic transition or melting temperature of the untreated sample was 331°C, whereas those of the AA and AA+silane grafted samples were shifted to temperatures of 334°C and 335°C, respectively. The grafted jute fiber had higher thermal stability because of the

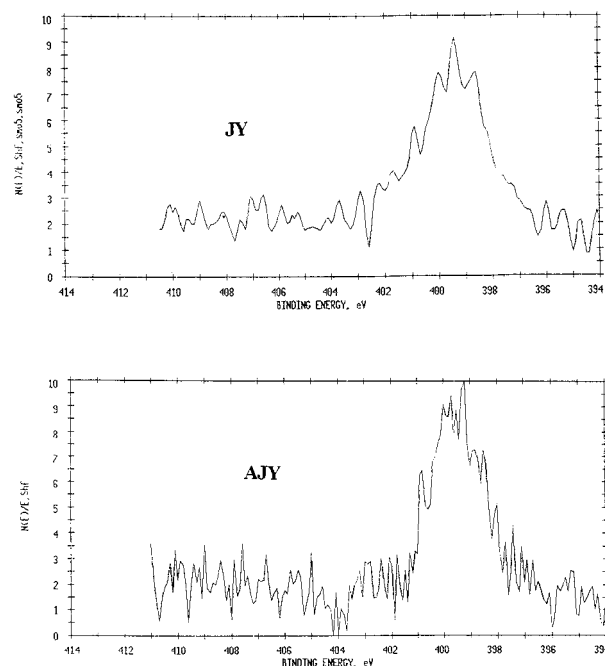


Figure 6 (a) XPS spectrum of nitrogen (N_{1s}) peaks of untreated jute yarn (JY); (b) XPS spectrum of nitrogen (N_{1s}) peaks of acrylamide-grafted jute yarn (AJY).

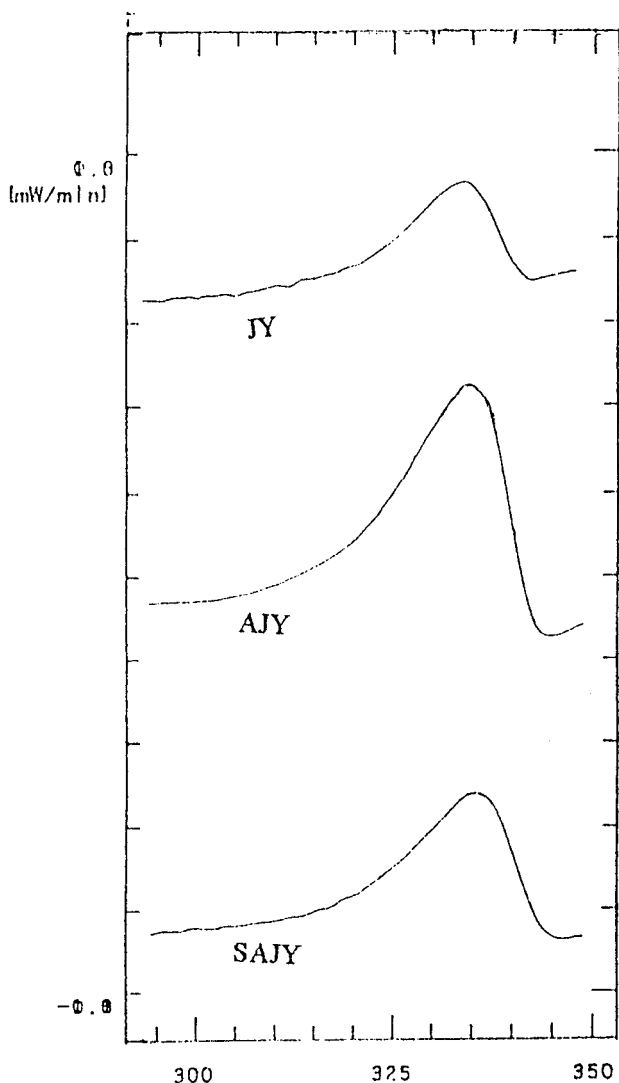


Figure 7 DSC curves of untreated (JY), acrylamide grafted (AJY), and acrylamide + silane grafted (SAJY) of jute yarn.

grafted and crosslinking moiety present in the grafted samples. The appearance of both endo- and exothermic peaks could be related to morphological changes that possibly occurred because of the decomposition of hemicellulose and cellulose, respectively.

Polymer loading

Polymer loading (PL) of the treated jute yarns, which was determined at different monomer concentrations, was accomplished by irradiation of varied duration under a UV lamp. For the AA+MeOH system, AA concentrations varied from 5% to 50%, and irradiation periods varied from 15 to 300 min. The PL values plotted against irradiation time as a function of monomer concentration are shown in Figure 8. It was observed that PL values increased sharply at the initial irradiation time, after which they decreased with any further increase in radiation time and monomer con-

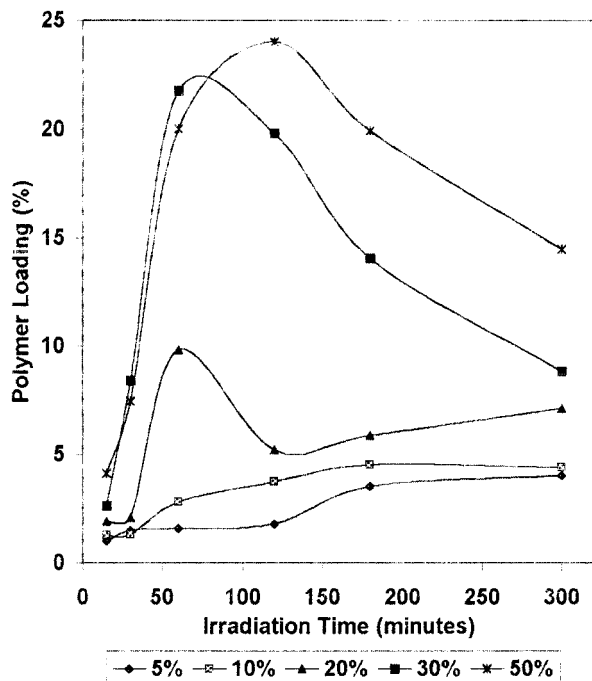


Figure 8 Effect of monomer concentration on polymer loading of jute yarn against irradiation time.

centration. The highest PL value (24%) was obtained at 120 min of irradiation time and 50% AA concentration. At a low concentration there was less polymer loading because vinyl monomers promote a rapid free-radical propagation reaction with the help of a photoinitiator, leading to a network polymer structure

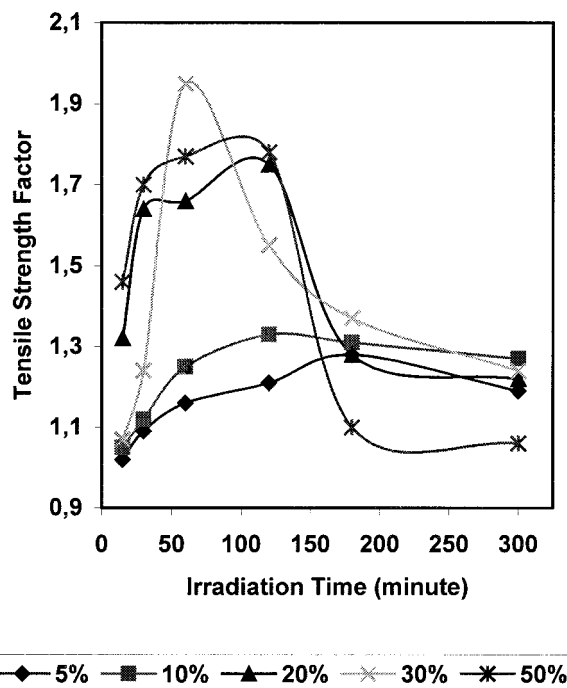


Figure 9 Effect of monomer concentration on tensile strength of jute yarn against irradiation time.

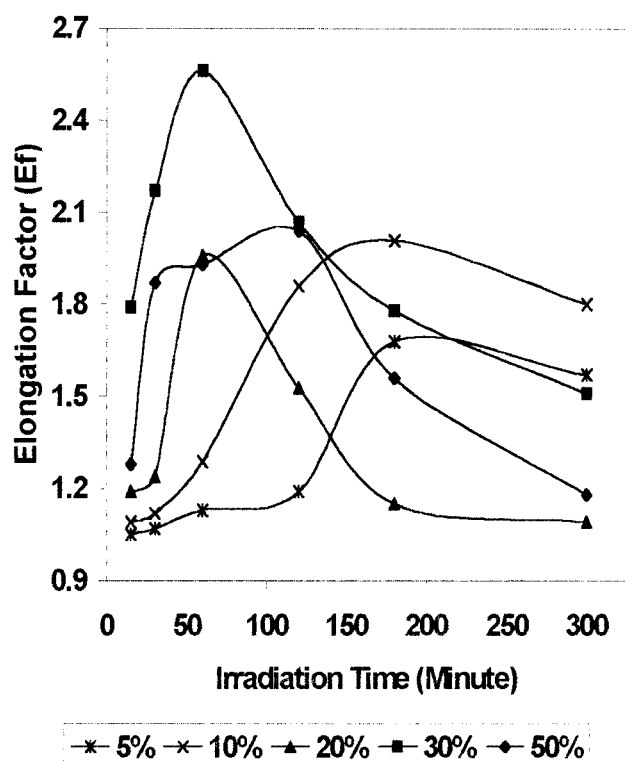


Figure 10 Effect of monomer concentration on elongation at break of jute yarn against irradiation time.

through a graft copolymerization reaction via their double bonds.¹⁷ As AA concentration increased, the amount of residual unsaturation was also increased, with the consequence of a faster rate of formation of a three-dimensional network structure, which caused restricted mobility. The decrease in PL values after attainment of the maximum PL values could be caused by the radiation degradation of higher UV doses.¹⁸ The decrease in PL values at higher monomer concentrations could be associated with the dominance of the homopolymerization reaction between monomers and monomer radicals rather than the monomer and jute cellulose reactions.

Tensile properties

The tensile strength factor (T_f) is the ratio of the tensile strength of the treated jute sample (TS_t) to that of the untreated sample (TS_o). Similarly, the elongation factor (E_f) is the ratio of the elongation at break of the treated jute sample (Eb_t) to that of the untreated virgin sample (Eb_o). The T_f values of AA-treated samples are shown in Figure 9, where T_f values are plotted against irradiation time as a function of AA concentration. Tensile strength increased with irradiation time up to 60 min and then decreased as irradiation time was increased. The highest tenacity factor (1.95) was obtained at 60 min of irradiation time and an AA concentration of 30%. It was observed that the highest T_f value was obtained with sam-

ples whose PL value was 22%. But for those with the highest PL value, the T_f value was 1.78. The decrease in T_f value with increase in PL value may be a result of the formation of excess three-dimensional crosslinked structures in the grafting zone of the cellulose, forming a brittle-type polymer product. The enhanced elongation of the AA-treated jute yarns is depicted in Figure 10, where elongation factor (E_f) is plotted against irradiation time as a function of AA concentration. The highest elongation factor (2.56) was attained by the sample treated with an AA concentration of 30%.

Effect of additive

A minute amount of silane (1%) was used as an additive in a 30% monomer concentration, and the samples were irradiated for 60 min under a UV lamp. The effects of the additive on PL, T_f , and E_f values are shown in Figures 11 and 12. It can be observed that incorporation of an additive into the 30% AA+MeOH system increased the PL, T_f , and E_f properties more than the corresponding properties of the jute samples treated with the AA+MeOH solution only. The PL, T_f , and E_f values increased to 25.4%, 2.18%, and 3.1%, respectively. Alkoxysilanes undergo hydrolysis, condensation, and a bond formation reaction in water-methanol. Silanol undergoes a transformation to polysiloxane through a polymerization reaction.¹⁹ The OH groups of polysiloxane react with the OH groups

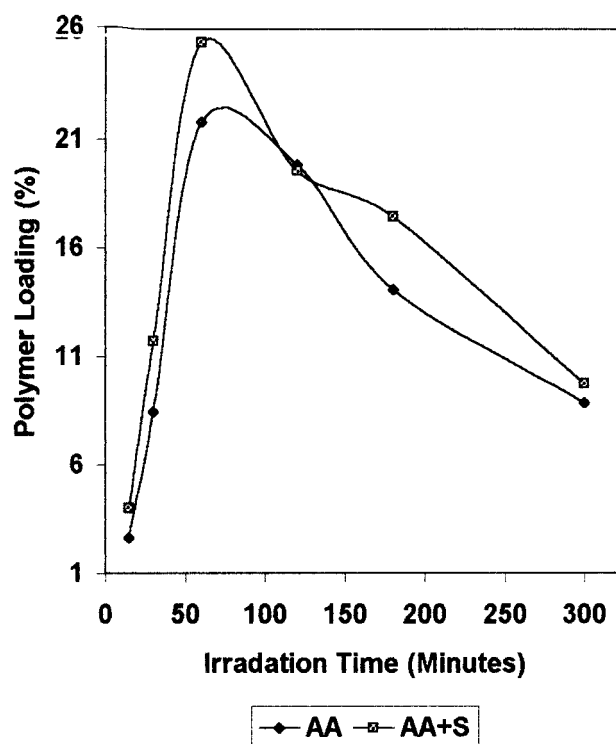


Figure 11 Effect of silane on polymer loading of jute yarn against irradiation time.

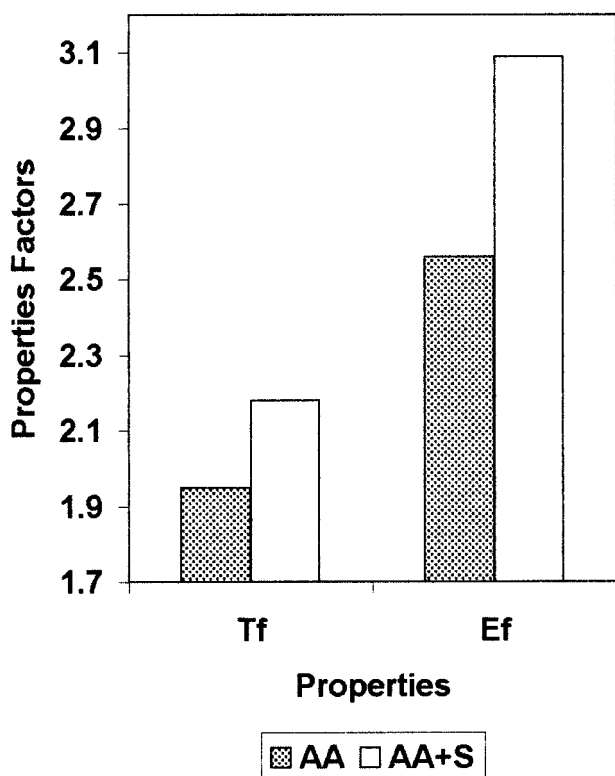


Figure 12 Effect of silane on mechanical properties of treated jute yarn.

of cellulose through a condensation reaction produced by silanized jute.²⁰ During radiation the silanized jute may be grafted with acrylamide, with the resulting three-dimensional network leading to better mechanical properties and grafting.

Simulating weathering effect

Jute samples, both those treated with AA and AA with silane (AA+S) and those untreated, were exposed to an accelerating weathering tester over 600 h to study the properties of the degradation resulting from the weathering. The samples' loss of weight, *TS*, and *Eb* from weathering are shown in Table III. The loss of *TS*

of the blank sample was about 11%, whereas that of the AA-treated sample was about 7% and of the AA+silane-treated sample was about 5%. Similarly, *Eb* loss was 74% in the blank sample and 41.5% and 33.5%, respectively, in the AA and AA+silane samples.

Soil degradation

Jute samples both treated (sample treated with 30% AA and with 30% AA with 1% silane at an irradiation time of 60 min) and untreated were buried in soil (25% water) for 16 weeks in order to study the effect of such an environmental condition on the degradability of the sample. The weight loss and tensile properties [tensile strength (*TS*) and elongation at break (*Eb*)] of the samples were periodically measured, and the results are tabulated in Table IV. The weight loss was minimum for the sample treated with additive (12.4%) compared with the sample treated without any additive (15.8%). But the untreated sample lost more than 36% of its weight, which is the maximum. *TS* and *Eb* loss from degradation was the minimum for the sample treated with silane (additive) at the maximum period of observation.

Water uptake

Water uptake by the treated and untreated samples was monitored at 25°C for 120 min. From the results of equilibrium water uptake against polymer loading, shown in Figure 13, it was observed that the water uptake decreased with an increase in polymer loading. The monomer AA reacted with the OH group of the cellulose backbone of the jute fiber through graft copolymerization. As a result, the hydrophilic nature of jute was reduced.

CONCLUSION

Jute yarns were treated with a single-monomer acrylamide in methanol solution under a low-capacity UV lamp. Optimized conditions such as monomer concen-

TABLE III
Loss of Weight and Mechanical Properties of Untreated and Treated Jute Because of Simulating Weathering

Weathering time (h)	Weight loss (%)			Loss of tensile strength (%)		Loss of elongation of break (%)			
	JY	AJY	SAJY	JY	AJY	SAJY	JY	AJY	SAJY
12	4	2.5	2	1.01	0.98	0.94	3.5	2	2
20	7.5	4	2.5	2.56	1.09	1.06	7	3	2
40	13	9	6.5	4.56	1.89	1.27	11.5	6.5	5.5
80	22	17	13	5.96	2.01	1.98	19.5	14	11.5
150	37	29	21.5	6.34	2.96	2.13	34.5	23.5	19
300	56	35	27.5	8.96	4.87	3.67	48.5	31	26.5
600	77	46	36	11.06	7.31	5.21	74.5	41.5	33.5

JY = virgin jute yarn; AJY = jute yarn treated with AA; SAJY = jute yarn treated with AA and silane.

TABLE IV
Loss of Weight and Mechanical Properties of Untreated and Treated Jute Because of Soil Degradation

Degradation time (week)	Weight loss (%)			Loss of tensile strength (%)			Loss of elongation at break (%)		
	JY	AJY	SAJY	JY	AJY	SAJY	JY	AJY	SAJY
1	8.74	2.75	2.01	11	9.5	5	13	11	6.5
2	13.9	5.98	3.95	26	18	13	24	21.5	15.5
4	21.4	9.63	5.09	41	32	23	39	33	25
8	27.3	13.9	8.91	62	47	31	64	51.5	36
16	36.7	15.8	12.4	83	61	45	86	64	49

JY = virgin jute yarn; AJY = jute yarn treated with AA; SAJY = jute yarn treated with AA and silane.

tration (30%) and radiation time (60 min) attained enhanced tensile strength by 95% and elongation at break by 156% with 22% polymer loading. The XPS studies of acrylamide-treated jute fiber surfaces indicated the presence of AA deposited on the surface of jute yarn. The ESEM and FTIR spectroscopic analyses revealed the presence of AA, which may be deposited or reacted with the cellulose backbone of jute yarn. To further improve the properties of the jute yarn, the additive silane (1%) was added in an optimized solution. The enhanced mechanical properties such as *TS* (118%) and *Eb* (209%) were produced by the addition

of silane. Degradation studies showed that the treated jute yarn, like synthetic products, could be easily degraded in mud without causing any environmental pollution. This will open a new area for finding diverse applications for jute yarn.

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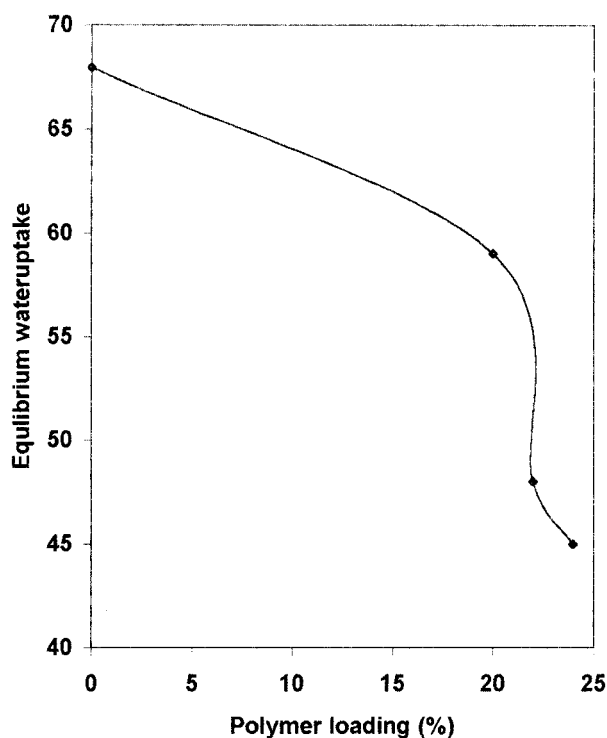


Figure 13 Equilibrium water uptake of jute yarn against polymer loading.