Structure and Physical Properties of Grafted Jute Fiber

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SYNOPSIS

Jute fiber was grafted by acrylonitrile and methylmethacrylate monomers in raw and bleached condition. It was also noted that optimum grafting takes place after about 4 h of grafting. It was also noted that in bleached jute fiber, the grafting percentage is higher at all stages of grafting but the tenacity value at the highest add-on is practically the same as that of raw fiber with much less add-on. The crystalline orientation of the grafted jute fiber was correlated with the fiber tenacity. The moisture regain (%) of the fiber after grafting showed a decreasing trend with an increase in grafting percentage. No structural change of the fiber occurred due to grafting. © 1995 John Wiley & Sons, Inc.

INTRODUCTION

The mechanical properties of grafted cellulosic fibers including cotton have been studied but no systematic work has been reported on the mechanical properties of grafted lignocellulosic fibers. Hence, this work was undertaken to examine the mechanical properties of grafted jute fiber in relation to their fine structure.

Kaizermann et al.[1] observed that in the case of cotton fabrics grafted with polyacrylonitrile, there was significant increase in tenacity after grafting. Iwakura et al.[2] showed that denier and elastic recovery increased with an increase of graft content when cellulosic fibers were grafted with glycidyl methacrylate. Blouin et al.[3] preirradiated cotton yarns and fabrics with gamma rays in nitrogen atmosphere and then treated them with monomer solution (vinyl acetate, acrylonitrile, AN, and methylmethacrylate, MMA) in 75–80% zinc chloride solution. They observed an exceptionally high degree of abrasion resistance for the grafted sample, but the improvement in tear and tensile strength was not appreciable. Prahl et al.[4] reported that the primary effect of grafting vinyl monomers on the cotton fabrics is a substantial increase in elongation and a moderate decrease in tensile strength, resulting in an increase in toughness.

Sakurada[5] and Tsuji and coworkers[6] studied the properties of cotton cloth grafted with polystyrene using the postirradiation technique and observed that there was some loss of strength of cotton fabric. And when the percent graft was higher than 40%, the grafted cotton fabric showed excellent thermoplasticity and the tearing strength of cotton fabric and flex life of cotton fiber were somewhat lowered by grafting. Sakurada et al.[7] grafted styrene onto high tenacity rayon by mutual irradiation in the absence of air and observed that the wet strength of the fiber dropped sharply at a 1.6 Mrad dose rate due to radiation damage. The elongation at break increased with increasing percent graft. Butadiene, butadiene-styrene, or butadiene-AN were also grafted onto cotton and rayon.[7] It was observed that the elasticity markedly improved. Das Gupta[8] reported a substantial increase in tensile strength for cotton fabric grafted with a styrene-MMA mixture by the irradiation technique. Kamagowa and Sekuja[9] grafted acryloamide to cotton fabric by the Ce^{+4} ion initiation technique. The amido group was then methylolated by reacting the grafted cotton fabrics with formaldehyde at pH 11. This group was then reacted with adjacent cellulose molecules through substances such as ethylene urea, melamine etc. They found that the fabrics grafted and cross-linked by this method showed better retention of tensile or tear strength than did ungrafted fabrics cross-linked with a similar cross-linking agent.

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EXPERIMENTAL

Sample Preparation

Raw and bleached olitorius jute were used in this study. The jute fiber was first dewaxed by soxhleting with an alcohol:benzene mixture (1 : 1 v/v). The temperature under the soxhlet apparatus was adjusted so that siphoning occurred within a 2–3 min interval. Each sample was treated for 8 h. The extracted fibers were rinsed with alcohol (to remove adhering benzene from the fibers), and then were washed several times with distilled water to remove other reaction products. The estimation of lignin was done by the TAPPI standard, according to which 15 mL of cold (12–15°C) 72% sulfuric acid was added to 1 g of an oven dry sample. This dissolved the carbohydrates, leaving an insoluble residue of lignin. The mass of this insoluble lignin was determined and hence its percentage.

A part of the dewaxed jute was then bleached by 0.5% volume aqueous hydrogen peroxide solution at 80°C for 2 h. The lignin content of the raw dewaxed jute was found to be 12%; with that of the bleached jute only 8%.

The redox system of grafting was employed to graft the raw dewaxed and bleached jute fibers. AN and MMA were used for the present study. Ceric ammonium sulfate in 1% sulfuric acid was taken as the initiator. The grafting reaction was carried out in an inert (nitrogen) atmosphere at 40°C. The fiber to monomer ratio was 1 : 50. The only parameter that was varied in the grafting reaction was the time of reaction; with increasing time the graft add-ons increased.

The single filaments were obtained after combing and hand cleaning the fiber samples. They were then cut into a length of 5 cm and conditioned at 60–70% relative humidity (RH) and at room temperature (25°C). The average linear density was determined by cutting and counting 500 pieces, 2 mm length, of conditioned fibers and weighing them in a microbalance.[10]

The single fibers were tested for filament tenacity and breaking extension in an INSTRON tensile strength tester in a controlled humidified room. The setting of the Instron tester was the same for all the samples, the rate of extension being 0.5 cm/min and the test length was 1 cm. The fibers were tested at 60–70% RH at 25°C. The number of fibers analyzed

Table I Mechanical Properties of Grated Jute

Sample	Time of Reaction (h)	Filament Tenacity (g/tex)	Breaking Extn (%)	Angle of Orientation
Raw Tossa	—	33.1	2.5	10°07′
14.8% AN grafted	2	44.5	2.4	9°17′
20.4% AN grafted	4	40.2	2.4	12°14′
21.9% AN grafted	6	26.2	2.5	13°00′
Bleached Tossa	—	30.8	2.1	11°25′
17.8% AN grafted	1	31.5	2.1	12°00′
26.8% AN grafted	2	45.6	2.0	9°30′
27.4% AN grafted	3	33.8	1.9	11°45′
29.0% AN grafted	4	27.9	1.9	12°30′
30.3% AN grafted	5	25.9	1.6	13°00′
Raw Tossa	—	47.2	2.5	9°25′
15.7% MMA grafted	1	45.4	2.3	10°00′
18.9% MMA grafted	2	44.6	2.3	10°25′
23.1% MMA grafted	4	41.2	2.1	11°20′
23.9% MMA grafted	5	40.1	2.2	12°30′
Bleached Tossa	—	40.2	2.0	11°30′
17.8% MMA grafted	1	41.3	1.9	11°00′
20.0% MMA grafted	2	40.9	1.8	10°30′
24.7% MMA grafted	3	40.5	1.9	11°20′
26.2% MMA grafted	4	37.6	2.0	13°30′
26.9% MMA grafted	5	35.2	1.9	14°00′

Table II Moisture Regain (%) of AN Grafted Tossa Jute Fiber

Sample	Moisture Regain (%)		
	65% RH	75% RH	90% RH
Raw dewaxed tossa	14	18	22
14.8% grafted	12	15	18
20.4% grafted	10	13	17
21.9% grafted	9	11	14

in each case was 100, which included only those fibers that broke at or near the middle of the specimen.

Determination of Moisture Regain

For determining the regain values of raw and grafted jute fibers at different RHs, duplicate samples, each weighing about 1 g were taken in different weighing bottles and dried over P_2O_5 in vacuum for 3 weeks. The weight of the dry samples were taken and the samples were then conditioned at progressively increasing humidity using sulfuric acid solution of suitable concentrations. From these, percentage regain values (i.e., moisture content calculated on the dry weight of the fiber) were determined. The investigation was carried out at room temperature.

Determination of Fine Structure

The experimental arrangement to obtain the X-ray photographs was based on the method followed by Hermans and Weidinger.[11] In the present work, Ni filtered CuK_α radiation from a Philips X-ray tube maintained at 40 kV and 20 mA was used. The bundle of fibers parallelized with a fine needle was mounted and kept taut in a stretching frame. Each bundle was of 2.5 cm length and 30 mg weight. The specimen to film distance was 30 cm. The specimen size, exposure time, and photographic technique were standardized as far as practicable.

Table III Moisture Regain (%) of AN Grafted Bleached Tossa Jute Fiber

Sample	Moisture Regain (%)		
	65% RH	75% RH	90% RH
Raw bleached tossa	16	18	21
17.8% grafted	15	17	19
20.8% grafted	13	16	18
27.4% grafted	12	15	17
29.0% grafted	9	13	15
30.3% grafted	8	12	14

Table IV Degree of Crystallinity of Grafted Jute Fiber

Sample	Degree of Crystallinity (%)	Degree of Crystallinity Corrected to Equal wt Cellulose (%)
Raw tossa jute	53.0	53.0
14.8% AN grafted	45.2	53.0
20.4% AN grafted	41.4	52.4
21.9% AN grafted	41.2	53.0
Raw bleached tossa	57.0	57.0
17.8% AN grafted	46.9	57.2
20.8% AN grafted	45.0	56.8
27.4% AN grafted	41.4	57.2
29.0% AN grafted	40.6	57.0
30.3% AN grafted	40.0	57.4
Raw tossa jute	55.0	55.0
15.7% MMA grafted	47.0	55.7
18.9% MMA grafted	44.6	54.9
20.4% MMA grafted	43.8	55.2
23.1% MMA grafted	42.3	55.0
23.9% MMA grafted	42.0	55.2
Raw bleached tossa	61.5	61.5
17.8% MMA grafted	46.0	61.9
20.0% MMA grafted	43.0	61.4
24.7% MMA grafted	41.0	61.2
26.2% MMA grafted	43.5	61.2
26.9% MMA grafted	46.8	61.6

RESULTS AND DISCUSSION

The results are tabulated in the tables. From Table I, it is seen that optimum grafting for both AN and

Table V Moisture Regain (%) of MMA Grafted Tossa Jute Fiber

Sample	Moisture Regain (%)		
	65% RH	75% RH	90% RH
Raw dewaxed tossa	14	18	22
15.7% grafted	13	17	22
18.9% grafted	13	17	21
20.4% grafted	13	16	20
23.1% grafted	12	15	19
23.9% grafted	11	14	19

Table VI Moisture Regain (%) of MMA Grafted Bleached Tossa Jute Fiber

Sample	Moisture Regain (%)		
	65% RH	75% RH	90% RH
Raw bleached tossa	16	18	21
17.8% grafted	16	18	21
20.0% grafted	15	17	20
24.7% grafted	15	17	20
26.2% grafted	14	16	19
26.9% grafted	13	15	18

MMA monomers takes place after 4 h of treatment. In bleached jute fiber the grafting percentage is higher at all stages of grafting. Trivedi and Mehta[12] obtained maximum grafting of 45% when lignin was extracted from 14 to 1.8% in jute fiber. Similar results were obtained by Majumdar and Rapson[13] in the case of grafting of jute by styrene. The increased grafting onto bleached jute fiber was attributed to its greater accessibility to monomers.

Table I also shows that for AN grafted jute fiber, there is deterioration in crystalline orientation with increase in add-on percentage, except for 14.8% grafted raw and 26.8% grafted bleached jute fibers. The crystalline orientation improves in these two cases and the improvement is also reflected in the tenacity values (Table I). However, for MMA grafted bleached jute fibers there is a constant fall in crystalline orientation with an increase in add-on percentage. Thus it appears that at a certain level of grafting by AN monomers, chain molecules in jute fibers are in a better position to realign themselves and at a higher level of grafting this process of realignment appears to be disturbed. For MMA grafting no such realignment appears to take place and this might be due to the fact that MMA is a rigid monomer. At a higher level of grafting, that is as more and more grafted molecules enter the fiber structure, the deterioration of crystalline orientation takes place. The fiber tenacity values as recorded in Table I are in agreement with fine structural changes.

Moisture Regain

The moisture regain values of raw and bleached jute fibers grafted by AN and MMA at 65, 75, and 90% RH are shown in Tables II and III (for AN) and Tables V and VI (for MMA).

As reported in the case of cotton and other cellulosic fibers,[15–17] the moisture regain values showed a decreasing trend with an increase in graft content. This is due to the fact that with the increase in graft add-on, more and more hydroxyl groups in the cellulose backbone chain are being used up for grafting, making the fibers more hydrophobic.

Fine Structure

From the crystallinity values of raw and differently grafted fibers (Table IV) it is observed that when these values are computed for grafted jute fibers for equal weight of cellulose, there is no appreciable change in these values. This observation is in agreement with the results reported by other workers.[2,14]

Determination of Unit Cell

Values of unit cell dimensions, for example a, b, c, and β for the raw and grafted jute fibers were determined from the relation applicable to monoclinic unit cell.

Table VII Dimensions of Unit Cell of Raw and Differently Grafted Jute Fiber

Sample	a (°A)	b (°A)	c (°A)	β (degrees)
Raw dewaxed tossa jute fiber	8.2	10.1	7.7	85
14.8% AN grafted tossa jute fiber	8.3	10.2	7.8	84
21.9% AN grafted tossa jute fiber	8.3	10.2	7.9	85
Raw bleached tossa jute fiber	8.5	10.2	7.7	86
17.8% AN grafted bleached tossa jute fiber	8.4	10.3	7.9	85
30.3% AN grafted bleached tossa jute fiber	8.4	10.3	7.8	84
15.7% MMA grafted tossa jute fiber	8.6	10.3	7.9	84
23.9% MMA grafted tossa jute fiber	8.3	10.2	7.9	85
17.8% MMA grafted bleached tossa jute fiber	8.2	10.0	7.8	84
26.9% MMA grafted bleached tossa jute fiber	8.3	10.1	7.7	85

$$d^{hkl} = \frac{1}{\left(\frac{h^2}{a^2\sin^2\beta} + \frac{l^2}{c^2\sin^2\beta} - \frac{2hl}{ac\sin^2\beta} + \frac{k^2}{l^2}\right)^{1/2}}$$

where d_{hkl} is the lattice spacing corresponding to hkl planes.

The unit cell dimensions computed from the above relation for raw and grafted jute fiber are shown in Table VII. It is observed that no appreciable change in unit cell dimensions occurs due to grafting indicating thereby that there is no structural change of the fibers as a result of the grafting process.

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