Graft Copolymerization of Nitrile Monomers onto Bleached Jute Fiber Using Potassium Persulfate System and Their Textile Characteristics

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ABSTRACT: Graft copolymerization of nitrile monomers, such as acrylonitrile and methacrylonitrile, onto bleached jute fiber was carried out by using $K_2S_2O_8/FeSO_4$ redox system in nitrogen atmosphere and their effect on the textile characteristics was also investigated. Percent graft yield increased with the increase of concentrations of monomer, initiator, and catalyst, reaction time, and reaction temperature up to a certain value, and, thereafter, it decreased. The effect of percent grafting efficiency was similar to that of percent graft yield, except for the monomer concentration. The increase of percent graft yield was dependent on the availability of jute-macroradicals as well as monomer radicals. Sometimes the predominancy of homopolymerization over grafting and the premature termination of growing grafted chains occurred because of the higher monomer

INTRODUCTION

Jute, a lignocellulosic fiber, is the most abundant renewable agricultural raw material and is transformed into multifarious products affecting every phase of our daily life. It is a bast fiber of rather coarse character. Unlike cotton fiber, jute is not uniform in chemical composition, which is evidenced by the multicellular structure of the fiber. However, jute fiber is facing tough competition from the synthetic fibers, as the latter offers certain advantages in terms of many useful properties, durability, and production economics. The survival of jute in competition with the synthetic fibers lies in the retention of jute as a preferred material in the eyes of the consumers; it must be constantly developed and improved to retain that status.

The graft copolymerization onto textile fibers is a challenging field of research having enormous future prospects. The synthetic graft copolymers of natural polymers, such as cotton and other cellulosic fibers, silk, wool, etc., exhibited excellent properties in addition to showing their original properties. Although a lot of work had been done on graft copolymerization radicals and excess primary radicals, $SO_4^{-\bullet}$ and ${}^{\bullet}OH$, from $K_2S_2O_8$ initiator. The percent graft yield of acrylonitrile and methacrylonitrile was 20.5 and 29.1%, respectively. Higher graft yield for methacrylonitrile might be due to the methyl group present in it. Infrared spectra at 2229–2235 cm⁻¹ of acrylonitrile- and methacrylonitrile-grafted jute strongly supported the graft formation. Grafting of jute fiber improved the thermal stability, protected from photooxidative degradation, and decreased swellability as well as dyeability, etc. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 92: 3622–3629, 2004

Key words: graft copolymerization; percent graft yield; nitrile monomer; jute fiber; tenacity

of vinyl monomers onto cellulosic, protein, and other textile fibers,^{1–3} comparatively much less was reported on grafting of vinyl monomers onto jute fiber. Therefore, graft copolymerization of vinyl monomers onto jute fiber has received considerable attention in the recent years. However, some work on graft copolymerization of methyl metacrylate,^{4,5} acrylamide,^{6,7} and acrylic acid⁸ onto jute fiber had been done. Trivedi and Metha⁹ reported grafting of acrylonitrile onto defatted and bleached jute fibers with ceric ion initiation. Again, Das et al.^{10,11} also studied kinetics of grafting of acrylonitrile onto jute fiber by using ceric ion initiators. Graft copolymerization of acrylonitrile onto jute fibers¹² and acetylated jute fiber¹³ was also reported by Ce⁴⁺-hippuric acid and V⁵⁺-cyclohexanone redox initiator systems, respectively. So, no information was available on the grafting of acrylonitrile and methacrylonitrile onto jute fiber by using potassium persulfate as redox initiator.

The present report investigates the graft copolymerization of nitrile monomers, such as acrylonitrile (AN) and methacrylonitrile (MAN), onto bleached jute fiber initiated by potassium persulfate and ferrous sulfate redox system under variable reaction conditions and the effect of grafting on the inherent properties of the jute fiber for better competitive use. The grafting was determined on the basis of increased weight of the fiber treated.

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EXPERIMENTAL

Materials

Raw jute fiber (*Corchorus olitorius*, Tossa variety) was collected from Rajshahi Jute Mills Ltd. (Bangladesh). A portion of jute fiber between 70 and 100 cm from the bottom was taken, combed, blended, and washed with 6.5 g soap flake and 3.5 g soda per liter at 75°C for 30 min.¹⁴ Then, the jute fiber was bleached with sodium chlorite solution of strength 0.5 wt % at pH 4 and at 85–90°C for 90 min.^{14,15}

Acrylonitrile (RDH, Germany) and methacrylonitrile (RDH, Germany) was used fresh after distillation under reduced pressure. All other chemicals [e.g., potassium persulfate ($K_2S_2O_8$), ferrous sulfate (FeSO₄), etc.] used were of analytical reagent (AR) grade and were used as such without further purification.

Method of grafting

The graft copolymerization of jute fiber was carried out in a 100-ml stoppered Erlenmeyer flask. Polymerization was done with 10–120% monomer, 0.2–2.0% potassium persulfate as initiator, and 0.2–2.0% ferrous sulfate as catalyst based on the weight of fiber at $30-100^{\circ}$ C for 0.5–4.0 h in the fiber–liquor ratio of 1 : 50. At the end of the desired reaction period, the jute fiber was thoroughly washed with boiling soap solution (2.0%) to remove the loosely adhering polymer, washed with distilled water, and dried.^{16–18} Percent graft yield and graft efficiency were calculated according to the formula

% graft yield =
$$\frac{B-A}{A} \times 100$$

% grafting efficiency = $\frac{B-A}{C} \times 100$

where *A* is the weight of ungrafted jute fiber before treatment, *B* is the weight of the grafted jute fiber after treatment, and *C* is the weight of total monomer used.

IR spectroscopy

Jute sample was mixed with KBr in a ratio of 1 : 100 and a pellet was prepared. Then, the IR spectra of this KBr pellet was recorded with an IR-470 infrared spectrometer (Shimadzu, Kyoto, Japan).

Thermogravimetric analysis

The thermogravimetric analysis (TGA) of the fabric sample was done by using a Mettler TG-50 (Shimadzu, Kyoto, Japan). The heating rate throughout the study was 10° C /min. The measurement was

Method of dyeing

Dyeing was carried out with 2.0% dye (Congo red or Direct blue 1) and 10.0% sodium chloride as electrolyte on the basis of fiber at the boiling point for 60 min in the fiber–liquor ratio of 1 : 30.¹⁵ Absorption of dye was determined colorimetrically as

Absorption of dye (%) =
$$\frac{D_o - D_e}{D_o} \times 100$$

where D_o and D_e are the original and exhausted dye bath concentration, respectively.

Method of measurement of tenacity

Breaking strength of jute fiber was measured by Torsee's Schopper Type OS-100 Tensile Tester. The length of each specimen (total length, 25 cm; weight, 0.5 g) between the jaws of the machine was maintained by 10 cm and a twist per 2 cm was given along the length of the fiber. The average breaking load (the mean of 25 readings) of the fiber was measured in kg/yarn.^{14,20} Tenacity was calculated by the formula²¹

Tenacity =
$$\frac{\text{Average breaking load}}{\text{Denier}} \times \text{g/denier}$$

where

Denier =
$$\frac{\text{Weight of the sample in grams}}{\text{Length of the sample in meters}} \times 9000$$

Method of sunlight exposure

The jute sample was exposed directly on a flat board under sunlight without any protection from weathering, but was protected from rain, snow, etc. At the same time and in the same place, bleached and grafted jute fibers were exposed under the sun for 7 h each day and continued for 350 h.

Method of heating

Jute sample was placed in an electric oven in the presence of air at 30–160°C for 3 h and its breaking strength was measured as above.

RESULTS AND DISCUSSION

Effect of monomer concentration

The effect of variation of monomer concentration in the presence of potassium persulfate and ferrous sulfate as redox initiator system on percent graft yield and grafting efficiency is shown in Figure 1. From Figure 1, it can be seen that the percent graft yield increased with the increase of concentrations of both monomers, AN and MAN. The optimum percent graft yield was 17.2 and 20.9% for 90% AN and 100% MAN, respectively, and the corresponding percent grafting efficiencies were 19.11 and 20.9%. The increase in the monomer concentration in the aqueous medium increased the number of monomer radical, which caused an increase in percent graft yield. Although the percent graft yield increased, the percent grafting efficiency decreased means at higher concentration monomer was less reactive toward graft formation. Again, the slight increase of percent graft yield above optimum monomer concentration might be due to the cross-termination of the reactive monomer or radical sites. In this case, the rate of combination and disproportionation of monomer radical itself was faster than that of their combination with jute radicals. A similar explanation for the enhancement of percent graft yield was observed by Hebeish et al.²²

Effect of initiator concentration

The percent graft yield and grafting efficiency versus initiator concentration are shown in Figure 2. The percent graft yield increased with the increase of potassium persulfate concentration up to 1.4% for AN and 1.6% for MAN and beyond which it decreased. At these concentrations of initiator, the percent graft yields were 20.2 and 24.8%, respectively, and the corresponding grafting efficiencies were 22.44 and 27.55%, respectively. The nature of the grafting efficiency curve was similar to that of percent graft yield. The enhancement of percent graft yield by the addi-



Figure 1 Effect of monomer concentration on percent grafting and grafting efficiency of nitrile monomers onto bleached jute fiber ($K_2S_2O_8$, 1.0%; FeSO₄, 1.0%; time, 60 min; temp., 100°C; ratio 1 : 50). Grafting: \bigcirc , acrylonitrile; \triangle , methacrylonitrile; grafting efficiency: \bullet , acrylonitrile; \blacktriangle , methacrylonitrile.



Figure 2 Effect of initiator $(K_2S_2O_8)$ concentration on percent grafting and grafting efficiency of nitrile monomers onto bleached jute fiber (monomer, 100.0%; FeSO₄, 1.0%; time, 60 min; temp., 100°C; ratio 1 : 50). Grafting: \bigcirc , acrylonitrile; \triangle , methacrylonitrile; grafting efficiency: \blacklozenge , acrylonitrile; \blacktriangle , methacrylonitrile.

tion of more initiator might be attributed to an increase in the number of active sites on the backbone of jute fibers arising from the attack of the diffused $K_2S_2O_8$ molecule on the polymer matrix. Again, during bleaching, cellulose–lignin linkages were broken and some of the functional groups of cellulose were freed. This created additional sites where $K_2S_2O_8$ might be preferentially adsorbed.

The percent graft and grafting efficiency decreased after 1.4 and 1.6% initiator concentrations for AN and MAN, respectively. This retarding effect of initiator on percent graft yield at higher concentration might be the cause of predominancy of homopolymerization over grafting and the premature termination of growing grafted chain by excess primary radicals, $SO_4^{-\bullet}$ and $^{\bullet}OH$ resulted from the decomposition of the excess of the initiator.¹⁵

Effect of catalyst concentration

The effect of variation of catalyst concentration on percent graft yield is shown in Figure 3. The percent graft yield increased with the increase of Fe(II) ion concentration in the reaction medium up to 1.2% for AN and 1.4% for MAN. At these concentrations of catalyst, the maximum percent graft yields were 20.4 and 25.6%, respectively. The higher concentration of ferrous ion in solution increased the Fe(II)–K₂S₂O₈ transient adduct, which underwent forced decomposition at the fiber surface, resulting in the production of jute macroradicals at a faster rate. Again, ferrous sulfate in the medium assisted primary radical formation as well as the formation of graft copolymer by swelling of the substrate, and also by acting as a



Figure 3 Effect of catalyst (FeSO₄) concentration on percent grafting and grafting efficiency of nitrile monomers onto bleached jute fiber (monomer, 100.0%; $K_2S_2O_8$, 1.0%; time, 60 min; temp., 100°C; ratio 1 : 50). Grafting: \bigcirc acrylonitrile, \triangle , methacrylonitrile; and Grafting efficiency: \bullet acrylonitrile, \blacktriangle methacrylonitrile.

catalyst in the hydrolysis of cellulose leading to uncoiling of the chains and hence, improving the monomer accessibility. Above 1.2 and 1.4% ferrous sulfate for AN and MAN, respectively, the decrease in percent grafting and grafting efficiency might be attributed to a detrimental factor arising from excess Fe(III) ions produced on oxidation of Fe(II), leading to retardation of rate through premature termination of the growing grafted chains. It may also be explained that a higher concentration of ferrous sulfate, however, might cause degradation of the backbone cellulose as well as of the graft chain.²³ So, ferrous sulfate enhanced both the graft formation as well as the homopolymer formation.

Effect of reaction time

The effect of reaction time on percent grafting is shown in Figure 4. It can be observed from Figure 4 that the percent graft yield of nitrile monomer onto jute fiber increased with the progress of reaction time up to 120–150 min and thereafter it remained almost same or slightly decreased. The maximum percent graft yield of AN and MAN obtained at 150 and 120 min was 20.5 and 28.9%, respectively. The rate of grafting increase with time was due to higher copolymerization reaction and a lower homopolymerization reaction. Sufficient time enhances swelling of the fiber and diffusion of both monomer and initiator, and thereby, gave rise to a substantial amount of graft formation. Again, the decrease of percent graft yield after optimum reaction time might be explained from the concept of the kinetics and statistics of random degradation. If the extent of reaction proceeded, then



Figure 4 Effect of reaction time on percent grafting and grafting efficiency of nitrile monomers onto bleached jute fiber (monomer, 100.0%; $K_2S_2O_8$, 1.0%; FeSO₄, 1.0%; temp., 100°C; ratio 1 : 50). Grafting: \bigcirc , acrylonitrile; \triangle , methacrylonitrile; grafting efficiency: \bullet , acrylonitrile; \blacktriangle , methacrylonitrile.

the extent of degradation occurred. After the optimum reaction time, the monomer was not assumed to form graft with fiber, but the excess monomer was used to form homopolymer.

Effect of reaction temperature

The effect of rise of temperature from 30 to 100°C on the percent graft yield is shown in Figure 5. From Figure 5 it can be seen that the percent graft yield increased with the increase of reaction temperature up



Figure 5 Effect of reaction temperature on percent grafting and grafting efficiency of nitrile monomers onto bleached jute fiber (monomer, 100.0%; $K_2S_2O_8$, 1.0%; FeSO₄, 1.0%; time, 60 min; ratio 1 : 50). Grafting: \bigcirc , acrylonitrile; \triangle , methacrylonitrile; grafting efficiency: \bullet , acrylonitrile; \blacktriangle , methacrylonitrile.

Monomers	Monomer	Initiator	Catalyst	Reaction	Reaction	
	conc. (%)	conc. (%)	conc. (%)	time (min)	temp. (°C)	
Acrylonitrile	90	1.4	1.2	150	50	
Methacrylonitrile	100	1.6	1.4	120	80	

 TABLE I

 Effective Optimum Conditions of Grafting of Acrylonitrile and Methacrylonitrile onto Bleached Jute Fiber

to 50°C for AN and 80°C for MAN and beyond which it decreased. At 50 and 80°C, the percent graft yield of AN and MAN were 20.5 and 29.1%, respectively. The increase in percent graft yield at higher temperature might be due to the increase in the rate of production of active free radicals, which increased the number of grafting sites at a higher rate as well as the rate of graft initiation by jute radicals. Again, the increase in temperature also increased the rate of diffusion of monomer into the fiber matrix where grafting was also initiated by complexed monomer. Similar phenomena were observed by Samal et al. in the grafting of AN onto nylon 6 and silk fibers.^{24,25} However, at ceiling temperature, the propagation and depropagation rates were equal when the net rate of polymer production was zero and monomer underwent termination by both coupling and disproportionation.

The decrease in percent graft yield beyond 50°C for AN and 80°C for MAN, respectively, might be the result of an increase in the formation of homopolymer and the premature termination of growing grafted chains by excess Fe(III) ions produced on oxidation of Fe(II) ions. On the other hand, beyond the ceiling temperature, the rate of polymer formation was less than that of polymer degradation.²⁶ Effective optimum conditions of graft copolymerization for AN and MAN are listed in Table I.

Reactivity of acrylonitrile and methacrylonitrile

Both AN and MAN have one double bond, but MAN contains one excess substituent methyl group (— CH_3) that is absent in AN. So, due to the presence of the methyl group, MAN might have the capability of producing more reactive sites than AN. Thus, grafting properties of MAN were better than that of AN. Initiator, catalyst, and other experimental conditions were sometimes enhanced to produce more radical sites.

IR spectroscopy

The infrared spectra of bleached, AN-, and MANgrafted fibers are shown in Figure 6. The absorption bands of hydroxyl and carboxyl groups of bleached jute fiber were around 3315–3455 and 1639–1725 cm⁻¹, respectively. However, an additional peak was observed for both AN- and MAN-grafted fibers at 2239–2235 cm⁻¹, which are characteristic of the nitrile group of AN and MAN monomers incorporated with bleached jute fibers.

Thermogravimetric analysis

The thermal behavior of bleached, AN-, and MANgrafted fibers was examined by a study of their TGA thermograms, and the loss in weight corresponding to decomposition temperatures and times are listed in Table II. From the table, it can be seen that the loss in the same amount of weight of AN-grafted jute fiber required a higher temperature and more time than ungrafted bleached jute fiber (i.e., for 60% weight loss, AN-grafted jute fiber required 519.4°C and 48.6 min, where as bleached jute fiber required 352.7°C and 38.6 min). However, in the case of MAN-grafted fiber, this effect was observed above 60% weight loss. From this study, grafting of nitrile monomers onto bleached jute fiber increased the thermal stability. Again, the thermal stability of AN-grafted fibers is much higher than MAN-grafted fiber.



Figure 6 FTIR spectrographs of bleached, AN-, and MANgrafted jute fibers. (a) Bleached jute fiber; (b) AN-grafted jute fiber; (c) MAN-grafted jute fiber.

Loss in weight (%)	Bleached jute fiber		AN-grafted jute fiber		MAN-grafted jute fiber	
	Temp. (°C)	Time (min)	Temp. (°C)	Time (min)	Temp. (°C)	Time (min)
0	26.7	0	26.5	0	27.3	0
-10	84.3	7.1	269.6	23.7	120.3	10.5
-20	283.8	26.6	315.9	28.4	250.0	23.6
-60	352.7	33.6	519.4	48.6	348.1	33.3
-80	438.7	42.1	703.2	67.1	479.6	46.3
-90	462.6	44.3	781.2	75.0	536.3	52.1

TABLE II Variation of Loss in Weight of Bleached, AN-, and MAN-Grafted Jute Fibers Against Decomposition Temperature and Time Recorded by TGA

Effect of heating temperature on tenacity of jute fiber

From Figure 7 it can be seen that tenacity or tensile strength of bleached (ungrafted), AN-, and MANgrafted jute fibers increased with the rise of heating temperature up to 100°C for 3 h, then it decreased with a further rise of temperature. The moisture present in the bleached and grafted fibers might be eliminated with the rise of temperature, thus causing increased tenacity of the dried fibers. The tenacity of bleached, AN-, and MAN-grafted fibers at 100°C was 2.04, 2.1, and 2.19 g/denier, respectively, which were 20.00, 14.13, and 15.26% higher to that of their control. In all cases, the tenacity of bleached and grafted jute fibers decreased above 100°C. Most probably, this was due to the presence of lignin in jute fiber. The lignin molecules started to melt at around 120°C; as a result, the rupture of the lignin incrustant occurred.²⁷ At 160°C, the losses in tenacity were 19.41, 15.76, and 16.32%, respectively. Again, the tenacity of grafted jute fibers was higher than that of bleached jute fiber (Fig. 7). This is caused by grafting of jute fiber with AN and

MAN; the monomer might mechanically bind the cellulosic chains or microfibrils conferring additional strength to jute fiber.²¹

Effect of sunlight exposure on tenacity of jute fiber

The tenacity of both bleached, AN-, and MAN-grafted fibers decreased with the progress of exposure time when they were exposed to open air under the sun, shown in Figure 8. The loss in tenacity of bleached, AN-, and MAN-grafted fibers for 300 h exposure was 27.05, 22.8, and 25.25%, respectively. The loss in tenacity was, probably, due to the presence of lignin content in jute. The photooxidation reaction was initiated by lignin and caused degradation of jute in all possible manners through the formation of hydrogen peroxide.²⁸ Again, the affinity of grafted jute fiber toward moisture was found to decrease with the increase of polymer loading onto jute fiber. So, it is thought that the reduction of the affinity of jute fiber toward moisture by graft copolymerization might render it less susceptible to the degrading action of light.²⁹ For this



Figure 7 Effect of heating temperature on the tenacity of bleached, AN-, and MAN-grafted jute fibers. \bigcirc , bleached jute; \triangle , acrylonitrile grafted jute; \diamondsuit , methacrylonitrile grafted jute.



Figure 8 Effect of sunlight exposure on the tenacity of bleached, AN-, and MAN-grafted jute fibers. \bigcirc , bleached jute; \triangle , acrylonitrile grafted jute; \diamondsuit , methacrylonitrile grafted jute.

Exposure	Fastness grade and color					
period (h)	Bleached jute	N-modified jute	MAN-modified jute			
0	5	5				
	White	Cream white	Cream white			
50	4	4–5	4–5			
	Slightly yellowish	Whiteness decreased	Whiteness decreased			
100	3–4	4	4–5			
	Slightly yellowish	Whiteness decreased	Whiteness decreased			
150	3	4	4			
	Slightly yellowish	Slightly yellowish	Slightly yellowish			
200	2–3	3–4	3–4			
	Slightly yellowish	Slightly yellowish	Slightly yellowish			
250	2–3	3–4	3–4			
	Yellowish	Slightly yellowish	Slightly yellowish			
300	2	3	3			
	Yellowish	Slightly yellowish	Slightly yellowish			
350	2	3	3			
	Yellowish	Slightly yellowish	Slightly yellowish			

TABLE III Color Fastness and Change in Color of Bleached, AN-, and MAN-Grafted Jute Fibers on Exposure to Sunlight in Air

reason, tenacity of grafted jute fibers was lower than that of bleached jute fiber.

with organic acid was good, whereas that of inorganic acid was unsatisfactory.

Dyeing characteristics

Bleached and grafted jute fibers were dyed in an aqueous medium with 2.0% direct dye (Congo red and Direct blue 1), 5% NaCl as electrolyte at 70°C for 60 min.³⁰ The absorptions of Congo red and Direct blue 1 by bleached, AN-, and MAN-grafted fibers were 98.38 and 79.05, 85.25 and 60.55, and 74.1 and 59.35%, respectively. The grafting of nitrile monomers increased the hydrophobicity of jute fiber, due to which lower dye absorption occurred in aqueous medium. Thus, the absorption of dye depended on the characteristics of dye and base fiber, and on the experimental conditions.

Color fastness characteristics

Change in color of bleached, AN-, and MAN-grafted jute fibers on exposure to sunlight in air are listed in Table III. It can be seen from Table III that bleached, AN-, and MAN-grafted jute fibers became yellow on exposure to sunlight in air and this yellowing effect was due to the photochemical degradation of lignin component in jute in the presence of oxygen or moisture, which resulted in the formation of quinonoid-type compounds.³¹ The modified jute showed lower yellowing rate because of the low rate of oxygen or moisture diffusion to the site of fading. There was no effect of 0.5% soap solution, and washing temperature up to boiling of bleached, AN-, and MAN-grafted jute fiber on change in color. Color fastness to spotting

CONCLUSION

Potassium persulfate initiated graft copolymerization of AN and MAN was studied onto bleached jute fiber. The increase or decrease of percent graft yield depends on the number of both jute-macroradicals and monomer radicals whose production also depended on the initiator, catalyst, and other parameter variables. Excess of monomer radicals or primary radicals had a detrimental effect. Thus, proper choice of initiator, catalyst, and parameter variables would give better yield. However, the graft copolymerization of vinyl monomers onto jute fiber provided additional properties and improved the textile performance.

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