

Grafting of Methyl Acrylate and Methyl Methacrylate onto Jute fiber: Physico-Chemical Characteristics of the Grafted Jute

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ABSTRACT

The graft copolymerization of methyl acrylate and methyl methacrylate vinyl monomers onto jute fiber using potassium persulfate and ferrous sulfate redox system was investigated in an aqueous medium. Optimized reaction conditions for the graft copolymerization were established for parameter variables, of monomer concentration, initiator concentration, catalyst concentration, reaction time, and reaction temperature. The grafting degrees were found to depend on the above mentioned variables. A maximum graft yield of 17.3% for MA and 19.4% for MMA was obtained under effective optimized conditions for grafting. The attachment of the monomer on the grafted fiber was confirmed by FTIR studies. Dyeing was affected by monomer characteristics, and due to the development of hydrophobic nature on jute fiber by grafting, a lower amount of dye absorption occurred. Grafting has a favorable influence on tenacity, color fastness, and other properties of jute fiber.

Keywords: Grafting; vinyl monomer; jute fiber; tenacity; color fastness.

INTRODUCTION

Natural cellulosic fibers provide a wide range of application in textile materials. The strongest potential for cellulose products originates from the combination of their low cost, biodegradability, renewability, and high specific strength, especially with bast fibers such as jute, hemp and flax. It is very likely that these fibers will be a major chemical resource for the future. Jute, a multicellular fiber, is transformed into multifarious products for making coarse woven fabrics, such as gunny sacks and bagging where cheapness is the main factor. But the major problem with using jute products is light-induced discoloration [1,2]. Recently it has been found that grafting improves the extensibility and susceptibility of jute to sunlight [3-5]. Therefore,

graft copolymerization of vinyl monomers onto jute fibers has been receiving considerable attention recently.

Chemical modification of cellulosic fibers by graft polymerization of vinyl monomers results in structural change and is a means for acquisition of new properties. Such properties include improved elasticity, sorbency, fastness, and thermal resistance depending on the solvent system and conditions of modification. As vinyl monomers, methyl acrylate and methyl methacrylate have the potential to graft onto jute fiber using different initiation systems, such as radical initiation [6-9], photo-initiation [10], and chemical initiation [11-15]. From the above three methods, chemical initiation of grafting involving oxidizing agents such as KMnO_4 , $\text{Na}_2\text{S}_2\text{O}_3$, $\text{K}_2\text{S}_2\text{O}_8$, Ce(IV) , V(V) are promising from the economic point of view and quite selective in nature. Among the initiators, persulfate ($\text{S}_2\text{O}_8^{2-}$) has an identity of its own, which is used for graft copolymerization onto various cellulose substrates [16-19].

In the present investigation, methyl acrylate (MA) and methyl methacrylate (MMA) were graft copolymerized onto jute fiber using $\text{K}_2\text{S}_2\text{O}_8$ as initiator in the presence of Fe(II) as catalyst. To make the bleached and modified jute fibers more attractive and increase their potential uses, they were dyed with reactive dyes. The assessment of color fastness and breaking strength of the fiber was also investigated.

EXPERIMENTAL

Materials

Jute (*Crochurus olitorius*) was collected from Rajshahi Jute Mills Ltd., Bangladesh. About 30 cm of those fibers were taken above 60 cm from the bottom of the stem. Vinyl monomers (MMA and MA) obtained from BDH (England) were freed from stabilizer by washing with 5% sodium hydroxide

followed by a water rinse. The fibers were then dried with anhydrous sodium sulfate and distilled water under reduced pressure in nitrogen before use without any purification [20]. All other chemicals and dyestuffs used were of C.P. grade.

Preparation of Jute Samples

The raw jute samples were initially subjected to scouring and bleaching prior to grafting and dyeing. The fibers were scoured at 70-75°C for 30 min in a solution containing 6.5 gm detergent and 3.5 gm Na₂CO₃ per liter [21]. The fibers were then bleached with 5 gm NaClO₂ per liter at 85-90°C for 90 min. During bleaching, the pH of the liquor was adjusted to 4 and buffered with 0.2 M acetic acid and 0.2 M sodium acetate [21,22]. After bleaching, the fibers were immersed in 0.2% sodium met bisulfite solution for 15 min to neutralize chlorine action and followed by water washing [23].

Grafting

Vinyl monomers (MA and MMA) were dissolved in warm distilled water by adding a few drops of 1 gm per liter soap solution. The graft copolymerization of jute fiber was carried out in a 100 ml stoppered Erlenmeyer conical flask. The flask was prepared with 20-110% monomer, 0.2-2.0% potassium persulfate (K₂S₂O₈) as initiator, (0-1.8%) ferrous sulfate as catalyst based on the weight of fiber at 30-90°C for 30-210 min in the fiber-liquor ratio of 1:30. The polymerization was done by the addition of jute fiber in a nitrogen atmosphere and stoppering of the flask, and the flask was placed in a thermo state control shaking water bath. At the end of the desired reaction period, the grafted fiber was thoroughly washed with hot distilled water and then extracted with acetone for 10 h in a Soxhlet apparatus to remove the loosely attached polymer [24]. A blank experiment was done for precision which gave eventual weight loss during modification. Percent graft yield was calculated according to the following formula [11]:

$$\text{Graft yield, \%} = (W_g - W_0) / W_0 \times 100$$

Where, W_g is the weight of the grafted fiber after modification and W₀ is the weight of bleached jute fiber before modification.

IR Spectroscopy

IR spectra of bleached, MA- and MMA-grafted jute fibers were recorded with a Shimadzu IR-8900 spectrophotometer (Shimadzu, Kyoto, Japan) using the KBr pellet technique. The dried fiber samples were crushed to a size of about 20 meshes before

pelleting with KBr. The test KBr pellet contained about 1% powdered fiber [16].

Dyeing

The reactive dyes; Reactive Orange 14 (C.I. No. 19138) and Reactive Brown 10 (C.I. No. 12225) were first made into a paste with little water and then dissolved by adding distilled water. A 2.5% dye and 5% aluminum sulfate (Al₂(SO₄)₃) as an electrolyte based on the weight of fiber in the fiber-liquor ratio of 1:30 was taken in a 50 ml Erlenmeyer stoppered conical flask. Dyeing was done by putting the flask in an Oscillating dyeing machine (Model TB700, Dysin, China) at 60°C for 60 min. After 30 min, 2% sodium carbonate solution was added to make the medium alkaline and to fix the dye on the fiber [23,25]. The dye uptake by both bleached and modified samples was measured by sampling the dye bath before and after the dyeing process. Dye exhaustion (%E) was determined colorimetrically [23] as

$$\%E = (D_0 - D_e) / D_0 \times 100$$

Where, D₀ and D_e are the original and exhausted dye bath concentration, respectively.

Color Fastness

Dyed and undyed jute samples were exposed directly under sunlight without any protection from weathering, but was protected from rain and snow for 6 h each day from June to August and continued for 300 h. After every 50 h the color fastness of the specimen was assessed by the Grey scales comparing with the control (26). Color fastness of the fiber on washing with 5 gm per liter soap solution at 40 to 100°C for 30 min in the fiber-liquor ratio of 1:50 was also evaluated using the Grey scales [27].

Measurement of Breaking Strength

Breaking strength of jute fiber was measured by a Torsee's Schopper OS-100 Tensile Tester. The breaking load of the fibers was measured in kg/yarn according to an international standard [28]. In each experiment, the mean of 25 specimens was taken as breaking strength. Then, the tenacity was calculated by the following formula [29].

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

RESULTS AND DISCUSSION

Grafting

The grafting was studied by changing the concentration of monomer is shown in *Figure 1*. The results show that the percent graft yield increased with an increase of monomer concentration up to

90% MA and 80% MMA and thereafter decreased. It is evident that the increase in monomer concentration in an aqueous medium increased the reaction sites and complexation of jute with MA and MMA enhanced reactivity due to the formation of donor-acceptor complex. But higher concentration of monomer in polymerization medium acted in favor of molecular collision, thereby enhancing polymerization in general or homopolymer formation in particular. Hence the graft yield decrease after a certain monomer concentrations suggests that homopolymerization prevails over grafting at higher MA and MMA concentration [16].

It is apparent from the *Figure 1* that the percent graft yield of MMA is greater than that of MA. This may be due to the higher molecular weight of MMA compared to that of MA. Again the reactive thermo-

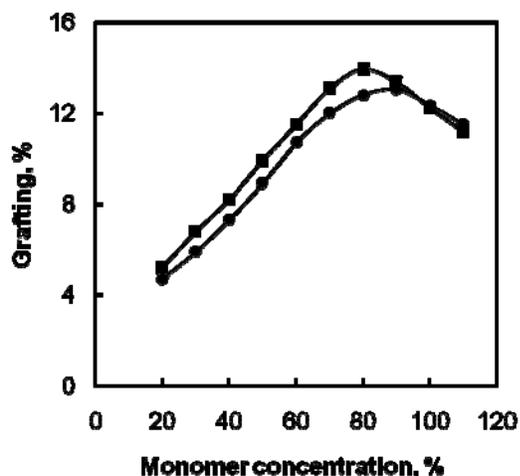


FIGURE 1. Effect of monomer concentration on grafting of MA and MMA onto bleached jute fibers: MA (●); MMA (■); $K_2S_2O_8$, 1.0%; $FeSO_4$, 1.0%; temp., 50°C; time, 60 min; fiber-liquor ratio, 1:50.

dynamic feasibility of a monomer varies, depending on the substituents present in it. Substituent increases the reactivity of a monomer towards radical attack. In a particular series of vinyl monomers, reactivity increases with the increase of the number of substituent present in monomer. Substituents composed of unsaturated linkages are most effective in resonance stabilization of loosely held π -electrons [30]. Both of the monomers MMA [$H_2C=C(CH_3)-COO-CH_3$] and MA [$H_2C=CH-COO-CH_3$] contain π -electron and the hydrogen atom of C_2 of MA is replaced by $-CH_3$ group giving the structure of MMA. Due to the presence of $-CH_3$ group in MMA, synthesized polymer is more resonance stable. As a result, higher percent graft yield was obtained in favor of MMA.

Figure 2 shows that the percent graft yield increased with the increase of $K_2S_2O_8$ concentration up to 1.0% and 1.4% for MA and MMA, respectively and then decreased. Initiator, $K_2S_2O_8$ generates activated monomers, which showed a sufficient affinity to combine with the cellulose macro radicals that are

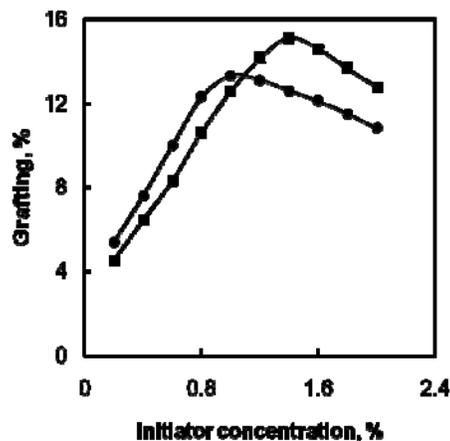


FIGURE 2. Effect of initiator ($K_2S_2O_8$) concentration on grafting of MA and MMA onto bleached jute fibers: MA (●), 90%; MMA (■), 80%; $FeSO_4$, 1.0%; temp., 50°C; time, 60 min; fiber-liquor ratio, 1:50.

produced by the action of $Fe(II)$ on the surface of jute fiber and enhance the grafting up to certain $K_2S_2O_8$ concentration. In higher $K_2S_2O_8$ concentration large number of activated monomer become available which turn to the formation of homopolymer causing the lackness of monomer available in the vicinity of cellulose macroradicals and hence the percent graft yield start to decrease.

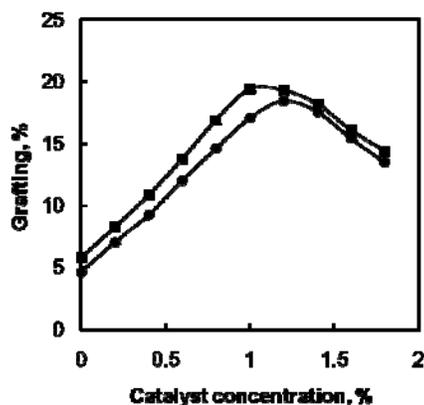


FIGURE 3. Effect of catalyst ($FeSO_4$) concentration on grafting of MA and MMA onto bleached jute fibers: MA (●), 90%; MMA (■), 80%; $K_2S_2O_8$, 1.0% for MA and 1.4% for MMA; temp., 50°C; time, 60 min; fiber-liquor ratio, 1:50.

The dependence of grafting on the amount of FeSO_4 concentration was studied at selected monomer and initiator concentration (Figure 3). Maximum graft yield was found at FeSO_4 concentration up to 1.2% and 1.0% for MA and MMA respectively. The graft yield decreased above those of Fe(II) concentration. As the increase in Fe(II) ion concentration increases $\text{Fe(II)-K}_2\text{S}_2\text{O}_8$ transient adduct, create more cellulose macroradicals which shows the greater tendency to combine with the activated monomers allowing the greater grafting. The graft yield decrease at greater Fe(II) concentration probably due to the formation of large numbers of Fe(II) ions which promote the premature termination of the growing grafted chains [31].

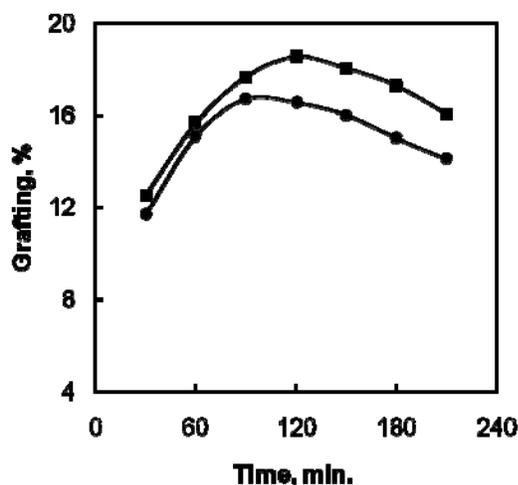


FIGURE 4. Effect of polymerization time on grafting of MA and MMA onto bleached jute fibers: MA (●), 90%; MMA (■), 80%; $\text{K}_2\text{S}_2\text{O}_8$, 1.0% for MA and 1.4% for MMA; FeSO_4 , 1.2% for MA and 1.0% for MMA; temp., 50°C; fiber-liquor ratio, 1:50.

The grafting reaction was carried out from 30 to 210 min and maximum percent graft yield was found at 90 min for MA and 120 min for MMA and is shown in Figure 4. After this period of time both the percent graft yield started to decrease. The rate of grafting increased with time at first very sharply due to maximum co-polymerization reaction between the reactive sites of jute and the activated monomers. For long reaction time, the percent graft yield decreased due to the partial dissolution of grafted fiber [16].

The graft yield increased with the rise of reaction temperature from 30 to 60°C both for MA and MMA, and then fell with further increase of temperature as shown in Figure 5. The increase in percent graft up to 60°C may be ascribed to the increase in the rate of production of active free radicals which increase the

number of grafting sites; the rate of initiation by jute radicals are thereby increased, and with the increase in temperature the rate of diffusion of monomers into the jute matrix increases where grafting is also initiated by complex monomer [30-32].

The decrease in percent graft yield beyond optimum temperature may be attributed to the increase in activation energy, increase in the rate of homopolymerization, and premature termination of growing grafted chains by excess Fe(III) ions produced on the oxidation of Fe(II) ions. The optimized conditions of grafting with MA and MMA onto jute fiber are listed in Table I.

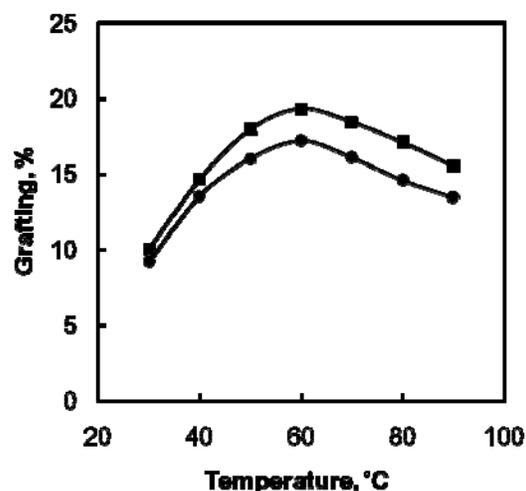


FIGURE 5. Effect of polymerization temperature on grafting of MA And MMA onto bleached jute fibers: MA (●), 90%; MMA (■), 80%; $\text{K}_2\text{S}_2\text{O}_8$, 1.0% for MA and 1.4% for MMA; FeSO_4 , 1.2% for MA and 1.0% for MMA; time, 90 min for MA and 120 min for MMA; fiber-liquor ratio, 1:50.

FT-IR Studies for the Conformation of Grafting Reaction

The IR spectra of bleached, MA- and MMA-modified jute fibers are shown in Figure 6. The spectra of the modified samples were more or less similar in nature to that bleached jute fiber, except at the peaks at 1727 and 1719 cm^{-1} for modified fibers. The additional peaks at 1727 and 1719 cm^{-1} correspond to the ester carbonyl groups of MA and MMA modified fibers respectively. The sharper and intense carbonyl peak of MMA modified fiber compared to that of MA modified fiber is due to the larger amount of MMA attached on to the jute macroradicals.

TABLE I. Effective optimum conditions of modification.

Vinyl monomers	Monomer conc., %	Initiator conc., %	Catalyst conc., %	Modification time, min	Modification temp., °C	Graft yield, %
MA	90	1.0	1.2	90	60	17.3
MMA	80	1.4	1.0	120	60	19.4

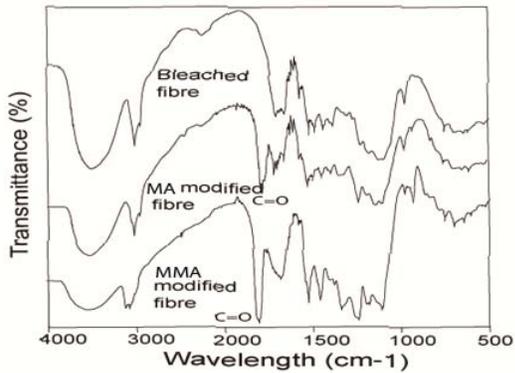


FIGURE 6. FT-IR spectra of bleached, methyl acrylate (MA) and methyl methacrylate (MMA) modified fibers.

Mechanism of Grafting Reaction

Jute cellulose has three hydroxyl groups per anhydroglucose unit. Ideally, these hydroxyl groups should offer potential reactive sites for graft copolymerization. In the presence of redox initiator, there are six possible points to form radical on each glucose unit of jute cellulose depending on reaction conditions [33]. The regions are at C2, C3 and C6. Three of these occur by the abstraction of hydrogen as carbon radical and the other three occur as oxygen radical of the cellulose backbone. These radicals lead to the formation of graft or block copolymers.

The proposed mechanism of graft copolymerization of vinyl monomers (MA and MMA) onto bleached jute fiber initiated by potassium persulfate ($K_2S_2O_8$) in the presence of Fe(II) may be pictured as involving generation of free-radical ($\cdot OH$ and $SO_4^{\cdot -}$) species and to some extent by Fe(III) ions on jute fibers. The free radical species and Fe(III) ions are produced from the redox reaction between $K_2S_2O_8$ and Fe(II). The macroradicals so formed subsequently attack the monomer leading to chain initiator. The termination of graft copolymerization may be due to the interaction with Fe(III) ions or mutual combination of grafted chains on different backbones [23].

Production of Primary Radicals and Fe(III) Ion

- $K_2S_2O_8 \rightleftharpoons 2K^+ + S_2O_8^=$
 - $FeSO_4 \rightleftharpoons Fe^{++} + SO_4^=$
- $S_2O_8^= + Fe(II) \rightleftharpoons \text{Adduct}$
 - $\text{Adduct} \longrightarrow Fe(III) + HO\cdot + 2SO_4^= \longrightarrow Fe(III) + 2SO_4^{\cdot -} + OH^-$
- $SO_4^{\cdot -} + H_2O \longrightarrow HSO_4^- + HO\cdot$
 - $Fe(III) + H_2O \longrightarrow Fe(II) + HO\cdot + H^+$

Formation of Macrojute Radicals

- $J-H + R\cdot \longrightarrow J\cdot + RH$ ($R\cdot = \cdot OH$ & $SO_4^{\cdot -}$)
- $J-H + Fe(III) \longrightarrow J\cdot + Fe(II) + H^+$

Oxidation



Initiation

- Graft Copolymerization

$$J\cdot + M \longrightarrow J-M\cdot$$
 (M = Monomer)
- Homopolymerization
 - $R\cdot + M \longrightarrow R-M\cdot$
 - $Fe(III) + M \longrightarrow M\cdot + Fe(II) + H^+$

Propagation

- $$J-M\cdot + M \longrightarrow J-M\cdot_1$$

$$\vdots$$

$$J-M\cdot_{n-1} + M \longrightarrow J-M\cdot_n$$
- $$M\cdot + M \longrightarrow M\cdot_1$$

$$\vdots$$

$$M\cdot_{n-1} + M \longrightarrow M\cdot_n$$

Termination

1. i) $J-M_n^{\bullet} + Fe(III) \longrightarrow$
Grafted jute copolymer + $Fe(II) + H^+$
- ii) $J-M_n^{\bullet} + J-M \longrightarrow$ Grafted jute copolymer
2. i). $M_n^{\bullet} + Fe(III) \longrightarrow$ Homopolymer
- ii) $M_n^{\bullet} + M_m^{\bullet} \longrightarrow$ Homopolymer

Dimerization

Two macrojute radicals might couple to yield a dimerized product.



Where, $S_2O_8^{2-}$, R^{\bullet} , JH , J^{\bullet} , M , M^{\bullet} are peroxydisulfate ion, free radical, jute molecule, jute macroradical, monomer, monomer radical, respectively.

Dyeing

The absorption of Reactive Orange 14 and Reactive Brown 10 by the fibers is relatively low and is listed in *Table II*. As known, all water soluble dyes are electrolyte and in aqueous solution dissociate into the dye color ions and the colorless compensating ions having an opposite charge to that of the former. The color ion of reactive dyes is negatively charged and the colorless compensating ion is positively charged. Again jute fiber, when immersed in dye solution, due to the presence of carboxyl group in jute, becomes

negatively charged, and a repulsive force between dye anion and fiber anion may develop [22]. But dye contains some impurities like salts and dextrin etc. These and dyeing temperature overcome some of the potential barriers. As a result, a comparatively lower amount of dye from the dye bath can attached with the fiber.

TABLE II. Effect of dye exhaustion on bleached and modified fibers with Reactive Orange 14 and Reactive Brown 10.

Types of sample		Dye exhaustion, %	
		Reactive Orange 14	Reactive Brown 10
Bleached fiber	jute	40.7	37.4
MA fiber	modified	36.8	30.8
MMA fiber	modified	37.9	33.4

From the *Table II*, it can be seen that the dye exhaustion of bleached jute fiber was greater than that of modified fiber. The variation of dye exhaustion is that dye absorption by the fiber depends on the availability of total external surface of the fiber pores or cavities in a fixed amount of fiber and the attractive forces between the fiber and dye ions. The bleached fiber has more available pore or cavities than modified fiber, which on grafted blocked. So dye exhaustion of bleached fiber is greater than modified jute fiber [30].

TABLE III. Color fastness of bleached and modified jute fiber on the exposure to sunlight.

Exposure period, hr	Fastness grade of jute fiber								
	Undyed			Dyed with Reactive Orange 14			Dyed with Reactive Brown 10		
	BF	MAF	MMAF	BF	MAF	MMAF	BF	MAF	MMAF
00	5	5	5	5	5	5	5	5	5
50	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
100	4	4-5	4	4-5	4-5	4-5	4	4-5	4-5
150	3-4	4	4	4	4	4	4	4	4-5
200	3-4	4	3-4	4	4	4	3-4	4	4
250	3	3-4	3	3-4	4	4	3	3-4	3-4
300	2-3	3-4	3	3-4	4	4	3	3-4	3-4

BF-Bleached jute fiber; MAF- MA modified fiber; MMAF- MMA modified fiber.

TABLE IV. Color fastness of bleached and modified jute fiber on washing.

Washing temperature °C	Fastness grade of jute fiber					
	Dyed with Reactive Orange 14			Dyed with Reactive Brown 10		
	BF	MAF	MMAF	BF	MAF	MMAF
Unwashed	5	5	5	5	5	5
40	4	4-5	4-5	4	4	4
60	3-4	4	4	3-4	4	3-4
80	3	4	3-4	3	3-4	3-4
100	2-3	3-4	3	2-3	3	3

BF-Bleached jute fiber; MAF- MA modified fiber; MMAF- MMA modified fiber.

Color Fastness

The light fastness ratings of bleached and grafted jute fibers after a 300 h exposure to sunlight in open air are nearly the same. As the grafted jute fibers show less affinity towards moisture can somehow protect the yellowing trends to fiber. Polymer loaded in case of MMA modified fiber was more, so somewhat good result was found as compared to that modified fiber with MA (Table III).

The color fastness of dyed jute fiber was studied at 40, 60, 80 and 100°C temperature in detergent solution. The results are tabulated in Table IV. It seems that at higher temperature, dissolution of the dye particles from fiber surface takes place and hence more dye was easily washed off from the fiber. In that case grafted jute fiber possesses a somewhat better color fastness in washing, because in grafting the fiber becomes hydrophobic in nature and grafted fiber is less shrinkable in water.

The tensile properties of bleached and grafted jute fibers after exposure to sunlight were determined by taking a minimum of 10 specimens for each fiber sample. The loss in tenacity for every 50 h are pictured in Figure 7. In the results grafted fibers showed lower loss in tenacity than bleached fiber. In grafting the affinity of jute fiber towards moisture decreased. Lower affinity towards moisture grafted jute fiber showed less susceptibility to the degrading action of light [11].

CONCLUSIONS

Modification of jute fibers with methyl acrylate and methyl methacrylate monomers was carried out in this study to obtain better surface properties of jute

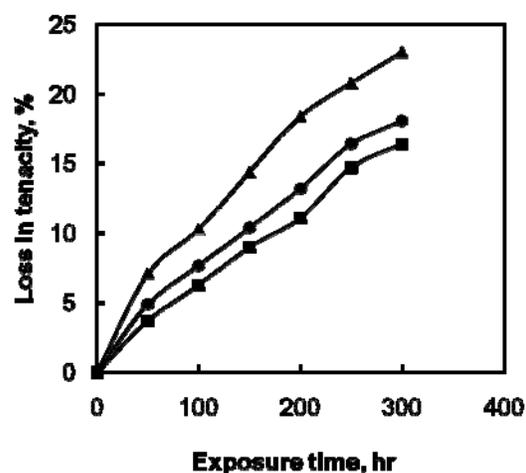


FIGURE 7. Effect of sunlight on loss in tenacity of bleached, MA and MMA modified jute fibers: Bleached jute fiber, (▲); MA, (●); MMA, (■).

fibers while maintaining good inherent mechanical properties. The increase or decrease of percent graft yield was found to depend on the number of both jute-macro radicals and monomer radicals whose production also depended on the initiator, catalyst, and other 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. Again, grafted fiber provides improved tensile properties by protecting photo-oxidative degradation, and color fastness properties towards sunlight and washing with soap solution.

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