

# Simultaneous Free Radical Polymerization and Acidic Polycondensation of Acrylamide–Formaldehyde Resin on Jute Fabric

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## SYNOPSIS

Acid catalyzed grafting, polycondensation, and crosslinking of acrylamide–formaldehyde (AMF) resin and simultaneous free radical graft copolymerization of the resin moieties on jute fabric were studied using a combination of  $MgCl_2$  and  $K_2S_2O_8$ , respectively, as the (dual) catalyst system. Resin application and finish on jute fabric were accomplished by padding at room temperature, drying at 80°C, and polymerization and curing at 150°C. Changes in chemical structure and surface morphology of jute fiber on resin treatment were examined by Fourier transform infrared (FT-IR) spectroscopy and scanning electron microscopy (SEM), respectively. Application of 8% AMF resin under the dual catalyst system in appropriate doses produced the most improved balance in the properties of jute fabric, including tenacity, modulus, breaking extension, stiffness, crease recovery angle, fiber shedding, moisture regain, and dyeability. Analysis indicated that all the major constituents of jute, namely,  $\alpha$ -cellulose, hemicellulose, and lignin reacted with the AMF resin system and that the resin finish turned jute thermally more stable. Effects of different degrees of removal of lignin and hemicellulose from jute on its AMF resin uptake were also studied and reported. © 1997 John Wiley & Sons, Inc.

## INTRODUCTION

Jute is a lignocellulosic bast fiber obtained from the bark of two cultivated species of genus *chorchorus*. Jute fiber has some inherent advantages<sup>1–4</sup> for its renewable nature, biodegradability, high strength and initial modulus, moderate moisture regain, easy dyeability, good heat and sound insulation properties, and low price. However, major disadvantages<sup>1,4–8</sup> of jute fabric lie in its coarseness, stiffness, low extensibility, low wet strength, harsh feel with poor drapability, moderate wash shrinkage, poor abrasion resistance, high fiber-shedding, photo-yellowing, ready susceptibility to microbial attack, and poor crease recovery.

Jute enjoyed a formidable position in bulk packaging until it started facing growing competition from synthetic fibers since the late 1950s because of the favorable properties profile and production cost structure for the synthetics. The position appears to be swinging back in favor of jute in view of a steep rise in the price of petroleum products and petrochemicals in recent decades. Fast depletion of petroleum reserves, growing consciousness about environmental preservation and pollution control, growing emphasis on the use of renewable resources, and energy efficient materials and adoption of related processings in the industrial sector are also contributing to this swing.

In tune with the developing trends, current research efforts in jute and allied fibers are directed toward not only reinstating them in their traditional application areas but also toward securing for them many new and diverse applications.

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A solution to minimize or even eliminate some of the major disadvantages of jute may be achieved by modifying jute fabric by suitable resin or polymer finishes with or without selective chemical pretreatments. Such modifications done with dimethylol derivatives of urea and substituted urea impart cross-linkages<sup>2-7</sup> in chain molecules of jute, causing noticeable improvements<sup>4,6-7</sup> in crease recovery properties, though with considerable loss of strength<sup>4-7</sup> and abrasion resistance<sup>4</sup> and enhancement in the stiffness character.<sup>4</sup>

Another approach in modifying jute may involve graft copolymerization of vinyl and related monomers.<sup>9-16</sup> It would be even more interesting if a modification process for jute could be developed whereby graft copolymerization of a vinyl monomer on the fiber system and optimum cross-linking of the fiber are accomplished simultaneously. It is with this objective that the present work relating to modification of scoured and bleached jute in fabric form using acrylamide-formaldehyde (AMF) or N-methylol acrylamide resin was undertaken.

## EXPERIMENTAL

### Materials

#### *Jute Fabric*

Ex-powerloom, plain weave, raw jute fabrics with yarns of 195 tex as warp and 214 tex as weft having 63 ends and 59 picks per dm and weighing 264 g/m<sup>2</sup> on the average were obtained from a local jute mill.

#### *Chemicals and Reagents*

Laboratory reagent (LR) grade sodium hydroxide, 50% hydrogen peroxide, acrylamide (AAm), formalin (40% formaldehyde), potassium persulphate, magnesium chloride, sodium chlorite, etc., from BDH or E-Merck, India were used in the experiments.

### Methods

#### *Preparation of Acrylamide Formaldehyde (AMF) Resin*

33 g (1.1 mol) of formaldehyde (83 cc of formalin solution) was initially neutralized using dilute NH<sub>4</sub>OH solution. 71 g (1 mol) of analytical reagent (AR) grade acrylamide dissolved in 100 ml water was mixed with it, and the pH of the mixture

was maintained at 9.0 with dropwise addition of a dilute NaOH solution. The mixture was allowed to react at 50°C under reflux for 2 h and was finally cooled to give a water-soluble acrylamide-formaldehyde (AMF) precondensate resin with 24–30% solid content.

For analysis and characterization purposes, the water-soluble AMF resin precondensate was extracted by shaking with warm ethyl-acetate. The ethyl-acetate solution of the AMF resin was then cooled to ice temperature; the precipitated resin was filtered, washed with acetone, and dried in vacuum at 40°C.

#### *Dewaxing, Scouring, and Bleaching of Jute Fabric*

Small pieces of raw jute fabric sample were dewaxed<sup>14</sup> when needed, by Soxhlet extraction for 6 h using an ethyl-alcohol–benzene mixture (1 : 2 vol/vol), followed by successive washing with fresh alcohol and distilled water.

The jute fabrics were scoured<sup>6,17</sup> with 4 g/l sodium carbonate solution and 1 g/l nonionic detergent by heating at 80°C for 30 min in a laboratory jigger using a fabric/liquor ratio of 1 : 5 (w/v). H<sub>2</sub>O<sub>2</sub> bleaching of scoured jute fabrics was carried out in a laboratory jigger using a fabric/liquor ratio of 1 : 6 (w/v) employing 3% H<sub>2</sub>O<sub>2</sub> on weight of fabric at pH 10–11, following well-established procedures.<sup>7,17</sup> After bleaching for 1 h at 90–95°C, each fabric sample was washed with water, neutralized with dilute acetic acid, washed again with water, and dried in air.

#### *Removal of Hemicellulose and Lignin from Dewaxed Jute Fabric*

Removal of hemicellulose to different extents was accomplished<sup>18-21</sup> by treating jute fibers/fabrics with NaOH solutions of different concentrations (5, 9, 13, and 18%) for 30 min, at room temperature, followed by successive washing with 1% acetic acid and distilled water and drying in air. Removal of lignin to different extents was accomplished<sup>20-29</sup> by treating jute fibers/fabrics with 0.7% NaClO<sub>2</sub> for different time periods (5, 10, 25, 50, and 100 min) at pH 4 at boiling temperature, followed by washing with 2% solution of NaHSO<sub>3</sub> at 50°C for 30 min and then with distilled water and finally drying in air.

#### *AMF Resin Application*

It was thought to be interesting to study the effects of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and MgCl<sub>2</sub> used as catalysts for inducing grafting and free radical chain polymer-

ization<sup>13,15</sup> and binding and curing<sup>5,24,25</sup> by a process of acidic polycondensation of the acrylamide-formaldehyde (AMF) resin applied on jute. In one set of experiments, the selected fabric sample was presoaked with a prescribed dose of  $K_2S_2O_8$  solution prior to AMF-resin application.  $K_2S_2O_8$  picked up by the fabric would be expected to effect radical graft copolymerization of the AMF resin moieties on the segments of different constituents of jute in the subsequent step of AMF resin application on the fabric by the pad-dry-cure process at a high temperature (80–150°C). Presoaking of  $K_2S_2O_8$  solution and AMF resin application on jute fabric were done using a laboratory two-bowl padding mangle (two dip, two nip) adjusting squeeze pressure to enable a wet pick up of 80%. Aqueous solutions of AMF resin of known concentration containing no  $MgCl_2$  or specific doses of  $MgCl_2$  (acidic curing catalyst) were used separately for padding of the jute fabric having or having not been presoaked with  $K_2S_2O_8$ , as stated above. The resin-treated fabrics were subjected to drying at optimum conditions (80°C, 10 min) and curing (2–20 min, at 100–160°C). The resin-finished fabric was then soap-washed at 50°C following the ISO-II method,<sup>26,27</sup> followed by washing with water at room temperature and drying in air.

#### ***Dyeing of Bleached and AMF Resin Treated Jute Fabrics***

Dyeing of bleached and AMF resin treated jute fabrics with an acid dye (CI Acid Red-73,  $\lambda_{max}$  = 480 nm) and a reactive dye (CI Reactive Red-11,  $\lambda_{max}$  = 540 nm) was accomplished following standard procedures<sup>28</sup> using a fabric/liquor ratio of 1 : 100 in each case.

#### ***Testing and Evaluation***

Tensile and other physical testings were performed taking test specimens in the warp direction unless otherwise stated. Breaking tenacity and breaking extension of the fabric samples ravelled to a strip of a specific size (50 × 20 mm) were determined following a method<sup>29</sup> described in IS 1969–1968 using a Zwick 1445 universal tensile testing machine at a traverse speed of 100 mm/min employing a pretension of 0.2 Newton. The initial modulus (modulus at 1% extension) values were also recorded.

Fabric stiffness as expressed by bending length was measured in a SASMIRA stiffness tester using the method<sup>30</sup> described in IS 6490-1971. Crease recovery (CR) angle of selected fabric samples was measured<sup>31</sup> using a SASMIRA crease re-

covery tester according to ASTM D 1295-67. The fiber shedding propensity of untreated and differently treated jute fabrics was evaluated by measuring the weight loss in mg on abrading a fixed area of selected jute fabric sample using another piece of the corresponding fabric as the abrader for a fixed number (100) of cycles of abrasion; an electronically controlled abrading instrument was used, and the method followed has been described elsewhere.<sup>4</sup> Moisture regain of selected oven-dried samples was determined<sup>31</sup> according to ASTM-629-59T. Copper number<sup>32,33</sup> and COOH group content<sup>34</sup> of jute, untreated or treated in different ways, were determined following appropriate standard methods.

The K/S value (Kubelka Munk-function<sup>26,28</sup>) viewed as an index of surface dye-uptake was estimated by measuring the surface reflectance of each dyed fabric sample at the wavelength of maximum absorbance ( $\lambda_{max}$ ) using a computer-aided Macbeth 2020-plus reflectance spectrophotometer following standard procedures.<sup>26,28,34</sup> Exhaustion of dye from residual dye bath liquor was also estimated spectrophotometrically following standard procedures<sup>35</sup> using a UV-2000 Hitachi absorbance spectrophotometer, and the bulk dye-uptake  $[D]_f$  value expressed in g of dye/kg of fiber was calculated using the relevant data.

The fiber samples taken from selected treated and untreated jute fabrics were also subjected to analysis by scanning electron microscopy and FTIR spectroscopy following appropriate standard technique and procedure in each case. Compositional analysis of jute was done following established (Tappi) methods<sup>36</sup> and isolation of different jute constituents ( $\alpha$ -cellulose, hemicellulose, and lignin) was done as and when required following standard procedures<sup>22,36</sup> in each case. AMF resin uptake by jute fabric or different constituents of jute was judged by nitrogen-content analysis of the relevant untreated and resin-treated substrates by the Kjeldahl method. Differential scanning calorimetric study for jute and AMF-resin-treated jute samples was carried out<sup>35,36</sup> under flowing nitrogen using a Shimadzu-DSC-50 equipment.

## **RESULTS AND DISCUSSION**

The results of the present studies on AMF resin application and finishes on jute fabric by pad-dry-cure technique are given in Tables I–III and Figures 1–8. The discussions and interpretations of observed effects and the structure property relationships will be better appreciated if

**Table I** Some Textile Related Physical Properties of Raw Jute Fabric and of Jute Fabric Treated Differently with AMF Resin in Pad-Dry-Cure Process Under the Catalytic Influence of  $\text{MgCl}_2$  and/or  $\text{K}_2\text{S}_2\text{O}_8$ <sup>a</sup>

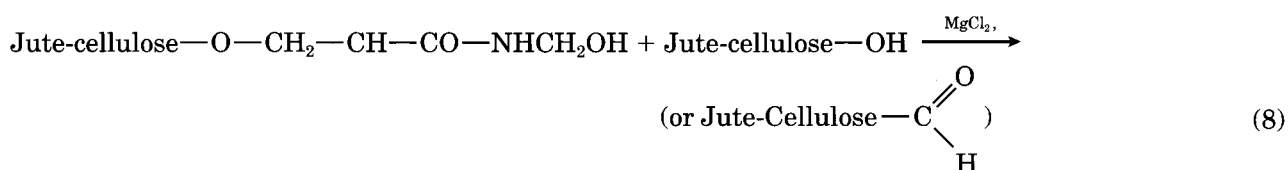
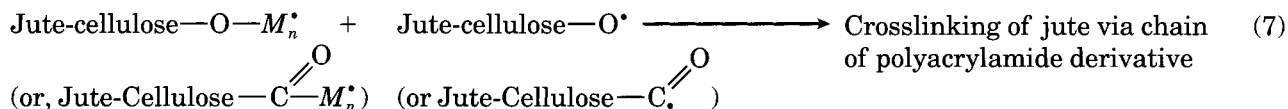
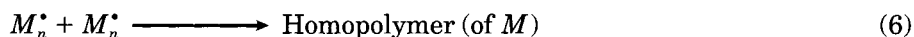
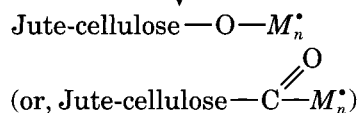
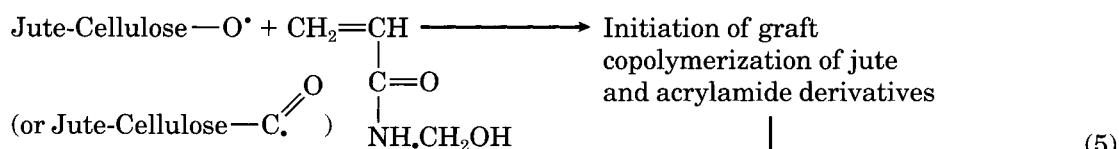
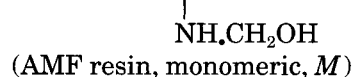
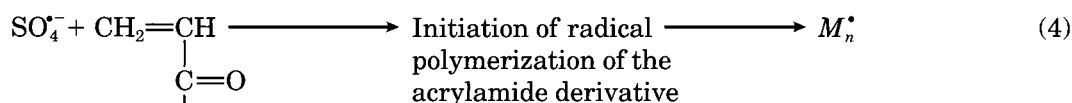
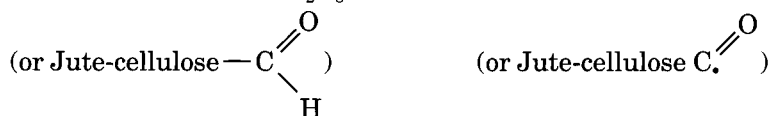
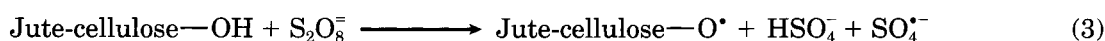
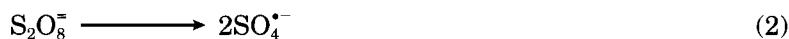
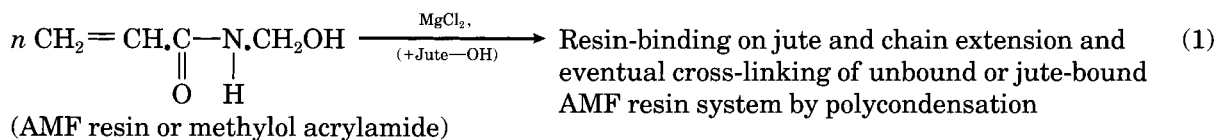
Jute Fabric Used	Treatment Given on Fabric	Weight Gain (+) or Loss (-) (%)	Tenacity (N/mm)	Breaking Extension (%)	Initial Modulus (N/mm)	Bending Length (cm)	Crease Recovery Angle (Degree) (Warp + Weft)	Fiber Shedding (mg)	Moisture Regain (%)
1. Raw	Nil	—	12.72	4.92	94.4	4.4	132	49.0	14.6
2. Raw	Scouring only	-1.60	12.60	5.24	86.1	4.2	128	48.0	15.0
3. Raw	Scouring and bleaching	-6.02	11.90	7.12	28.1	4.0	133	40.0	16.0
4. Scoured and bleached	Heated at 150°C using 2% $\text{MgCl}_2$ only without AMF resin.	—	10.67	6.96	25.7	4.0	134	46.0	15.4
5. Scoured and bleached	Heated at 150°C on 8% AMF resin application with 2% $\text{MgCl}_2$ .	+5.16	9.30	6.56	34.3	4.10	169	38.0	14.3
6. Scoured and bleached	$\text{K}_2\text{S}_2\text{O}_8$ (0.5%) presoaked fabric heated at 150°C without AMF resin.	—	10.77	4.91	24.1	4.0	125	39.1	16.4
7. Scoured and bleached	$\text{K}_2\text{S}_2\text{O}_8$ (0.5%) presoaked fabric heated at 150°C with 8% AMF resin.	+4.69	10.89	7.88	34.6	3.8	152	20.4	18.4
8. Scoured and bleached	$\text{K}_2\text{S}_2\text{O}_8$ (0.5%) presoaked fabric heated at 150°C with 8% AMF resin and 2% $\text{MgCl}_2$ .	+6.10	9.60	6.70	37.9	4.0	207	28.4	17.6

<sup>a</sup> Predrying condition: 80°C, 10 min; curing condition: 150°C for 4 min; after-wash condition: soap wash by ISO-II method.

the mechanistic of the chemical changes incorporated on AMF resin application under the influence of different catalysts are considered in the beginning.

### Mechanism of Chemical Changes Incorporated

The reactions involved during pad-dry-curing of AMF resin on mildly bleached jute under the prescribed condition may be described as follows.



Cross-linking of jute via acidic condensation of —NH— or methylol groups of jute-bound AMF resin moieties and —CHO or —OH groups of (bleached) jute-cellulose

Reaction (1) involves primarily AMF resin binding on jute and AMF resin based (—NH)—methylol or methylol—methylol polycondensation leading to chain extension and ultimate cross-linking; this sequence of reaction is catalyzed by MgCl<sub>2</sub> (the acidic curing catalyst) effectively at 150°C. Radical reactions (2)–(7) initiated by S<sub>2</sub>O<sub>8</sub><sup>2-</sup> are effective even at room temperature, as

well as at the drying (80°C) and curing (150°C) temperatures; the rate of reaction is expectedly higher at a higher temperature. Sequence of reactions (2), (4), and (6) may lead to formation of homopolymers of AMF resin, i.e., methylolated acrylamide (M); while reactions (3), followed by (5) and (7), would finally lead to the formation of AMF-resin-modified/cross-linked fiber system.

**Table II Data Showing AMF Resin Uptake in Terms of Weight Gain (%) and N-content (%) for the Resin Treated Jute Fabric and Different Major Constituents of Jute<sup>a</sup>**

Sample	Weight Gain (%)	N-Content (%)	
		Calculated (From Weight Gain)	Observed
1. Raw jute fabric	—	—	0.13
2. Only AMF resin	—	—	13.40
3. Scoured and bleached jute fabric	(-) 6.02 <sup>b</sup>	—	0.10
4. 8% AMF resin cured (K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> + MgCl <sub>2</sub> ) on fabric as in Sample 3	6.10	0.82	0.85
5. Delignified jute cured with 8% AMF in presence of K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> and MgCl <sub>2</sub>	7.52	1.00	1.08
6. $\alpha$ -cellulose isolated from bleached jute and cured with 8% AMF in presence of K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> and MgCl <sub>2</sub>	6.56	0.88	0.92
7. Lignin isolated (powder) from bleached jute and cured with 8% AMF in presence of K <sub>2</sub> S <sub>2</sub> O <sub>8</sub> and MgCl <sub>2</sub>	5.01	0.68	0.71

<sup>a</sup> Resin concentration = 8% AMF. Catalysts used = 0.5% K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and 1.6% MgCl<sub>2</sub>. Predrying condition = 80°C, 10 min. Curing condition = 150°C, 4 min. After-wash condition = soap wash by ISO-II method.

<sup>b</sup> (—) indicates a loss in weight.

Furthermore, reactions between alcoholic groups present in chain molecules of jute and the methylol or —NH— groups of the AMF resin moieties (at different stages of their growth and unbound or bound to jute as in reactions 5 and 7) via MgCl<sub>2</sub> catalyzed acidic condensation reaction, as shown in reaction (8), may also play significant roles in development of cross-linking or intercross-linking of the fiber and resin system and, thus, in modifying the properties of jute fiber so treated.

#### Comparison of Effects of Use of MgCl<sub>2</sub> and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as Catalysts Separately or in Mutual Combination

The differences in the observed properties of pad-dry-cured jute fabrics using AMF resin (8% application) under the influence of (1) only MgCl<sub>2</sub> (acidic catalyst leading to resin binding, chain extension, and curing by polycondensation), (2) only K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (free radical catalyst inducing graft copolymerization), and (3) selected combination of MgCl<sub>2</sub> and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> leading to modifications including vinyl grafting on and curing of jute fabric are highlighted in Table I.

The use of only K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> to cause vinyl polymerization of the AMF moieties picked up by jute fabric results in a relatively high level of tenacity retention and prominent improvement in resistance to fiber shedding, in addition to producing

a relatively low stiffness value (bending length) and a high breaking extension. In comparison, the use of MgCl<sub>2</sub> only does not apparently produce many improved effects. The use of the dual catalyst system (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> + MgCl<sub>2</sub>) results in an improved balanced effects, considering tensile properties, stiffness level, crease recovery, moisture regain, and resistance to fiber shedding (Table I). All subsequent experiments were therefore done using a combination of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and MgCl<sub>2</sub> as catalysts for pad-dry-cure of jute fabric. The total crease recovery angle improves prominently under the effect of the dual catalyst system. The effects of MgCl<sub>2</sub> or K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as lone catalyst under otherwise comparable conditions in the absence of AMF resin were also evaluated separately, and they are shown in Table I for comparison.

#### Variation of Dose of MgCl<sub>2</sub> at a Fixed Dose of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>

Effect of variation of doses of MgCl<sub>2</sub> used as the (acidic) curing catalyst<sup>6,24,25</sup> under otherwise fixed or comparable conditions is shown in Figure 1. Variation in the doses of MgCl<sub>2</sub> is likely to cause different degrees of chain extension via random condensation between methylol or —NH— functional groups of the AMF resin moieties and the alcoholic (—OH) or aldehyde

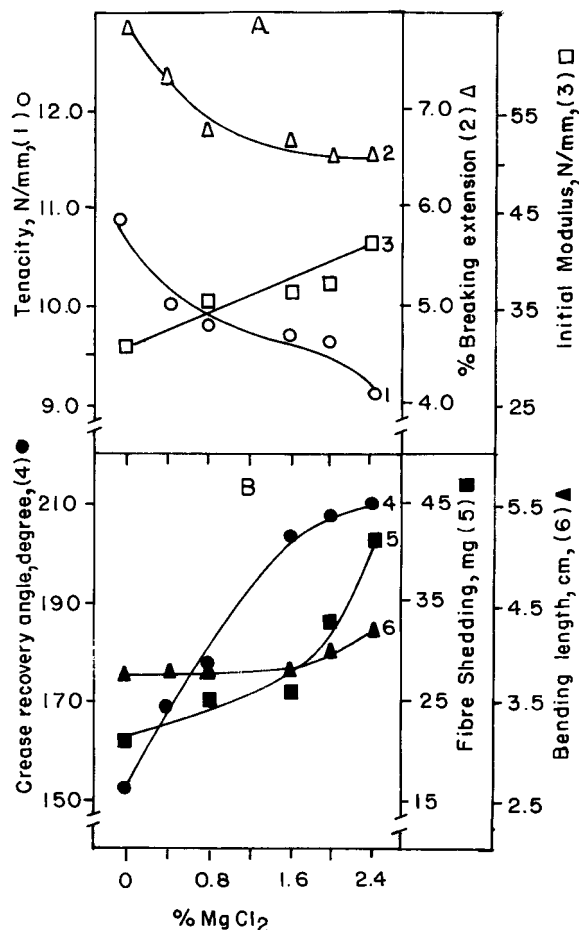
**Table III Effect of Progressive Removal of Hemicellulose and Lignin Separately from Dewaxed Jute (Prior to 8% AMF Resin Application) on Change of its Functional Groups (—CHO Group and —COOH Group Content) Pattern and AMF Resin Uptake (Estimated by % N-content)**

Reagent and Treatment Condition	Weight Loss (%)	Major Chemical Constituents of Jute, % (on Dry Weight of Dewaxed Jute)			Functional Groups Pattern		
		$\alpha$ -Cellulose	Hemicellulose	Lignin	Copper Number	—COOH Group Content (meq/100 g)	N-content (%)
1. Dewaxed jute	—	60.9	23.8	13.4	2.06	9.8	0.15 (0.78) <sup>a</sup>
2. Scoured and bleached jute	6.02	59.4	21.6	11.8	2.92	20.2	0.10 (0.85)
3. Progressive removal of hemicellulose from dewaxed jute using NaOH solution of different concentrations at room temperature for 30 min.							
5% NaOH	8.70	60.3	18.6	13.1	1.90	15.4	— (0.67)
9% NaOH	11.42	60.1	15.8	12.9	1.65	12.6	— (0.50)
13% NaOH	12.21	59.8	11.6	12.6	1.50	9.3	— (0.75)
18% NaOH	13.41	59.8	10.8	12.4	1.40	6.6	— (0.98)
4. Progressive removal of lignin from dewaxed jute using 0.7% NaClO <sub>2</sub> solution on boiling at pH 4.0 for the following time periods.							
10 min	4.11	60.1	23.4	10.10	3.12	14.4	— (0.69)
25 min	7.20	59.8	23.2	6.20	3.46	18.5	— (0.74)
50 min	11.43	59.5	22.8	4.90	4.01	21.6	— (0.90)
100 min	15.42	59.1	22.5	2.40	4.75	23.9	— (1.06)

<sup>a</sup> Values in the parentheses indicate the corresponding N-content data for 8% AMF resin cured and washed samples showing an estimate of AMF resin uptake.

(—CHO) groups in the macromolecular chains in the (bleached) jute fiber (cellulosic, hemicellulosic, and lignin constituents), leading ultimately to occasional cross-linking. With increase in MgCl<sub>2</sub> content, initial modulus follows a monotonously increasing trend, while tenacity and breaking extension follow dropping trends as

shown in Figure 1(A). The crease recovery angle follows a sharp increasing trend with increase in MgCl<sub>2</sub> dose until about 1.6% MgCl<sub>2</sub>, beyond which a levelling off trend follows [Fig. 1(B)]. An increase in MgCl<sub>2</sub> dose, however, tends to enhance the fiber shedding character at a relatively low rate initially until about 1.6% MgCl<sub>2</sub> [Fig.

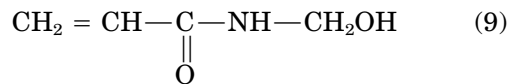


**Figure 1** Plots showing the effect of variation of dose of  $\text{MgCl}_2$  at a fixed dose (0.5%) of  $\text{K}_2\text{S}_2\text{O}_8$  for treating jute fabric with 8% AMF resin by pad-dry-cure process. (Other conditions: drying at  $80^\circ\text{C}$  for 10 min, and curing at  $150^\circ\text{C}$  for 4 min.)

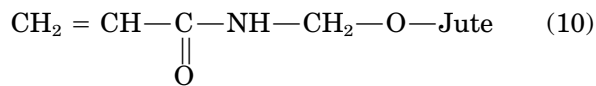
1(B)]. The bending length taken as an index of stiffness of the fabric remains practically unchanged on increasing  $\text{MgCl}_2$  dose up to about 1.6%; beyond this critical dose of  $\text{MgCl}_2$ , a relatively sharp upward turn follows in both the stiffness property (bending length) and the fiber shedding property [Fig. 1(B).] Results in Figure 1 clearly indicate that a dose of  $\text{MgCl}_2$  around 1.6% produces optimum effects.

#### Variation of Dose of $\text{K}_2\text{S}_2\text{O}_8$ at a Fixed Dose of $\text{MgCl}_2$

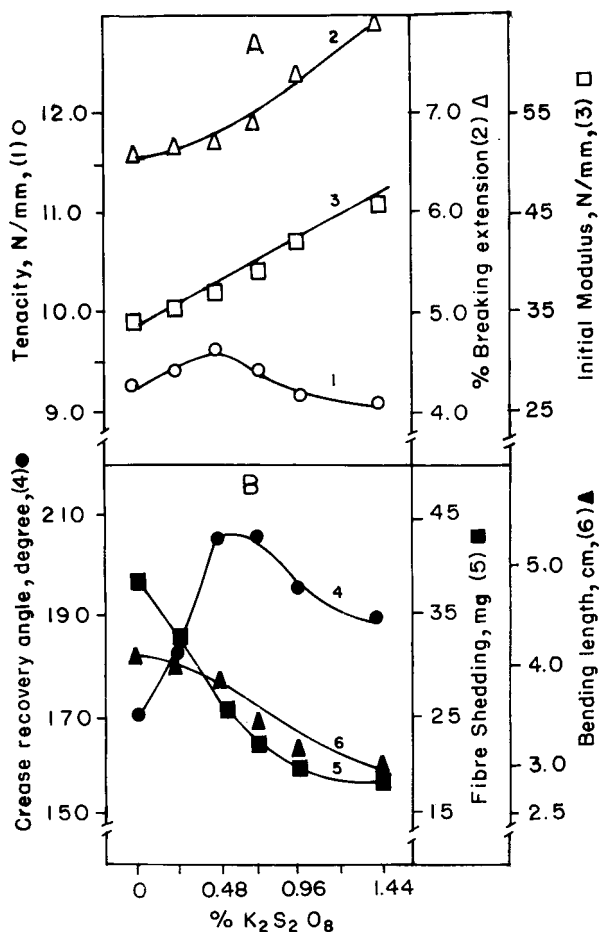
The effects of different doses of  $\text{K}_2\text{S}_2\text{O}_8$  as a free radical catalyst<sup>17,18</sup> for the vinyl polymerization of the (unbound or jute-bound) AMF resin moieties (Schemes 9 and 10) were also investigated.



or

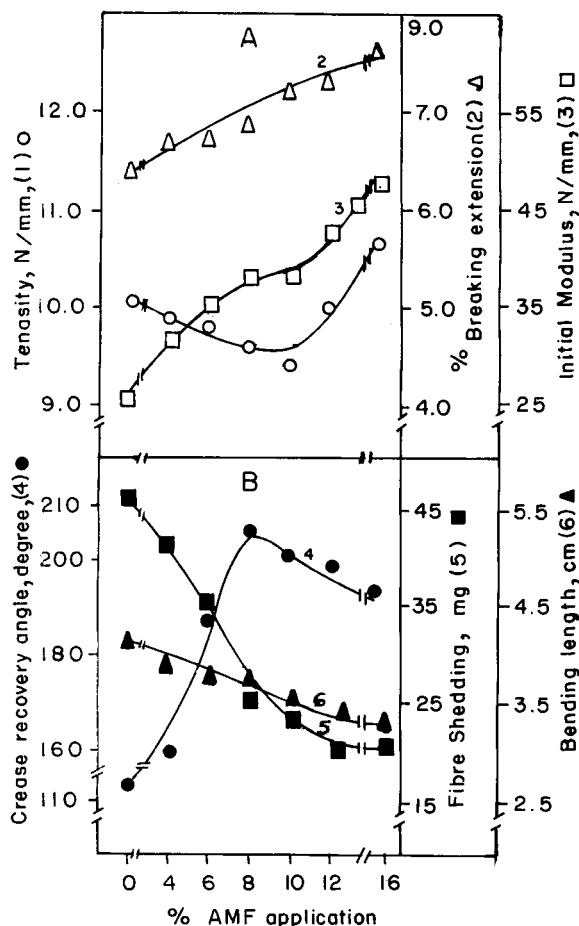


This polymerization leads to chain extension and occasional cross-linking and, consequently, to changes in the properties of the jute fabric used. Relevant results are graphically shown in Figure 2. With respect to initial modulus, variations in  $\text{MgCl}_2$  dose and  $\text{K}_2\text{S}_2\text{O}_8$  dose produce similar trends of change [Fig. 1(A) and 2(A)]; however, the trends of change in breaking extension and tenacity stand in contrast in the two cases. The



**Figure 2** Plots showing the effect of variation of dose of  $\text{K}_2\text{S}_2\text{O}_8$  at a fixed dose (1.6%) of  $\text{MgCl}_2$  catalyst for treating jute fabric with 8% AMF resin by pad-dry-cure process. (Other conditions: drying at  $80^\circ\text{C}$  for 10 min, and curing at  $150^\circ\text{C}$  for 4 min.)





**Figure 3** Plots showing the effect of variation of application level of AMF resin applied on jute fabric by pad-dry-cure process. (Other conditions are as follows. Catalyst: 0.5%  $K_2S_2O_8$  and 1.6%  $MgCl_2$ . Drying at 80°C for 10 min; curing at 150°C for 4 min.).

tenacity shows an initial increasing trend followed by a decrease beyond the use of 0.5%  $K_2S_2O_8$ , while breaking extension shows an increase with an increase in  $K_2S_2O_8$  dose [Fig. 2(A)]. The effects of an increase in the doses of  $MgCl_2$  is much similar to that produced by an increase in the  $K_2S_2O_8$  dose with respect to improvement in the crease recovery angle [Fig. 1(B) and 2(B)]. The peak in the crease recovery angle in Figure 2(B) corresponds to the use of 0.5%  $K_2S_2O_8$ . Trends of changes in fiber shedding and bending length (stiffness) properties consequent to an increase in the dose of  $K_2S_2O_8$  and  $MgCl_2$ , on the other hand, are opposite in nature. Higher  $K_2S_2O_8$  dose produces prominent lowering of fiber shedding and stiffness character of the AMF resin finished jute fabrics [Fig. 2(B)]. Analysis of data presented in Figure 2 indicates that the use of 0.5%  $K_2S_2O_8$  as the catalyst for free radical graft copolymerization

produces optimum effects with respect to properties modification of jute during AMF resin finish.

#### Effect of Variation of Application Level of AMF Resin

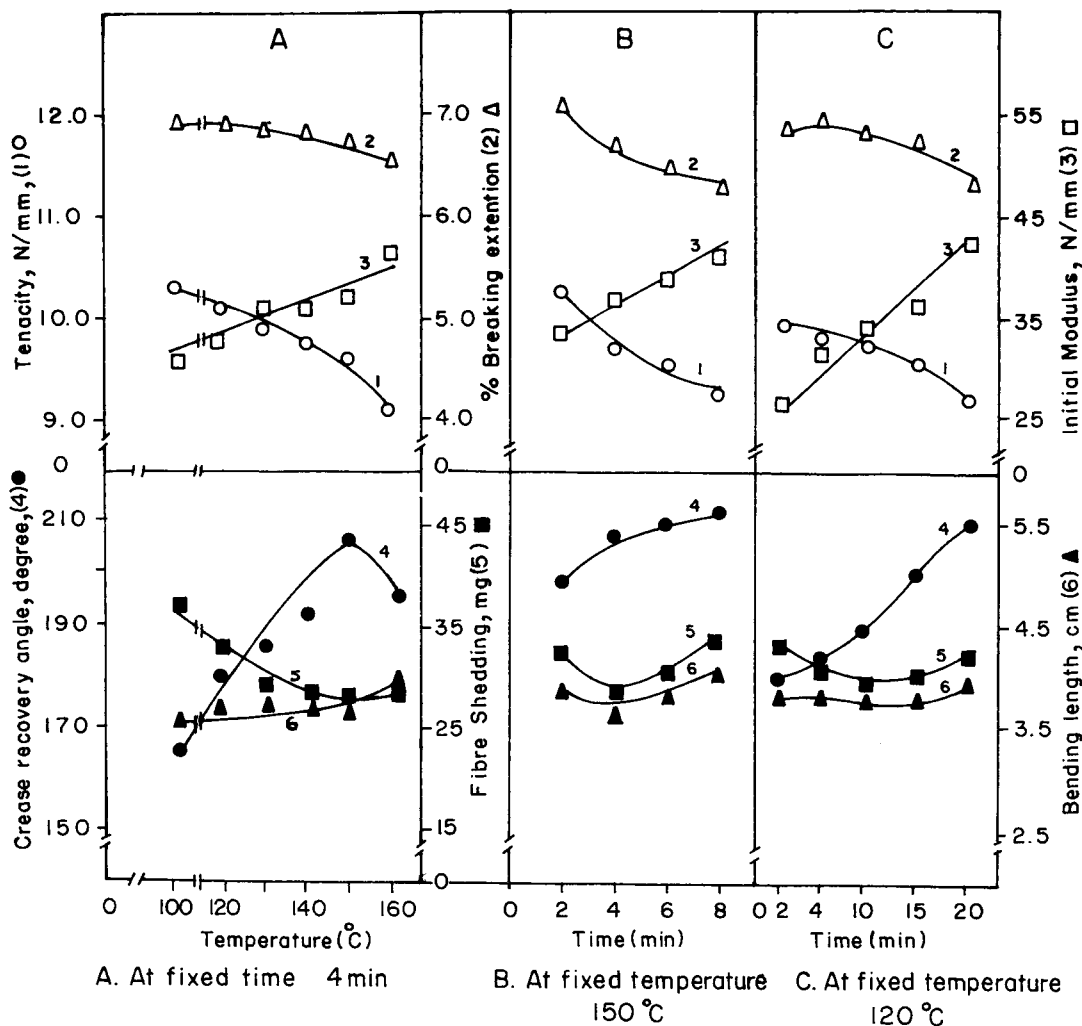
Increase in the level of AMF resin application also produces increasing trends in the initial modulus and breaking extension, as shown in Figure 3(A), while the tenacity value follows an initial dropping trend up to about 10% AMF resin application, followed by a sharply increasing trend thereafter. Trends of changes in fiber shedding, crease recovery, and stiffness characters of the treated jute fabrics with variations in AMF resin application dose, on the one hand [Fig. 3(B)], and in  $K_2S_2O_8$  dose, on the other [Fig. 2(B)], are very much similar. 8% AMF resin application resulting in about 6.0% resin uptake (weight gain data as shown in Table II), on the average, produces optimum effects.

#### Optimum Drying Condition

Drying prior to curing of the AMF-resin-treated jute fabrics was studied at different conditions, and physical/textile related properties were evaluated in each case (data not shown here), which indicate that drying at 80°C for 10 min produces optimum effects.

#### Effect of Variation of Curing Conditions

The effects of variation of curing temperature for a low time of cure (4 min) and of variation of time of cure at two selected temperature (120 and 150°C) are shown in Figure 4. Considering the range of properties such as tenacity, breaking extension, modulus, crease recovery, fiber shedding, and stiffness character (bending length) observed under different sets of conditions, as shown in Figure 4, it may be said that padding of scoured and bleached jute fabric with 8% AMF resin and curing of the resin padded fabric (subsequent to drying at 80° for 10 min), for only 4 min at 150°C or for 15 min at 120°C under the dual catalytic influence of  $MgCl_2$  (1.6%) and  $K_2S_2O_8$  (0.5%) produced the most balanced improvements in textile-related physical properties of the fabric [Fig. 4(A-C)]. Though curing at 120°C for 15 min produced comparable properties development, it showed lower crease recovery improvement than curing at 150°C for 4 min. Hence, all subsequent work was done by curing AMF resin on jute at 150°C for 4 min.



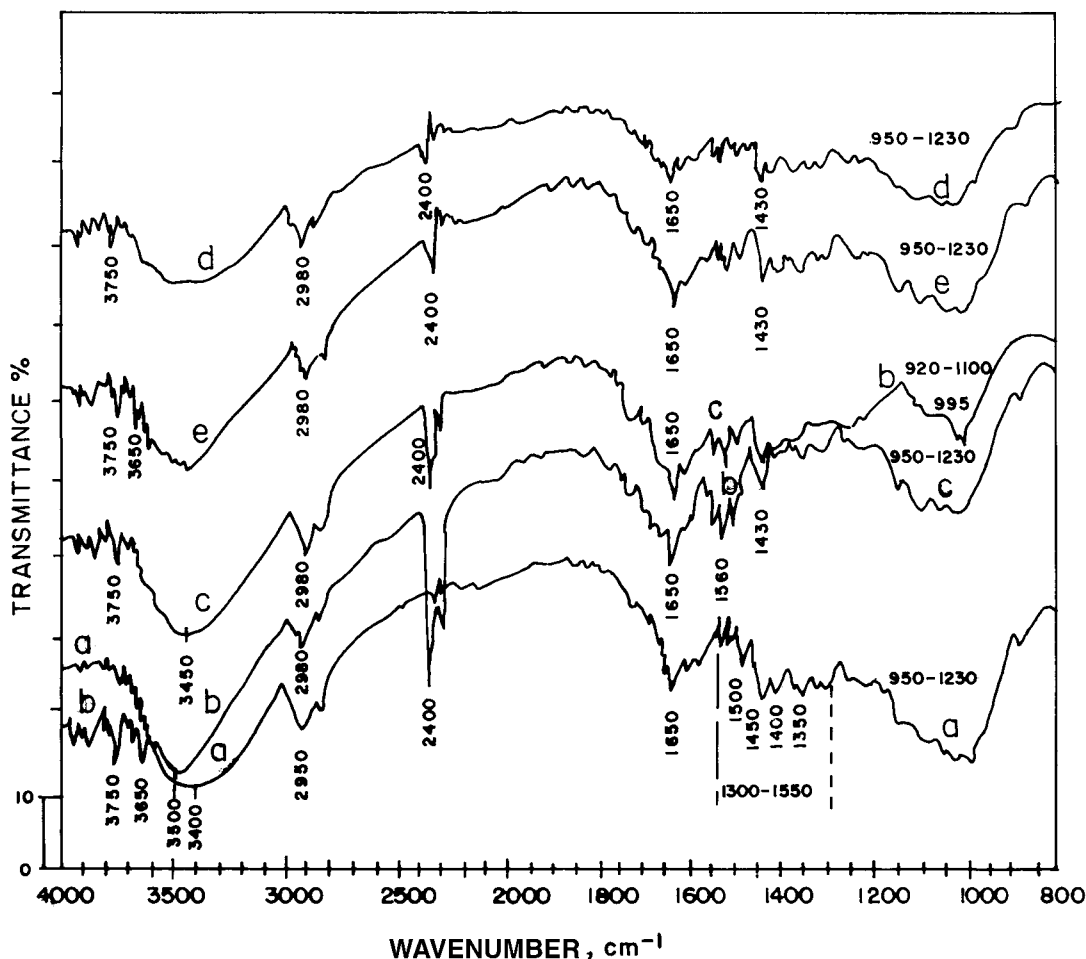
**Figure 4** Plots showing effect of variation of curing conditions (time and temperature) for treating 8% AMF resin on jute fabric by pad-dry-cure process. (Other conditions are as follows. Catalysts: 0.5%  $K_2S_2O_8$  and 1.6%  $MgCl_2$ . Drying: 80°C for 10 min.)

### FTIR Spectra

FTIR spectra of bleached jute<sup>37-40</sup> and AMF resin powder, are shown in Figure 5(a) and (b). Relevant spectra of bleached jute<sup>37,38</sup> treated differently with AMF resin, namely, using only  $MgCl_2$  as the curing catalyst [Fig. 5(c)], using only  $K_2S_2O_8$  as the catalyst for graft copolymerization [Fig. 5(d)], and using  $K_2S_2O_8$  and  $MgCl_2$  combination as the dual catalyst system for simultaneous radical chain polymerization and acidic polycondensation and curing [Fig. 5(e)] are also shown. Each spectrum [Fig. 5(a)–(e)] shows a broad band over 3200–3650  $cm^{-1}$  ascribed to O—H stretching and hydrogen bonded O—H stretching in jute-alcohol groups and resin-methylol groups and to adventitious moisture<sup>16</sup> present in the fiber/resin substrates. The small and sharp absorp-

tion bands over 3700–3800  $cm^{-1}$  for AMF resin and AMF resin treated jute samples correspond primarily to N—H stretching of the amide moieties in the resin residues.

The broad medium band over 2800–3100  $cm^{-1}$  corresponds to C—H stretching in the major constituents of jute and in the resin residues, including C—H stretching in the —CHO groups generated on jute during bleaching. In the spectra of AMF resin [Fig. 5(b)] and of resin treated jute fibers [Fig. 5(c)–(e)], presence of a relatively sharp band at 2400  $cm^{-1}$  corresponding to C=C stretching of the residual vinyl units is notable; the said peak is, however, much subdued in the spectra of the AMF-resin-treated fiber samples due to low (6% weight gain) resin content in them, particularly where the resin treatment and curing were done in the presence of  $K_2S_2O_8$  causing substan-



**Figure 5** FTIR spectra of AMF resin powder and differently treated jute fibers: (a) 3%  $\text{H}_2\text{O}_2$  bleached jute; (b) Acrylamide resin powder; (c) 8% AMF resin cured on bleached jute in presence of 1.6%  $\text{MgCl}_2$  as only catalyst; (d) 8% AMF resin cured on bleached jute, in presence of 0.5%  $\text{K}_2\text{S}_2\text{O}_8$  as the only catalyst; and (e) 8% AMF resin cured on 0.5%  $\text{K}_2\text{S}_2\text{O}_8$  presoaked bleached jute and in presence of 1.6%  $\text{MgCl}_2$ , i.e., under a dual catalyst system. (For AMF resin curing of jute fabric, other conditions were as given in Number 8 in Table I were employed.)

tial loss of alkyl unsaturation due to radical vinyl polymerization [Fig. 5(d) and (e)].

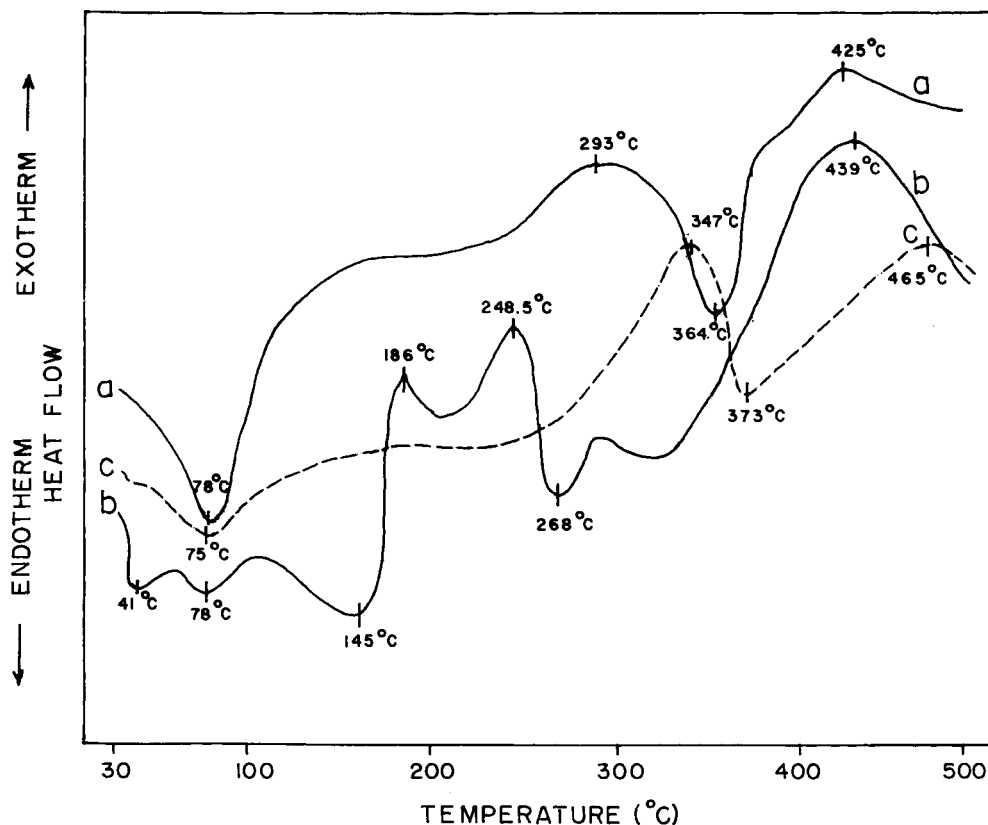
A broad and relatively sharp band ( $1550\text{--}1700\text{ cm}^{-1}$ ) having a sharp peak at  $1650\text{ cm}^{-1}$  is notable in the spectra of AMF resin and the resin-treated jute fiber samples. This band is believed to be the results of coupling of N—H deformation with C=O stretching and mesomeric C—N stretching in AMF resin moieties. In the spectra of bleached jute fiber samples (treated and untreated), a band in the  $1600\text{--}1750\text{ cm}^{-1}$  region with a peak at  $1650\text{ cm}^{-1}$  also appears consequent to overlapping of absorption due to C=O stretching in the aldehydic, carboxylic<sup>39</sup> (generated during bleaching of jute), and ester groups likely to be present in the untreated and oxidized (bleached) cellulose/hemi-

cellulose constituents and in the quinonoid groups resulting from oxidation of phenolic moieties of lignin in jute.<sup>16,40</sup>

#### Differential Scanning Calorimetric (DSC) Analysis

DSC thermograms of dewaxed jute, acrylamide formaldehyde resin powder, and AMF-resin-treated jute under the influence of the dual catalyst system ( $\text{K}_2\text{S}_2\text{O}_8 + \text{MgCl}_2$ ) are shown in Figure 6(a)–(c). Each of the three DSC curves [Fig. 6(a)–(c)] shows a broad endotherm with a peak around  $75\text{--}78^\circ\text{C}$ . The said endotherms correspond to loss of absorbed moisture/water on heating the substrates.<sup>22</sup>

The DSC curve of dewaxed jute is characterized

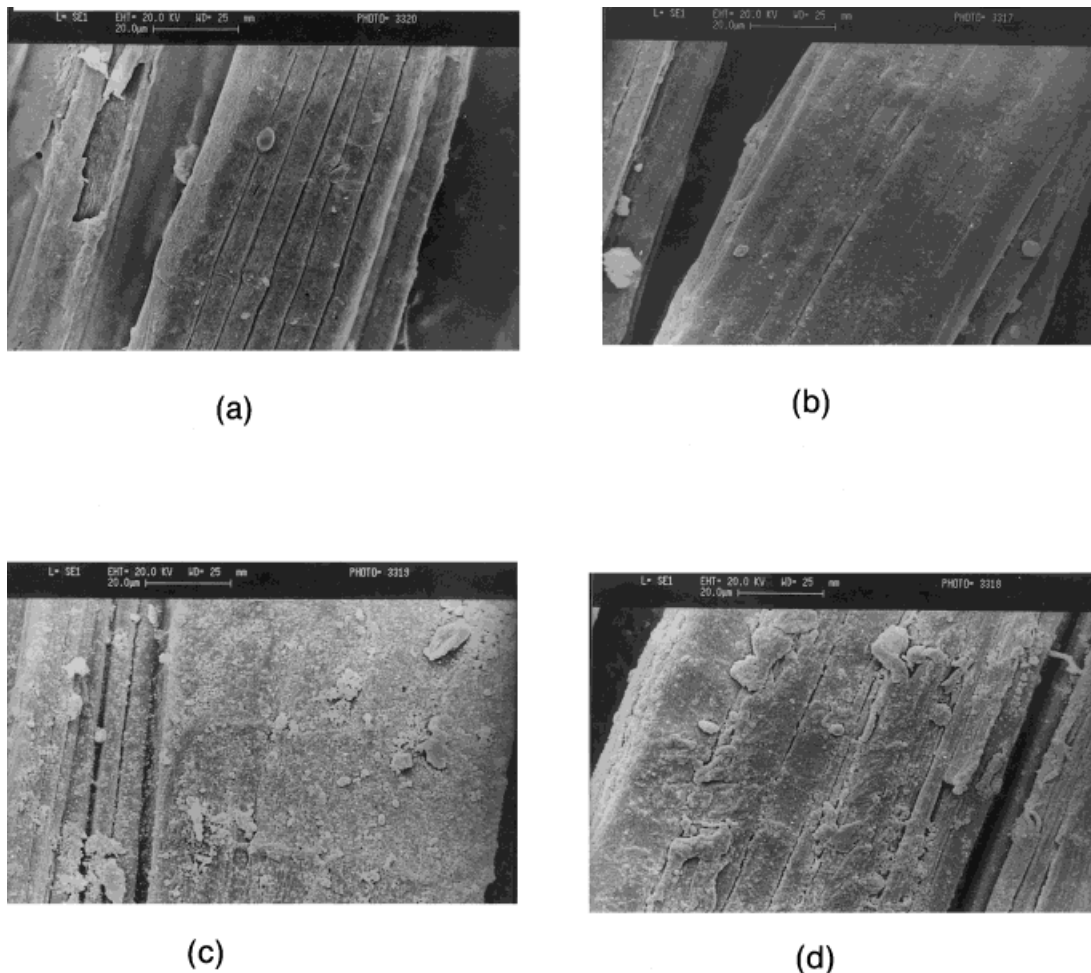


**Figure 6** DSC thermograms of fiber/resin samples under flowing nitrogen: (a) de-waxed jute; (b) AMF resin powder; and (c) 8% AMF resin cured on bleached jute in presence of 0.5%  $K_2S_2O_8$  + 1.6%  $MgCl_2$  as a dual catalyst system. (Other conditions: drying at 80°C for 10 min, and curing at 150°C for 4 min.)

by two exothermic peaks at 293 and 425°C that correspond to thermal decomposition of the hemicellulose and lignin constituents of jute respectively [Fig. 6(a)], and an endothermic peak for jute at 364°C corresponds to the thermal decomposition of  $\alpha$ -cellulose.<sup>14,37,38</sup>

The curve for AMF resin powder shows an additional weak endotherm in the low temperature region, i.e., at about 41°C; this is ascribed to loss of absorbed free formaldehyde. The endotherms and exotherms over the temperature range of 100–300°C, (with two exotherms at 186 and 248.5°C and two endotherms at 145 and 268°C) for AMF resin are likely to be associated with thermally initiated complex reactions leading to further chain extension, self-condensation, and cross-linking via methylol-methylol or methylol-residual-NH condensation and thermally initiated chain polymerization of acrylamide (vinyl moieties), when the AMF resin is heated during DSC analysis. The exotherm at 439°C [Fig. 6(b)], is apparently related to ultimate thermal decomposition of the resin system. However, absence of

the above said exotherms and endotherms over the temperature zone of 100–300°C for DSC trace of AMF-resin-treated jute sample [Fig. 6(c)] is due primarily to its low resin content; it is also indicative of the fact that reactions of AMF-resin with the constituents of jute favorably compete with the thermally initiated self-cross-linking reactions of the resin. The curve for AMF resin treated jute (having only 6% resin uptake) under the influence of the dual catalyst ( $K_2S_2O_8$  +  $MgCl_2$ ) system, is significantly different from those of jute and AMF resin. Notable cross-linking of all the major constituents of jute ( $\alpha$ -cellulose, hemicellulose, and lignin) by reaction with the AMF resin is a distinct possibility. The cross-linking of jute constituents via AMF resin segments is expected to enhance stability toward thermal decomposition of each of the above major constituents of jute. This is clearly highlighted by the DSC thermogram of the AMF resin treated jute where the exothermic peaks for the decomposition of hemicellulose (347°C) and lignin (465°C) and the endothermic peak for the decomposition of  $\alpha$ -cellulose



**Figure 7** SEM micrographs showing surface morphology of bleached and AMF-resin-finished jute under different catalyst system: (a) 3% H<sub>2</sub>O<sub>2</sub> bleached jute; (b) 8% AMF resin cured on bleached jute using only 0.5% K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as catalyst; (c) 8% AMF resin cured on bleached jute using only 1.6% MgCl<sub>2</sub> as catalyst; and (d) 8% AMF resin cured on 0.5% K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> presoaked bleached jute fabric, and in presence of 1.6% MgCl<sub>2</sub>, i.e., under a dual catalyst system. (For AMF resin curing of jute fabric, other conditions were as given in Number 8 of Table I were employed.)

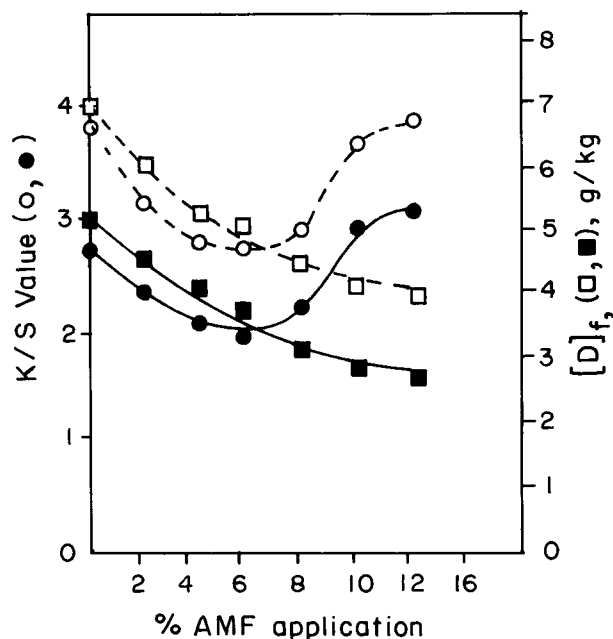
(373°C) appear at significantly higher temperatures when compared with the corresponding peaks for dewaxed jute [Fig. 6(a)]. Thus, AMF resin treatment apparently makes jute thermally more stable, as indicated by the results of DSC analysis.

#### Study of Surface Morphology of AMF-resin-treated Jute Fibers

The scanning electron micrograph of scoured and bleached jute fiber is shown in micrograph in Figure 7(a). The effect of 8% AMF resin application on the jute fiber under the influence of 0.5% K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, 1.6% MgCl<sub>2</sub>, and a combination of 0.5%

K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and 1.6% MgCl<sub>2</sub> are shown in Figure 7(b)–(d), respectively. A comparison of surface morphologies of the treated fibers reveals some interesting features; use of only K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> as the catalyst effects prominent coverage of the surfaces of the strands of the multicellular jute fibers with a cohesive film [Fig. 7(b)], apparently arising as a result of formation of polyacrylamide (PAAm) derivatives through radical vinyl polymerization of the AMF resin moieties grafted on the reactive sites or otherwise deposited on the jute fiber.

Use of MgCl<sub>2</sub> along with K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> results in a higher overgrowth of long chain polymers of AMF resin much as a consequence of additional reactions involving chain extension of the AMF resin



**Figure 8** Plots showing K/S value (index of surface dye uptake) and bulk dye uptake ( $[D]_f$  in grams of dye per kilogram of fiber) for acid dyeing and reactive dyeing of different degree of modified jute fabric employing varying percentages of AMF resin. (Open symbols are for acid dye; close symbols are for reactive dye).

system via significant methylol-methylol condensation as well as condensation between  $-\text{NH}-$  or methylol groups of the AMF resin moieties and the aldehydic or the alcoholic groups of cellulose and related constituents of (bleached) jute under the influence of the additional acidic curing catalyst,  $\text{MgCl}_2$ . The additional effect of use of  $\text{MgCl}_2$  on surface morphology of the film shown in Figure 7(d) may be compared with the features of Figure 7(b), showing the effect of use of only  $\text{K}_2\text{S}_2\text{O}_8$ ; the difference between the two is glaringly distinctive.

Use of only  $\text{MgCl}_2$  as the catalyst for resin-binding, chain extension, and curing produces an effect which is also distinctively different from that produced by use of only  $\text{K}_2\text{S}_2\text{O}_8$  as the catalyst for graft copolymerization by radical mechanism. The surface features in the former case revealed in Figure 7(c) partly resemble those in the latter case revealed in Figure 7(d); there is a common character between the reactions involved during resinification under the pad-dry-cure technique under the above two specified conditions, the common reactions being chain extension of the AMF resin system and cross-linking of the cellulosic and related chains of the jute fiber through the AMF resin moieties. The distinct cohesive film coverage

apparently due to formation of polyacrylamide (PAAm) derivatives formed by radical polymerization of AMF when  $\text{K}_2\text{S}_2\text{O}_8$  is used fully or partly as the catalyst is not effectively visible in Figure 7(c). It is indicated therefore that formation of cohesive film forming polyacrylamide derivatives by radical polymerization of the AMF resin is by and large inconsequential under the prevalent time and temperature conditions when only  $\text{MgCl}_2$  is used as the curing catalyst.

#### Extent of AMF Resin Fixation on Different Major Constituents of Jute

The major constituents of jute were isolated and AMF-resin was applied on them separately under the prescribed conditions and the finished/cured products were appropriately collected after thorough washing and drying. In each case, weight gain on resin finish was noted and N-content of the resin-finished products were estimated, and relevant data are shown in Table II. It can be readily concluded from data in Table II that during the prescribed AMF-resin finish (pad-dry-cure) of jute under the given condition, the resin is likely to react not only with the cellulosic constituents ( $\alpha$ -cellulose and hemicellulose) but also with the lignin part of the fiber. The extent of AMF resin fixation or consequent weight gain (Table II) was found to be in the following order: delignified jute (containing hemicellulose and  $\alpha$ -cellulose of the fiber)  $>$   $\alpha$ -cellulose  $>$  lignin. When the (bleached) jute fiber was cured with 8% AMF resin under the given condition using the dual catalyst ( $\text{K}_2\text{S}_2\text{O}_8 + \text{MgCl}_2$ ) system, all the major constituents of jute reacted with the resin and turned thermally more stable on resin-finish (cross-linking) as revealed by the results of DSC analysis presented earlier in the paper.

#### Effect of Different Degrees of Removal of Lignin and Hemicellulose Separately from Jute

Different degrees of removal of hemicellulose (by the action of different concentrations of  $\text{NaOH}$  under a specified condition) or of lignin (by the action of 0.7%  $\text{NaClO}_2$  under specified condition for different time periods) from dewaxed jute fabric was separately achieved. Effects of such part removal of the two constituents of jute separately on changes in the functional group pattern ( $-\text{CHO}$  and  $-\text{COOH}$  group content), on weight loss, and on subsequent AMF resin pickup (estimated by N-content analysis and weight gain data; Table III) during pad-dry-curing of the jute fiber/fabric

using AMF resin under the optimum conditions were studied.

The percentages of the major constituents of jute and their changes consequent to the said treatments for removal of hemicellulose or lignin separately as shown in Table III are all based on the initial dry weight of dewaxed jute. Changes in the functional group pattern ( $-\text{CHO}$  group content expressed in terms of copper number and  $-\text{COOH}$  group content expressed in meq/100 g of fiber) in the jute fiber consequent to the said chemical treatments are also shown in Table III. Progressive removal of hemicellulose (Table III) expectedly cause lowering of copper number (due to loss of reducing  $-\text{CHO}$  end groups with solubilization of hemicellulose). For low loss of hemicellulose using relatively dilute NaOH solution (5%), the  $-\text{COOH}$  content is relatively high but for higher removal of hemicellulose using higher concentrations of NaOH, the  $-\text{COOH}$  group content drops down. The result is indicative of ready oxidative/hydrolytic generation/liberation of  $-\text{COOH}$  groups particularly from esterlink on hemicellulose constituents on alkali attack, along with progressive loss of hemicellulose causes the  $-\text{COOH}$  content to fall after an initial buildup. Similar results were also indicated in earlier reports on hemicellulose removal from jute for other purposes.<sup>19-21</sup>

For treatment with  $\text{NaClO}_2$  under acidic condition (pH = 4) as in Table III for removal of lignin, there are monotonous increases in both  $-\text{CHO}$  group content and  $-\text{COOH}$  group content. Loss in lignin content and hydrolytic and oxidative attacks contribute to this overall effect.

It may be seen from Table III that on progressive lowering of hemicellulose content, the AMF resin pick up as given by nitrogen content passes through a minimum corresponding to about 16% hemicellulose content (the hemicellulose content of the initial dewaxed jute being 23.8%). However, a notable point is that the N-content of the AMF-resin finished jute fabric corresponding to a relatively low hemicellulose content (about 10.8–11.6%) is comparable to or measurably higher than that of the resin finished fabric having suffered no removal of hemicellulose. The trend of results as above may be understood on the basis of the following considerations.

NaOH treatment of jute causing hemicellulose removal brings two opposing effects to play—(1) mass loss, thus lowering of the more readily accessible resin-binding sites ( $-\text{OH}$  and  $-\text{CHO}$  groups in hemicellulosic constituents) and (2) swelling, resulting in creation of more voids and

enhancement of accessibility of the hydrophilic resin to the initially inaccessible or less accessible resin-binding sites. The former effect predominates until lowering of the hemicellulose content to about 16% (Table III), thereby causing a dropping trend in resin pick up, whereas the latter effect becomes overwhelming beyond this point, causing progressively enhanced resin pick up more than compensating the initial lowering trends or effects. Increased grafting of a mixture of acrylamide and methyl methacrylate monomers on hemicellulose-depleted jute using  $\text{K}_2\text{S}_2\text{O}_8$  initiator was also observed<sup>29</sup> in our laboratory. Moreover, cotton, jute, or jute blended fabrics pretreated with strong NaOH solution showed more favorable responses to subsequent methylolated resin finish showing increased resin pick up.<sup>8,41,42</sup> The effect of lignin removal as shown in Table III are very much similar, and the reasons for reversal of trend in resin pick up with progressive removal of lignin may also be attributed to the two opposing effects as detailed above; however, action of boiling dilute  $\text{NaClO}_2$  causes progressive increase in copper number in the residual jute simultaneous to weight loss as a consequence of progressive lignin removal. The enhancement in the resin pick up after some removal of lignin may also be additionally ascribed to the progressive lowering of the inhibitory effect<sup>14,16,40,43,44</sup> of lignin for free radical polymerization/graft copolymerization; moreover, notable enhancement in the reducing  $-\text{CHO}$  group content on delignification makes jute expectedly<sup>28,45</sup> more responsive to graft copolymerization utilizing the vinyl groups of AMF resin in the presence of  $\text{K}_2\text{S}_2\text{O}_8$ , much as a consequence of redox activation of the peroxy initiator.

#### Dye-Uptake of AMF Resin Treated Jute

Dyeability data in terms of K/S value (an index for estimating surface dye uptake) for different percentages of AMF resin application indicate an initial lowering in the K/S value or surface dye uptake for up to 6–8% AMF resin application in case of both reactive dye (CI Reactive Red-11,  $\lambda_{\text{max}} = 540$  nm) and acid dye (CI Acid Red-73,  $\lambda_{\text{max}} = 480$  nm) (Fig. 8); above 8% AMF resin application, interestingly, the surface dye-uptake follows an increasing trend that finally tends to level off at or just beyond 10–12% AMF resin application (Fig. 8).

Bulk dye uptake ( $[D]_f$ ) of the untreated and AMF resin treated jute fabrics (expressed in terms of grams of dye pick up per kilogram of the

fiber as indirectly determined from dye exhaustion data obtained by examination of spent dye-bath liquor in a spectrophotometer), however, follows a monotonously decreasing trend for both acid and reactive dyes with increase in AMF-resin application level. The lowering of both surface and bulk dye uptakes by the fabrics with increase in AMF resin application is due probably to (1) higher degree of cross-linking of AMF resin (2) higher degree of grafting of AMF resin moieties to jute-constituents, and (3) higher degree of masking of the fiber surfaces consequent to higher extents of film formation on the fiber due to polymerization/cross-linking effects, all impairing penetration of dye within the fibers. Independent experiments with cross-linked/polymerized AMF resin (in absence of jute) indicated good degree of dye-binding for both type of dyes by the resin itself under comparable conditions of dyeing. Since the resin binding on jute and masking effect of the film forming polymer from the AMF resin system is located mostly on the fiber surface, the surface dye uptake of the fiber, i.e., K/S value, shows an upward turn after the initial falling trend even though the bulk dye uptake continues to show the falling trend monotonously. The minimum in the surface dye-uptake apparently corresponds to about 6–8% of AMF resin application.

## CONCLUSION

8% AMF resin application on scoured and bleached jute fabrics in pad-dry (80°C, 10 min)–cure (150°C, 4 min) process under the influence of an optimum combination of 0.5%  $K_2S_2O_8$  and 1.6%  $MgCl_2$  as the dual catalyst system allowed simultaneous acidic polycondensation and free radical polymerization/graft copolymerization of AMF resin on jute fibers. This ultimately led to some cross-linking of the fiber systems and infusion of a much improved balance in their textile related properties. Jute fabrics so treated showed reduced fiber shedding, good moisture regain, and improved flexibility with much desired improved crease recovery despite some drop in tenacity.

It was indicated that all the major fractions of jute, namely, the cellulosic constituents (hemicellulose,  $\alpha$ -cellulose) and lignin reacted with the AMF resin moieties and the order of extent of AMF resin fixation was delignified jute >  $\alpha$ -cellulose > lignin. AMF resin treatment apparently made jute thermally more stable. Different degrees of removal of lignin or hemicellulose from jute fabric separately prior to AMF resin treat-

ment led to an initial lowering trend in resin uptake up to 6–8% resin application followed by an upward turn thereafter. Such chemical processing and resin-finish of jute fabrics enhanced their prospects for consideration for application in diverse, nontraditional areas such as crease resistant upholstery, furnishing, and apparel.

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