Finishing of jute using methacrylic acid in presence of tetrassodium pyrophosphate and potassium persulphate as catalysts under thermal treatment

Debasish Das*  
Institute of Jute Technology, 35 B C Road, Kolkata 700 019, India  
and  
Rajiv Munshi  
International Institute of Fashion Design, 20 A Lindsay Street, Kolkata 700 085, India

*To whom all correspondence should be addressed.
Present address: National Institute of Fashion Technology, Salt Lake City, Kolkata 700 098, India.  
E-mail: drdebasishdas@yahoo.co.in

Revised received 26 May 2008; accepted 15 July 2008

Jute fabric has been modified using methacrylic acid as the finishing agent in the presence of \( K_2S_2O_8 \) and \( Na_4P_2O_7 \) catalysts separately or in selected combinations, employing a pad-batch-dry-cure technique. It is found that the treatment with 100g/L methacrylic acid at 30°C and \( p\text{H} \) 7 using batching time of 60-75 min at 30°C followed by drying of the batched fabric at 95°C for 5 min and curing of the dried fabric at 140°C for 5 min under the catalytic influence of \( K_2S_2O_8 \) and \( Na_4P_2O_7 \) produces most balanced improvement in the textile-related properties, such as wrinkle recovery angle, flexibility, abrasion resistance, elongation-at-break and resistance to photo-yellowing with retention of strength.

Keywords: Cellulose, Esterification, Finishing, Graft copolymerization, Jute, Methacrylic acid

1 Introduction

In recent years, consciousness about environmental preservation, control of pollution and emphasis on use of energy efficient material and processing in the industrial sector have renewed interest in renewable resources including jute for use not only in their traditional application areas but also in many new and diverse application areas. Among the different non-traditional applications envisaged, jute decorative and furnishing fabrics, coated jute textile, floor mats, nonwoven, geotextiles and jute reinforced plastics are rated high in importance.\(^1\)\(^-\)\(^3\) Exploitation of jute in these application areas necessitates improvements in various properties of related fibres and textile. There remains ample scope for balanced improvement in wrinkle recovery, flexibility, strength, light fastness (for bleached and dyed jute), fibre shedding, fire resistance and abrasion resistance. The present work relates to modification of jute fibre by reaction with methacrylic acid (MAA), leading to notable esterification, control degree of cross-linking of jute and graft copolymerization of MAA onto the fibre substance.

2 Materials and Methods

2.1 Materials

2.1.1 Fabric

Plain weave, 100% jute fabric with 63 ends/dm, 64 picks/dm, 191 tex warp, 194 tex weft, and 262 g/m² average area density was used for the study.

2.1.2 Chemicals

Textile grade 1, 3-dimethylol, 4, 5-dihydroxy-ethylene urea (DMDHEU), obtained from Ahura Chemicals Ltd. (Ahuramine – YX), India, was used. Commercial grade methacrylic acid (MAA), obtained locally in Calcutta, was used without any treatment or purification. All other chemicals (E. Merck, India) used were of laboratory reagent grade.

2.2 Methods

2.2.1 Scouring

Scouring of jute fabric was performed\(^3\) using a solution containing 2 g/L sodium carbonate and 1 g/L a nonionic detergent with fabric-to-liquor ratio of 1 : 20 (w/v) at 60°C for 30 min, followed by washing of fabric with water and then drying in air.
2.2.2 Bleaching

Bleaching of jute fabric was performed in aqueous media using a solution containing 0.5% hydrogen peroxide (H₂O₂), 1.66% sodium silicate, 0.42% trisodium phosphate (Na₃PO₄), 0.08% sodium hydroxide (NaOH), and 0.08% nonionic detergent in a laboratory jigger machine at a fabric-to-liquor ratio of 1:6 (w/v) for 1 h at 80°C. After bleaching, the fabric was washed with distilled water, neutralized with dilute acetic acid, and finally washed with distilled water until the wash liquor was neutral.

2.2.3 Dyeing

Unmodified jute fabric and jute fabrics duly modified with MAA monomer system were selectively dyed using Bismark Brown (CI Basic Brown 331) at 4% shade in lab open beaker dyeing equipment at 80°C for 1.5 h under stirring condition; pH of the dye bath was adjusted at 4. After completion of dyeing, the fabrics were washed with distilled water and dried in air.

2.3 Application of Polymer or Resin Finishes on Jute

2.3.1 Methacrylic Finish

For finishing of bleached jute fabric with methacrylic acid, the jute fabric was pretreated with an aqueous solution containing 5g/L of potassium persulphate following a padding technique in a laboratory two-bowl padding mangle maintaining 100% wet pick-up. Application of aqueous methacrylic monomer formulations on persulphate pretreated jute fabric was performed thereafter also by the padding technique in the laboratory two-bowl padding mangle (two-dip-two-nip). After each of two successive dipping in the methacrylic formulation, the pressure between the squeezing rollers was adjusted to enable a wet pick-up of 100%. The monomer solutions were neutralized to pH 7 by the addition of a required dose of sodium carbonate. The aqueous formulation of MAA also contained a known dose of Na₄P₂O₇ as the esterification catalyst. The padded, squeezed fabrics were allowed to stand at room temperature 30°C (Batching temperature) for different time periods (0-75 min, known as batching time) before they were subjected to drying in an oven at 95°C for 5 min. The dried fabrics were then oven-cured at 140°C for 5 min.

2.3.2 DMDHEU Resin Finish

In the case of DMDHEU resin finish, the fabric was likewise padded with aqueous resin containing 120g/L DMDHEU and 40g/L MgCl₂ under controlled squeeze pressure in order to allow a wet pick-up of 100%. The resin treated fabric was then dried at 95°C for 5 min and subsequently cured at 140°C for 5 min.

The jute fabrics treated with acrylic monomers and DMDHEU resin were soap-washed following ISO-II method, rinsed with water and dried in air.

2.3.3 Determination of Moisture Regain and Weight gain

Moisture regain of jute fabrics was determined following a standard procedure. For the determination of weight gain upon different finishing treatments, the fabric samples treated with only MAA were first soap-washed; the fabrics were then extracted under reflux in a water bath for 8-10 h using water, methanol-water mixture, tetrahydrofuran and dimethylformamide (DMF) respectively, in order to ensure removal of unreacted monomers and polymeric acrylic moieties remaining unbound with the chain molecules of jute samples. The extracted fabric samples were then oven dried.

2.3.4 Measurement of Tensile Properties

Tenacity and elongation-at-break of different fabric samples were measured according to a method prescribed by IS: 1969-1968 using a Zwick 1445 CRT universal tensile testing machine. The results obtained were based on an average of 10 tests in the warp direction of each sample. The test strip specimens were raveled to a size of 50 × 20 mm between the jaws of the machine, the tests were performed with a traverse speed of 100 mm/min at a pretension of 0.5 N.

2.3.5 Determination of Wrinkle Recovery Angle

Dry wrinkle recovery angle (warp + weft) of selected fabric samples was determined according to a method prescribed by ASTM-D-1295-67 using a SASMIRA wrinkle recovery tester with a specimen size of 25 × 200 mm.

2.3.6 Measurement of Fabric Stiffness (Bending Length)

Fabric stiffness as expressed in terms of bending length was measured according to IS: 6490-1971 (Cantilever Test) in a SASMIRA stiffness tester with a specimen size of 25 × 200 mm.

2.3.7 Determination of Abrasion Resistance

Comparative evaluation of accelerated abrasion resistance of selected fabrics was done in an electronic abrading instrument (Crockmeter) using a
standard (IS: 715) paper abrader for a certain wear following AATCC- 93-974. The abrasion resistance of the fabric samples was given by digital display of the number of cycles of accelerated abrasion corresponding to the appearance of the wear (first appearance of a hole on the fabric by wear and tear of the yarns).

2.3.8 Measurement of Dyeability, Whiteness Index and Specular Reflectance

To determine dyeability, the \( K/S \) value of the treated and untreated fabrics dyed with a basic dye were examined in a Macbeth 2020+ reflectance spectrophotometer interfaced with a computer. The reflectance values \( R \) of the dyed fabric at the wavelength of maximum absorption \( \lambda_{\text{max}} = 540 \text{ nm} \) were converted to the corresponding \( K/S \) value using Kubelka – Munk equation.\(^{4}\)

The whiteness index (WI) in the Hunterlab scale of the selected fabric samples was obtained following a standard approach and procedure\(^{6}\) (ASTM E 313-1976/1973) with the help of Macbeth 2020+ reflectance spectrophotometer. Specular reflectance (surface gloss) of the selected fabrics (surface reflectance at 45°) was also measured using the same spectrophotometer.

2.3.9 Measurement of Fibre Shedding

Fibre shedding level of the jute fabrics was estimated\(^{3}\) by measuring the weight loss in mg on abrading a fixed area of a selected fabric sample of 10 cm length with a stretched piece of the corresponding fabric on a drum for affixed number of cycles using an electronically controlled standard abrading instrument (SDL – Crockmeter).

2.3.10 Determination of Acid Value and Ester Value

The COOH group content and ester value of selected jute samples were determined following the methods described elsewhere.\(^{2}\) The fabric samples were made (metallic) cation free by dipping in the dilute HCl for 2 h and subsequently made HCl - free by washing with CO\(_2\) - free distilled water prior to the acid value and ester value determination.

2.3.11 Infrared Spectroscopy

Infrared (IR) spectra of unmodified and selectively modified jute samples were obtained following the KBr pellet technique as detailed elsewhere\(^{2}\) using Shimadzu IR 440 spectrophotometer. The dried fibre samples were crushed to a size finer than 20 mesh before pelletizing with KBr. The test KBr pellets contained about 1% powdered fibre.

3 Results and Discussion

3.1 Role of Dual Catalyst System for Methacrylic Acid Finishing of Jute

Jute fabric has been modified by treatment with MAA under the influence of (i) Na\(_2\)P\(_2\)O\(_7\) as the esterification catalyst and (ii) K\(_2\)S\(_2\)O\(_8\) as the catalyst separately and in combination to induce free-radical polymerization and graft copolymerization of MAA on jute (Scheme 1). The sequence of reactions, as shown in Scheme 1, would be expected to take place, ultimately leading to notable weight gain and changes in the chemical nature of jute during the overall process. Na\(_2\)P\(_2\)O\(_7\) is known to be an efficient esterification catalyst \(^{7}\) and the reaction [Scheme 1 (a)] producing MAA ester of jute would be the direct consequence of its catalytic action. However, the MAA ester of jute then reacts with the excess MAA, leading to thermal polymerization of the monomeric acid as revealed by reaction [Scheme 1 (b) (i)] and to subsequent crosslinking of jute as shown by reaction [Scheme 1 (b) (ii)] during the drying and curing step. Direct consequence of esterification under the influence of Na\(_2\)P\(_2\)O\(_7\) is therefore unlikely to enhance –COOH group content of jute, even though it would lead to the enhancement in the ester value of the product. However, uncatalysed thermal polymerization of MAA ultimately leads to grafting of poly(methacrylic acid) chains on the chain molecules of jute and ultimate crosslinking [Scheme 1(b)] potentially occurs in addition to peroxodisulphate (S\(_2\)O\(_8^2\)) induced free-radical polymerization of MAA [Scheme 1, reaction 2(a)-(c)], if K\(_2\)S\(_2\)O\(_8\) is also included in the catalyst system.

The thermal graft copolymerization and crosslinking [Scheme 1(b) (i) and (ii)] would cause measurable enhancements in the –COOH content of the product from which removal of the unbound (not chemically bound to jute) monomeric and polymeric MAA is ensured by solvent extractions.

On the other hand, potassium persulphate is capable of initiating either photo and redox graft copolymerization\(^{8,9}\) of jute at ambient temperature (30°C) or photo, redox and thermal graft polymerization of lignocellulosic fibre at a high temperature (>40°C). Direct influence of the free-radical catalyst K\(_2\)S\(_2\)O\(_8\) over the batching time at room temperature and at higher temperatures during drying and curing would cause substantial enhancement in the –COOH
content of the modified jute, as a consequence of graft copolymerization without enhancement in the ester value. However, some purely thermal self-catalyzed esterification involving –OH groups in the chain molecules of the jute and finishing agent MAA during drying (95°C) and curing (140°C) process, even in the total absence of esterification catalyst Na₄P₂O₇, cannot be altogether precluded. Any enhancement in the ester value of the MAA modified jute in the absence of esterification catalyst should thus be taken as a consequence of this self (MAA) catalysed thermal esterification.

Under the influence of two catalysts taken together (Na₄P₂O₇ and K₂S₂O₈) for the curing of the jute, all the reactions shown and discussed above are likely to take place simultaneously or successively, leading to a weight gain for the fibre – fabric system from which unreacted MAA and the homopolymer of MAA have been removed by solvent (water) extraction as well as enhancement in its COOH group content (acid value) and ester value. Moreover, additional reactions leading to further esterification and complex network formation may also take place due to limited establishment of ester linkages between the carboxylic (–COOH) groups of a poly-carboxylic chain bound to a particular jute molecule and alcoholic (–OH) groups of the same or other neighbouring jute cellulose molecules.
3.2 Effect of Variation in Batching Time

The effect of variation in batching time (0, 15, 30, 45, 60 and 75 min) on fabric properties is shown in Table 1. There is a notable increase in the weight gain, abrasion resistance and wrinkle recovery angle (WRA) of the fabric. On the other hand, the tenacity value decreases with the increase in batching time. The drop in tenacity of finished jute fabrics from 0 min to 75 min batching time at 30°C is found to be ≤20%. Batching for an extended time distinctly favours the higher incorporation of MAA moieties on jute by K₂S₂O₈ – induced graft copolymerization.

The carboxylic content appears to drop and the ester value tends to increase as the batching time is increased from 0 min to 60 min. There is little likelihood of the establishment of ester linkages at ambient temperature over the different periods of batching. Initial S₂O₈²⁻ induced graft copolymerization or homopolymerization of MAA, to increasing extents over increasing batching time periods, at ambient temperature (30°C) and further polymerization of free MAA and jute bound MAA moieties during the subsequent drying period at 95°C cause an overall change in environment and proximity of the hydroxyl groups of jute and carboxyl groups of the unbound or jute bound MAA or poly(methacrylic acid) moieties in a manner that finally causes an enhanced degree of esterification during curing at 140°C under the influence of Na₄P₂O₇ catalyst.

The esterification reaction that assumes more prominence at the high processing temperature (140°C) in the final stage appears to be somewhat dependant on the initial batching time. A higher batching time favours improved transformation of the grafted-on MAA or poly(methacrylic acid) units to ester moieties at the high curing temperature (140°C) under the influence of the esterification catalyst in the final stages of processing. Optimum batching time (60-75 min) also allows improved diffusion / penetration of the finishing agent MAA within the chain molecules of jute.

This may well explain the trends of change in the acid and ester values with the change in batching time as shown in Table 1. However, beyond an optimum batching period (60-75 min), the initial accumulation of poly(methacrylic acid) on jute fabric by grafting and some degree of crosslinking of jute cellulose may become very high indeed, even prior to heating at the curing temperature (140°C). The trends of change in the carboxyl content and ester value get significantly perturbed on curing in the subsequent step, even though the tenacity value, abrasion resistance and wrinkle recovery of the fabrics remain at the same level or are slightly improved (Table 1) due to favourable curing or crosslinking effect. In the absence of Na₄P₂O₇ esterification catalyst, simple persulphate action expectedly leads to limited graft copolymerization and little esterification over the

<table>
<thead>
<tr>
<th>Catalyst*</th>
<th>Batching time, min</th>
<th>Weight gain, %</th>
<th>-COOH value, meq/100g</th>
<th>Ester value</th>
<th>Tenacity N/cm at-break %</th>
<th>Elongation-angle (W+F), deg</th>
<th>Abrasion resistance, No. of cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>K₂S₂O₈</td>
<td>Na₄P₂O₇</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>+</td>
<td>+</td>
<td>0</td>
<td>7.38</td>
<td>29.32</td>
<td>12.48</td>
<td>101.07</td>
<td>7.21</td>
</tr>
<tr>
<td>+</td>
<td>+</td>
<td>15</td>
<td>7.92</td>
<td>28.61</td>
<td>16.21</td>
<td>99.26</td>
<td>7.84</td>
</tr>
<tr>
<td>+</td>
<td>+</td>
<td>30</td>
<td>8.27</td>
<td>27.14</td>
<td>18.13</td>
<td>96.11</td>
<td>8.72</td>
</tr>
<tr>
<td>+</td>
<td>+</td>
<td>45</td>
<td>8.80</td>
<td>26.34</td>
<td>22.31</td>
<td>89.33</td>
<td>9.37</td>
</tr>
<tr>
<td>+</td>
<td>+</td>
<td>60</td>
<td>9.21</td>
<td>24.28</td>
<td>25.11</td>
<td>82.41</td>
<td>9.97</td>
</tr>
<tr>
<td>+</td>
<td>+</td>
<td>75</td>
<td>9.87</td>
<td>27.79</td>
<td>22.38</td>
<td>84.63</td>
<td>10.22</td>
</tr>
<tr>
<td>-</td>
<td>+</td>
<td>0</td>
<td>7.26</td>
<td>14.38</td>
<td>11.35</td>
<td>98.94</td>
<td>7.65</td>
</tr>
<tr>
<td>-</td>
<td>+</td>
<td>60</td>
<td>8.06</td>
<td>27.77</td>
<td>13.52</td>
<td>100.00</td>
<td>7.78</td>
</tr>
<tr>
<td>+</td>
<td>-</td>
<td>60</td>
<td>6.32</td>
<td>34.47</td>
<td>7.36</td>
<td>118.65</td>
<td>7.98</td>
</tr>
</tbody>
</table>

Only Bleached jute

- - - - - - - - 12.36 5.87 104.48 6.32 51 144

* K₂S₂O₈ = 5g/L and Na₄P₂O₇ = 150g/L; '+' and '-' signs indicate presence and absence of respective catalyst during application of methacrylic acid.
optimum batching period at 30°C. Subsequent thermal curing ultimately produces a finished fabric having much enhanced carboxyl content and poor ester value for which the observed tenacity value is even higher than that of the untreated (bleached) jute. The abrasion resistance is also the highest for this finished fabric, even though the improvement in the wrinkle recovery angle is not high enough. Data in Table 1 clearly show that the retention or improvement in the functional group pattern and weight gain, as well as textile related properties is optimal on pad-batch-dry-cure of bleached jute with MAA at pH 7 under the influence of dual catalyst system following a batching time of 60-75 min. For finishing of jute fabric with methacrylic acid under the influence of free radical polymerization catalyst and esterification catalyst, batching of padded fabric is an integral part of the methacrylic acid application technique that cannot be eliminated, if one aims at improving textile-related properties of jute fabric including wrinkle recovery angle. Introduction of such batching process for 60-75 min however makes the application technique semi-continuous.

3.3 Effect of Variation in pH

Table 2 shows the effect of variation in pH (5, 7, 8 and 10) of the MAA finishing formulation on the properties of the finished fabrics. It is found that under neutral condition (pH 7), the optimum grafting and esterification, leading to much improved wrinkle recovery angle (WRA), abrasion resistance and substantial weight gain, are achieved with nearly 80% retention of tenacity of the initial fabric. Under slightly alkaline condition (pH 8, 10), the improvement in wrinkle recovery angle is only marginal or poor, even though the improvement in abrasion resistance is notable and there is even 5-10% enhancement in the tenacity of the finished fabric. A slightly acidic condition (pH 5) results in poor retention of tenacity (<45%) and abrasion resistance (<30%) despite substantial weight gain much as a consequence of weakening of the fibre in the fabric by acid attack. Hence, neutral pH 7 apparently provides the most optimum condition for the finishing process.

3.4 Application Level of Methacrylic Acid

Table 3 shows the effect of MAA concentration (0, 20, 40, 80, 100 and 120 g/L) under the influence of dual catalyst system at pH 7. It is observed that the optimum weight gain and balance of textile-related properties for the finished fabric are obtained for the application of 100g/L MAA. The finished jute fabric, so obtained, also shows a substantial enhancement in the dye uptake (K/S) value. Improvement in basic dye uptake of the above finished jute fabric is the direct consequence of the incorporation of carboxyl acid group under the influence of finishing condition employed. Whiteness index of the bleached jute fabric practically remains undiminished on finishing with MAA.

A comparison between MAA-finished and DMDHEU-finished jute fabrics against the unfinished jute fabric with respect to their property parameters (Table 4) clearly indicates that MAA produces much desired improvement in the fabric quality. MAA specifically imparts a lower order of stiffness (bending length), a higher abrasion resistance with higher retention of strength, improved resilience and fibre shedding quality and high moisture regain and light fastness rating for the fabric dyed with a basic dye.

Table 2 — Effect of variation in pH on property of bleached jute fabrics treated with methacrylic acid at constant batching time of 60 min

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Application pH</th>
<th>Weight gain, %</th>
<th>-COOH meq/100g</th>
<th>Ester value</th>
<th>Tenacity N/cm</th>
<th>Elongation - at-break %</th>
<th>Abrasion resistance</th>
<th>Wrinkle recovery angle (W+F), deg</th>
</tr>
</thead>
<tbody>
<tr>
<td>K₂S₂O₈</td>
<td></td>
<td>5</td>
<td>8.88</td>
<td>24.98</td>
<td>23.56</td>
<td>47.05</td>
<td>7.55</td>
<td>29</td>
</tr>
<tr>
<td>Na₄P₂O₇</td>
<td></td>
<td>7</td>
<td>9.21</td>
<td>24.28</td>
<td>25.11</td>
<td>82.41</td>
<td>9.97</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>+</td>
<td>8</td>
<td>9.75</td>
<td>30.04</td>
<td>22.21</td>
<td>107.54</td>
<td>8.86</td>
<td>51</td>
</tr>
<tr>
<td></td>
<td>+</td>
<td>10</td>
<td>10.22</td>
<td>32.66</td>
<td>11.25</td>
<td>116.28</td>
<td>8.65</td>
<td>58</td>
</tr>
<tr>
<td>Only bleached jute</td>
<td></td>
<td>-</td>
<td>-</td>
<td>12.36</td>
<td>5.87</td>
<td>104.48</td>
<td>6.32</td>
<td>51</td>
</tr>
</tbody>
</table>

a K₂S₂O₈ = 5g/L and Na₄P₂O₇ = 150g/L; ‘+’ sign indicates presence of respective catalyst during application of methacrylic acid.
It has been observed that the whiteness index of the bleached jute practically remains undiminished on MAA finish but it suffers some lowering on finishing with DMDHEU for nearly comparable improvement in the WRA.

3.5 Photo-yellowing of Jute

Bleached, MAA treated jute and DMDHEU resin treated jute fabrics were exposed to UV light in an MBTL light fastness tester and changes in the whiteness index after different periods of UV exposure at room temperature (30°C) were observed (Fig. 1). It is observed from the figure that the trend of lowering of whiteness index on UV exposure is common to each treated jute sample; the rate of lowering being more severe initially in each case. Both the rate and degree of lowering of whiteness index on UV exposure are in the order: DMDHEU treated jute > bleached jute >> MAA treated jute. Photo exposure of jute is known to cause some discoloration or yellowing of the fabric as a consequence of (i) scissions of occasional bonds between lignin and cellulose/hemicellulose chains and (ii) methoxyl splitting from the lignin constituents leading ultimately to the formation of orthoquinones and related chronophers, thus causing intensification of colour or yellowing. Control estrification of jute such as by acetylation has earlier been reported to minimize or prevent its yellowing on exposure to light. Improved effect on whiteness index and retention for jute treated with MAA as observed in the present studies is in tune with the reported effect of acetylation. Photo splitting of ester groups found on jute via MAA or acetic anhydride treatment is believed to cause some bleaching effect, consequent to associated in-situ oxidation of photo split acid residues to peroxy moieties. The additional bleaching effect of ester photo splitting of MAA treated jute partly compensates for the yellowing effect due to simultaneous photo splitting of the lignin constituents; the net effect being a relatively high retention of whiteness index for the MAA treated jute fibre on UV exposure.

3.6 Specular Reflectance of Jute Fabric

Figure 2 provides information about the relative rating of bleached jute fabric and differently finished jute fabric with DMDHEU resin and MAA monomer system with respect to surface gloss or specular reflectance at 45°. It is clear from the specular reflectance data that MAA finished bleached jute attains substantially improved gloss; the order being MAA finished jute fabric > DMDHEU finished jute fabric > bleached jute fabric. Higher specular

### Table 3 — Effect of methacrylic acid treatment at pH 7 on bleached jute

<table>
<thead>
<tr>
<th>MAA Conc. g/L</th>
<th>Weight gain, %</th>
<th>Basic dye uptake (K/S)</th>
<th>Wrinkle recovery angle (W+F), deg</th>
<th>Tenacity N/cm</th>
<th>Abrasion resistance No. of cycle</th>
<th>Whiteness index</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>-</td>
<td>12.33</td>
<td>144</td>
<td>104.48</td>
<td>51</td>
<td>59.25</td>
</tr>
<tr>
<td>20</td>
<td>1.608</td>
<td>14.28</td>
<td>183</td>
<td>101.22</td>
<td>41</td>
<td>59.38</td>
</tr>
<tr>
<td>40</td>
<td>3.92</td>
<td>18.54</td>
<td>193</td>
<td>95.55</td>
<td>45</td>
<td>59.52</td>
</tr>
<tr>
<td>80</td>
<td>6.86</td>
<td>22.02</td>
<td>223</td>
<td>83.65</td>
<td>50</td>
<td>59.47</td>
</tr>
<tr>
<td>100</td>
<td>9.21</td>
<td>23.14</td>
<td>234</td>
<td>82.41</td>
<td>55</td>
<td>59.82</td>
</tr>
<tr>
<td>120</td>
<td>10.88</td>
<td>24.87</td>
<td>241</td>
<td>77.89</td>
<td>58</td>
<td>59.86</td>
</tr>
</tbody>
</table>

### Table 4 — Comparison of properties between MAA – finished jute and conventional DMDHEU – finished jute fabrics

<table>
<thead>
<tr>
<th>Type of finish</th>
<th>Wrinkle recovery angle (W+F), deg</th>
<th>Bending length cm</th>
<th>Abrasion resistance No. of cycle</th>
<th>Tenacity N/cm</th>
<th>Elongation at-break %</th>
<th>Fibre shedding mg</th>
<th>Moisture regain %</th>
<th>Light fastness ratinga</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nil</td>
<td>144</td>
<td>3.8</td>
<td>51</td>
<td>104.48</td>
<td>6.32</td>
<td>34.69</td>
<td>16.87</td>
<td>1</td>
</tr>
<tr>
<td>MAA (100 g/L)</td>
<td>234</td>
<td>2.6</td>
<td>55</td>
<td>82.41</td>
<td>9.42</td>
<td>9.88</td>
<td>19.21</td>
<td>3</td>
</tr>
<tr>
<td>DMDHEU (120 g/L)</td>
<td>246</td>
<td>4.3</td>
<td>36</td>
<td>66.28</td>
<td>4.35</td>
<td>36.88</td>
<td>9.88</td>
<td>1</td>
</tr>
</tbody>
</table>

*a Fabric dyed with a Bismark Brown (CI Basic 331).
reflectance is indicative of deposition of smooth and cohesive film of poly(methacrylic acid) on the fabric (fibre) surface with wide coverage on completion of the finishing process.

3.7 IR Analysis

The infrared spectra of unmodified scoured and bleached jute fibre [spectrum (a)] and of the jute fibre differently modified with MAA treatment using only K$_2$S$_2$O$_8$ as the free radical polymerization catalyst [spectrum (b)], only Na$_4$P$_2$O$_7$ as the esterification catalyst [spectrum (c)] and both K$_2$S$_2$O$_8$ and Na$_4$P$_2$O$_7$ catalysts together [spectrum (d)] are shown in Fig. 3. A broad absorption band over 3500-3000 cm$^{-1}$, characteristic of hydrogen bonded O-H stretching vibration, is found to be common in all the four spectra. Two notable absorption bands in the regions 1558-1572 cm$^{-1}$ and at 1742 cm$^{-1}$ appearing in different intensities are characteristics of carboxylate (anion) and ester stretching vibrations respectively. The MAA finish of jute in the presence of K$_2$S$_2$O$_8$ [Fig. 3(b)] results in the appearance of a new weak absorption band at 1788 cm$^{-1}$, characteristic of vinyl ester stretching and an intensification of the absorption band at 1572 cm$^{-1}$ for the carboxylate anion. MAA finish in the presence of esterification catalyst (Na$_4$P$_2$O$_7$) only, however, results in an intensification of the absorption band at 1788 cm$^{-1}$ for vinyl ester stretching consequent to incorporation of MAA moieties in the fibre by esterification; weak and medium absorption bands at 1742 cm$^{-1}$ and 1558 cm$^{-1}$ for ester stretching and carboxylate anion stretching respectively appear in the said spectrum. However, the MAA finish on jute under the dual catalysts (K$_2$S$_2$O$_8$ + Na$_4$P$_2$O$_7$) system expectedly results in substantial weakening of 1788 cm$^{-1}$ (vinyl ester) band with sharp intensification of the band at 1742 cm$^{-1}$ (due to ester stretching) and good retention of the band corresponding to 1558 cm$^{-1}$ for carboxylate (anion) stretching. The results of the IR analysis are in tune with the mechanism proposed.

4 Conclusions

4.1 Methacrylic acid finish on scoured and bleached jute fabric renders the fabric suited for many
diverse applications, such as furnishing, upholstery, draperies, soft luggage and blended textiles for apparels, in view of certain property advantages imparted. The major property advantages that can be derived from MAA (100g/L) finishes by following a pad-batch-dry-cure process under the catalytic influence of $K_2S_2O_8$ (5 g/L) and $Na_4P_2O_7$ (150 g/L) are substantial improvements in the wrinkle recovery angle from 144° to 234°, moisture regain from 16.87% to 19.21%, light fastness rating from 1 to 3 of dyed jute with notable improvement in basic dye uptake, surface gloss or reflectance, flexibility, whiteness index, abrasion resistance and resistance to photo-yellowing. The MAA finished jute fabric also shows nearly 80% retention of tenacity with substantial gain in elongation-at-break and much subdued fibre shedding character.

4.2 Mechanistic considerations, IR spectroscopy and specular reflectance data indicate that changes or improvements in properties are the direct consequences of different degrees of esterification of jute cellulose, grafting of film forming poly (methacrylic acid) onto the jute cellulose substrate and notable crosslinking of cellulosic chain molecules of jute fibre through long segments of the poly (methacrylic acid) formed during pad-batch-dry-cure process.

**Industrial Importance:** The above process of MAA modification appears to have practical importance for the industry aiming at producing jute and/or jute blended upholstery and furnishing products with improved property profiles, following formaldehyde – free route as mentioned above.

**References**