Moisture sorption characteristics of jute crosslinked with dimethylol dihydroxyethylene urea

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The moisture sorption characteristics of jute crosslinked with dimethylol dihydroxyethylene urea (DMDHEU) using metal salt catalysts (MgCl₂ and ZnCl₂) and acid catalyst (acetic acid) by the pad-dry-cure method have been assessed. Crosslinking treatment reduces the hygroscopicity of jute fibre at any level of relative humidity. The concentrations of DMDHEU (4-12%) and catalyst and the curing conditions (2-10 min) also affect the moisture sorption characteristics of crosslinked jute. Preswelling treatment with sodium hydroxide (16.5% w/w) prior to crosslinking further reduces the hygroscopicity of jute fibre.

Keywords: Crosslinking, Dimethylol dihydroxyethylene urea, Jute fibre, Moisture sorption characteristics

1 Introduction

When vegetable textile fibres are kept exposed to any specific humidity, they exchange moisture with the surrounding atmosphere by adsorption and desorption until equilibrium is reached. The origin of the hygroscopicity of fibre may be traced to the hydrophilic nature of its constituent molecules. In pure cellulose, the hydrophilic groups are mainly the hydroxyls and in jute the constituents have hydroxyl and phenolic groups. The constituent molecules are held together through different types of bonds. Water molecules cause swelling of cellulose¹ by breaking the intra- and inter-molecular hydrogen bonds between the polymer molecules and form hydrogen bonds with them thus pushing apart the adjacent chains. Many reports²-⁷ are available on the mechanism and determination of moisture sorption of cotton fibre. The first systematic study on the relationship of moisture uptake and nature of raw jute was made by Powrie and Speakman⁸. Later, Macmillan et al.⁹ studied the moisture adsorption and heat of adsorption of raw jute fibre and found that jute is also a highly hygroscopic fibre.

The sorption characteristics of crosslinked cellulose fibre were studied by several workers¹⁰-¹⁶ who observed that crosslinking treatment affects the moisture regain and other properties. Gill and Steele¹⁴ observed that dry curing of cotton and rayon fabrics reduces the moisture sorption in cotton whereas wet curing increases the sorption of moisture in both the cases. Singh et al.¹⁵ found that crosslinking treatment reduces the moisture content of cellulosic fibre. Honold and Grant¹⁶ observed that in pad-dry-cure process, the moisture regain of cotton is reduced due to the crosslinking treatment and it becomes progressively less as the nitrogen content is increased, whereas in wet fix process, a final increasing trend is observed for cotton having more than 1.4% nitrogen. Jute is a highly hygroscopic fibre and receptive to crosslinking treatment. In view of our earlier studies¹⁷, showing improved crease recovery properties of jute fabrics through crosslinking with a few resins in presence of catalysts, and studies¹⁸ on preswelled jute fabrics, it was felt worthwhile to study the moisture adsorption behaviour of jute fibre after crosslinking with DMDHEU resin in presence of different types of catalyst. This paper reports the results of these studies.

2 Materials and Methods

2.1 Materials

2.1.1 Fibre

Tossa-daise (TD₁-ISI standard) jute fibre, cleaned by extraction with alcohol-benzene solvent (1:1) in soxhlet for 4 h, was used.

2.1.2 Fabric

Plain weave 100% jute fabric having the following structural characteristics was used: warp—60 ends/dcm (count, 340 tex); weft—40 ends/dcm
The fabric was cleaned before use by scouring with 2% sodium carbonate (on fabric weight) at 80°C for 30 min. Bleaching was carried out in a laboratory jigger by alkaline hydrogen peroxide. This was followed by washing with water, treatment with acetic acid and finally with water.

2.1.3 Chemicals
Ahuramine YX, an aqueous product containing 51% solid 1,3-dimethylol-4,5 dihydroxyethylene urea was used as crosslinking agent. Magnesium chloride hexahydrate, zinc chloride and acetic acid, all of analytical grade, were used as catalysts.

2.2 Methods
The moisture adsorption properties of the following samples were studied:

1. Raw jute, tossa TD3 variety.
2. Jute fibre (sample No. 1) treated with 16.5% sodium hydroxide solution (w/w) for 30 min at 22°C followed by washing and neutralization with acetic acid and finally washed and air dried.
3. (a) Jute fibre obtained from jute fabric crosslinked by padding DMDHEU (12%) resin and magnesium chloride (3%) catalyst, dried at 80°C for 7 min, cured at 150°C for 5 min and washed.
   (b) Same as (a) without any catalyst.
   (c) Same as (a) using zinc chloride catalyst (2.4%).
   (d) Same as (a) using acetic acid catalyst (pH 2.5).
4. (a) Jute fibre obtained from jute fabric crosslinked by padding 2% DMDHEU resin and 0.5% magnesium chloride catalyst, dried and cured as in the case of (3(a)).
   (b) Same as (4(a)) without any catalyst.
5. (a) Jute fibre obtained from jute fabric crosslinked by padding 4% DMDHEU resin and 1% magnesium chloride catalyst, dried and cured as in the case of (3(a)).
   (b) Same as (5(a)) without any catalyst.
6. Jute fibre obtained from jute fabric crosslinked by padding 6% DMDHEU resin and 1.5% magnesium chloride catalyst, dried and cured as in the case of (3(a)).
7. (a) Jute fibre obtained from jute fabric crosslinked by padding 8% DMDHEU resin and 2% magnesium chloride catalyst, dried and cured as in the case of (3(a)).
   (b) Same as (7(a)) without any catalyst.
8. (a) Jute fibre obtained from jute fabric crosslinked by padding 10% DMDHEU resin and 2.5% magnesium chloride catalyst, dried and cured as in the case of (3(a)).
   (b) Same as (8(a)) without any catalyst.
9. Same as (3(a)) without any catalyst.
10. (a) Jute fibre obtained from jute fabric crosslinked by padding 12% DMDHEU resin and 3% magnesium chloride catalyst, dried at 80°C for 7 min, cured at 150°C for 2.5 min and washed.
    (b) Same as (10(a)) cured for 5 min.
    (c) Same as (10(a)) cured for 7.5 min.
    (d) Same as (10(a)) cured for 10 min.
11. (a) Jute fibre obtained from jute fabric crosslinked by padding 12% DMDHEU and 2.4% zinc chloride catalyst, dried at 80°C for 7 min, cured at 150°C for 2.5 min and washed.
    (b) Same as (11(a)) cured for 5 min.
    (c) Same as (11(a)) cured for 7 min.
    (d) Same as (11(a)) cured for 10 min.
12. (a) Sample No. 2 treated with 12% DMDHEU and 3% MgCl₂ catalyst, dried at 80°C for 7 min, cured at 150°C for 5 min and washed.
    (b) Same as (12(a)) using acetic acid catalyst (pH 2.5).

2.2.1 Measurement of Moisture Regain
Jute fibres from each of the crosslinked fabric samples were collected by taking out the yarns and then untwisting them to separate the single fibre strands. About 1 g of each of the above mentioned samples was placed separately in a previously weighed bottle. The bottles were kept in an oven and heated at 105°C until a constant weight was registered, indicating that there is no moisture in the samples. The oven-dry samples were then kept over distilled water in a desiccator for a week. The bottles were weighed at intervals of few days until constant weights were obtained, indicating the attainment of equilibrium. The samples in the bottles were then conditioned at 35% (saturated solution of potassium chromate), 65% (saturated solution of sodium nitrite), 50% (saturated solution of magnesium chloride) relative humidity and the equilibrium weights of all the fibre samples were recorded.

The moisture regain values of the fibre samples were then calculated as follows

\[
\% \text{ Moisture regain} = \frac{\text{Equilibrium weight of fibre} - \text{oven-dry weight}}{\text{Oven-dry weight}} \times 100
\]

The percentage of nitrogen in the untreated and crosslinked jute was determined by the Kjeldahl method.
3 Results and Discussion

Jute differs from cotton in chemical composition. While cotton fibre is purely cellulosic, jute contains cellulose (62-64%), hemicellulose (21-23%) and lignin (12-14%) as major constituents.

Water adsorption properties of the jute, preswelled jute fibre, lignin and jute cellulose at different relative humidities are shown in Fig. 1. The figure shows that hygroscopicity increases as the relative humidity increases. The swelling treatment of jute fibre with caustic soda reduces the hygroscopicity—a phenomenon opposite to that encountered with cotton, which shows a considerable increase in hygroscopicity after the preswelling treatment by caustic soda. Mukherjee et al. observed that extracted jute cellulose shows an increase in hygroscopicity like cotton (though less) after caustic soda treatment. This difference in hygroscopicity is attributed to the removal of hemicellulose by caustic soda treatment.

Water adsorption properties of crosslinked jute fibre using different types of catalyst are shown in Table 1 and Fig. 2, which give an insight to the accessibility character of the crosslinked fibre. Swelling of the jute structure is inhibited by crosslinking treatment. The moisture adsorption characteristics of jute fibre, however, did not show any significant difference due to the use of different catalysts. The moisture regain of jute after crosslinking treatment is lower in the case of magnesiuim chloride catalyst at all levels of relative humidity. This supports our earlier observation that MgCl₂ is the best catalyst (of those used) for crosslinking of jute with DMDHEU.

The extent of crosslinking of DMDHEU with jute fibre is reflected in the changes in moisture regain. The moisture regain values of samples treated with different concentrations of DMDHEU and MgCl₂ catalyst are shown in Table 2 and Fig. 3. The difference in crosslinking reaction with different concentrations of DMDHEU using magnesium chloride catalyst is indicated by bound nitrogen value. Table 1 shows that control fabric and fabric treated with DMDHEU without catalyst give almost identical water content at different levels of relative humidity. In our earlier studies it was found that there is very mild crosslinking reaction between DMDHEU and jute in absence of any catalyst. The accessibility of water molecules to the cellulose hydroxyls in the samples treated with DMDHEU without any catalyst does not reduce significantly.

Table 1—Effect of catalyst on moisture regain of crosslinked jute

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Catalyst</th>
<th>Moisture regain (%) at</th>
</tr>
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<tbody>
<tr>
<td></td>
<td></td>
<td>30% RH</td>
</tr>
<tr>
<td>Control</td>
<td>—</td>
<td>7.20</td>
</tr>
<tr>
<td></td>
<td>MgCl₂</td>
<td>6.95</td>
</tr>
<tr>
<td>12% DMDHEU</td>
<td>ZnCl₂</td>
<td>7.07</td>
</tr>
<tr>
<td></td>
<td>Acetic Acid</td>
<td>7.10</td>
</tr>
</tbody>
</table>
Table 2—Effect of DMDHEU concentration on moisture regain of crosslinked jute

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Catalyst</th>
<th>Moisture regain (%) at</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>30% RH</td>
</tr>
<tr>
<td>Control</td>
<td>-</td>
<td>7.20</td>
</tr>
<tr>
<td>2% DMDHEU</td>
<td>0.5% MgCl₂</td>
<td>7.18</td>
</tr>
<tr>
<td>4% DMDHEU</td>
<td>1% MgCl₂</td>
<td>7.26</td>
</tr>
<tr>
<td>6% DMDHEU</td>
<td>1.5% MgCl₂</td>
<td>7.15</td>
</tr>
<tr>
<td>8% DMDHEU</td>
<td>2% MgCl₂</td>
<td>7.05</td>
</tr>
<tr>
<td>10% DMDHEU</td>
<td>2.5% MgCl₂</td>
<td>6.95</td>
</tr>
<tr>
<td>12% DMDHEU</td>
<td>3% MgCl₂</td>
<td>6.95</td>
</tr>
</tbody>
</table>

Fig. 3—Relationship between moisture adsorption properties of jute fabric and DMDHEU add-on at different levels of relative humidity.

Fig. 3 shows that as the crosslinking reaction of DMDHEU with jute intensifies there is a gradual decrease in moisture content of the treated sample at different levels of relative humidity. Since the extent of crosslinking reaction is reflected on bound nitrogen values of the fabric, a relationship between bound nitrogen (of the crosslinked fabrics) and moisture content is expected. This relationship was actually obtained and is shown in Fig. 4, which shows the systematic decrease of moisture content with increase in bound nitrogen, the decrease being more pronounced at higher relative humidities.

Reduction in moisture regain of crosslinked jute fibre is also dependent upon the reaction conditions. Exposure of fibre in different curing conditions affects the moisture adsorption properties. The effect of curing time in crosslinking reaction between DMDHEU and jute fibre using metal salt catalyst on moisture adsorption property at different relative humidities is shown in Fig. 5.
The figure shows that except at 100% RH the moisture regain drops considerably at 2.5 min curing time and thereafter it levels off irrespective of further increase in curing time. In other words, maximum drop in moisture regain takes place within 2-5 min curing time.

Unlike cotton, jute fibre preswelled with caustic soda shows reduction in hygroscopicity due to the molecular rearrangement of fibre. The preswelled jute fibres crosslinked with DMDHEU using metal salt catalyst, such as magnesium chloride, and free acid catalyst, such as acetic acid, were assessed for moisture adsorption properties. The results are shown in Fig. 6. The hygroscopicity of mercerised cotton is not affected by the crosslinking treatment but the hygroscopicity of preswelled jute fibre is further reduced by the crosslinking treatment. The reduction in moisture regain is more in preswelled crosslinked jute fibre compared to crosslinked jute (untreated) fibre. This result also corroborates our earlier finding that the preswelled jute undergoes better crosslinking with DMDHEU than untreated jute.

It is expected that due to the preswelling treatment by sodium hydroxide, a considerable amount of non-crystalline hemicellulose is removed and so jute becomes less hygroscopic. When the swollen jute is crosslinked the empty places are partly occupied by the resin to make it even less hygroscopic than the unswelled jute.

4 Conclusion
Crosslinking treatment of jute with DMDHEU makes it less hygroscopic. Reduction in hygroscopicity increases with increase in crosslinking, i.e. with increase in DMDHEU content (bound nitrogen), curing time and curing temperature.

The alkali-swollen jute retains less moisture than unswelled jute. This may be due to the removal of large quantity of hemicellulose, which is non-crystalline and consequently more moisture absorbing component. Crosslinking of alkali-swollen jute makes it even less hygroscopic.

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