List of Publications:

*Research Journals*:

1. "Degumming of ramie: the role of the individual constituents of the gummy material"

2. "Kinetic Studies of ramie fiber degumming using caustic soda"

3. "Degumming of decorticated ramie: Effects of alkalis on gummy components vis-à-vis their properties"
   In *Journal of The Textile Institute*, In Press

4. "Characterization of degummed ramie fiber"
   In *Indian Journal of Fiber & Textile Research*, Communicated

*Paper Presented in Conference / Seminars*:

1. "Eco-friendly pretreatment processes on ramie fiber"
   At International Conference on "Emerging Trends in Polymers and Textiles"
   7–8 January 2005, I. I. T., Delhi, India (In Personal)

2. "Ramie: The Valued Fiber for Future"
   At National Conference on "Emerging Trends in Textile, Fiber & Apparel Engineering"
   18–19 March 2006, Berhampore, West Bengal (In Absentia)

*Research Papers to be Communicated*:

1. Studies of structure–property relationship of decorticated & degummed ramie fibers

2. Degumming of ramie: Effects of gum content on dyeing behavior
inetic studies of Ramie Fibre degumming using caustic soda

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Abstract
Non-cellulosic components of decorticated ramie were heterogeneous in nature. Kinetics of caustic soda degumming reaction was studied with conventional (weight loss) and spectroscopic methods. Such study manifests that caustic soda degumming could not remove non-cellulosic components uniformly from decorticated fibre. Rate of removal of gummy material was found to be faster compared to hemicelluloses and that of pectin was the least. Removal of various non-cellulosics impurities depend on the severity of process conditions.

roduction
ECORTICATED-ramie fibre contains high amount of gummy material (19-30%), but only 2-6% of residual is recommended for its good utility as textiles.1 Degumming of this fibre is well established commercially using caustic soda. Role of the main individual contents of gummy material on fibre characteristics and its degumming at 70°C was reported earlier. Kinetic studies of such degumming reaction at different temperatures, taking into consideration of overall gummy material as well as individual components was not available in to of extensive research. Such studies give an insight understanding about the rate and mechanism of degumming reaction incurred during and in-situ with gummy substances. The present investigation was aimed to study kinetics of caustic soda degumming reaction of ramie fibre and the role of each component played therein. This will facilitate to judge the progress of reaction at specific conditions.

Experimental
Materials
Decorticated ramie fibre (variety R-1449) was obtained from Ramie Research Station, ICAR, Sorbhog, Assam (India). Fibres were cut into small length (10 cm) and were used throughout the work. All the chemicals used were of laboratory grade reagent.

Degumming procedure
One gram of decorticated fibre was treated with 2% (w/v) caustic soda solution at three different tempera-
Chemical analysis of ramie fibre

Chemical analysis of the constituents of the decorticated fiber was carried out using the method as suggested by Turner and Doree.*9*

Determination of percentage weight loss

The percentage weight loss of different sample was determined by finding the difference in weight before and after degumming and always after conditioning as prescribed earlier.*18*

Determination of hemicellulose and pectin content

The amount of hemicellulose and pectin present in the degummed liquor was determined as per the standard spectroscopic method.*8*

Kinetic studies of degumming reaction

Kinetics of degumming reaction was studied in terms of rate curves from removal of three components, namely, gummy material (as one component taken from weight loss), hemicellulose and pectin (determined by spectral analysis). Further, the rate constant ($K_d$) of degumming reaction at three different temperatures was calculated using second order rate equation for three main components.*10* Activation energy was also determined using Arhenius equation for various components.*10*

Results and discussion

Chemical compositions and rate of degumming of decorticated ramie fibre

Decorticated ramie fibre (Variety R-1449) contained 27.27% (owf) gummy material and its composition was analyzed and reported in Table 1. Effectivity of degumming can be assessed by the removal of gummy material. A minimum amount of residual gum (2 to 6%) is recommended for good textile application. Therefore, degumming of the fibre was carried out with 2% (w/v) caustic soda solution using various process conditions. Degumming reaction rate with respect to overall gummy material (as one component), hemicellulose and pectin individually and separately were studied at different temperatures

as shown in Figures 1 to 3. From all these figures it was observed that the degumming was effective as the temperature increase vis-à-vis with time. Removal of overall gummy material was hardly affected beyond 70°C. The scenario was different when individual components, namely, hemicellulose and pectin were considered with respect to their extent of removal. At all the temperatures, initial removal of hemicellulose was quite high and progressively increased with severity of the process. Pectin removal was comparatively lower with respect to hemicellulose and gummy material at initial stage but suddenly increased at higher temperature. The exact mechanism of removal of different components was not well understood and the heterogeneous nature of the gummy material was thought to be responsible.

Half-time values of degumming reaction

Differential behavior of caustic soda reaction on various non-cellulosic components of gummy material can also be evaluated by their half-time values of degumming reaction (Table 2). Half time of various homogeneous reactions may reveal more comparative information about progress of reactions at specific conditions. Half time values of various components differ considerably. The half time values of caustic soda reaction of various components were higher at 50°C and decreased with increase of temperatures. Hydrolysis of pectin to half extent required more time at all temperatures compared to other components. Hemicellulose can easily be hydrolyzed and its removal was found higher i.e. next to overall gummy material. Pectin, the second major non-cellulosic compo-

<table>
<thead>
<tr>
<th>Name of the component</th>
<th>Percentage (owf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble component</td>
<td>5.29</td>
</tr>
<tr>
<td>Hemicellulose</td>
<td>15.93</td>
</tr>
<tr>
<td>Pectin</td>
<td>4.86</td>
</tr>
<tr>
<td>Lignin</td>
<td>0.79</td>
</tr>
<tr>
<td>Fats and Waxes</td>
<td>0.40</td>
</tr>
<tr>
<td>Cellulose</td>
<td>72.73</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Samples description</th>
<th>t&lt;sub&gt;1/2&lt;/sub&gt; (Min.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overall gummy material (Fig 1)</td>
<td>56</td>
</tr>
<tr>
<td>Hemicellulose (Fig 2)</td>
<td>93</td>
</tr>
<tr>
<td>Pectin (Fig 3)</td>
<td>500</td>
</tr>
</tbody>
</table>
nent present in gummy material hydrolyzed to lesser extent and required drastic process conditions.

Rate constant and activation energy values of degumming reaction of ramie fibre

Removal of various components of gummy material from decorticated fibre was found to be non-uniform and this was further observed by its rate constant and activation energy values. Rate constant of caustic soda hydrolysis of various components increased with temperature (Table 3). Rate constant for overall gummy material revealed that as the temperature of degumming reaction increases, rate of removal becomes faster, but above 70°C, it increases at lower extent. Hemicellulose removal constantly increased but its rate increased with temperature. Hydrolysis of pectin was slower at 50°C and its rate increased at 90°C. Rate constant of component 'a' i.e. overall gummy material was less than the summation of rate constants (K) of component 'b' i.e. hemicellulose and 'c' i.e. pectin but higher than the individual component at initial stage. As temperature increases, K of component 'a' was less than the summation of K of component 'b' and 'c' but also less than the individual component.

This indicates that at lower temperature hemicellulose with other non-cellulosic components (except pectin) hydrolyzed easily. At higher temperature hemicellulose and pectin, both get hydrolyzed to significant extent. This may be the reason for the lowering of K of 'a' with respect to their individual component together as well as individually at later stage i.e. under drastic conditions.

Activation energy of degumming reaction was also measured with respect to the three components i.e. a, b

Table 3: Rate constant values (K, min⁻¹) of caustic soda degumming reaction at different temperatures of ramie fibre

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Time (min.)</th>
<th>50°C</th>
<th>70°C</th>
<th>90°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>a x 10⁻²</td>
<td>b x 10⁻²</td>
<td>c x 10⁻²</td>
<td>a x 10⁻²</td>
</tr>
<tr>
<td>1</td>
<td>30</td>
<td>6.64</td>
<td>6.78</td>
<td>7.90</td>
</tr>
<tr>
<td>2</td>
<td>45</td>
<td>6.01</td>
<td>6.02</td>
<td>5.72</td>
</tr>
<tr>
<td>3</td>
<td>60</td>
<td>8.27</td>
<td>6.04</td>
<td>5.29</td>
</tr>
<tr>
<td>5</td>
<td>120</td>
<td>9.01</td>
<td>5.99</td>
<td>4.56</td>
</tr>
<tr>
<td>6</td>
<td>150</td>
<td>8.32</td>
<td>6.85</td>
<td>5.28</td>
</tr>
<tr>
<td>7</td>
<td>180</td>
<td>7.42</td>
<td>6.04</td>
<td>5.72</td>
</tr>
<tr>
<td>8</td>
<td>240</td>
<td>6.44</td>
<td>6.52</td>
<td>5.00</td>
</tr>
<tr>
<td>10</td>
<td>360</td>
<td>7.51</td>
<td>6.78</td>
<td>4.42</td>
</tr>
</tbody>
</table>

Average: 7.15 6.43 5.35 11.98 8.93 7.63 12.34 12.69 21.24

a = Gummy Material (Overall) b = Hemicellulose Removed c = Pectin Removed
and c and values were 9.57, 19.14 and 30.63 KJ respectively (ΔE was expressed in KJ as the mol.wt. of the impurities are unknown). Activation energy also confirmed slower removal of pectin in gummy material.

Hemicellulose, the major non-cellulosic component removed faster, next to overall gummy material. The heterogeneity of distribution and chemical reactivity of various components in gummy material in the fibre strand were responsible for their differential reaction to caustic soda. Non-cellulosic cementing material present in decorticated ramie was distributed unequally throughout the fibre strand. Major proportion of hemicellulose was found to be available on cell wall. Pectic substances are mainly present on walls of the soft cell surrounding the fibre bundles in the stem, which are in the interior layers. Hemicelluloses, which is present in the outer layer, easily accessible to caustic soda even at milder processing conditions. Removal of outer layer of gummy material opens space for reaction with pectin and results in higher extent of removal of this component at later stage.

Conclusions

Non-cellulosic components of decorticated ramie were heterogeneous in nature. Rate of removal of various components by caustic soda differs considerably. Half time values for degumming reaction of non-cellulosic components were 56, 43, and 38 minutes at 50, 70 and 90°C respectively. The same for hemicellulose were 93, 84 and 58 minutes and pectin were 500, 261 and 100 minutes at respected temperatures. At all level of degumming the removal of gummy material was higher than that of individual constituents and degumming rate increased with temperatures as expected. This was not so at higher extent of degumming (say 90°C). This is so because of the heterogeneous nature of the gummy material, where an individual constituent behaves differently. Activation energy value of overall gummy material was the lowest and followed by hemicellulose and pectin. Heterogeneous nature of the constituents of gummy material to caustic soda reaction was also confirmed from the rate studies.

References

Degumming of ramie: the role of the individual constituents of the gummy material

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The stepwise removal of the components of gummy material found on ramie fibres was used to determine the effects of the individual constituents on the whiteness and yellowness indices of the fibres. The main components were found to be hemicellulose, pectin and lignin and these had the greatest effect on the whiteness and yellowness indices. A spectroscopic method for assessing the hemicellulose and pectin content of the degumming bath was investigated and found to be comparable to the results of chemical analysis. The spectroscopic method was used to study the removal of hemicellulose and pectin continuously during degumming, and the data obtained were used to calculate rate constants for the degumming process.

Introduction

Ramie, the bast fibre, is obtained from the plant Boehmeria nivea after decortication. Different varieties contain different amounts (10-30%) of gummy material [1-5]. To make the fibre suitable for textile use the gum content should be reduced to ca. 2-6% in order to improve its properties, such as its strength on wetting, lustre, dyeing properties, fineness and anti-microbial properties [6-18]. The degumming of ramie fibre can be carried out by two methods, namely, chemical degumming using various alkalis, and microbial degumming [19-23]. Among these, caustic soda degumming is most widely practiced commercially and a large number of literature references are available pertaining to this method [6,10,24]. In these references, the performance of caustic soda degumming was mainly assessed with respect to fibre weight loss, changes in fibre fineness and physico-mechanical properties, along with other fibre properties. The chemical composition of the gummy material in ramie is also well established [25,26]. However, the role of the main individual components of the gummy material, hemicellulose and pectin, during caustic soda degumming has not been reported. In this paper degumming performance has been studied taking into consideration these two components separately. An attempt has also been made to study the degumming reaction rate using a weight loss method as well as by a novel technique utilizing spectroscopic analysis.

Experimental

Materials

Decorticated ramie fibre (variety R-1449) was obtained from the Ramie Research Station, Assam, India. Long strands of fibre were cut into small lengths (ca. 10 cm) and used throughout the work. Hemicellulose (xylan) was supplied by Loba Chemie (India) and pectin was obtained from S D Fine Chemicals Pvt. Ltd (India). All other chemicals used were of laboratory reagent grade.

Degumming of decorticated ramie

Samples of decorticated fibre (1 g) were degummed with caustic soda solutions of 2, 4 and 6% (w/v) at 70 ± 1 °C over a covered water bath for 2 h at a liquor-to-goods ratio of 50:1. After degumming the fibre was washed five times in distilled water, neutralised with acetic acid (0.5% v/v) and washed thoroughly with distilled water.

To study the rate of degumming, 1 g of decorticated fibre was degummed with 2% (w/v) caustic soda at 70 ± 1 °C for different periods (0.5, 0.75, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 and 6.0 h) at a liquor-to-goods ratio of 50:1. The samples were washed as described above. The degumming liquors were preserved for subsequent analysis of the individual components. The rate constant for the degumming reaction was calculated by determining the weight loss by a spectroscopic method.

Analysis

Chemical analysis

Chemical analysis of the constituents of the gummy material was carried out using the method of Turner and Doree [27].

Determination of percentage weight loss

The percentage weight loss of the degummed samples was determined by measuring the difference in weight of the samples before and after degumming. The samples were conditioned at 27 ± 2 °C and 65 ± 2% relative humidity (RH) [28].

Determination of Methylene Blue exhaustion

Ramie fibre samples (0.2 g) were treated with Methylene Blue (CT Basic Blue 9) dye solution (0.25% owf) with occasional stirring at room temperature for 18 h at pH 8-8.5 (adjusted with soda ash) at a liquor-to-goods ratio of 50:1. After the stipulated period, the fibre was squeezed, washed and dried. The dye exhaustion (%E) was calculated from the optical density of the dye solution by standard
spectroscopic methods at a wavelength of 660 nm using a Systronic (India) model 119 ultraviolet–visible (UV-vis) spectrophotometer.

**Determination of moisture regain**

Moisture regain values were determined by exposing fibre samples to 65% RH at 21 °C for five days. The samples were weighed in the conditioned state, then dried at 110 °C for 12 h in an oven and weighed again.

**Determination of whiteness and yellowness indices**

The Hunter whiteness and ASTM-313 yellowness indices of the samples were measured on a Premier Colour Scan System (India) Spectra Scan 5100 spectrophotometer by making a uniform web of parallel fibres.

**Determination of hemicellulose and pectin content by a spectroscopic method**

A spectroscopic method has been developed to determine the amount of hemicellulose in the bath after degumming. Initially, the optical density of hemicellulose of two different concentrations (0.5 and 1 g/l) was measured over the wavelength range 250–700 nm on UV-vis spectrophotometer and the wavelength of maximum absorption \( \lambda_{max} \) was determined. The \( \lambda_{max} \) was found to be at 340 nm for both the concentrations of hemicellulose and a calibration curve was plotted by measuring the optical density at different hemicellulose concentrations. This calibration curve was used to determine the hemicellulose content in the degumming bath. A similar procedure was adopted for pectin and the \( \lambda_{max} \) was found to be 410 nm. As the two components have distinct \( \lambda_{max} \) values, the content of each could be measured separately at the appropriate wavelength. The hemicellulose and pectin contents were expressed as percentages of the weight of the material.

**Determination of the rate constant for degumming**

The rate of the degumming reaction was determined from the weight loss after degumming with caustic soda, as described earlier. The order of the degumming reaction was determined mathematically and the reaction was found to be second order. From the weight loss values and using the second order rate equation, the rate constant of the degumming reaction \( K \) was calculated using Eqn 1 [29]:

\[
K = \frac{W_t}{t \times (W_0 - W_t)}
\]

where \( W_t \) is the weight of the fibre before the degumming reaction (i.e. at time \( t = 0 \)) and \( W_t \) is the weight of the degummed fibre after degumming for time \( t \).

Similarly, the rate constants of the degumming reaction for hemicellulose and pectin removal separately were also determined. The amounts of hemicellulose and pectin in the degumming bath were determined spectrophotometrically as described above and the rate constants were calculated using Eqn 1, with the weight of the hemicellulose or pectin on the fibre substituted for the weight of the fibre.

**Results and Discussion**

**Effect of degumming and caustic soda concentration on fibre properties**

The degumming of decorticated ramie fibre is required for better processability. A certain amount of residual gum (2–6%) is recommended for its use as a textile fibre [6]. The moisture regain of the degummed ramie was lower than that of the decorticated ramie. The values did not change drastically with increasing caustic soda concentration. Moisture regain has some bearing on processing of ramie fibre and it gives an indication of the removal of gummy material [7].

Methylene Blue exhaustion on the degummed fibre has been used as a tool to indicate the extent of degumming. Methylene Blue dye, being cationic, has no affinity for cellulose, therefore exhaustion of the dye onto the fibre indicates the presence of gummy materials, mainly pectin. The decrease in Methylene Blue exhaustion with increasing caustic soda concentration indicates that the extent of the removal of gummy material is greater at higher concentrations of caustic soda, as shown above by the weight loss results.

As the degree of degumming increased with an increase in the concentration of caustic soda, the whiteness of the fibre improved and the yellowness index decreased. This phenomenon has been observed before with cellulotic fibres [30]. The Hunter whiteness index increased from 38.3 before degumming to 52.43 after degumming, and similarly the yellowness index decreased from 52.03 before degumming to 46.80 after degumming, as shown in Table 1.

**Table 1** Effect of degumming caustic soda concentration on ramie fibre properties

<table>
<thead>
<tr>
<th>NaOH conc. (% w/v)</th>
<th>Weight loss (%)</th>
<th>Residual gum content (%)</th>
<th>Moisture regain (%)</th>
<th>%R</th>
<th>Whiteness index</th>
<th>Yellowness index</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 ( a )</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>23.80 ( b )</td>
<td>4.19</td>
<td>4.59</td>
<td>69.00</td>
<td>31.32</td>
<td>22.17</td>
</tr>
<tr>
<td>4</td>
<td>24.80 ( b )</td>
<td>3.48</td>
<td>4.08</td>
<td>53.20</td>
<td>31.72</td>
<td>22.87</td>
</tr>
<tr>
<td>6</td>
<td>25.96 ( b )</td>
<td>1.31</td>
<td>4.03</td>
<td>46.80</td>
<td>32.43</td>
<td>20.72</td>
</tr>
</tbody>
</table>

\( a \) Degumming conditions: temperature, 70 °C; time, 2 h; liquor ratio 50:1

\( b \) Data obtained from raw decorticated ramie fibre
to 20.72 after; but the change in whiteness and yellowness with increasing caustic soda concentration was very small. This indicates that there is a limit to how much the whiteness or yellowness can be improved by degumming using caustic soda. Although the whiteness index did not improve as the degumming conditions became more harsh, a sufficient degree of whiteness was obtained with a soft feel to the fibres.

Effect of individual components of gummy material on Methylene Blue exhaustion, whiteness and yellowness

Although the Methylene Blue exhaustion values shown in Table 1 indicate the extent of degumming they do not show the role of the two main gummy materials, hemicellulose and pectin, during the degumming process. Consequently, each component of the gummy material was removed individually using the method of Turner and Doree [27]. The effect of each component on the Methylene Blue exhaustion, whiteness index and yellowness index is shown in Table 2.

The largest decrease in Methylene Blue exhaustion on the decorticated ramie was seen after the removal of pectin and hemicellulose and after removal of the lignin. Pectin and hemicellulose are both polysaccharides but the chief constituent of pectin is polygalacturonic acid, which contains many carboxy groups. Hemicellulose is similar in structure to cellulose, but without projecting methylol groups on the C5 atoms. Therefore, the Methylene Blue exhaustion mainly indicated the pectin content of the gummy material. Lignin, being an amorphous polymeric material, has no reactivity with Methylene Blue. The very low exhaustion of Methylene Blue after the removal of lignin is therefore an indication of the character of the fibre becoming more cellulosic, i.e. indicating the removal of nearly all the gummy material. As the stepwise removal of the different components of the gummy material proceeded the whiteness index value increased and reached a maximum (73.08) after the removal of lignin. Reasonably good whiteness was obtained when pectin (43.82) and hemicellulose (55.49) were removed. Similarly, the yellowness index of the samples decreased as the components were removed, reaching a minimum after the removal of lignin.

Composition of the gummy material

It has been observed that the individual components of the gummy material have a great influence on the characteristics of the fibre. The relevant amounts of the constituents of the gummy material on the decorticated fibre and samples degummed with 2, 4 or 6% (w/v) of caustic soda were determined and are shown in Table 3. The largest constituent of the gummy material was hemicellulose (15.93%), followed by pectin (4.86%), lignin (0.79%) and fats and waxes (0.40%). It can be seen that the removal of the gummy material was not uniform, e.g. after degumming with 2% w/v caustic soda, the amount of pectin removed was ca. 37%, the amount of hemicellulose removed was ca. 91% while the removal of the other components was greater than 95%. The percentage of pectin removed during degumming increased with

### Table 2: Influence of individual constituents of gummy material on ramie properties

<table>
<thead>
<tr>
<th>Sample description</th>
<th>Whiteness index</th>
<th>Yellowness index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Decorticated ramie</td>
<td>78.20</td>
<td>38.30</td>
</tr>
<tr>
<td>WSC removed</td>
<td>77.80</td>
<td>39.20</td>
</tr>
<tr>
<td>WSC and F&amp;W removed</td>
<td>77.40</td>
<td>42.06</td>
</tr>
<tr>
<td>WSC, F&amp;W and pectin removed</td>
<td>49.00</td>
<td>43.82</td>
</tr>
<tr>
<td>WSC, F&amp;W, pectin and HC removed</td>
<td>45.60</td>
<td>55.49</td>
</tr>
<tr>
<td>WSC, F&amp;W, pectin, HC and lignin removed</td>
<td>45.00</td>
<td>73.08</td>
</tr>
</tbody>
</table>

- WSC = water soluble component
- F&W = fats and waxes
- HC = hemicellulose

### Table 3: Composition of decorticated and degummed ramie fibres

<table>
<thead>
<tr>
<th>Component (%)</th>
<th>Ramie fibre</th>
<th>WSC&lt;sup&gt;a&lt;/sup&gt;</th>
<th>F&amp;W&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Pectin</th>
<th>HC&lt;sup&gt;c&lt;/sup&gt;</th>
<th>Lignin</th>
<th>Cellulose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Decorticated&lt;sup&gt;d&lt;/sup&gt;</td>
<td>5.28 (6.1)</td>
<td>0.40 (0.3)</td>
<td>4.66 (2.1)</td>
<td>15.93 (14.4)</td>
<td>0.79 (0.7)</td>
<td>72.73 (79.2)</td>
<td></td>
</tr>
<tr>
<td>Degummed, 2% NaOH&lt;sup&gt;c&lt;/sup&gt;</td>
<td>0.10</td>
<td>0.02</td>
<td>3.13</td>
<td>1.50</td>
<td>0.03</td>
<td>95.30</td>
<td></td>
</tr>
<tr>
<td>Degummed, 4% NaOH&lt;sup&gt;c&lt;/sup&gt;</td>
<td>0.04</td>
<td>0.01</td>
<td>1.52</td>
<td>1.39</td>
<td>0.01</td>
<td>97.03</td>
<td></td>
</tr>
<tr>
<td>Degummed, 6% NaOH&lt;sup&gt;c&lt;/sup&gt;</td>
<td>0.01</td>
<td>0.01</td>
<td>0.62</td>
<td>1.10</td>
<td>0.01</td>
<td>98.05</td>
<td></td>
</tr>
</tbody>
</table>

- WSC = water soluble component, F&W = fats and waxes, HC = hemicellulose
- Values in parentheses are typical values [25]
- Caustic soda concentrations in w/v

increasing caustic soda concentration, while the removal of hemicellulose was largely unaffected. Following degumming the fibres became more cellulosic in nature, with a cellulose content of ca. 95–98%, compared to a cellulose content of ca. 72% for the raw decorticated fibre.

The distribution of the different constituents of the gummy material in the ramie fibre is not well defined. It has been reported that lignin, an amorphous polymeric material, is present in the space between the cells and enveloping the cellulosic strands [31]. After degumming with 2% w/v caustic soda most of the lignin was removed and the strands of the fibres loosened. Hemicellulose was present in varying amounts in different parts of the ramie fibre. Hemicellulose in the cell wall was found to be less resistant to acids and alkalis and can be removed easily during degumming. Hemicellulose present in the core of the fibre may not be hydrolysed as easily. The exact amount of hemicellulose present in these two positions is not well defined, but this might explain why after degumming with 2% w/v caustic soda 91% of the hemicellulose was removed, but after degumming with 6% w/v caustic soda 93% was removed. It has been reported that pectic substances present in ramie fibres are heterogeneous in nature and mainly present in the walls of the soft cells surrounding the ramie bundles in the stem and in the walls of the cells of the fibre. The pectic substances were found mainly in the interior layers of the cell wall and this may be the reason that the percentage of pectin removed was lower than that of the other components [32].

Spectroscopic analysis of the degumming bath

To gain an understanding of the roles of hemicellulose and pectin during degumming, an attempt was made to monitor this process continuously by spectroscopic analysis. Spectroscopic analysis is used for the quantitative analysis of various hydrocarbons, vitamins, steroids and other compounds [33]. The results obtained by spectroscopic analysis are shown in Table 4.

Table 4 Spectral analysis of the degumming liquor

<table>
<thead>
<tr>
<th>NaOH conc. (NaOH%)</th>
<th>Hemicellulose (%)</th>
<th>Pectin (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMa</td>
<td>CA6</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>10.89</td>
<td>14.43</td>
</tr>
<tr>
<td>4</td>
<td>15.00</td>
<td>14.54</td>
</tr>
<tr>
<td>6</td>
<td>16.30</td>
<td>14.83</td>
</tr>
<tr>
<td>Average</td>
<td>11.96</td>
<td>8.93</td>
</tr>
</tbody>
</table>

a SM = spectroscopic method
b CA = chemical analysis, results calculated from data in Table 3

c The results obtained with spectral analysis were in proximity (in the range of 80–95%) of the results obtained by chemical analysis. Therefore, the spectroscopic method was considered viable for determination of the levels of these two components in the degumming liquor, in order to study the progress of the degumming reaction.

Determination of the degumming reaction rate

Ramie fibre was degummed for varying periods of time, using 2% w/v caustic soda. The hemicellulose and pectin content of the degumming bath was determined by spectroscopic analysis, and the total weight loss of the fibres was calculated. The rate of the degumming reaction, with respect to the total weight loss and the removal of hemicellulose and pectin individually was calculated using Eqn 1. The results are shown in Table 5.

The results show that the rate for the overall removal of the gummy material was greater than the rates for hemicellulose and pectin when considered separately. No clear indication about the individual rate constants for hemicellulose and pectin was obtained because their values were almost the same. However, the initial hemicellulose content is ca. four times that of the pectin and this may have an impact on the rate of the overall degumming reaction.

Table 5 Rate constants for the degumming of ramie fibre with 2% w/v caustic soda

<table>
<thead>
<tr>
<th>Conc. in degumming bath (% owf)</th>
<th>Rate constant, K (mm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Hemicellulose</td>
</tr>
<tr>
<td>Time [h]</td>
<td>Weight loss x 10⁻²</td>
</tr>
<tr>
<td>0.50</td>
<td>11.10</td>
</tr>
<tr>
<td>0.75</td>
<td>14.65</td>
</tr>
<tr>
<td>1.00</td>
<td>17.30</td>
</tr>
<tr>
<td>1.50</td>
<td>20.32</td>
</tr>
<tr>
<td>2.00</td>
<td>23.00</td>
</tr>
<tr>
<td>2.50</td>
<td>25.29</td>
</tr>
<tr>
<td>3.00</td>
<td>25.51</td>
</tr>
<tr>
<td>3.50</td>
<td>25.65</td>
</tr>
<tr>
<td>4.00</td>
<td>25.85</td>
</tr>
<tr>
<td>4.50</td>
<td>24.41</td>
</tr>
<tr>
<td>Average</td>
<td>11.96</td>
</tr>
</tbody>
</table>

a Degumming temperature, 70 °C; liquor ratio, 50.1
b HEC = hemicellulose

degumming reaction. This is supported by the fact that the rate constant for hemicellulose and pectin when considered together was $4.92 \times 10^{-2} \text{ min}^{-1}$. From this it may be safely said that though these two components governed the degumming reaction effectively, their heterogeneous nature in the gummy material made the overall degumming rate much faster. Beyond this no other inferences can be made at this stage, but work in this area is in progress.

Conclusions

Compared with the decorticated (un-degummed) fibre, the degummed ramie fibre exhibited weight loss, a decrease in moisture regain, a decrease in Methylene Blue exhaustion, an increase in the whiteness index value and a decrease in the yellowness index value. Increasing the caustic fibre concentration in the degumming process caused a further increase in the fibre weight loss and a further decrease in the Methylene Blue exhaustion, indicating that Methylene Blue can be used as a tool to measure the extent of degumming. Increasing the caustic soda concentration had little effect on the moisture regain, the whiteness index value and the yellowness index value. This shows that there is a limit to how much the whiteness can be improved, and the yellowness decreased, by the degumming process.

The roles of the individual constituents of the gummy material on the whiteness index, yellowness index and Methylene Blue exhaustion were studied by the stepwise removal of each constituent. It was observed that each constituent had an influence on the degumming performance, but the removal of pectin, hemicellulose and lignin components had the greatest impact on the whiteness and yellowness indices. The removal of pectin and hemicellulose during degumming was measured by spectroscopic analysis. This technique was quite comparable with the standard chemical analysis process [27]. The spectroscopic technique, together with the conventional weight loss method, was utilised to study the rate of the degumming reaction.

References

32. H I Parsona, Rev. Text. Prog., 83 (1956) 44.
Dear shailish,

I am pleased to tell you that your work has now been accepted for publication in The Journal of the Textile Institute.

It was accepted on 13/07/2006

Comments from Reviewers and me can be found below.

Thank you for submitting your work to this journal.

Kind Regards

David P. Buchanan
Editor-in-Chief
The Journal of the Textile Institute

Comments from the Editor and Reviewer: