Brightening of grey jute—Mechanism and application

S K Chakrabarti, B S Ghosh, D Adhikari & A B Kundu
Indian Jute Industries' Research Association, 17 Taratola Road, Calcutta 700 088, India
Received 21 April 1994; accepted 22 September 1994

Ferric iron in combination with phenolic groups of tannin and phenolic acid lignin imparts grey colour in jute. Treatment with EDTA removed almost all the Fe(III), improving the brightness significantly but caused over-yellowing of fibres. Evidences presented suggest that the removal of metal ions bound with phenolic groups generated free phenolic—OH which on aerial oxidation caused the over-yellowing. Reducing action of dithionite minimized such oxidation and prevented over-yellowing. Besides, dithionite reduced Fe(II) to soluble Fe(II), thereby enhanced the brightening process. Thus, the combined action of EDTA and dithionite induced a high brightness and restored the natural yellow colour of jute. Brightening of jute under submerged condition proved uneconomical due to the expenses of post-treatment drying. Treatment at a low level of moisture eliminated the above. This was done by spraying brightening chemicals on fibres before piling. The prolonged piling time and inherent acidity of fibres favoured the brightening reactions. The resultant fibres were brightened appreciably and the yarn spun thereof was found to be superior in quality compared to control.

Keywords: Diaminocyclohexane tetraacetic acid, Dithionite, Ethylene diamine tetraacetic acid, Jute fibre

1 Introduction

The fibre of jute (Chorchorus olitorius) assumes golden yellow colour if retted in slow flowing clean water. Lack of such ideal retting facilities forces the farmer to steep green stems of jute in stagnant, muddy and shallow water for retting. The resultant fibres often turn dull grey which degrades their quality and value. Indeed, the bulk of our national produce lacks the desired colour and brightness due to such faulty retting. The grey fibres thus generated need treatment for brightening before being processed with well-retted bright fibres.

Earlier work has shown that insoluble complexes of iron and tannin, encrusting the fibre surfaces, are responsible for grey colour in jute. Suggested applications of phosphate, oxalate or mineral acids for removal of grey colour were discarded because all these agents deteriorate other properties of fibres including the colour and strength. We have shown earlier that treating jute with ethylene diamine tetraacetic acid (EDTA) eliminates the above difficulties, improving the brightness of grey fibres significantly but not to the level of well-retted bright jute. It appears that EDTA along with a reducing agent, a recipe commonly used in the brightening of paper pulps, would be more effective than EDTA alone for removing grey colour of jute. The present investigation defines such a formulation for brightening jute and elucidates the functions of both the sequestrant and the reducing agent in the brightening process. A brightening formulation suited for jute industry is proposed and its advantages in terms of fibre and yarn quality improvements are discussed.

2 Materials and Methods

2.1 Materials

EDTA and diaminocyclohexane tetraacetic acid (CDTA), products of Sigma Chemical Co, USA, were used. Grey jute and well-retted bright jute, graded as TD4 and TD2 respectively, were used throughout the experiments.

2.2 Brightening Treatments

Brightening treatments were carried out under optimized conditions by steeping fibres (100 g, 20 cm length) in a solution (500 ml) containing 0.1% EDTA and 0.15% sodium dithionite for 30 min at 60°C and at pH 5.5. After the treatment, the fibres were washed with demineralized hot water and then dried under vacuum over fused calcium chloride. Although slightly higher brightness was achieved under alkaline condition (pH 8.0), we preferred acidic pH, which is automatically attained due to acidity of jute, to ensure faster reaction and to cut down the cost of alkali which would otherwise be required to neutralize the acidity of jute in large-scale application.
Brightening treatments in mills (New Central Jute Mills, Budge Budge, and Wellington Jute Mills, Rishra) were carried out on 1 tonne of fibre (TD4) by spraying the requisite amounts of brightening chemicals (EDTA and dithionite), mixed in oil emulsion, during the usual passage of fibres through softener machine. The fibres released from softener contained 45% moisture, 4% oil and brightening chemicals. These were then piled under a cover for 20 h and finally spun into yarns in a flyer spinning system.

An aerobic brightening treatment was carried out in an evacuated jar containing aqueous alkaline pyrogallol as the scavenger of residual oxygen.

2.3 Iron Estimation
Iron contents in fibres were estimated by the method given in ATCC monograph. Both acid-extractable iron (AEI) and total iron contents were determined. The acid extract was prepared by treating 2 g of fibres with warm (60°C) hydrochloric acid (10 ml, 0.1 N) for 2 h in N₂ environment. To 5 ml of diluted extract were added in sequence the freshly prepared solutions of KMnO₄ (0.05 ml, 0.1 N), HNO₃ (1 ml, 0.1 N) and KSCN (1.5 ml, 1.5 N) with stirring and the pink-red colour developed was read at 490 nm. This gave the estimate of total AEI containing both Fe(II) and Fe(III). The Fe(III) fraction present in AEI was estimated directly by adding KSCN without prior oxidation. The Fe(II) was then calculated by subtracting Fe(III) content from total AEI. The total iron content of fibres, comprising soluble and insoluble fractions, was estimated by extracting ashed fibres (5 g) in HCl and then following the above method.

2.4 Determination of Phenolic Groups and Acidity
Free phenolic hydroxyl groups were estimated by the method of Yang et al. and as adopted in jute by Kundu et al. Acidity and tannin contents in jute fibres were determined by the methods of Das et al. and Ghosh et al. respectively.

2.5 Spectral Characteristics of Fibres
The spectral characteristics of fibres were examined by a computer-aided spectrophotometer (Match Scan II, Milton-Ray, USA) using D65 illuminant. Brightness and yellowness index were measured following ASTM standards E313-73 and E308-90 respectively.

2.6 Fibre Testing
The physical properties of fibres and yarns were tested following the standard methods.

3 Results and Discussion
3.1 Mechanism of Brightening
Early work has shown that insoluble complexes of iron and tannin, encrusting the surfaces of fibres, impart grey colour in jute. Nodder et al. indicated that out of the two ionic forms, the ferric iron is responsible for grey colour since it can only combine with tannins to form insoluble dark complexes. Sengupta et al., on the other hand, showed that ferric iron in combination with phenolic acid lignin contributes to the darkening of jute. Brightening of grey jute is, therefore, related to the changes in either tannin or iron contents. To identify the relationship, we determined the differences in iron and tannin contents after treating the grey jute with efficient brightening agents like EDTA and dithionite; such estimates along with the spectral characteristics of raw and brightened samples are given in Table I. The results show that grey jute contains a large amount (241 ppm) of acid-extractable iron (AEI), out of which 43% is present as Fe(III) and 57% as Fe(II). Treatment with EDTA resulted in 53.2% loss in AEI and 11.9 points gain in brightness. Analysis of residual iron (113 ppm) in EDTA-treated sample shows that it contains mainly Fe(II) (98.8%) and a negligible amount (1.2%) of Fe(III). Therefore, it can be inferred that EDTA predominantly chelated Fe(III) and its removal enhanced the brightness. Similarly, treatment with dithionite also removed the Fe(III), probably by reducing them into soluble Fe(II), and induced a high gain (10.1 points) in brightness. To identify the exact function of dithionite, grey jute was treated first with EDTA to ensure the removal of all Fe(III) and then with dithionite to see whether it could remove the residual Fe(II). The results show that residual Fe(II) was retained, indicating that dithionite acted only on Fe(III). In the above treatment when dithionite preceded EDTA, almost all Fe(II) and Fe(III) were removed, indicating that EDTA can chelate the residual Fe(II) in jute. Combined (at a time) application of EDTA and dithionite is therefore expected to remove iron more exhaustively than the individual treatment. Indeed, we have found so and achieved a high brightness (48.3) which is even higher by 4.1 points than the brightness (44.2) of TD2 jute. Such removal of iron resulted in a large portion of tannins to be dissolved since these two components remain as insoluble complexes in grey jute. Exhaustive removal of both iron (98%) and tannin (84%), as achieved by combined application, is desirable because residues, if present in large amounts, may recombine and darken the bright samples. Further, the results show that the combined formulation can be used for improving the brightness (by 4.3 points) of TD2 jute as well. It
may be mentioned here that AEI, which represents 68% of total iron in grey jute, is responsible for darkening of fibres. The acid-insoluble fraction which was found to be present almost in same proportion (about 120-140 ppm) in both grey (TD4) and bright (TD2) fibres probably do not contribute in the darkening process.

Table 1 shows that when a potent sequestrant, CDTA, rendered the grey jute very bright (Brightness, 47.8) and almost free from iron, a subsequent treatment with dithionite further increased the brightness by about 1.8 points. This shows that additional brightness induced by dithionite is not related to the removal of iron. It seems that dithionite increased the brightness either by preventing the oxidation of phenolic hydroxyl groups generated de novo, or by reducing the existing quinonoids of lignin into phenol. If the hypothesis is true, both the reactions should increase phenolic groups and, consequently, the acidity of jute. To verify this, we determined the phenolic groups and acidity in raw and treated jute. The phenolic groups were measured by an enzymatic method described earlier. The method involves assay of the enzyme peroxidase bound with free phenolic hydroxyl groups of fibres, thereby giving a relative estimate of the latter. The results in Table 2 show that the sample treated in the absence of air contains more phenolic groups (by 7%) and exhibits higher acidity (by 0.4 mol/100 g) than the aerobically-treated sample. Moreover, anaerobic treatment induced slightly higher brightness (by 0.9 point). This proves free phenolic groups undergo aerial oxidation and possibly form quinonoids colour structures, which reduced the brightness. Evidently, such phenolic groups were denied oxidation by removing air. It is believed that the reducing action of dithionite serves this purpose, minimizing the formation of chromophoric quinonoids. It is, however, not clear from above experiments whether dithionite could reduce the existing quinonoids of lignin into phenol. Such a possibility exists because a number of studies have clearly shown that quinonoid colour structures are reduced by the action of dithionite.
3.2 Colour of Jute

The depth of yellow shade in brightened grey jute was found to vary depending on the kind of treatment given. The choice of brightening agent, therefore, depends on how efficiently it restores the natural yellow colour of jute. This has been judged by comparing the yellowness of treated fibres with that of TD2 jute. The results (Table 1) show while the treatment with EDTA or CDTA rendered the grey fibres more yellow (YI, 40.2 and 40.7 respectively), the dithionite turned it less yellow (YI, 37.7) compared to TD2 jute (YI, 38.4). On the other hand, yellowness achieved by the combined application of sequestrant and dithionite is similar to that of TD2 jute. Thus, the combined formulation is proved most efficient in restoring natural yellow colour. Since CDTA is very expensive, we preferred to use EDTA in the subsequent experiments, particularly for brightening trials conducted in mills.

The differences in colour shade are more clearly revealed by comparing the colour of fibres in CIELAB colour space. Fig. 1 shows the relative positions of treated samples in the colour coordinate having grey jute at its origin. It can be seen that EDTA-treated sample is not only more yellow but also more reddish in colour compared to others. The reddish shade, in all probability, was induced by the oxidation of free phenolic groups into quinonoid colour structures. Evidently, the reddish shade faded on addition of dithionite because it protected the free phenolics from oxidation.

The difference in the depth of colour shades can also be revealed from the relative positions of samples in Lightness-Darkness scale shown in Fig. 1. Clearly, the colour achieved by treating with combined formulation is lighter in shade than the others. The light yellow shade so achieved not only resembles the colour of TD2 jute but also has an attractive aesthetic appeal.

3.3 Practical Application

Any method, however efficient in improving quality of jute, has to be inexpensive and must fit in the existing processing system so that the cost of products remains competitive. Our method of brightening jute is efficient but involves the undesired expenses of post treatment drying. Hence, we explored and located a situation within the normal processing chain that would accommodate the brightening treatment without additional cost. Spraying of brightening solution, containing EDTA and dithionite, on raw fibres during its passage through softener machine proved most feasible and convenient. Since the fibres released from softener are piled for hours (20 h) the chemicals get time to react. And because the microbial growth in a pile depletes oxygen and generates heat, creating a heated (55-65°C) anaerobic environment, the reactions of brightening are favoured. By this, a substantial gain in brightness was achieved in spite of the fact that an uneven spray and an inadequate moisture in pile restricted the reactions. The results given in Table 3 show that such treatment not only improved the brightness (by 6.3 points) but also increased the fineness of fibres without deteriorating the strength. When such treated and control fibres were spun, the quality of treated yarn was found to be improved (Table 4) compared to control. The high tenacity and reduced

<table>
<thead>
<tr>
<th>Property</th>
<th>Raw jute</th>
<th>Brightened jute</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linear density, tex</td>
<td>2.2</td>
<td>1.9</td>
</tr>
<tr>
<td>Bundle tenacity, mN/tex</td>
<td>160</td>
<td>162</td>
</tr>
<tr>
<td>Brightness</td>
<td>33.5</td>
<td>39.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Yarns from</th>
</tr>
</thead>
<tbody>
<tr>
<td>Property</td>
</tr>
<tr>
<td>Linear density, tex</td>
</tr>
<tr>
<td>Yarn tenacity, mN/tex</td>
</tr>
<tr>
<td>Strength CV%</td>
</tr>
<tr>
<td>Irregularity (diameter CV%)</td>
</tr>
<tr>
<td>Brightness</td>
</tr>
</tbody>
</table>

Table 3—Properties of raw and brightened jute fibres (TD4) (Mill experiment)

Table 4—Properties of jute yarn spun from grey and brightened jute (TD4) fibres (Mill experiment)
strength CV\% and diameter irregularity of treated yarns as compared to that of control clearly show that the improvements achieved are substantial.

The increase in fibre fineness, as achieved by brightening treatment (Table 3), is not unusual. We have shown earlier that calcium and magnesium pectates, the cementing materials in jute, are removed on treating jute with EDTA, separating the strands of fibres into finer filaments.

References