In-situ peracetic acid bleaching of jute

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Bleaching of jute with peracetic acid formed in situ in bleach bath has been studied. It is observed that in an unbuffered system, the bleach bath pH drops from its initial set value. This drop in pH is found to be maximum for initial bath pH of 7. This suggests that a buffer medium is required for the bleaching of jute with peracetic acid developed in situ by the reaction of acetic anhydride and hydrogen peroxide. Excellent bleaching of jute with relatively higher loss in strength is possible by peracetic acid bleaching followed by bleaching with unreacted hydrogen peroxide of in-situ peracetic acid bleach bath. A low temperature (30°C) peracetic acid bleaching using 68 m moles/L acetic acid and 74 m moles/L hydrogen peroxide (30%) shows quite high degree of whiteness in jute with lesser damage.

Keywords: Bleaching, Jute, Peracetic acid, Whiteness index

1 Introduction
In the year 1902, Freer and Novy reported the preparation and bactericidal properties of peracetic acid. Initially, it was more popular because of its antimicrobial and disinfecting properties. Due to the recent stringent environmental regulations, peracetic acid is now being popularized as an alternative to conventional sodium hypochlorite bleaching.

The popularity of hypochlorite is because of its low cost and excellent bleaching power, even at a low temperature. However, the formation of highly toxic chlorinated organic byproduct (AOX) during the conventional sodium hypochlorite bleaching has restricted its use over the last few years. On the other hand, the peracetic acid is environmentally safe. The commercial peracetic acid is an equilibrium solution of hydrogen peroxide, acetic acid, peracetic acid and water. For bleaching purposes, either commercial peracetic acid or in-house prepared or in-situ developed peracetic acid is used. In India, it is difficult to get commercial peracetic acid for industrial application. Moreover, the stability of commercial peracetic acid is poor. It slowly decomposes to acetic acid and oxygen. Bleaching of textiles with peracetic acid developed in situ has, therefore, attracted textile chemists to avoid wasteful decomposition of readymade product.

Peracetic acid bleaching is generally done in the presence of stabilizers, like sodium hexametaphosphate and sodium tetraborate. Cai and David compared the effect of stabilizers, like borax and tetraborax (TSPP), on in-situ peracetic acid bleaching of jute fabric at neutral pH. TSPP was found to provide a far more superior stabilizing effect on jute bleaching than borax; the borax being recognized as suitable in the peracetic acid bleaching of cotton.

In recent years, there has been considerable interest in low-temperature bleaching with peracetic acid. Woods showed that the addition of a small amount of chelating agent (a transition metal cation of atomic number 24-29) and an anionic surfactant catalyzes bleaching of cotton fibres with peracetic acid (formed in-situ by the reaction of acetic anhydride and sodium perborate) to such an extent that the excellent bleaching could be obtained even at 49°C. Among the various chelating agents, 2,2'-bipyridine was found to be most effective and the cobaltous ion is the most...
with either hypochlorite or hydrogen peroxide or peracetic acid could not produce adequate whiteness, whereas a sequential bleaching process, i.e. peracetic acid bleaching followed by hydrogen peroxide bleaching, was found to be an ecofriendly and suitable alternative to the conventional hypochlorite – hydrogen peroxide bleaching for jute.

The present work was, therefore, aimed at investigating a suitable low-temperature single-bath peracetic acid (formed in-situ) bleaching method for jute to get a satisfactory level of whiteness.

2 Materials and Methods

2.1 Materials

2.1.1 Fabric

Grey jute fabric having the following specifications was used for the study:

- Weave: Plain
- Warp count: 8 lb
- Weft count: 8 lb
- Ends/cm: 6.7
- Picks/cm: 6.3

2.1.2 Chemicals

Sodium carbonate and Amolan FBOL (commercial grade, BASF) were used for jute fabric scouring. Acetic anhydride (Merek) and commercial hydrogen peroxide (30%) were used for in-situ peracetic acid preparation. Tetrasodium pyrophosphate \(\text{Na}_4\text{P}_2\text{O}_7\cdot10\text{H}_2\text{O}\), sodium tetraborate \(\text{Na}_2\text{B}_4\text{O}_7\cdot10\text{H}_2\text{O}\), sodium hexameta phosphate, ethylenediamine tetraacetic acid and sodium metasilicate were used as stabilizers. \(\text{Na}_3\text{HPO}_4\) and citric acid were used as pH buffer. All the chemicals used were of LR grade.

2.2 Methods

2.2.1 Scouring

The scouring was carried out in a laboratory winch machine with 2 g/L sodium carbonate and 2 g/L Amolan FBOL at 80°C for 45 min, maintaining the material-to-liquor ratio at 1:30. After scouring, the fabric was thoroughly washed, neutralized and dried.

2.2.2 Bleaching

All the bleaching operations were performed using peracetic acid prepared in-situ in the bleach bath. The bleaching solutions were prepared by adding 34 m moles/L acetic anhydride to a solution of 37 m moles/L hydrogen peroxide (30%) and water. Sodium metasilicate (0.2 g/L) was added to the above solutions and the solutions were stirred for 5-7 min to allow complete perhydrolysis of the acetic anhydride. Bleaching of the scoured fabric samples was done at the liquor-to-material ratio of 50:1. The effect of varying pH for two levels of temperature was examined for selecting a suitable pH for further study. The bleaching effect was examined under three bleaching techniques, namely normal bleaching, one-bath two-step bleaching and concentrated bleaching.

2.2.2.1 Bleaching at Varying pH Level

Bleaching of jute fabric samples was performed at varying pH levels (7, 8, 9 & 10) adjusted by adding sodium metasilicate in the bleach bath. The experiment was carried out separately at low temperature (30°C) and high temperature (70°C) for 90 min in an unbuffered medium. After the end of bleaching, the change in pH of the bath was measured using a laboratory pH analyzer. The fabric samples were tested for improvement in whiteness.

2.2.2.2 Normal Bleaching

Normal bleaching was done using peracetic acid prepared in-situ in the bleach bath. The bleach bath pH was adjusted at 7 by adding sodium metasilicate and the bleaching was carried out for 50 min at low temperature (30°C) in buffer medium using 0.2 M disodium hydrogen phosphate and 0.1 M citric acid as buffer.

2.2.2.3 One-bath Two-step Bleaching

In this process, after a similar treatment as reported in section 2.2.2.2, the pH of the solution was increased to 10 using sodium carbonate, temperature was gradually raised to 85°C and the bleaching was continued for a further period of 50 min in the same bath.

2.2.2.4 Concentrated Bleaching

Bleaching in this process was carried out in the same fashion as reported for normal bleaching with the exception that the molar concentrations of acetic anhydride and hydrogen peroxide (30%) were doubled and the time of treatment was increased to 100 min.

All the fabric samples were conditioned at a constant temperature of 70 ± 2°F and relative humidity of 65 ± 2% RH for 24 h and then used for physical testing.
2.2.3 Estimation of Peracetic Acid Content in Bleach Bath

For assessing the decomposition of peracetic acid in the bleach bath in the absence of fabric samples, the acetic anhydride (34 m moles/L) and hydrogen peroxide (37 m moles/L) were mixed to form 100 ml bleaching solution. Sodium metasilicate (0.2 g/L) was added to the above solution and then stirred for 5-7 min. The pH was adjusted at 7 by further addition of sodium metasilicate. The solution was buffered using 0.2 M Na2HPO4 and 0.1 M citric acid solutions. The peracetic acid and hydrogen peroxide contents of the solution were measured at 30°C in a constant temperature bath for different time intervals (0, 2, 5, 10, 15, 20, 30, 40, 50 and 60 min) as per the method suggested earlier. The percent decomposition of peracetic acid and reduction in free hydrogen peroxide content at different time intervals were further calculated.

2.2.4 Evaluation of Fabric Properties

The tensile strength of the samples was measured as per the IS : 1969-1968 method using 1445 CRT Zwick universal tensile testing equipment. Flex abrasion test was carried out using C.S.I abrasion tester. Fabric stiffness (expressed as bending length) was measured as per the IS : 6490-1971 (Cantilever test) using SASMIRA stiffness tester.

The whiteness of the bleached samples was evaluated using the Hunter Lab scale, employing Macbeth spectrophotometer (2020+, Colour Eye) interfaced with computer colour matching system.

The surface morphology of scoured and bleached jute fibres was examined using a scanning electron microscope (model: Stereo Scan 360, Cambridge Instrument Limited, UK). The photographs of the samples were taken at a magnification of ×2000.

3 Results and Discussion

Preliminary experiments on in-situ peracetic acid bleaching of jute using various stabilizers like tetrasodium pyrophosphate, sodium hexametaphosphate, borax, sodium metasilicate and ethylenediamine tetraacetic acid showed the excellent result for sodium metasilicate and further experiments were, therefore, conducted using sodium metasilicate as stabilizer. It was also found that the bleaching below pH 7 could not produce adequate whiteness in jute.

3.1 Effect of Initial pH on Residual pH of Bleach Bath

When bleaching was conducted at different initial pH (7, 8, 9 and 10), it was found that at the end of bleaching operation the pH of the bleach bath dropped in case of initial pH 7 and 8, whereas there was very little or no drop in pH in the case of initial pH 9 and 10 (Table 1). The drop in pH is because of the decomposition of peracetic acid into acetic acid. As the drop in pH is maximum at initial pH 7, it can be inferred that the maximum peracetic acid decomposition takes place at this pH. Table 1 also shows that at 70°C, the pH 7 is reduced by 1.7 unit whereas at 30°C the drop in pH is 0.44 unit. The increase in temperature has, therefore, an accelerating effect on peracetic acid decomposition.

3.2 Effect of pH and Temperature on Whiteness

Table 2 shows that the increase in pH from 7 to 10 drastically decreases the level of whiteness of jute fabric for bleaching at 30°C, whereas the fabric whiteness improves in case of pH 9 and 10 for the bleaching at 70°C. The results obtained at 30°C treatment prove that the peracetic acid shows maximum bleaching action at pH 7. The substantial improvement in whiteness for pH 9 and 10 in case of 70°C treatment is due to the bleaching action of unreacted hydrogen peroxide left in the bath which remains inactive at lower temperature.

Thus, pH 7 was selected for rest of the bleaching treatments performed at 30°C. Reduction in pH during the experiments suggested the use of a buffer system. Further bleaching treatments were, therefore, conducted in buffered medium using 0.2 M Na2HPO4 and 0.1 M citric acid.

3.3 Decomposition of Peracetic Acid in Bleach Bath

The decomposition of peracetic acid in the bleach bath at 30°C was assessed both in the absence and

<table>
<thead>
<tr>
<th>Initial bath pH</th>
<th>Final bath pH</th>
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<tr>
<td>30°C</td>
<td>70°C</td>
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<tr>
<td>7</td>
<td>6.9</td>
</tr>
<tr>
<td>8</td>
<td>6.9</td>
</tr>
<tr>
<td>9</td>
<td>8.9</td>
</tr>
<tr>
<td>10</td>
<td>9.9</td>
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</table>

<table>
<thead>
<tr>
<th>Bleach bath temperature, °C</th>
<th>Whiteness index</th>
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<tr>
<td>7.0°C</td>
<td>53.9</td>
</tr>
<tr>
<td>8.0°C</td>
<td>51.3</td>
</tr>
<tr>
<td>9.0°C</td>
<td>43.4</td>
</tr>
<tr>
<td>10.0°C</td>
<td>42.7</td>
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</table>

*Initial bleach bath pH
presence of fabric sample by measuring the peracetic acid content of the bath at different time intervals. The reduction in free hydrogen peroxide content of the bath was also measured simultaneously at the same time intervals.

Both peracetic acid and hydrogen peroxide contents of the bath are found to be reduced with the time in the presence and absence of fabric sample. It is observed from Fig. 1 that the presence of fabric enhances the decomposition of peracetic acid. The initial gradual reduction in hydrogen peroxide content (Fig. 2) proves the in-situ peracetic acid formation in the bleach bath and a part of the free hydrogen peroxide gets thus consumed. It is observed that after 50 min bleaching the decomposition of peracetic acid reduces. This may be due to the shift of pH towards acidic side due to the gradual generation of acetic acid by peracetic acid decomposition.

These results directed further research towards an interesting avenue, i.e. the utilization of residual hydrogen peroxide in the bath by increasing the pH to 10 and continuing bleaching at 85°C for further 50 min after the initial treatment of 50 min at 30°C. This is called as one-bath two-step bleaching.

As the peracetic acid content was found to be stabilized after a treatment of 50 min, in the subsequent process the one-bath two-step bleaching was compared with the low temperature (30°C) bleaching carried out for 50 min, i.e. normal bleaching. The main aim of this work was to search a suitable method for low-temperature bleaching of jute using in-situ peracetic acid. The effectiveness of the bleaching was also studied by doubling the molar concentrations of both acetic anhydride and hydrogen peroxide for low-temperature (30°C) bleaching at pH 7 which is named as concentrated bleaching.

3.4 Effect of Bleaching Techniques on Whiteness and Fabric Properties

Table 3 shows that a high degree of bleaching is possible at 30°C by doubling the molar concentrations of acetic anhydride and hydrogen peroxide. An excellent bleaching effect can be obtained by one-bath two-step technique also. The concentrated process saves energy and the one-bath two-step process saves chemicals. Among the three techniques, one-bath two-step process shows maximum whiteness with a comparatively higher losses in strength and abrasion resistance.

To understand the effect of different bleaching techniques on the surface morphology of the fibre, the scanning electron photomicrographs of samples bleached by normal (Fig. 3b), one-bath two-step

<table>
<thead>
<tr>
<th>Bleaching technique</th>
<th>Whiteness index</th>
<th>Strength loss %</th>
<th>Abrasion resistance (No. of cycles)</th>
</tr>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Warp</td>
</tr>
<tr>
<td>Control</td>
<td>41.8</td>
<td>—</td>
<td>286</td>
</tr>
<tr>
<td>Normal</td>
<td>64.1</td>
<td>8.6</td>
<td>254</td>
</tr>
<tr>
<td>One-bath two-step</td>
<td>74.7</td>
<td>18.5</td>
<td>216</td>
</tr>
<tr>
<td>Concentrated bleaching</td>
<td>71.2</td>
<td>11.7</td>
<td>233</td>
</tr>
</tbody>
</table>

*Scoured sample without any bleaching treatment
Fig. 3—Scanning electron photomicrographs of jute fibres [(a) control, (b) bleached through normal bleaching, (c) bleached through one-bath two-step bleaching, (d) bleached through concentrated bleaching, (e) treated only in the second step of one-bath two-step bleaching, and (f) treated in the first step of one-bath two-step bleaching with all chemicals except acetic anhydride]

(Fig. 3c) and concentrated bleaching (Fig. 3d) techniques were compared with the control scoured sample (Fig. 3a). To realize which stage of one-bath two-step techniques is responsible for relatively higher strength and abrasion resistance losses, the scanning electron photomicrographs of an equal weight fresh sample bleached at the second step of the two-step process (Fig. 3e) and a sample treated for 50 min at 30°C with hydrogen peroxide (same molar concentration as used in in-situ normal process), water and sodium metasilicate (Fig. 3f), were also recorded in buffer medium at pH 7. Fig. 3e shows
clearly that maximum surface damage in two-step technique is because of the second step which is alkaline treatment at higher temperature, whereas the presence of hydrogen peroxide in the first step causes no appreciable damage.

4 Conclusions

Commercial peracetic acid suffers from two major problems, viz. not easily available and storage instability. Bleaching of textiles by peracetic acid formed in-situ within the bleach bath is, therefore, seems to be a viable alternative to commercial peracetic acid application for ecofriendly bleaching.

4.1 A buffered peracetic acid bleach bath results in reduction in bath pH from its initial pH value. This reduction is maximum for a starting pH of 7. The drop in pH is more for bleaching at 70°C compared to that for bleaching at 30°C.

4.2 The increase in bleach bath pH from 7 to 10 causes reduction in whiteness of the bleached fabric for a low-temperature (30°C) bleaching treatment, whereas bleaching at 70°C causes improvement in whiteness for pH 9 and 10.

4.3 Peracetic acid and hydrogen peroxide contents of the bath are found to be reduced with time. The peracetic acid decomposition is accelerated by the presence of fabric sample.

4.4 Excellent bleaching of jute textile with a relatively higher loss in strength and abrasion resistance is possible by using one-bath two-step method.

4.5 A high degree of whiteness with comparatively lesser damage can be achieved at 30°C using 68 mmoles/L acetic acid and 74 mmoles/L hydrogen peroxide (30%) at pH 7.

References