Short sequence environment friendly bleaching of wheat straw pulp

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In India, wheat straw is used as a potential raw material for the manufacture of writing and printing paper. In the present study, wheat straw pulp made through soda process and after oxygen delignification, was subjected to various conventional bleaching processes involving elemental chlorine and short sequence elemental chlorine free (ECF) bleaching processes. It was observed that \(D_{50}E_{50}\) sequence gives 84.8% brightness (Elrepho) and excellent strength properties of pulp and generates lower pollution load, both in terms of COD and TOCl, in comparison to the conventional bleaching processes involving elemental chlorine.

Keywords: ECF bleaching, Oxygen delignification, Short sequence, Wheat straw

IPC Code: D21C9/10

Introduction

Conventional bleaching processes cannot take the brightness of pulp to higher level (beyond 80%) without sacrificing the mechanical properties of pulp. This is achievable by usage of hydrogen peroxide at alkali extraction stage. Higher brightness pulps can be produced by short sequence bleaching process when the incoming kappa number is reduced by oxygen delignification. Studies highlight the multiple advantages of oxygen delignification prior to bleaching of pulp.

Due to limited availability of conventional forest based paper making raw materials like wood and bamboo, use of alternative raw materials mainly agro residues is increasing day by day. Wheat straw is a cheap substitute for wood raw material and can be used for manufacturing writing and printing paper with a required brightness level (> 80%). But it has a major limitation of poor strength properties, which are reduced further in conventional bleaching processes using elemental chlorine and hypochlorite. In the present study, pulp made from wheat straw through soda process was subjected to oxygen delignification. After that, the oxygen delignified low kappa number pulp was bleached through various conventional and short elemental chlorine free (ECF) bleaching sequences. Comparison of different bleaching sequences has been done on the basis of optical and physical strength properties of pulp and pollution parameters like COD and TOCl of the combined effluent from various stages of a particular sequence.

Materials and Methods

Locally available wheat straw was collected, washed with water, air dried, grinded and the fraction passing through a 45 mesh but retained over 85 mesh, was selected. Extractive content and other chemical analysis were performed as per TAPPI methods. Proximate analysis of air dried wheat straw is as follows: moisture, 11.9; alcohol-benzene solubility, 3.9; hot water extractive, 7.9; 1% NaOH solubility, 39.2; lignin, 17.9; ash, 7.6; and holocellulose, 66.7%.

Pulping of Wheat Straw

High quality pulp could be produced when NaOH is used as cooking agent. Four sets of pulping experiments were carried out in the laboratory WEVERK digester (20 l). The pulp was refined and then screened after washing. Pulp obtained after cooking of raw material with the conditions as mentioned under experiment number 4 was used for further investigation (Table 1). All the calculations have been done on O.D. basis.

Oxygen Delignification

Oxygen delignification of wheat straw pulp was carried out in same laboratory digester under following conditions: consistency, 10; NaOH, 2.0; MgSO\(_4\), 0.1; and brightness (Elrepho), 38%; kappa number, 16; \(O_2\) pressure, 7.5 bar; temp, 120°C; and retention time, 60 min. Pulp, after oxygen delignification, was subjected to following ten
bleaching sequences – CEHH, CEHD, CED, D$_{50}$EP, D$_{50}$EP$\textsubscript{P}$, D$_{70}$EP$\textsubscript{P}$, D$_{70}$ED$_{30}$, D$_{50}$ED$_{50}$, D$_{70}$EP$\textsubscript{P}$D$_{30}$, D$_{50}$EP$\textsubscript{P}$D$_{50}$. All the bleaching experiments were carried out in the laboratory, using batch vessels immersed in constant temperature bath (Table 2).

**Chlorination (C)**

Bleach liquor was added to the pulp in the chlorination vessel to generate chlorine gas at pH less than 2 (1.5-1.8). At the end of retention time, pulp was washed and subjected to alkali extraction. For CEHH and CEHD sequences, total chemical charge was calculated and 70 percent of the total chlorine demand was utilized in the C stage, while 15 percent of the total chlorine demand was fed in each H stage.

**Extraction (E)**

Chlorinated lignin derivatives were extracted out by addition of NaOH (conc, 45 gpl). After the end of retention time, the pulp was washed.

**Hypochlorination (H)**

The same bleach liquor as used in the C stage was used for hypochlorination with different pH (10–11). Washing of pulp was performed between each stage.

**Treatment with Chlorine Dioxide (D)**

Sodium chlorite solution was used in this stage and the pH was maintained between 4-5. This stage was used to replace both chlorination and hypochlorination stages. For CEHD and CED sequences, 15 percent and 30 percent of the total chlorine demand was fed to D stage, respectively. For elemental chlorine free sequences, the suffix to D indicates the percentage of total chlorine demand charged in that stage.

**Oxidative Extraction Stage (E$\textsubscript{p}$)**

Oxidative extraction has proved to be a very effective approach to achieve higher brightness pulp. In the present investigation, 0.5 percent hydrogen peroxide (H$_2$O$_2$) was added along with the alkali in some of the ECF bleaching sequences.

**Peroxide Stage (P)**

To reduce generation of chlorinated compounds in some of the ECF sequences, H$_2$O$_2$ was used in the final stage of bleaching sequence replacing H or D.

**Determination of COD and TOCl**

Chemical oxygen demand (COD) of the combined effluent generated from various bleaching sequences, were estimated by open reflux method. TOCl for all the sequences has been calculated using the following empirical correlation:

\[
TOCl = 0.1[\text{Active (Cl}_2\text{)+ Active (ClO/2) + Active (ClO}_2/5\text{)]}
\]

which gave a qualitative measure of the chloroorganics generated in different bleaching processes.

**Determination of Optical and Strength Properties of Bleached Pulp**

Handsheets were prepared in British sheet former under standard pressing and drying conditions and optical and physical strength properties of all the

<table>
<thead>
<tr>
<th>Stage</th>
<th>C</th>
<th>E</th>
<th>H</th>
<th>E$_{p}$</th>
<th>D</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Consistency, %</td>
<td>4</td>
<td>10</td>
<td>7</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Temp, °C</td>
<td>40</td>
<td>60</td>
<td>70</td>
<td>70</td>
<td>70</td>
<td>85</td>
</tr>
<tr>
<td>Time, min</td>
<td>45</td>
<td>70</td>
<td>90</td>
<td>60</td>
<td>60</td>
<td>120</td>
</tr>
<tr>
<td>pH</td>
<td>2</td>
<td>10.5-11.0</td>
<td>10.5-11.0</td>
<td>10.5-11.0</td>
<td>4.5</td>
<td>11</td>
</tr>
<tr>
<td>Peroxide, %</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.5</td>
<td>-</td>
<td>1.5 in D$_{50}$EP</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.5 in D$_{50}$EP$\textsubscript{P}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.0 in D$_{50}$EP$\textsubscript{P}$</td>
</tr>
</tbody>
</table>
bleached pulps were evaluated as per BIS-1848 and IS: 1060 specifications (Table 3).

**Results and Discussion**

Experiment number 4 resulted in higher kappa number pulp with maximum yield (Table 1). Oxygen delignification resulted in reduction in pulp kappa number (43%). This in turn will reduce the chemical requirements in the bleaching processes and thus pollution load generated will be significantly low. Increasing the number of stages in bleaching process to get higher brightness not only increases cost of bleaching but also the level of pollution and reduces pulp strength. In the present investigation, the number of stages has been limited to three only. The commonly practiced CEHH and CEHD sequences have been studied to compare the optical and strength properties of pulp obtained through various ECF processes (Table 3). Although conventional bleaching of chemical pulp using chlorine is the most economic one, it is not possible to achieve higher brightness without sacrificing pulp strength. Hypochlorite extensively degrades the cellulose. Replacement of H – stages from CEHH sequence by D – stage as in CED sequence shows improvement (23%) in both tensile and tear strength of wheat straw pulp for same level of brightness achieved. Higher values of tensile index and tear index were achieved in ECF sequences (Table 3). DED sequences were found to give good strength pulp but failed to reach the target brightness. Inclusion of $E_P$ stage has shown considerable improvement in brightness level in all the ECF sequences studied. Among the sequences studied, $D_{50}E_P$, $D_{70}E_PD_{30}$ and $D_{50}E_PD_{50}$ were found to give brightness above 80 percent (Fig. 1). The conventional CEHH sequence almost reached the target brightness at the cost of drastic reduction in strength properties.

For writing and printing paper, opacity is important in order to avoid show-through of the printed image to the reverse side. The $D_{50}E_PD_{50}$ bleaching sequence (94.6% opacity) gave 6 percent more opacity than the conventional CEHH sequence (89.1% opacity).

Another major concern with regard to bleach plant effluent is the COD load and chloroorganics. In comparison to the conventional CEHH sequence in $D_{50}E_PD_{50}$ bleaching process (84.8% brightness),

<table>
<thead>
<tr>
<th>Sl No</th>
<th>Sequence</th>
<th>Brightness, (Elrepho)</th>
<th>Opacity, %</th>
<th>Tensile Index, Nm/g</th>
<th>Tear Index, mNm²/g</th>
<th>COD, kg/ton</th>
<th>TOCl, kg/ton</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CEHH</td>
<td>79.7</td>
<td>89.1</td>
<td>43.2</td>
<td>2.0</td>
<td>36.4</td>
<td>4.14</td>
</tr>
<tr>
<td>2</td>
<td>CEHD</td>
<td>77.5</td>
<td>90.0</td>
<td>49.4</td>
<td>2.2</td>
<td>34.6</td>
<td>3.62</td>
</tr>
<tr>
<td>3</td>
<td>CED</td>
<td>79.4</td>
<td>97.1</td>
<td>56.6</td>
<td>2.6</td>
<td>33.9</td>
<td>3.14</td>
</tr>
<tr>
<td>4</td>
<td>$D_{50}EP$</td>
<td>76.0</td>
<td>91.2</td>
<td>62.1</td>
<td>2.8</td>
<td>23.7</td>
<td>0.42</td>
</tr>
<tr>
<td>5</td>
<td>$D_{50}EP$</td>
<td>79.0</td>
<td>85.6</td>
<td>72.1</td>
<td>2.8</td>
<td>24.9</td>
<td>0.42</td>
</tr>
<tr>
<td>6</td>
<td>$D_{50}EP$</td>
<td>82.2</td>
<td>94.1</td>
<td>80.1</td>
<td>3.2</td>
<td>25.7</td>
<td>0.58</td>
</tr>
<tr>
<td>7</td>
<td>$D_{70}ED_{50}$</td>
<td>66.8</td>
<td>95.0</td>
<td>70.8</td>
<td>3.3</td>
<td>25.9</td>
<td>0.84</td>
</tr>
<tr>
<td>8</td>
<td>$D_{90}ED_{50}$</td>
<td>69.9</td>
<td>96.0</td>
<td>81.4</td>
<td>3.1</td>
<td>25.2</td>
<td>0.84</td>
</tr>
<tr>
<td>9</td>
<td>$D_{50}EPD_{50}$</td>
<td>83.4</td>
<td>91.8</td>
<td>74.6</td>
<td>2.9</td>
<td>27.1</td>
<td>0.84</td>
</tr>
<tr>
<td>10</td>
<td>$D_{50}EPD_{50}$</td>
<td>84.8</td>
<td>94.6</td>
<td>73.7</td>
<td>3.4</td>
<td>26.2</td>
<td>0.84</td>
</tr>
</tbody>
</table>

Fig. 1 — Comparison of brightness and strength properties of pulps from different bleaching sequences: ○, Brightness; □, Tensile Index; △, Tear Index
Conclusions

Though oxygen delignification prior to bleaching is capital intensive, higher brightness pulp (about 85%) with reduced load of COD and TOCl may be achieved through only three stage bleaching process. Higher values of tensile index and tear index were achieved in all the ECF bleaching sequences. Inclusion of $E_p$ stage has shown considerable improvement in brightness level in all the ECF bleaching sequences and sequences with $E_p$ stage could cross the target of 80 percent brightness. The $D_{50}E_pD_{50}$ elemental chlorine free bleaching sequence is very much effective in achieving brightness level (nearly 85%) and in comparison to the conventional CEHH sequence, reduction in TOCl level (80%) and COD load (28%) has been observed. This short, three stage bleaching sequence, which improves strength properties substantially, is quite promising for oxygen delignified wheat straw pulp.

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References