Studies on use of lignocellulosic residues of palma rosa grass after steam distillation for the production of chemical grade pulp

Dharm Dutta*, C H Tyagi, A K Upadhyay & A P Garg

aDepartment of Paper Technology, Indian Institute of Technology Roorkee
Saharanpur Campus, Saharanpur 247 001, India
bAbhishek Industries Ltd., Dhaula, Mansa Road, Barnala, India
cDepartment of Microbiology, CCS University, Meerut, India
Email: dharm_dutt@yahoo.com

Received 3 April 2006; revised received 11 January 2007; accepted 8 March 2007

The study aims at using of lignocellulosic residues of Cymbopogon martini to develop value added quality papers by eco-friendly pulping and bleaching techniques. C. martini grass is exclusively used to extract important geranium oil by distillation in crude direct–fired stills. Anatomical, morphological and chemical studies indicate its similarities with hardwoods and its suitability for the production of chemical grade pulp. Molecular oxygen, which is specific oxidizing agent for lignin has an immediate drawback to its use, is the low solubility in cooking liquor. The diffusion problem of dissolved oxygen is reduced to insignificance because of looser and open structure in the lignocellulosic residues of C. martini after distillation. Distillation process makes the C. martini suitable for alkali-O₂ delignification process by bridging over the problem of mass transfer of O₂, which is far less than C. martini without distillation. C. martini produces 44.73% screened pulp yield at kappa number 22.12 by soda cooking process and screened pulp yield of 45.80% at kappa number of 20.47 by alkali-O₂ delignification process. The addition of 0.1% AQ in alkali-O₂ process reduces kappa number to 17.35 and improves pulp yield by 1.20%. The unbleached pulp shows good response towards bleaching, which can be bleached up to 85.95% by OCEHH bleaching sequence.

Keywords: Cymbopogon martini, Steam distillation, Lignocellulosic residues, Soda and alkali-O₂ pulping, Bleaching

IPC Code(s): D21C3/00, C08L97/02

Indian paper makers are facing a serious lack of better quality of fibrous raw materials. The inadequate supply of better quality fibrous material has forced the paper makers to spend heavily on imported wood fiber. With other associated multifarious problems like, tough competition from imports, obsolete technology, soaring environmental costs, depreciation of money, high manufacturing cost, minimum profit level due to less selling price of end products in such a competitive atmosphere, the paper technocrats have to think seriously on sustainable availability of cost-effective raw materials.

To overcome this serious lack of good quality of fiber including their sustainable availability along with cost-effectiveness non-woody plant fiber may play an important role in the global scenario for pulp and papermaking. Besides agro-based residues, plenty of other non-woody plants are available abundantly in India. It has been proven that by selecting an approximate mixture of non-woody plant fibers and appropriate pulping process, any quality of paper and paper-board can be manufactured with or without addition of any wood pulp.

C. martini is a tall perennial sweet-scented grass, 5-8 ft. high occurring in the drier localities of India, from Kashmir through Punjab hills to Almora, Garhwal and Singhbhum and extending to Bombay and south India. It grows in lower altitudes and valleys, in shady nallas, and dense moist areas with poor drainage. C. martini is commonly known as Palma rosa or Rusa grass and is cultivated for the extraction of valuable geranium or palma rosa oil. An acre of grass yields 15-20 lb. oil. The leaves of C. martini grass contain about 1.4% oil (dry basis) and stalks contain the least quantity (0.03%) of oil.

Although, it is accepted that molecular oxygen is a specific oxidizing agent for lignin, an immediate drawback to its use is the low solubility in cooking liquors. Oxygen is three phase system. Oxygen must cross the gas liquid interface, fiber, and finally diffuse
into fiber wall before reacting. The mass transfer problem is aggravated by the low solubility of oxygen in aqueous alkali solution. This limits a problem of mass transfer in a heterogeneous chemical process, such as wood pulping. Even applying very high dose of oxygen, useful transfer of the delignifying agent (molecular oxygen) into the fiber walls, where the reaction should take place, is difficult to obtain in one stage pulping. Many efforts have been made in the development of two stage alkali-oxygen delignification of wood to give pulps comparable to kraft, in yield and quality. These generally involve a mild cooking stage followed by mechanical defiberation. The coarse pulp thus obtained is very suitable for subsequent alkaline treatment in the presence of oxygen due to its higher exposed surfaces; but unfortunately the two-stage pulping process is rather cumbersome and likely to be high in capital costs. Afterwards, instead of wood chips, thermo mechanical fibers have been used for alkali oxygen pulping process to develop one-stage oxygen pulping, but some strength deficiencies have taken place, originating from mechanical damage caused in the pressurized refining, which itself is an operation with high energy consumption. However, the problem of oxygen mass transfer is far less than woods because of the looser and open plant structure. Bio-soda and bio-alkali-O₂ pulping of solid waste of *C. martini* has been found to be suitable for the development of value added quality papers. The pulp of solid waste of *C. martini* shows good response towards CEHH bleaching sequence. *C. martini* has low lignin and more open and looser structure, which makes it suitable for biopulping.

In view of the above facts, efforts were made to use lignocellulosic residues of *C. martini*, which is available in ample quantity after steam distillation for the production of chemical grade pulp. The study is conducted with the concept that distillation of *C. martini* removes oil ducts and lignocellulosic residues becomes more open and looser and makes the lignocellulosic residues of *C. martini* suitable for alkali-O₂ delignification process by bridging over the problem of mass transfer of O₂, which is far less than *C. martini* without distillation. In this way, the lignocellulosic residues can be used to develop value added quality papers by eco-friendly pulping and bleaching techniques.

### Experimental Procedure

**Raw materials**

*C. martini* was collected from Punjab Agriculture University, Jalandhar. The raw materials were hand-chopped and sun-dried for 20 days before distillation. Geranium oil was extracted from the grass by distillation in crude iron direct-fired stills having false bottom over which the grass is charged. The lignocellulosic residues after extraction was air dried and kept in polythene bags.

**Anatomical studies**

A number of transverse sections (T.S.) of stem and leaves of *C. martini* before and after distillation of thickness 25 μm were cut on Leitz base sladge microtome 1300. The glass slides for microscopic examination were prepared as per BIS method 5285-1969. The microphotographs of the projected images of T.S. and L.S. of *C. martini* were taken at a magnification of 200 X.

**Morphological and proximate chemical analysis**

The culms of *C. martini* were hand-chopped and boiled with concentrated solution of HCl to separate cellulosic fibers by dissolving middle lamella. The fibers were washed and stained with Hertzberg stain. Morphological characteristics of *C. martini* were determined with the help of microscope and compared with *Picca abies* and *Pinus kesiya*. Hand-chopped *C. martini* before and after extraction was disintegrated separately into powder in wood mill and a fraction passed through +48 mesh size but retained on +80 mesh size was used for proximate chemical analysis.

**Pulping studies**

Hand-chopped lignocellulosic residues of *C. martini* were cooked in WEVERK rotary digester of capacity 0.02 m³ by soda, soda-AQ, alkali-O₂, and alkali-O₂-AQ pulping processes. The pulp was washed, screened and crumbled. The pulp was evaluated for screened pulp yield, screening rejects and kappa number. The black liquor generated during pulp squeezing was analyzed for COD using Dralange Lasa photometer in each pulping stage.

**Bleaching studies**

To achieve the target brightness CEHH and OCEHH bleaching sequences were conducted. For the estimation of pulp strength, the viscosity was determined by capillary tube viscometer using Cupriethylenediamine solution (CED).
Results and Discussion

Microphotograph A (Fig. 1) shows T.S. of *C. martini* oil ducts, vascular bundles and ground tissue at a magnification of 200 X without distillation. The anatomical structure of *C. martini* shows very strong outer zone of fibrous material, surrounded by sclerified tissue of parenchyma cells. The minor bundles are concentrated close to the epidermis and form an almost continuous ring of fibrous tissue. In the central portion of the stem (internodes), the pith, vascular bundles are scattered. The parenchyma cells gradually increase in size towards the centre of the stem, whereas the wall thickness of the cells is decreasing. Oil ducts are scattered in pith as well as in cortical region of the stalk of *C. martini*. Microphotographs B and C show empty oil ducts, vascular bundles and ground tissue after distillation. Distillation process makes the *C. martini* suitable for alkali-O\(_2\) delignification process because the problem of useful mass transfer of O\(_2\) is far less than with
virgin *C. martini* grass due to looser and open structure. Microphotograph D shows the T.S. of leaf of *C. martini*, which consists of mesophyll cells. The secretary cells lining cavities are thin walled parenchyma with dense protoplasm. These cells are elongate with the long dimension extending parallel with the long dimension with the duct. These ducts secrete important palma rosa oil. Microphotographs E and F show L.S. of *C. martini* after and before distillation of oil.

Table 1 shows the morphological and chemical characteristics of *C. martini* before and after distillation. The hot water soluble is 15.90%, which reduces to 0.1% after distillation. It means that hot water-soluble mainly contains important geranium oil with other acids and pigments. Alcohol-benzene soluble and one percent NaOH soluble remains unaffected after and before distillation. *C. martini* contains lignin, pentosan and holocellulose 13.67, 25.38 and 74.51%, respectively. It clearly indicates that *C. martini* produces high pulp yield and requires lesser cooking chemicals and shorter cooking cycle. However, silica contents are slightly higher i.e. 2.16%.

The average fiber length of *C. martini* is 0.96 mm, which resembles to those of hard woods. The fiber diameter, cell wall thickness and lumen diameter of *C. martini* is 30.53, 6.42 and 20.58 μm, respectively. The flexibility coefficient and slenderness ratio of *C. martini* is less than *Picca abies* and *Pinus kesiya*, it means that the fibers of *C. martini* are readily converted into double ribbons during pressing. Due to low slenderness ratio, it is expected that such type of fibers is having high degree of collapsibility and conformability. Therefore, such type of paper gives denser and less opaque sheet with low tear, porosity and bulk. The runkel ratio of *C. martini* resembles very closely with *Pinus kesiya*. The runkel ratio is

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Parameters</th>
<th><em>C. martini</em> Before distillation</th>
<th><em>C. martini</em> After distillation</th>
<th><em>Picca abies</em></th>
<th><em>Pinus kesiya</em></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cold water soluble, %</td>
<td>12.18</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td>Hot water soluble, %</td>
<td>15.90</td>
<td>0.10</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>3</td>
<td>Alcohol-benzene soluble, %</td>
<td>5.36</td>
<td>5.32</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>4</td>
<td>1% NaOH soluble, %</td>
<td>37.85</td>
<td>37.83</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>5</td>
<td>Lignin, %</td>
<td>13.67</td>
<td>13.66</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>6</td>
<td>Pentosan, %</td>
<td>25.38</td>
<td>25.32</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>7</td>
<td>Holocellulose, %</td>
<td>74.51</td>
<td>74.48</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>8</td>
<td>Ash, %</td>
<td>4.52</td>
<td>4.48</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>9</td>
<td>Silica</td>
<td>2.16</td>
<td>2.18</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>10</td>
<td>Fibre length, (L) mm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>0.96</td>
<td>—</td>
<td>2.25</td>
<td>2.32</td>
</tr>
<tr>
<td></td>
<td>Variation</td>
<td>0.35-2.25</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>11</td>
<td>Fibre width (D), μm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>30.53</td>
<td>—</td>
<td>41.70</td>
<td>40.70</td>
</tr>
<tr>
<td></td>
<td>Variation</td>
<td>12.50-40.00</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>12</td>
<td>Average lumen diameter, (d), μm</td>
<td>20.58</td>
<td>—</td>
<td>35.70</td>
<td>34.75</td>
</tr>
<tr>
<td>13</td>
<td>Average cell wall thickness (w), μm</td>
<td>6.42</td>
<td>—</td>
<td>6.00</td>
<td>5.85</td>
</tr>
<tr>
<td>14</td>
<td>Flexibility coefficient (d/D) × 100</td>
<td>67.41</td>
<td>—</td>
<td>85.54</td>
<td>85.62</td>
</tr>
<tr>
<td>15</td>
<td>Slenderness ratio L/D</td>
<td>31.44</td>
<td>—</td>
<td>53.96</td>
<td>57.00</td>
</tr>
<tr>
<td>16</td>
<td>Rigidty coefficient 2w/D</td>
<td>0.42</td>
<td>—</td>
<td>0.29</td>
<td>0.29</td>
</tr>
<tr>
<td>17</td>
<td>Wall fraction (2w/D) × 100</td>
<td>41.20</td>
<td>—</td>
<td>29</td>
<td>29</td>
</tr>
<tr>
<td>18</td>
<td>Runkel ratio 2w/d</td>
<td>0.62</td>
<td>—</td>
<td>0.34</td>
<td>0.51</td>
</tr>
</tbody>
</table>
related with fiber diameter, flexibility and degree of collapseness both of which control the degree of conformability within the paper sheet and as such the size and number of inter fiber bonds are improved in case of *C. martini*. The runkel ratio is related to fiber density\(^{22,23}\), which determines breaking length, bursting strength and double fold of paper. However, the mechanical properties along with other properties of paper related to wet plasticity may be increased by fibrillation and high pentosan content (25.38%). Based on morphological characteristics, it is expected that all the mechanical strength properties except tear strength of *C. martini* should be slightly better compared to strength properties of hard woods.

Table 2 shows the experimental conditions and results of soda pulping of lignocellulosic residues of *C. martini* at different alkali doses i.e. 10 to 20% (as Na\(_2\)O). The screened pulp yield increases with increasing alkali dose from 10 to 14%, when *C. martini* is delignified at the same H-Factor i.e. 553.21. Further, on increasing alkali dose screened pulp yield decreases from 44.73 to 38.92%, whereas kappa number first drops from 30.95 to 22.12 and then only insignificant increase is noticed, while keeping other variables constant like bath ratio 1:5, maximum cooking temperature 150°C and maximum pulping time 3 h.

Table 3 shows the effect of temperature on pulp yield and kappa number during soda pulping of lignocellulosic residues of *C. martini*, while keeping all variables constant, like alkali dose 14% (as Na\(_2\)O), bath ratio 1:5 and maximum cooking temperature 150°C and maximum pulping time 4.5 h. The screened pulp yield increases from 41.98 to 44.73% up to H-factor 553.21 and kappa number drops from 28.42 to 22.12. Further, on increasing H-factor from 1212.24 to 3033.98 screened pulp yield drops sharply, while kappa number decreases slowly.

Table 4 shows the effect of time on pulp yield and kappa number during soda pulping of lignocellulosic residues of *C. martini*, while keeping all variables constant, like alkali dose 14% (as Na\(_2\)O), bath ratio 1:5 and maximum cooking temperature 150°C. The screened pulp yield increases from 41.98 to 44.73% up to H-factor 553.21 and kappa number drops from 32.45 to 22.12. Beyond that screened pulp yield drops sharply, while kappa number remains almost constant.

Table 5 shows the experimental conditions and results of soda-AQ pulping of lignocellulosic residues of *C. martini*. The screened pulp yield increases from 44.73 to 45.45%, while kappa number drops from 22.12 to 21.00 when *C. martini* is delignified to H-Factor 553.21 at AQ dose varied from 0.0 to 0.1%, while keeping other variables constant, i.e. alkali dose 14% (as Na\(_2\)O), bath ratio 1:5, maximum cooking temperature 150°C and maximum pulping time 3 h. When soda-pulping process is compared with soda-AQ process, the addition of 0.1% AQ in soda-AQ process reduces kappa number by 1.12 units and increases pulp yield by 0.72%.

Table 6 shows the effect of O\(_2\) during alkali-O\(_2\) pulping of lignocellulosic residues of *C. martini*. The screened pulp yield increases from 44.73 to 45.80%, while kappa number drops from 22.12 to 20.47, when *C. martini* is delignified to H-Factor 553.21 at O\(_2\) pressure varied from 0.0 to 5 kg/cm\(^2\), while keeping other variables constant i.e. alkali doses, 14% (as Na\(_2\)O), bath ratio 1:5, maximum cooking temperature 150°C and maximum pulping time 4.5 h.

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Set-I</th>
<th>Set-II</th>
<th>Set-III</th>
<th>Set-IV</th>
<th>Set-V</th>
<th>Set-VI</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NaOH charged, %</td>
<td>10.0</td>
<td>12.0</td>
<td>14.0</td>
<td>16.0</td>
<td>18.0</td>
<td>20.0</td>
</tr>
<tr>
<td>2</td>
<td>Bath ratio</td>
<td>1:5</td>
<td>1:5</td>
<td>1:5</td>
<td>1:5</td>
<td>1:5</td>
<td>1:5</td>
</tr>
<tr>
<td>3</td>
<td>Max cooking temp, °C</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>4</td>
<td>Temp raising time, h</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>5</td>
<td>Temp keeping time, h</td>
<td>3.0</td>
<td>3.0</td>
<td>3.0</td>
<td>3.0</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>6</td>
<td>Whole cycle H-factor</td>
<td>553.21</td>
<td>553.21</td>
<td>553.21</td>
<td>553.21</td>
<td>553.21</td>
<td>553.21</td>
</tr>
<tr>
<td>7</td>
<td>Screened pulp yield, %</td>
<td>40.46</td>
<td>43.41</td>
<td>44.73</td>
<td>43.66</td>
<td>42.86</td>
<td>38.92</td>
</tr>
<tr>
<td>8</td>
<td>Screening rejects, %</td>
<td>5.78</td>
<td>2.80</td>
<td>1.98</td>
<td>0.64</td>
<td>0.34</td>
<td>0.20</td>
</tr>
<tr>
<td>9</td>
<td>Unscreened pulp yield, %</td>
<td>46.24</td>
<td>46.21</td>
<td>46.71</td>
<td>44.30</td>
<td>43.23</td>
<td>39.12</td>
</tr>
<tr>
<td>10</td>
<td>Kappa no.</td>
<td>30.95</td>
<td>26.28</td>
<td>22.12</td>
<td>21.52</td>
<td>20.62</td>
<td>19.95</td>
</tr>
</tbody>
</table>
Table 3 — Effect of temperature during soda cooking of lignocellulosic residues of *C. martini*

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Cooking conditions and results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Set-VII</td>
</tr>
<tr>
<td>1</td>
<td>NaOH charged, %</td>
<td>14.0</td>
</tr>
<tr>
<td>2</td>
<td>Bath ratio</td>
<td>1:5</td>
</tr>
<tr>
<td>3</td>
<td>Max cooking temp, °C</td>
<td>140</td>
</tr>
<tr>
<td>4</td>
<td>Temp raising time, h</td>
<td>1.5</td>
</tr>
<tr>
<td>5</td>
<td>Temp keeping time, h</td>
<td>3.0</td>
</tr>
<tr>
<td>6</td>
<td>Whole cycle H-factor</td>
<td>223.42</td>
</tr>
<tr>
<td>7</td>
<td>Screened pulp yield, %</td>
<td>41.98</td>
</tr>
<tr>
<td>8</td>
<td>Screening rejects, %</td>
<td>3.78</td>
</tr>
<tr>
<td>9</td>
<td>Unscreened pulp yield, %</td>
<td>45.76</td>
</tr>
<tr>
<td>10</td>
<td>Kappa no.</td>
<td>28.42</td>
</tr>
</tbody>
</table>

Table 4 — Effect of time during soda cooking of lignocellulosic residues of *C. martini*

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Cooking conditions and results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Set-VII</td>
</tr>
<tr>
<td>1</td>
<td>NaOH charged, %</td>
<td>14.0</td>
</tr>
<tr>
<td>2</td>
<td>Bath ratio</td>
<td>1:5</td>
</tr>
<tr>
<td>3</td>
<td>Max cooking temp, °C</td>
<td>150</td>
</tr>
<tr>
<td>4</td>
<td>Temp raising time, h</td>
<td>1.5</td>
</tr>
<tr>
<td>5</td>
<td>Temp keeping time, h</td>
<td>2.0</td>
</tr>
<tr>
<td>6</td>
<td>Whole cycle H-factor</td>
<td>327.96</td>
</tr>
<tr>
<td>7</td>
<td>Screened pulp yield, %</td>
<td>40.56</td>
</tr>
<tr>
<td>8</td>
<td>Screening rejects, %</td>
<td>4.78</td>
</tr>
<tr>
<td>9</td>
<td>Unscreened pulp yield, %</td>
<td>45.34</td>
</tr>
<tr>
<td>10</td>
<td>Kappa no.</td>
<td>32.45</td>
</tr>
</tbody>
</table>

Table 5 — Effect of AQ during soda cooking of lignocellulosic residues of *C. martini*

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Cooking conditions and results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Set-XV</td>
</tr>
<tr>
<td>1</td>
<td>NaOH charged, %</td>
<td>14.0</td>
</tr>
<tr>
<td>2</td>
<td>AQ charged, %</td>
<td>0.0</td>
</tr>
<tr>
<td>3</td>
<td>Bath ratio</td>
<td>1:5</td>
</tr>
<tr>
<td>4</td>
<td>Max cooking temp, °C</td>
<td>150</td>
</tr>
<tr>
<td>5</td>
<td>Temp raising time, h</td>
<td>1.5</td>
</tr>
<tr>
<td>6</td>
<td>Temp keeping time, h</td>
<td>3.0</td>
</tr>
<tr>
<td>7</td>
<td>Whole cycle H-factor</td>
<td>553.21</td>
</tr>
<tr>
<td>8</td>
<td>Screened pulp yield, %</td>
<td>44.73</td>
</tr>
<tr>
<td>9</td>
<td>Screening rejects, %</td>
<td>1.98</td>
</tr>
<tr>
<td>10</td>
<td>Unscreened pulp yield, %</td>
<td>46.71</td>
</tr>
<tr>
<td>11</td>
<td>Kappa no.</td>
<td>22.12</td>
</tr>
</tbody>
</table>
Na$_2$O), bath ratio 1:5, maximum cooking temperature 150°C and maximum pulping time 3 h. The screened pulp yield drops to 44.23% further on increasing O$_2$ pressure, while kappa reduces to 18.50.

Table 7 shows the effect of AQ on pulp yield and kappa number at different O$_2$ pressures, while keeping other variables constant, i.e. bath ratio 1:5 and whole cycle H-factor 553.21. Figures 2 and 3 indicate that 0.1% AQ shows an improvement in pulp yield and kappa number at different O$_2$ pressure during alkali-O$_2$ delignification process. The relative screened pulp yield was improved by 2.25% in case of alkali-O$_2$ delignification process and the kappa number was reduced by 3.67 units than soda pulping.

In order to get target brightness soda pulp of lignocellulosic residues of C. martini was bleached by CEHH bleaching sequence at kappa factor 0.17. The brightness of pulp improves to 82.90% (ISO), opacity 80.50% and pulp viscosity 415 cm$^3$/g. The COD load at different stages is 48.2, 219.3, 17.6 and 10.2 kg/T, respectively. Table 9 indicates that pulp of lignocellulosic residues of C. martini shows no improvement in brightness and opacity but pulp viscosity slightly improves to 435 cm$^3$/g compared to soda pulp.

Table 6 — Effect of O$_2$ during alkali-O$_2$ cooking of lignocellulosic residues of C. martini

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Cooking conditions and results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Set-XIX</td>
</tr>
<tr>
<td>1</td>
<td>NaOH charged, %</td>
<td>14.0</td>
</tr>
<tr>
<td>2</td>
<td>O$_2$ pressure, kgf/cm$^2$</td>
<td>0.0</td>
</tr>
<tr>
<td>3</td>
<td>Bath ratio</td>
<td>1:5</td>
</tr>
<tr>
<td>4</td>
<td>Max cooking temp, °C</td>
<td>150</td>
</tr>
<tr>
<td>5</td>
<td>Temp raising time, h</td>
<td>1.5</td>
</tr>
<tr>
<td>6</td>
<td>Temp keeping time, h</td>
<td>3.0</td>
</tr>
<tr>
<td>7</td>
<td>Whole cycle H-factor</td>
<td>553.21</td>
</tr>
<tr>
<td>8</td>
<td>Screened pulp yield, %</td>
<td>44.73</td>
</tr>
<tr>
<td>9</td>
<td>Screening rejects, %</td>
<td>1.98</td>
</tr>
<tr>
<td>10</td>
<td>Unscreened pulp yield, %</td>
<td>46.71</td>
</tr>
<tr>
<td>11</td>
<td>Kappa no.</td>
<td>22.12</td>
</tr>
</tbody>
</table>

Table 7 — Effect of AQ at different O$_2$ pressures during alkali-O$_2$ cooking of lignocellulosic residues of C. martini

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Cooking conditions and results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Set-XXIII</td>
</tr>
<tr>
<td>1</td>
<td>NaOH charged, %</td>
<td>14.0</td>
</tr>
<tr>
<td>2</td>
<td>O$_2$ pressure, kgf/cm$^2$</td>
<td>0.0</td>
</tr>
<tr>
<td>3</td>
<td>AQ dose</td>
<td>0.1</td>
</tr>
<tr>
<td>4</td>
<td>Bath ratio</td>
<td>1:5</td>
</tr>
<tr>
<td>5</td>
<td>Max cooking temp, °C</td>
<td>150</td>
</tr>
<tr>
<td>6</td>
<td>Temp raising time, h</td>
<td>1.5</td>
</tr>
<tr>
<td>7</td>
<td>Temp keeping time, h</td>
<td>3.0</td>
</tr>
<tr>
<td>8</td>
<td>Whole cycle H-factor</td>
<td>480.20</td>
</tr>
<tr>
<td>9</td>
<td>Screened pulp yield, %</td>
<td>45.45</td>
</tr>
<tr>
<td>10</td>
<td>Screening rejects, %</td>
<td>1.02</td>
</tr>
<tr>
<td>11</td>
<td>Unscreened pulp yield, %</td>
<td>46.47</td>
</tr>
<tr>
<td>12</td>
<td>Kappa no.</td>
<td>21.00</td>
</tr>
</tbody>
</table>
Table 8 — Experimental conditions and results for CEHH bleaching for soda pulp of lignocellulosic residues of *C. martini* at optimum cooking condition

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>Bleaching conditions and results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
<td>E</td>
</tr>
<tr>
<td>1</td>
<td>Initial Kappa number</td>
<td>22.1</td>
</tr>
<tr>
<td>2</td>
<td>Kappa factor</td>
<td>0.18</td>
</tr>
<tr>
<td>3</td>
<td>Pulp consistency, %</td>
<td>3</td>
</tr>
<tr>
<td>4</td>
<td>Temperature, °C</td>
<td>Ambient</td>
</tr>
<tr>
<td>5</td>
<td>Chemical charged, %</td>
<td>3.98 as avail Cl₂</td>
</tr>
<tr>
<td>6</td>
<td>Retention time, h</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td>Initial pH</td>
<td>2.5</td>
</tr>
<tr>
<td>8</td>
<td>Final pH</td>
<td>1.8</td>
</tr>
<tr>
<td>9</td>
<td>Buffer, % NaOH of hypo</td>
<td>—</td>
</tr>
<tr>
<td>10</td>
<td>Sulphamic acid buffer, %</td>
<td>—</td>
</tr>
<tr>
<td>11</td>
<td>Brightness, % (Elrepho)</td>
<td>40.50</td>
</tr>
<tr>
<td>12</td>
<td>Opacity, %</td>
<td>—</td>
</tr>
<tr>
<td>13</td>
<td>Viscosity, cm³/g</td>
<td>776</td>
</tr>
<tr>
<td>14</td>
<td>COD, kg/T</td>
<td>48.2</td>
</tr>
<tr>
<td>15</td>
<td>Brightness at 35 °SR, % (Elrepho)</td>
<td>—</td>
</tr>
<tr>
<td>16</td>
<td>Opacity, %</td>
<td>—</td>
</tr>
</tbody>
</table>

Table 10 shows experimental conditions and results of CEHH bleaching of alkali-O₂ delignified pulp of lignocellulosic residues of *C. martini* at optimum cooking condition. The pulp brightness improves to 84.07% (ISO) and viscosity to 448 cm³/g. COD load at different stages reduces to 46.2, 19.3, 12.6 and 8.5 kg/t, respectively.

Table 11 shows the results of O₂ treated pulp of unbleached alkali-O₂-AQ delignified pulp of lignocellulosic residues of *C. martini*. The kappa number reduces to 49.29% and pulp brightness improves from 31.20 to 46.40% ISO.

Table 12 shows the results and conditions of OCEHH bleaching of alkali-O₂-AQ delignified pulp of lignocellulosic residues of *C. martini* in order to achieve target brightness of 85.95% (ISO). OCEHH bleaching shows an improvement in brightness 3.05%, viscosity 36 cm³/g. Figure 4 gives a comparison between viscosity and brightness for different pulping processes for pulp of waste of...
Table 9 — Experimental conditions and results for CEHH bleaching for soda-AQ pulp of lignocellulosic residues of *C. martini* at optimum cooking condition

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>C</th>
<th>E</th>
<th>H</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Initial kappa number</td>
<td>18.78</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td>Kappa factor</td>
<td>0.18</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>3</td>
<td>Pulp consistency, %</td>
<td>3</td>
<td>10</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>4</td>
<td>Temperature, °C</td>
<td>Ambient</td>
<td>65±2</td>
<td>45±2</td>
<td>45±2</td>
</tr>
<tr>
<td>5</td>
<td>Chemical charged</td>
<td>3.38 as avail Cl₂</td>
<td>3.0</td>
<td>1.5</td>
<td>0.6</td>
</tr>
<tr>
<td>6</td>
<td>Retention time, h</td>
<td>60</td>
<td>45</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>7</td>
<td>Buffer, % NaOH of hypo</td>
<td>—</td>
<td>—</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>Sulphamic acid buffer, %</td>
<td>—</td>
<td>—</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>9</td>
<td>Initial pH</td>
<td>2.6</td>
<td>11.5</td>
<td>11.6</td>
<td>11.4</td>
</tr>
<tr>
<td>10</td>
<td>Final pH</td>
<td>1.9</td>
<td>10.8</td>
<td>9.8</td>
<td>10.2</td>
</tr>
<tr>
<td>11</td>
<td>Brightness, % (Elrepho)</td>
<td>39.40</td>
<td>50.16</td>
<td>78.50</td>
<td>82.85</td>
</tr>
<tr>
<td>12</td>
<td>Opacity, %</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>80.30</td>
</tr>
<tr>
<td>13</td>
<td>Viscosity, cm³/g</td>
<td>789</td>
<td>486</td>
<td>458</td>
<td>435</td>
</tr>
<tr>
<td>14</td>
<td>COD, kg/T</td>
<td>49.2</td>
<td>20.3</td>
<td>14.6</td>
<td>10.2</td>
</tr>
<tr>
<td>15</td>
<td>Brightness at 35 °SR, % (Elrepho)</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>81.50</td>
</tr>
<tr>
<td>16</td>
<td>Opacity</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>79.97</td>
</tr>
</tbody>
</table>

Table 10 — Experimental conditions and results of CEHH bleaching for alkali-O₂ delignified pulp of lignocellulosic residues of *C. martini* at optimum cooking condition

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>C</th>
<th>E</th>
<th>H</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Initial kappa number</td>
<td>16.98</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td>Kappa factor</td>
<td>0.18</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>3</td>
<td>Pulp consistency, %</td>
<td>3</td>
<td>10</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>4</td>
<td>Temperature, °C</td>
<td>Ambient</td>
<td>65±2</td>
<td>45±2</td>
<td>45±2</td>
</tr>
<tr>
<td>5</td>
<td>Chemical charged</td>
<td>3.06 as avail Cl₂</td>
<td>3.0</td>
<td>1.5</td>
<td>0.6</td>
</tr>
<tr>
<td>6</td>
<td>Retention time, h</td>
<td>60</td>
<td>45</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>7</td>
<td>Buffer, % NaOH of hypo</td>
<td>—</td>
<td>—</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>Sulphamic acid buffer, %</td>
<td>—</td>
<td>—</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>9</td>
<td>Initial pH</td>
<td>2.8</td>
<td>11.6</td>
<td>11.6</td>
<td>115</td>
</tr>
<tr>
<td>10</td>
<td>Final pH</td>
<td>1.8</td>
<td>10.5</td>
<td>9.6</td>
<td>9.8</td>
</tr>
<tr>
<td>11</td>
<td>Brightness, % (Elrepho)</td>
<td>44.60</td>
<td>55.16</td>
<td>82.30</td>
<td>84.07</td>
</tr>
<tr>
<td>12</td>
<td>Opacity, %</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>81.43</td>
</tr>
<tr>
<td>13</td>
<td>Viscosity, cm³/g</td>
<td>881</td>
<td>496</td>
<td>462</td>
<td>448</td>
</tr>
<tr>
<td>14</td>
<td>COD, kg/T</td>
<td>46.2</td>
<td>19.3</td>
<td>12.6</td>
<td>8.5</td>
</tr>
<tr>
<td>15</td>
<td>Brightness at 35 °SR, % (Elrepho)</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>82.52</td>
</tr>
<tr>
<td>16</td>
<td>Opacity</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>80.72</td>
</tr>
</tbody>
</table>
C. martini when bleached with CEHH and OCEHH bleaching sequences. Alkali–O_2-AQ pulp shows higher brightness with an improvement in pulp viscosity.

**Conclusions**

Lignocarbohydrates residue of C. martini left after steam distillation can successfully be used for the production of chemical grade pulp. The distillation process makes the C. martini suitable for alkali-O_2 delignification process by bridging over the problem of mass transfer of O_2, which is far less than C. martini without distillation. The lignocellulosic

---

**Table 11 — Oxygen treatment of unbleached alkali-O_2-AQ delignified pulp of lignocellulosic residues of C. martini**

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Parameters</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Unbleached pulp kappa number.</td>
<td>17.35</td>
</tr>
<tr>
<td>2</td>
<td>Unbleached pulp brightness, % ISO</td>
<td>31.20</td>
</tr>
<tr>
<td>3</td>
<td>Unbleached pulp viscosity, cm³/g</td>
<td>778</td>
</tr>
<tr>
<td>4</td>
<td>NaOH,% (o.d. pulp basis)</td>
<td>2.0</td>
</tr>
<tr>
<td>5</td>
<td>Oxygen pressure, kgf/cm²</td>
<td>5.0</td>
</tr>
<tr>
<td>6</td>
<td>Consistency, %</td>
<td>12</td>
</tr>
<tr>
<td>7</td>
<td>Retention time, min</td>
<td>60</td>
</tr>
<tr>
<td>8</td>
<td>Kappa number</td>
<td>8.8</td>
</tr>
<tr>
<td>9</td>
<td>Kappa reduction, %</td>
<td>49.29</td>
</tr>
<tr>
<td>10</td>
<td>Viscosity, cm³/g</td>
<td>718</td>
</tr>
<tr>
<td>11</td>
<td>Brightness, % ISO</td>
<td>46.40</td>
</tr>
<tr>
<td>12</td>
<td>Pulp yield, % ISO</td>
<td>97.5</td>
</tr>
</tbody>
</table>

**Table 12 — Experimental conditions and results of CEHH bleaching for alkali-O_2-AQ delignified pulp of lignocellulosic residues of C. martini at optimum cooking condition**

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Particulars</th>
<th>C</th>
<th>E</th>
<th>H</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Initial kappa number</td>
<td>8.8</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td>Kappa factor</td>
<td>0.18</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>3</td>
<td>Pulp consistency, %</td>
<td>3</td>
<td>10</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>4</td>
<td>Temperature, °C</td>
<td>Ambient</td>
<td>65±2</td>
<td>45±2</td>
<td>45±2</td>
</tr>
<tr>
<td>5</td>
<td>Chemical charged</td>
<td>1.59 as avail Cl₂</td>
<td>3.0</td>
<td>1.5</td>
<td>0.6</td>
</tr>
<tr>
<td>6</td>
<td>Retention time, h</td>
<td>60</td>
<td>45</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>7</td>
<td>Buffer, % NaOH of hypo</td>
<td>—</td>
<td>—</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>Sulphamic acid buffer, %</td>
<td>—</td>
<td>—</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>9</td>
<td>Initial pH</td>
<td>2.6</td>
<td>11.6</td>
<td>11.6</td>
<td>11.4</td>
</tr>
<tr>
<td>10</td>
<td>Final pH</td>
<td>2.0</td>
<td>10.2</td>
<td>9.8</td>
<td>10.2</td>
</tr>
<tr>
<td>11</td>
<td>Brightness, % (Elrepho)</td>
<td>45.10</td>
<td>55.46</td>
<td>83.30</td>
<td>85.95</td>
</tr>
<tr>
<td>12</td>
<td>Opacity, %</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>82.02</td>
</tr>
<tr>
<td>13</td>
<td>Viscosity, cm³/g</td>
<td>889</td>
<td>492</td>
<td>468</td>
<td>451</td>
</tr>
<tr>
<td>14</td>
<td>COD, kg/T</td>
<td>45.8</td>
<td>18.1</td>
<td>10.2</td>
<td>8.3</td>
</tr>
<tr>
<td>15</td>
<td>Brightness at 35 °SR, % (Elrepho)</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>83.48</td>
</tr>
<tr>
<td>16</td>
<td>Opacity</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>81.02</td>
</tr>
</tbody>
</table>

---

**Fig. 4 — Effect of pulping processes on brightness and viscosity of C. martini**

---
residues of *C. martini* produce 44.73% pulp of kappa number 22.12 by soda pulping process at H-factor 553.21, maximum cooking time 3 h, maximum cooking temperature 150°C, alkali dose 14% (as Na₂O) and bath ratio 1:5. The addition of 0.1% AQ improves pulp yield by 0.72% and reduces kappa number by 1.12 units. A 5 kg/cm² O₂ pressure improves pulp yield by 1.07% and reduces kappa number by 1.65 units during alkali-O₂ pulping of lignocellulosic residues of *C. martini*. Further, addition of 0.1% AQ improves pulp yield by 2.25% and reduces kappa number by 3.64 units than soda pulping. Front end extended delignification of lignocellulosic residues of *C. martini* is carried out with O₂ pressure 5 kg/cm², which increases pulp brightness from 31.20 to 46.40% ISO with a kappa number reduction of 49.29%. CEHH bleaching of lignocellulosic residues of *C. martini* produces pulp of brightness 82.90, 82.85, and 84.07% ISO with soda, soda-AQ and alkali-O₂ pulping processes at kappa factor 0.17, respectively. The alkali-O₂-AQ pulp produces pulp of brightness of 85.95% (ISO) by OCEHH bleaching sequence.

**References**