CHEMIMECHANICAL PULPING OF COTTON STALKS

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ABSTRACT

Egyptian cotton stalks were pulped in a blender, using cold soda and alkaline peroxide chemimechanical (APMP) processes. Overall yields for both processes were low, but comparable to values found for semichemical pulping in the literature. Strength values were lower than for chemical pulping but comparable to literature values for semichemical pulping. For the cold soda process, the alkali concentration during soaking decreased the yield for both the soaking and refining stages, but it had little effect on fiber development during and sheet properties after secondary refining. A similar effect was found for soaking time. Higher soaking temperature increased the freeness level at a given secondary refining time and improved tear strength, but it had no effect on tensile or burst strength. An increase in primary refining time caused a decrease in tear strength. Pulps produced from the APMP process were 50% brighter and slightly weaker than for the cold soda process, and they required significantly less secondary refining to reach a given freeness level. An increase in the concentration of alkali and peroxide caused a decrease in both soaked and refined yields. Increased chemical concentration slightly reduced the secondary refining time required to reach a given freeness level, but it had no effect on strength at a given freeness. An increase in the alkali/peroxide ratio during soaking caused a decrease in screened rejects and an increase in screened yield. Higher ratios resulted in slightly higher freeness values at a given secondary refining time, and they also produced slightly higher tensile and moderately lower tear strength. Brightness was decreased as the ratio increased.

INTRODUCTION

Nonwood raw materials account for 5-7% of the worldwide production of paper pulp (1). The production of this type of pulp has increased more rapidly than that of pulp from wood in the last two decades, by a factor of about two in Latin America and three in Africa and the Middle East (2). In countries such as Egypt which have no forests, cotton stalk is one of the agricultural residues available for pulping and papermaking. Cotton stalks are available in large quantities in several parts of the world. In Egypt, 1.9 million tons are produced annually. The stalks contain a substantial percentage of pith cells which, together with the dark-colored outer bark, create problems in both pulping and papermaking processes.

Two major problems have hindered commercial utilization of cotton stalks for production of pulp and paper. The first problem is that of transportation of the raw materials, which are bulky in nature. This problem can be solved to some degree with densification techniques. The second problem is that of debarking, which is made difficult due to the fact that cotton stalks are thinly-branched, bushy plants. In the last few years, many efforts have been directed towards the exploitation of lignocellulosic materials by new pulping systems with two main objectives in mind: (1) to avoid or reduce the use of chemical agents which, alone or interacting with the raw materials, could produce a serious environmental impact, such as in the case of sulfate (kraft) and sulfite methods; 2) to achieve a selective separation of the main components (cellulose, hemicellulose, and lignin) in a non-degraded form, with subsequent processing according to different methods (3).

High-yield chemimechanical pulping can meet these objectives to some degree. Processes such as the cold soda process can be used to produce pulps with fair papermaking properties, but without the capital and operating costs associated with a full chemical process. Modifications to this process, such as the Alkaline Peroxide Mechanical Pulping Process (APMP), can produce pulps of even higher quality while maintaining overall process simplicity and pulp yield.

Cold soda pulping involves the treatment of the raw material with sodium hydroxide at atmospheric conditions (up to 100 C), followed by mechanical defibratation or refining (4). The action of the alkali causes uneven swelling in the
fiber wall, inducing stresses that cause the primary and outer secondary fiber walls to be shed during mechanical refining. The resulting exposed S2 layer has the potential for good interfiber bonding. Only a small amount of lignin is removed in the process, with the bulk of the yield loss associated with extractives and hemicellulose. For wood, cold soda pulps have better strength properties compared to groundwood, while retaining the advantages of good opacity and printability.

The use of alkali and hydrogen peroxide together, called the Alkaline Peroxide Mechanical Pulping (APMP) process, has been demonstrated to produced pulps of high quality and brightness from a variety of nonwood materials (5).

Few references exist in the literature regarding the use of cold soda or other chemimechanical pulping processes on cotton stalk.

METHODS AND MATERIALS

Raw Material

The cotton stalks used in this work were of the type Egyptian Giza 75. The stalks were cut into pieces about 2.5-4.0 cm in length by hand. No debarking was used. Samples of both stalks were milled in a Wiley mill, using a 0.4 mm screen. The milled samples were subjected to standard compositional analysis, according to TAPPI standards.

Cold Soda Pulping

All chemimechanical pulping was carried out at bench scale. To permit the soaking of small quantities of stalk without loss of fines and other small components, soaking was carried out by placing 100 OD grams of stalk material into a nylon bag with a mesh size of approximately 50. The bag was then submerged in a 10-liter controlled-temperature water bath filled with a solution of sodium hydroxide of the appropriate concentration. Distilled, deionized water was used to make up all solution. At the end of the desired reaction time, the bag was withdrawn from the bath and rinsed thoroughly with distilled water to remove any surface chemical. The bag was then placed in an oven at 105 C for at least 12 hours. The net dry weight of the contents was then obtained. In this manner, the total yield for the chemical treatment was determined.

Primary refining was carried out in a large commercial blender (Waring Model 37BL19) equipped with a 4-liter container. The rotor of the blender was as supplied by the manufacturer. The soaking process described previously was repeated. At the end of soaking for the second trial, the contents of the bag were drained briefly and then transferred to the blender container. Based on the yield numbers obtained from the first trial, enough distilled water at 25 C was added to the container to bring the consistency to 2.5 %. The blender was then energized on the lowest setting. A clamp-on ammeter was attached to the power cord for the blender, and motor load readings were taken every 5 seconds and totalized as the refining progressed. The blender refining was stopped when the total net specific refining input energy (the blender motor no load value was previously determined and subtracted from each recorded load value) reached a target value of 900 kW*Hr/MT. For most of the trials, the total refining time was approximately 12 minutes. The temperature of the contents before and after refining was measured. The pH of the blender contents was measured after refining.

One trial was conducted with a longer primary refining time (17.5 minutes versus 12 minutes).

The resulting coarse pulp was screened in a Voith laboratory flat screen equipped with 0.010-inch slots. The screen rejects were oven-dried at 105 C and weighed. The accepts were washed thoroughly with cold tap water in a screen box with a 150-mesh wire. The washed accepts were centrifuged, fluffed, and stored in a sealed plastic bag for at least 12 hours. The total weight and consistency were then determined.

Secondary refining was carried out in a smaller commercial blender (Waring Model 700). The blender was equipped with a 1-liter container with a cooling water jacket. The screen accepts were diluted to 1 % consistency with cold tap water. The blender was then energized at the only available speed for varying times, with cold tap water run through the jacket to prevent heating of the contents. After refining, the pulp was tested for Canadian
Standard Freeness and then made into standard handsheets (1.2 g OD). After conditioning, the handsheets were tested for various properties, according to TAPPI methods.

The variables for these trials were sodium hydroxide concentration, soaking temperature, and soaking time.

**Alkaline Peroxide Mechanical Pulping**

Alkaline peroxide mechanical pulping (APMP) was carried out using a mesh bag soaking and blender refining technique similar to that described for cold soda pulping. For each trial, the cotton stalks were first soaked for 30 minutes at 70 °C in a solution of the appropriate concentration of sodium hydroxide, along with magnesium sulfate and DTPA at a fixed concentration of 0.5 gpl. At the end of the soaking time, the hydrogen peroxide was added, and the soaking was continued for another 90 minutes. Distilled, deionized water was used to make up all solutions.

The variables for these trials were sodium hydroxide and hydrogen peroxide concentration, including the ratio of one to the other.

**RESULT AND DISCUSSION**

**Raw Material Analysis**

The results of chemical analysis of both types of cotton stalk are shown in Table 1, along with some values from the literature.

<table>
<thead>
<tr>
<th></th>
<th>Giza 75 Cotton Stalks</th>
<th>Literature (6-8)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ash, %</td>
<td>1.84</td>
<td>3.0 – 4.36</td>
</tr>
<tr>
<td>Silica, g/kg</td>
<td>4.92</td>
<td>0.6</td>
</tr>
<tr>
<td>Hot water solubility, %</td>
<td>10.77</td>
<td>6.6 – 13.7</td>
</tr>
<tr>
<td>Alcohol-benzene solubility, %</td>
<td>2.93</td>
<td>5.5 – 7.85</td>
</tr>
<tr>
<td>1 % NaOH solubility, %</td>
<td>39.60</td>
<td>34.85 – 44.6</td>
</tr>
<tr>
<td>Alpha cellulose, %</td>
<td>48.83</td>
<td>43.7</td>
</tr>
<tr>
<td>Pentosans, %</td>
<td>17.45</td>
<td>12.4 – 20.1</td>
</tr>
<tr>
<td>Lignin %</td>
<td>22.50</td>
<td>19.5 – 21.0</td>
</tr>
<tr>
<td>Cellulose/lignin ratio</td>
<td>2.17</td>
<td>---</td>
</tr>
<tr>
<td>Pith, %</td>
<td>4.17</td>
<td>---</td>
</tr>
<tr>
<td>Bark, %</td>
<td>30.81</td>
<td>---</td>
</tr>
</tbody>
</table>

Given the wide range of values found in the literature (6-8), the values obtained were considered reasonable. The high value obtained for NaOH solubility indicated that the yields for alkaline chemimechanical pulping would be lower than those for wood.

**Cold Soda Chemimechanical Pulping**

The first trials were intended to examine the effect of increasing sodium hydroxide concentration at a fixed soaking time of 2 hours and a fixed temperature of 70 °C. Table 2 contains the data from these trials (data for an NaOH concentration of 80 gpl were omitted due to errors discovered in the measurement).

<table>
<thead>
<tr>
<th>NaOH Conc, gpl</th>
<th>Soaked Yield, %</th>
<th>Temperature Before Refining, °C</th>
<th>Temperature After Refining, °C</th>
<th>pH After Refining</th>
<th>Total Refined Yield, %</th>
<th>Screened Refined Yield, %</th>
<th>Screened Rejects, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>85.07</td>
<td>29</td>
<td>60</td>
<td>11.9</td>
<td>55.66</td>
<td>53.75</td>
<td>1.91</td>
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<tr>
<td>120</td>
<td>82.80</td>
<td>29</td>
<td>61</td>
<td>12.4</td>
<td>52.33</td>
<td>50.93</td>
<td>1.40</td>
</tr>
<tr>
<td>160</td>
<td>80.11</td>
<td>29</td>
<td>58</td>
<td>12.4</td>
<td>50.93</td>
<td>47.01</td>
<td>3.92</td>
</tr>
</tbody>
</table>
The yield losses after soaking were 15-20%, significant but far lower than predicted by the NaOH solubility numbers for the milled material. Clearly, accessibility of the stalk to the alkali was lower than for the meal. As expected, an increase in the concentration of the alkali during soaking decreased all measured yield values.

Primary refining lowered the total yield after soaking by another 30%, indicating that a significant quantity of material was being either solubilized or converted into fines capable of passing through the 200-mesh screen used to catch the screen accepts. The total yield values were only about 15% higher than those obtained when the same stalks were pulped chemically in a previous trial (9). The values obtained were similar to those found for semichemical pulping (10-11).

Handsheet strength and optical properties of the pulps after secondary refining are shown in Table 3. As shown in Figure 1, freeness development during secondary refining was almost identical for all the pulps, showing that the concentration of alkali during soaking had no effect. As shown in Figures 2 and 3, tensile and burst strength were also unaffected by alkali concentration. Figure 4, however, shows that the higher alkali concentrations produced lower tear strength values.

<table>
<thead>
<tr>
<th>NaOH Conc, gpl</th>
<th>Refining Time, min</th>
<th>Freeness, CSF</th>
<th>Basis Weight, g/m²</th>
<th>Breaking Length, km</th>
<th>Burst Index, kPa*m²/g</th>
<th>Tear Index, mN*m²/g</th>
<th>Brightness, % ISO</th>
<th>TAPPI Opacity, %</th>
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<tr>
<td>40</td>
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<td>415</td>
<td>65.0</td>
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<td>11.0</td>
<td>38.3</td>
<td>93.3</td>
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<td>40</td>
<td>75</td>
<td>421</td>
<td>65.9</td>
<td>3.93</td>
<td>1.88</td>
<td>10.1</td>
<td>38.4</td>
<td>93.2</td>
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<tr>
<td>80</td>
<td>25</td>
<td>419</td>
<td>63.8</td>
<td>5.91</td>
<td>3.03</td>
<td>5.19</td>
<td>39.3</td>
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<td>75</td>
<td>283</td>
<td>63.3</td>
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<td>1.47</td>
<td>10.6</td>
<td>38.8</td>
<td>93.1</td>
</tr>
<tr>
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<td>25</td>
<td>452</td>
<td>64.1</td>
<td>4.40</td>
<td>3.88</td>
<td>10.3</td>
<td>39.2</td>
<td>93.4</td>
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<td>428</td>
<td>65.5</td>
<td>4.39</td>
<td>2.45</td>
<td>10.2</td>
<td>39.4</td>
<td>93.1</td>
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<tr>
<td>160</td>
<td>25</td>
<td>452</td>
<td>66.4</td>
<td>4.08</td>
<td>1.49</td>
<td>5.10</td>
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<td>80</td>
<td>142</td>
<td>63.4</td>
<td>5.16</td>
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<td>39.1</td>
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<td>62.2</td>
<td>3.68</td>
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<td>190</td>
<td>61.1</td>
<td>4.37</td>
<td>1.99</td>
<td>6.80</td>
<td>38.7</td>
<td>94.4</td>
<td></td>
</tr>
</tbody>
</table>

Opacity values for all the pulps were quite high, which was considered surprising in light of the low yields obtained. Cold soda pulps are normally valued for their high opacity, due in part to the amount of lignin left in the cell wall.

When the same stalks were pulped chemically (9), tensile and burst strength values were significantly greater than for the cold soda pulps. The tear strength values for the chemical pulps fell in between the cold soda tear values for low and high alkali concentrations.

The strength values obtained for the cold soda pulps were very similar to those obtained in the literature for the sulfite (12) and soda (10-11) semichemical pulping of cotton stalk.

The second series of trials were intended to show the effect of soaking time at a fixed alkali concentration of 80 gpl and a fixed temperature of 70°C. Table 4 shows the data obtained from these trials.

As expected, longer soaking times caused higher yield losses for the soaking step and lower total yields after the primary refining step. Higher soaking times sharply decreased the amount of screened rejects, but the overall dissolution of material still resulted in lower screened yields. The yield values obtained were similar to those obtained for the first series of trials.
Table 4. Data for soaking of cotton stalks at 70°C and 80 gpl alkali concentration, varying soaking times

<table>
<thead>
<tr>
<th>Soaking Time, hours</th>
<th>Soaked Yield, %</th>
<th>Temperature Before Refining, C</th>
<th>Temperature After Refining, C</th>
<th>pH After Refining</th>
<th>Total Refined Yield, %</th>
<th>Screened Refined Yield, %</th>
<th>Screened Rejects, %</th>
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<tr>
<td>1</td>
<td>83.10</td>
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<td>56</td>
<td>12.2</td>
<td>57.17</td>
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<td>4.52</td>
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<tr>
<td>2</td>
<td>78.09</td>
<td>28</td>
<td>59</td>
<td>12.3</td>
<td>56.92</td>
<td>54.77</td>
<td>2.15</td>
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<tr>
<td>3</td>
<td>77.41</td>
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<td>12.2</td>
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<td>12.3</td>
<td>51.22</td>
<td>50.44</td>
<td>0.78</td>
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</table>

Handsheet strength and optical properties for the second series of trials are shown in Table 5.

As shown in Figure 5, the soaking time had no significant effect on freeness development during secondary refining. Figures 6 and 7 show a similar effect for tensile and burst strength, while Figure 8 shows that the longest soaking time did produce lower tear strength values.

Table 5. Handsheet properties after secondary refining for cotton stalk soaked for various times

<table>
<thead>
<tr>
<th>Soaking Time, hours</th>
<th>Refining Time, min</th>
<th>Freeness, CSF</th>
<th>Basis Weight, g/m²</th>
<th>Breaking Length, km</th>
<th>Burst Index, kPa*m²/g</th>
<th>Tear Index, mN*m²/g</th>
<th>Brightness, % ISO</th>
<th>TAPPI Opacity, %</th>
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<td>1</td>
<td>20</td>
<td>446</td>
<td>65.2</td>
<td>3.62</td>
<td>1.22</td>
<td>9.67</td>
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<td>324</td>
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<td>36.8</td>
<td>94.6</td>
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<td>4.40</td>
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<td>5.73</td>
<td>37.4</td>
<td>96.0</td>
<td></td>
</tr>
</tbody>
</table>

The third series of trials was intended to show the effect of soaking temperature at a fixed alkali concentration of 80 gpl and a fixed time of 2 hours. Table 6 shows the data obtained from these trials.

Table 6. Data for soaking of cotton stalks at 2 hours soaking time and 80 gpl alkali concentration, varying soaking temperatures

<table>
<thead>
<tr>
<th>Soaking Temp, C</th>
<th>Soaked Yield, %</th>
<th>Temperature Before Refining, C</th>
<th>Temperature After Refining, C</th>
<th>pH After Refining</th>
<th>Total Refined Yield, %</th>
<th>Screened Refined Yield, %</th>
<th>Screened Rejects, %</th>
</tr>
</thead>
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<td>90</td>
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<td>12.3</td>
<td>50.54</td>
<td>48.14</td>
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</table>

As expected, increasing the soaking temperature reduced all the measured yield values.

Handsheet strength and optical properties for the third series of trials are shown in Table 7.
Table 7. Handsheet properties after secondary refining for cotton stalk soaked at various temperatures

<table>
<thead>
<tr>
<th>Soaking Temp, °C</th>
<th>Refining Time, min</th>
<th>Freeness, CSF</th>
<th>Basis Weight, g/m²</th>
<th>Breaking Length, km</th>
<th>Burst Index, kPa*m²/g</th>
<th>Tear Index, mN*m²/g</th>
<th>Brightness, % ISO</th>
<th>TAPPI Opacity, %</th>
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</thead>
<tbody>
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<td>425</td>
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<td>4.80</td>
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<td>91.8</td>
</tr>
<tr>
<td>30</td>
<td>200</td>
<td>62.1</td>
<td>4.25</td>
<td>2.20</td>
<td>5.20</td>
<td>44.2</td>
<td>92.8</td>
<td></td>
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<tr>
<td>72</td>
<td>74</td>
<td>61.6</td>
<td>4.82</td>
<td>2.67</td>
<td>5.42</td>
<td>44.7</td>
<td>93.6</td>
<td></td>
</tr>
<tr>
<td>70</td>
<td>25</td>
<td>419</td>
<td>63.3</td>
<td>3.64</td>
<td>1.47</td>
<td>10.6</td>
<td>38.8</td>
<td>93.1</td>
</tr>
<tr>
<td>39</td>
<td>283</td>
<td>64.1</td>
<td>4.40</td>
<td>3.88</td>
<td>10.3</td>
<td>39.2</td>
<td>93.4</td>
<td></td>
</tr>
<tr>
<td>75</td>
<td>150</td>
<td>60.5</td>
<td>4.39</td>
<td>2.45</td>
<td>10.2</td>
<td>39.4</td>
<td>93.1</td>
<td></td>
</tr>
<tr>
<td>90</td>
<td>410</td>
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<td>3.70</td>
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<td>246</td>
<td>62.3</td>
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<td>2.14</td>
<td>7.07</td>
<td>35.1</td>
<td>95.2</td>
<td></td>
</tr>
<tr>
<td>85</td>
<td>138</td>
<td>61.9</td>
<td>4.94</td>
<td>2.67</td>
<td>6.33</td>
<td>37.3</td>
<td>95.6</td>
<td></td>
</tr>
</tbody>
</table>

Unlike the other variables, temperature had a significant effect on the refining response. As shown in Figure 9, increasing the soaking temperature produced a higher freeness at a given secondary refining time. This effect was most likely due to improved fiber separation and less damaged material generation at the higher temperatures, which in turn was probably due to improved softening and alkali consumption.

In general, the effect of temperature on freeness development did not translate into similar effects on strength. As shown in Figures 10 and 11, tensile and burst strength were not dependent on soaking temperature. This result was surprising, since it was expected that higher temperatures would improve fiber development and thus paper strength. As shown in Figure 12, tear strength values for the lower two temperatures were practically identical. At 70°C, however, the values rose to a significantly higher level. When the temperature was raised further to 90°C, the tear strength values decreased to a level slightly higher than for the lower temperatures. This behavior, with an apparent maximum tear strength at a soaking temperature of 70°C, could not be explained. It was possible that some testing error occurred for the 70°C data, and re-testing was recommended.

In a final simple trial, it was desired to understand the effect of an increased primary refining time. Two samples of stalk were soaked in 80 gpl NaOH for 2 hours at 70°C. One sample was refined for 11 minutes and 43 seconds, the time required to achieve the primary stage energy target of 900 kW*Hr/MT. The other was refined for a longer time of 17 minutes and 34 seconds. Both pulps were then subjected to secondary refining. Table 8 contains the results.

Table 8. Handsheet properties after secondary refining, two different primary refining times (soaking in 80 gpl NaOH for 2 hours at 70°C)

<table>
<thead>
<tr>
<th>Primary Refining Time min:sec</th>
<th>Refining Time, min</th>
<th>Freeness, CSF</th>
<th>Basis Weight, g/m²</th>
<th>Breaking Length, km</th>
<th>Burst Index, kPa*m²/g</th>
<th>Tear Index, mN*m²/g</th>
<th>Brightness, % ISO</th>
<th>TAPPI Opacity, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>11:43</td>
<td>25</td>
<td>419</td>
<td>63.3</td>
<td>3.64</td>
<td>1.47</td>
<td>10.6</td>
<td>38.8</td>
<td>93.1</td>
</tr>
<tr>
<td>39</td>
<td>283</td>
<td>64.1</td>
<td>4.40</td>
<td>3.88</td>
<td>10.3</td>
<td>39.2</td>
<td>93.4</td>
<td></td>
</tr>
<tr>
<td>75</td>
<td>150</td>
<td>60.5</td>
<td>4.39</td>
<td>2.45</td>
<td>10.2</td>
<td>39.4</td>
<td>93.1</td>
<td></td>
</tr>
<tr>
<td>17:34</td>
<td>15</td>
<td>405</td>
<td>65.1</td>
<td>4.22</td>
<td>1.68</td>
<td>5.26</td>
<td>36.0</td>
<td>95.3</td>
</tr>
<tr>
<td>25</td>
<td>276</td>
<td>65.5</td>
<td>4.69</td>
<td>1.95</td>
<td>5.24</td>
<td>36.5</td>
<td>95.4</td>
<td></td>
</tr>
<tr>
<td>48</td>
<td>112</td>
<td>64.2</td>
<td>5.82</td>
<td>2.99</td>
<td>5.61</td>
<td>36.4</td>
<td>95.6</td>
<td></td>
</tr>
</tbody>
</table>

Figures 13 and 14 show that the primary refining time had no significant effect on either tensile or burst strength development during secondary refining (the one high burst value was considered an anomaly). For tear strength,
however, the longer primary refining time did produce uniformly-lower values, as shown in Figure 15. This effect was most likely due to fiber shortening during the increased time under the harsh primary refining conditions.

Alkaline Peroxide Mechanical Pulping

The first series of trials was intended to show the effect of alkali and peroxide concentration at a fixed soaking temperature of 70 C, a fixed soaking total soaking time of 2 hours, and a fixed 1:1 ratio of alkali to peroxide concentrations. Table 9 shows the data obtained from these trials.

Table 9. Data for soaking of cotton stalk in alkaline peroxide solutions, varying alkali and peroxide concentrations (soaking for 2 hours at 70 C)

<table>
<thead>
<tr>
<th>NaOH, gpl</th>
<th>H2O2, gpl</th>
<th>Soaked Yield, %</th>
<th>Screened Yield, %</th>
<th>Screen Rejects, %</th>
<th>Temp Before Refining C</th>
<th>Temp After Refining C</th>
<th>pH After Refining</th>
<th>H2O2 Consumed, gpl</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.0</td>
<td>12.0</td>
<td>90.10</td>
<td>54.73</td>
<td>8.33</td>
<td>26</td>
<td>65</td>
<td>10.5</td>
<td>5.54</td>
</tr>
<tr>
<td>14.0</td>
<td>14.0</td>
<td>90.84</td>
<td>55.76</td>
<td>7.00</td>
<td>28</td>
<td>72</td>
<td>10.7</td>
<td>6.44</td>
</tr>
<tr>
<td>16.5</td>
<td>16.5</td>
<td>88.52</td>
<td>56.50</td>
<td>5.07</td>
<td>27</td>
<td>71</td>
<td>10.4</td>
<td>7.39</td>
</tr>
</tbody>
</table>

In general, soaked yield decreased with increasing chemical concentration. Screened yield increased with increasing chemical concentration, due to a reduction in screened rejects. The screened yield values were very similar to those obtained for cold soda pulping, and again the values were considered quite low for a chemimechanical process.

The handsheet data are shown in Table 10. Freeness development is plotted versus secondary refining time in Figure 16. While the higher chemical charges did produce higher freeness values at a given refining time, overall the differences were considered insignificant. It was evident that the presence of peroxide caused a significant decrease in required refining times, as compared to the cold soda process. This decrease was attributed to improved fiber separation and development.

Figure 17 shows tensile strength as a function of freeness. Chemical concentration had no significant effect on tensile strength development. Data for the cold soda trials are shown for comparison. Over the range of freeness values obtained, the cold soda process produced slightly higher tensile strength. The same results were obtained for burst and tear strength, as shown in Figures 18 and 19.
The brightening power of the peroxide was evident. Brightness values for the APMP pulps were, on average, 48% higher than for the cold soda pulps.

The second series of trials was intended to determine the effect of the alkali/peroxide ratio on the pulping performance and sheet properties. The peroxide concentration was kept at a constant value of 18 gpl, and the NaOH concentration was varied at 10.8, 14.4, and 23.4 gpl levels, which translated into ratios of 0.57, 0.8, and 1.3, respectively. The data are shown in Table 11.

Table 11. Data for soaking of cotton stalk in alkaline peroxide solutions, varying alkali/peroxide ratios (70°C, total soaking time 2 hours)

<table>
<thead>
<tr>
<th>NaOH, gpl</th>
<th>H2O2, gpl</th>
<th>Soaked Yield, %</th>
<th>Screened Yield, %</th>
<th>Screen Rejects, %</th>
<th>Temp Before Refining, °C</th>
<th>Temp After Refining, °C</th>
<th>pH After Refining</th>
<th>H2O2 Consumed, gpl</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.8</td>
<td>18</td>
<td>87.97</td>
<td>56.33</td>
<td>2.01</td>
<td>28</td>
<td>69</td>
<td>10.9</td>
<td>14.4</td>
</tr>
<tr>
<td>14.4</td>
<td>18</td>
<td>89.49</td>
<td>58.65</td>
<td>1.87</td>
<td>28</td>
<td>70</td>
<td>10.9</td>
<td>14.0</td>
</tr>
<tr>
<td>23.4</td>
<td>18</td>
<td>92.93</td>
<td>58.51</td>
<td>0.38</td>
<td>26</td>
<td>64</td>
<td>11.6</td>
<td>16.6</td>
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</tbody>
</table>

An increase in the alkali/peroxide ratio appeared to increase the total yield of material. This result was contrary to expectation, since a higher alkali charge should have resulted in the loss of more material. It is suspected that the water soaking time was inadequate prior to oven-drying of the initial (yield) trial samples, permitting absorbed chemicals to remain with the dried material. An increased ratio reduced screen rejects and therefore improved screened yield. Overall, a higher alkali charge caused more peroxide to be consumed.

Handsheet strength properties for the pulp are shown in Table 12. Freeness development during secondary refining is shown in Figure 20. The higher alkali/peroxide ratios produced slightly higher freeness values at a given refining time.

Table 12. Handsheet properties after secondary refining for cotton stalk soaked at different alkali/peroxide ratios (soaking for 2 hours at 70°C)

<table>
<thead>
<tr>
<th>NaOH/ Peroxide Ratio</th>
<th>Refining Time, min</th>
<th>Freeness, CSF</th>
<th>Basis Weight, g/m²</th>
<th>Breaking Length, km</th>
<th>Burst Index, kPa*m²/g</th>
<th>Tear Index, mN*m²/g</th>
<th>Brightness, % ISO</th>
<th>TAPPI Opacity, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.57</td>
<td>7.5</td>
<td>347</td>
<td>66.6</td>
<td>3.67</td>
<td>1.84</td>
<td>4.62</td>
<td>64.4</td>
<td>87.7</td>
</tr>
<tr>
<td>12</td>
<td>246</td>
<td>66.7</td>
<td>4.07</td>
<td>2.16</td>
<td>5.22</td>
<td>65.5</td>
<td>88.6</td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>135</td>
<td>68.5</td>
<td>5.69</td>
<td>2.57</td>
<td>5.08</td>
<td>64.1</td>
<td>88.4</td>
<td></td>
</tr>
<tr>
<td>0.80</td>
<td>6</td>
<td>394</td>
<td>65.3</td>
<td>3.87</td>
<td>1.43</td>
<td>4.37</td>
<td>61.0</td>
<td>89.1</td>
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<tr>
<td>19.5</td>
<td>230</td>
<td>64.1</td>
<td>4.31</td>
<td>2.57</td>
<td>4.71</td>
<td>60.0</td>
<td>88.7</td>
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</tr>
<tr>
<td>35</td>
<td>95</td>
<td>64.0</td>
<td>4.50</td>
<td>2.39</td>
<td>4.88</td>
<td>60.7</td>
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<tr>
<td>1.3</td>
<td>3.5</td>
<td>369</td>
<td>63.7</td>
<td>4.44</td>
<td>1.71</td>
<td>5.04</td>
<td>58.2</td>
<td>86.4</td>
</tr>
<tr>
<td>9</td>
<td>265</td>
<td>64.4</td>
<td>5.09</td>
<td>2.22</td>
<td>5.17</td>
<td>58.3</td>
<td>86.8</td>
<td></td>
</tr>
<tr>
<td>24</td>
<td>133</td>
<td>63.3</td>
<td>6.29</td>
<td>2.84</td>
<td>5.27</td>
<td>58.4</td>
<td>86.9</td>
<td></td>
</tr>
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</table>

Tensile, burst, and tear strength are plotted versus freeness in Figures 21-23. The highest ratio of 1.3 produced slightly higher tensile strength and moderately lower tear strength. Burst strength was not affected by the ratio.

Increasing the alkali/peroxide ratio caused a decrease in handsheet brightness.
CONCLUSIONS

Cold Soda Pulping

1. Total yield values were 80-85 % after alkali soaking and 51-56 % after refining. About half of the yield loss occurred during the primary refining step. Increasing alkali concentration during soaking decreased the soaked and refined yield values.
2. The concentration of alkali during the soaking step had no effect on freeness development during secondary refining or on tensile and burst strength at a given freeness level. Higher alkali concentration during soaking did cause lower tear strength values.
3. Strength values were comparable to those in the literature for semi-chemical pulping of cotton stalk. Tensile and burst strength values were significantly lower than for the same stalks were pulped chemically, but tear strength values were comparable.
4. Longer soaking times at a given alkali concentration caused lower soaked yield, lower refined yield, and lower screened yield.
5. Longer soaking times at a given alkali concentration had no effect on freeness development during secondary refining or on tensile and burst strength at a given freeness level. The highest soaking time produced lower tear strength values.
6. Higher soaking temperatures at a fixed alkali concentration and soaking time decreased the soaked yield, refined yield, and screened yield.
7. Higher soaking temperatures at a fixed alkali concentration and soaking time produced higher freeness values at a given secondary refining time. Tensile and burst strength were not affected by soaking temperature, but tear strength was improved at higher temperatures.
8. An increase in the primary refining time had no effect on tensile and burst strength, but it did cause a decrease in tear strength.

Alkaline Peroxide Mechanical Pulping

1. Soaked yield and screened rejects decreased with increasing concentration of alkali and peroxide. Screened yield improved with increasing chemical concentration.
2. A higher chemical concentration caused a slight increase in the freeness at a given refining time. Secondary refining times to reach a given freeness level were significantly lower than for the cold soda process.
3. Chemical concentration had no significant effect on tensile, tear, or burst strength at a given level of freeness. All strength properties were higher at a given level freeness for the cold soda process.
4. Brightness values for the APMP pulps were about 50 % higher than for the cold soda pulps.
5. An increase in the alkali/peroxide ratio caused a decrease in screened rejects and an increase in screened yield. More peroxide was consumed at the higher alkali concentrations.
6. The higher alkali/peroxide ratios produced slightly higher freeness values at a given secondary refining time.
7. The highest alkali/peroxide ratio produced slightly higher tear strength and moderately lower tear strength. Burst strength was unaffected by the ratio.
8. An increase in the alkali/peroxide ratio caused a decrease in brightness.

REFERENCES


Figure 1. Freeness development during cold soda pulping of cotton stalks, varying alkali concentration

Figure 2. Tensile strength versus freeness for cotton stalk cold soda pulping, varying alkali concentration

Figure 3. Burst strength versus freeness for cotton stalk cold soda pulping, varying alkali concentration
Figure 4. Tear strength versus freeness for cotton stalk cold soda pulping, varying alkali concentration

Figure 5. Freeness development during cold soda pulping of cotton stalks, varying soaking times

Figure 6. Tensile strength versus freeness for cotton stalk cold soda pulping, varying soaking times
Figure 7. Burst strength versus freeness for cotton stalk cold soda pulping, varying soaking times

Figure 8. Tear strength versus freeness for cotton stalk cold soda pulping, varying soaking times

Figure 9. Freeness development during cold soda pulping of cotton stalks, varying soaking temperatures
Figure 10. Tensile strength versus freeness for cotton stalk cold soda pulping, varying temperatures

Figure 11. Burst strength versus freeness for cotton stalk cold soda pulping, varying soaking temperatures

Figure 12. Tear strength versus freeness for cotton stalk cold soda pulping, varying soaking temperatures
Figure 13. Tensile strength versus freeness for cotton stalk cold soda pulping, different primary refining times

Figure 14. Burst strength versus freeness for cotton stalk cold soda pulping, different primary refining times

Figure 15. Tear strength versus freeness for cotton stalk cold soda pulping, different primary refining times
Figure 16. Freeness development during alkaline peroxide pulping of cotton stalks, varying alkali and peroxide concentrations

Figure 17. Tensile strength versus freeness for alkaline peroxide pulping of cotton stalks, varying alkali and peroxide concentrations

Figure 18. Burst strength versus freeness for alkaline peroxide pulping of cotton stalks, varying alkali and peroxide concentrations
Figure 19. Tear strength versus freeness for alkaline peroxide pulping of cotton stalks, varying alkali and peroxide concentrations

![Tear strength versus freeness graph](image)

Figure 20. Freeness development during alkaline peroxide pulping of cotton stalks, varying alkali/peroxide ratios

![Freeness development graph](image)

Figure 21. Tensile strength versus freeness for alkaline peroxide pulping of cotton stalks, varying alkali/peroxide ratios

![Tensile strength versus freeness graph](image)
Figure 22. Burst strength versus freeness for alkaline peroxide pulping of cotton stalks, varying alkali/peroxide ratios

Figure 23. Tear strength versus freeness for alkaline peroxide pulping of cotton stalks, varying alkali/peroxide ratios