PRODUCTION OF HIGH-YIELD PULPS
FROM ASPEN BY MILD TREATMENTS
WITH SODIUM HYDROXIDE

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PRODUCTION OF HIGH-YIELD PULPS FROM ASPEN
BY MILD TREATMENTS WITH SODIUM HYDROXIDE

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Summary

In experiments at the U. S. Forest Products Laboratory, aspen (Populus tremuloides) chips were given mild treatments with sodium hydroxide solutions at atmospheric pressure and then mechanically fiberized to produce pulps in the yield range of 82 to 95 percent. Treatments were made with caustic solutions in concentrations of 24 and 63 grams per liter. Treating time varied from 15 minutes to 5 hours and temperature from 25° to 90° C.

The optimum treating condition from the standpoints of the treating variables, pulp yield, and pulp strength was a 2-hour treatment at 25° C. with a sodium hydroxide solution having a concentration of 24 grams per liter. Chemical consumption for this treatment was about 5.5 percent sodium hydroxide, based on the weight of the wood, and a pulp yield of about 91 percent. Pulp made under these conditions was converted into corrugating board that compared favorably with commercial hardwood semichemical corrugating board in strength properties.

The effects of the individual treatment variables are discussed.

2Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

Report No. 1774
Introduction

High-yield pulping has been receiving increasing attention in recent years in the endeavor to produce pulps in yields near those of groundwood pulps but with improved properties and wider uses. Its objective is closely tied in with those of wood conservation and the utilization of wood wastes and little-used species. The purpose of the work reported here was to obtain information on high-yield pulping in the range of pulp yields of 85 to 95 percent. This range is higher than that usually understood for semichemical pulping, and also differs basically from the latter in that partial delignification is preferably avoided. The present report is a continuation of work reported recently on steam and water cooking of wood chips (2). It gives the results of a systematic investigation of certain treatment variables involved in the experimental production of high-yield pulps from quaking aspen by a process consisting of a mild treatment of the chips with caustic soda, followed by their mechanical fiberization.

Although sodium hydroxide is the oldest of the commercial pulping agents, investigations reported in the literature on its use have mostly referred to the process of soda pulping, in which conditions are designed for a substantially complete removal of the lignin from the wood. In one study of the use of sodium hydroxide as a semichemical pulping agent, however, it was found that the pulps were much weaker than those made with sulfate pulping liquor (1).

Experimental Work

Peeled air-dry quaking aspen was used in these experiments. The wood was made into standard 5/8-inch chips for pulping. The chips had a moisture content of approximately 10 percent.

Except for one experiment, the caustic soda treatments were conducted in a 0.5-cubic foot, steam-jacketed, tumbling autoclave. One treatment was made in a 14-cubic foot, stainless-steel-lined, tumbling digester. After treatment in the autoclave, the chips were fiberized in an 8-inch diameter, single-rotating-disk attrition mill. The treated chips obtained in larger-scale experiments were fiberized in a 36-inch double-rotating-disk attrition mill. This pulp was converted into corrugating board on the Laboratory's 12-inch Fourdrinier paper machine.

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The digestions were conducted with a liquor-to-wood ratio of 5 to 1. For those experiments made at higher than room temperature, the liquor was heated to the treating temperature in the autoclave before the chips were added, thus eliminating the usual period of temperature rise with chips present. The treated chips were fiberized immediately after the caustic treatment after washing. The pulp yields were determined on the basis of the fiberized pulps. The pulps were beaten for strength development in a Valley beater by TAPPI standard methods and the 115-pound (weight per ream of 500 sheets, each 25 by 40 inches) test sheets made from the pulps were tested for strength by the standard methods. The pulps were also analyzed for their content of lignin, holocellulose, and alpha cellulose by standard methods.

In one experiment, the 5/8-inch chips were partially disintegrated before being given the caustic soda treatment, by passing them through the 8-inch attrition mill equipped with coarse breaker plates. This action broke down the chips mainly in the lengthwise direction and reduced them mostly to pieces having a maximum dimension of 1/8 inch in the longitudinal and radial directions. There was present, however, a small percentage of fragmented material and small pieces less than 5/8 inch in the grain direction. The disintegrated chips with a moisture content of 50 percent were then treated with a sodium hydroxide solution of 22.3 grams per liter concentration, and with a liquor-to-wood ratio of 6.3 to 1, for 2 hours at 25° C. The treated material was fiberized and handled as described above.

Discussion of Results

Effect of Treatment Variables

The three independent treatment variables, time, temperature, and alkali concentration, had considerable effect on the chemical consumed, the yield of pulp, and the strength properties and chemical composition of the pulps.

Chemical Consumption

The effects of the treatment variables on sodium hydroxide consumption are shown in figure 1. Chemical consumption increased with increases in time, temperature, and alkali concentration. The rate of chemical consumption was very rapid at the start of the treatment; 50 percent or
more of the total consumption in the 5-hour treating period was obtained in the first 15 minutes of that period. The rate was, however, very slow toward the end of the treating period. The lowered rate probably means that most of the hemicelluloses, which were readily soluble under the particular conditions of treatment, had by this time been removed from the wood.

The rate of chemical consumption increased with temperature at any given chemical consumption within the range of the experiments. This is obvious from the slope of tangents drawn on the consumption curves in figure 1 at a constant chemical consumption. By application of the Arrhenius law, the rate was found to be increased by approximately 175 percent for a temperature increase of 10°C.

The initial chemical consumption and the rate of consumption also increased with the stronger treating solution, as shown in figure 1.

Pulp Yield

The effects of the treatment variables on pulp yield are shown in figure 2. The rate of pulping, as measured in terms of reduction in pulp yield, was rapid during the first stages of the treatment and became very slow toward the end of the treatment. This again may have meant that the readily soluble material had been removed in the early stages of the treatments. The trend of the yield curves with respect to treating conditions paralleled that of chemical consumption with relation to treating conditions shown in figure 1. The relation between pulp yield and chemical consumption shown by figure 3 departed a little from a linear trend and indicated a chemical consumption which, for a given reduction in yield, was higher in the range above about 88-percent yield than in the range below that yield. This relation was independent of temperature and chemical concentration. A similar close relationship between pulp yield and chemical consumption in conventional soda pulping has also been reported (3).

Chemical Composition of the Pulps

The effect of the alkali treatment, as measured by the sodium hydroxide consumption, on the main chemical composition of the pulps is shown in figure 4. Neither the lignin nor the alpha cellulose content of the pulps, apparently, was affected by the treatments within the range of the experiments. The holocellulose content of the pulps, on the other hand,
decreased linearly with increase in alkali consumption. The scatter of points about the average line shows that this relationship is independent of temperature and alkali concentration. This meant that the loss in yield was due mainly to a loss in hemicellulose.

Strength Properties

Because the bulkiness of the pulps tended to increase with increasing yield and to decrease with the higher treating temperature, it was difficult to compare the strength properties of the more bulky pulps made in the highest yields at 25° C. with those of the denser ones made in the lowest yields at 90° C. on the basis of the same physical processing (measured by freeness) and equal test-sheet density. The strength data were therefore plotted on both bases, and the resulting curves are shown in figures 5 and 6. The relatively sharp maximums in bursting and tensile strength for the pulps made in yields near 91 or 92 percent with the weaker treating solution of 24.1 grams per liter, as shown by the curves plotted on an equal density basis (fig. 5), may be partly due to bonding caused by the hydration produced by the beating of these pulps. On the other hand, when the variations in these strength properties are considered on the basis of equal freeness (fig. 6), a constant value is approached at a yield near 90 percent, even though the test sheets from the higher-yield pulps are much lower in density than those from the lower-yield pulps. In either case, a maximum in bursting and tensile strength appears to have been reached in pulps made in yields near 90 percent and no appreciable increase in these strength properties was associated with yields below this point. The tearing strength of the pulps also appeared to approach a maximum at this yield, although the results are not as outstanding as for bursting and tensile strength. A possible explanation for these maximums is that this point also corresponds to the point of maximum swelling of the wood under the conditions used. At this point, the swelling might have weakened the fiber bonds in the wood, thereby permitting a relatively easy mechanical separation of the fibers without the physical disintegration obtained in a similar fiberizing of raw wood. Although hemicelluloses generally are related to pulp strength, their decrease with decreasing yield below 90 percent did not affect pulp strength within the range of the experiments.

These results indicate that the strength properties of the pulps made with the lower concentration of treating alkali were independent of the temperature of the treatment. The use of the higher concentration of 63 grams per liter for the treating solution caused a displacement of the maximums of the strength-yield relation toward a lower yield of roughly 88 percent.
This displacement may have resulted because a greater loss of hemicelluloses occurred before the pulp attained maximum swelling than with the less concentrated treating solution.

From considerations of pulp yields and strength properties within the range of the experiments, it is indicated that pulp No. 735 (table 1) would be optimum for this process. This pulp was produced in a yield near 90 percent with a sodium hydroxide liquor of about 25 grams per liter and a treating time of 2 hours at 25°C. Although the important factor of energy for fiberizing the treated chips was not investigated in this study, the observation was made that the chips treated at 60° and 90° C. were more easily fiberized than those treated at 25°C. Obviously, the optimum combination of treating and fiberizing conditions and chemical, steam, and energy consumption must be determined in order to obtain the best results from the process.

The pulps made by the mild alkaline treatment under the above optimum conditions were 25 to 50 percent stronger than the strongest pulps made in a previous investigation with a steam cooking procedure (2). The pulp made under the mildest treating condition in these experiments (15 minutes at 25°C. with a caustic solution concentration of 24.1 grams per liter) was produced in a yield approximately the same as that obtained by fiberizing raw wood. The pulp made by the procedure described was, however, much stronger than that from raw wood (pulp No. 731 and "raw wood," table 1).

**Effect of Chip Size**

The standard 5/8-inch chips, which are approximately 5/8 inch in the grain direction, 1/8 inch thick, and 1/2 to 1 inch wide, were not completely penetrated when treated for as long as 5 hours at 25°C. with caustic soda solution in concentrations of 24.1 and 63.0 grams per liter. Small areas in the centers of a large number of the treated chips were bright and dry. Complete penetration was obtained, however, with the material that had been given a preliminary disintegration in the attrition mill (No. 812, table 1). The chemical consumption was higher than that for practically the same treatment with the standard chips (No. 735), and there was a corresponding decrease in pulp yield. The rate of chemical consumption was thus higher with the reduced-size material and was nearly the same as that obtained with the standard chips treated with the more concentrated alkali solution of 63 grams per liter (No. 806, table 1). The strength properties of the pulp from the reduced-size chips...
were also much the same as those of the pulp made in the same yield (No. 805) with the more concentrated treating liquor. Apparently reducing the size of the chips made the hemicelluloses more available for solution. The results of the experiment with the reduced-size chips indicated that the lack of complete penetration of the standard chips was not serious with respect to strength.

It was observed that, after treatment, the reduced-size chips were more easily fiberized than standard chips comparably treated. Thus it is possible that there would be a saving in total energy consumption by preliminary partial disintegration, since the energy consumed in this step is probably also low.

Application of the Process to Corrugating Board

The treating conditions considered to be optimum produced pulp of a quality in the range of that used for corrugating board. These conditions were applied in a larger-scale experiment with the purpose of producing a sufficient amount of pulp for a run of this kind of board on the experimental paper machine. The treatment was made with chips of standard size (No. 5446, table 1). The fiberizing of the treated chips in a machine of commercial size required about 26 horsepower-days per ton of pulp to produce a pulp with a freeness of 450 cubic centimeters (Canadian Standard). This energy value is well within the range of commercial practice. The yield of pulp was slightly higher than that obtained under the same conditions in the small-scale experiments, although the chemical consumption was much the same (Nos. 5446 and 735, table 1). The pulp strength was also in line with that of the small-scale pulp.

The properties of the experimental corrugating board are compared in table 2 with average test values for commercial hardwood semichemical corrugating board. The experimental board compared favorably in almost every respect with the commercial board. It was also relatively light in color and had a brightness value of 46.1 percent. A corrugating test under commercial conditions, conversion of the material into container board, and tests on the containers would, of course, be needed for a complete evaluation of the material. The interesting point is that this high-yield pulp, made under relatively mild conditions at room temperature and pressure, has shown possibilities of being used in products normally made from lower-yield materials or from long-fibered woods. Undoubtedly, there are also other uses than corrugating board for pulps made by this process.
Literature Cited

(1) Bray, M. W., and Martin, J. S.  

1949. TAPPI 32, No. 10:440-8, October.

(3) Wise, L. F.  
### Table 1. Conditions and results of mild treatments of aspen chips with sodium hydroxide

<table>
<thead>
<tr>
<th>Digestion/Treatment No.</th>
<th>Temperature</th>
<th>Caustic consumed</th>
<th>Pulp yield</th>
<th>Strength Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>% of</td>
<td>of</td>
<td>Lignin</td>
<td>Hemicellulose</td>
</tr>
<tr>
<td>731</td>
<td>25</td>
<td>1/4</td>
<td>25.3</td>
<td>2.90</td>
</tr>
<tr>
<td>732</td>
<td>25</td>
<td>1/2</td>
<td>33.4</td>
<td>3.83</td>
</tr>
<tr>
<td>733</td>
<td>25</td>
<td>1</td>
<td>40.3</td>
<td>4.63</td>
</tr>
<tr>
<td>734</td>
<td>25</td>
<td>1-1/2</td>
<td>44.5</td>
<td>5.16</td>
</tr>
<tr>
<td>735</td>
<td>25</td>
<td>2</td>
<td>47.4</td>
<td>5.46</td>
</tr>
</tbody>
</table>

**Treatments with 24.1 grams per liter solution:***

| 5,178                  | 25   | 5   | 48.7 | 5.50 | 89.8 | 16.0 | 70.4 | 47.7 | 0.52 | 0.73 | 4,000 | 380 | 0.28 | 0.88 | 3,000 | 0.48 |
| 807                    | 60   | 1/4 | 40.0 | 4.67 | 91.5 | 17.3 | 70.3 | 49.3 | 0.62 | 0.80 | 4,100 | 380 | 0.28 | 0.88 | 3,000 | 0.48 |
| 808                    | 60   | 1/2 | 46.0 | 5.29 | 90.5 | 17.1 | 70.0 | 48.5 | 0.62 | 0.81 | 4,950 | 550 | 0.40 | 0.96 | 3,800 | 0.53 |
| 809                    | 60   | 1   | 52.0 | 5.66 | 90.2 | 17.2 | 69.6 | 48.9 | 0.67 | 0.95 | 4,500 | 650 | 0.41 | 0.99 | 4,300 | 0.58 |
| 810                    | 60   | 2   | 55.2 | 6.34 | 90.2 | 17.2 | 69.6 | 48.9 | 0.67 | 0.95 | 4,000 | 730 | 0.35 | 0.93 | 4,200 | 0.66 |

**Treatments with 63.0 grams per liter solution:***

| 811                    | 90   | 1/4 | 55.5 | 5.28 | 89.9 | 16.0 | 70.3 | 48.3 | 0.52 | 0.71 | 3,850 | 380 | 0.28 | 0.88 | 3,000 | 0.48 |
| 812                    | 90   | 1   | 65.9 | 7.04 | 84.3 | 17.0 | 63.6 | 48.8 | 0.68 | 0.98 | 2,800 | 740 | 0.34 | 0.93 | 3,600 | 0.54 |
| 813                    | 90   | 2   | 69.9 | 8.04 | 84.3 | 17.0 | 63.6 | 48.8 | 0.68 | 0.98 | 2,800 | 740 | 0.34 | 0.93 | 3,600 | 0.54 |

**Treatment with disintegrated chips and 22.3 grams per liter solution:***

| 812                    | 25   | 2   | 46.2 | 6.36 | 88.7 | 17.7 | 78.6 | 48.6 | 0.52 | 0.85 | 4,900 | 580 | 0.37 | 0.88 | 3,500 | 0.52 |

**Fiberized, untreated chips (raw wood):***

| 95.0                    | 17.7 | 78.6 | 48.6 | 0.046 | 0.15 | 0.50 | 0.32 |

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*Ream size of 500 sheets - 25 by 40 inches.

*Chemical ratios of 11.5, 13.8, and 30.0 percent of wood for treatments with 24.1, 22.3, and 63.0 grams per liter solutions of sodium hydroxide, respectively.

*213-cubic foot digester used.

*Data by interpolation.

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*2 M 84055 p
Table 2. --Properties of experimental boards made of aspen treated with caustic soda, as compared with a commercial corrugating board

<table>
<thead>
<tr>
<th>Properties of corrugating board:</th>
<th>Caustic-treated aspen</th>
<th>Commercial hardwood corrugating board</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulp yield, percent:</td>
<td>91.6</td>
<td></td>
</tr>
<tr>
<td>Weight, lb. per 1,000 sq. ft.:</td>
<td>25.4</td>
<td>27.4</td>
</tr>
<tr>
<td>Caliper, 0.001 in.:</td>
<td>8.3</td>
<td>10.0</td>
</tr>
<tr>
<td>Density, gm. per cc.:</td>
<td>.59</td>
<td>.53</td>
</tr>
<tr>
<td>Bursting strength:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mullen, pts.:</td>
<td>45.0</td>
<td>36.3</td>
</tr>
<tr>
<td>Unit, pts. per lb. per ream:</td>
<td>.51</td>
<td>.38</td>
</tr>
<tr>
<td>Tearing strength, gm. per lb. per ream:</td>
<td>.97</td>
<td>1.00</td>
</tr>
<tr>
<td>Tensile strength, lb. per in. width:</td>
<td>40.0</td>
<td>30.1</td>
</tr>
<tr>
<td>Stretch, percent:</td>
<td>2.1</td>
<td>2.0</td>
</tr>
<tr>
<td>Compression resistance, lb.:</td>
<td>50.9</td>
<td>49.6</td>
</tr>
</tbody>
</table>

1—Machine run 3177.
2—Average of five commercial hardwood semichemical corrugating boards.
3—25 x 40 - 500 ream.
Figure 1. -- Effect of time and temperature on sodium hydroxide consumption in the pulping of aspen chips.
Figure 2. --Effect of time and temperature on yield of pulp from aspen treated with sodium hydroxide.
Figure 3. --Relation between yield of pulp from aspen and consumption of sodium hydroxide under several treating conditions.
Figure 4. -- Relation between chemical composition of aspen pulps and consumption of sodium hydroxide under several treating conditions.
Figure 5. -- Relation between pulp strength and yield of aspen pulp made by mild sodium hydroxide treatments at different temperatures and alkali concentrations.
Figure 6. --Relation of yield and strength properties of aspen pulps made by sodium hydroxide treatment, when at 700 milliliters freeness (Schopper-Riegler).