SULPHATE PULPING OF SOUTHERN YELLOW PINES
Part 2: Effect of Growth Variables on Yield and Pulp Quality
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UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE
FOREST PRODUCTS LABORATORY
Madison, Wisconsin
In Cooperation with the University of Wisconsin
Introduction

In an earlier report of similar title it was shown that pulp quality is closely related to the conditions of growth, and that the quality of southern pine is quite variable. In fact wider variations sometimes exist between portions of wood from a single tree than are otherwise noticeable between trees belonging to different species, but grown under like conditions.

Obviously, confirmation of these findings requires numerous checks and re-checks, involving the taking of a large amount of data, examination of woods from different localities and of different species. The work reported here is of this nature, and to a certain degree repetitive. Two species of pines were obtained from a locality differing somewhat in climatic conditions from the source of samples which had been used in previous studies. These specimens were examined by methods developed in the earlier work. In addition, special attention was given to the possibility of using top-cut material in the production of strong kraft pulps.

Selection and Evaluation of the Raw Material

The wood, consisting of fourteen 4-foot top, middle, and butt bolts from one shortleaf (Pinus echinata) and two loblolly (Pinus taeda) pine trees and also two top bolts from each of these two species, was selected as representative of the material commonly used in Arkansas for pulping. A description of the several samples of raw material, together with their physical and chemical data is given in table 1. The two top bolts, C-1 and D-1, were taken from trees, the lower portions of which were saw logs for lumber production. No limb-wood was included in any of the material. The physical and chemical properties of the 4-foot bolts taken from different heights in the tree were determined according to standard methods at the Forest Products Laboratory.

1 Published in Southern Pulp & Paper Journal, November 1939.
The physical data, recorded in table 1, in general agree with the trends previously obtained for several members of the southern yellow pine group, namely, with increase in elevation in the tree there is an increase in springwood content and a decrease in the density of the wood. The total and alpha-cellulose contents in trees A and B noticeably decrease with height in tree, whereas the lignin content increases slightly. In tree E, however, the results are irregular with no particular trend indicated. Except for bolts C-1 and D-1, which showed evidence of decay, the chemical analysis of the logs indicated freedom from wood-destroying organisms.

In considering single trees, the decreased density of the top logs would result in lower pulp yields on a volume basis. On an oven-dry weight basis, the yields of pulp would also probably be slightly lower in the top wood because of the lower cellulose content. Chipping losses would probably be greater with top wood because of the increased number of knots in that part of the tree. The relation of the position of the log in the tree to its pulping characteristics, however, is probably secondary to the physical and chemical properties of the wood.

Pulping Experiments

Preparation of the Material

The 4-foot bolts were hand-peeled and chipped in a two-knife, 48-inch in diameter chipper, set to produce a 5/8-inch chip while running at a speed of 500 revolutions per minute. The chips were screened on a shaker screen, where the large knots, oversized material, and sawdust were rejected. All good chips from a definite location in the tree were thoroughly mixed and stored in air-tight cans placed in a room maintained at 40°F. and 93 percent relative humidity. Previous to pulping, the chips were sampled for moisture content.

Digestion Procedure

Thirty-three digestions were made on the two species in spherical, steam-jacketed autoclaves of 3.7 gallons capacity. All digestions were made in triplicate. The 5/8-inch chips were cooked with a total of 20 percent of their oven-dry weight of caustic soda and sodium sulphide in the ratio of two parts by weight of the former to one part of the latter. Thus the total sodium oxide content of the cooking liquors was 15.63 percent of the weight of oven-dry wood charged and the total active or "effective" alkalinity expressed as caustic soda was 16.75 percent. This latter figure is the sum of the caustic soda added as such and that resulting from the hydrolysis of the sodium sulphide according to the equation,

$$Na_2S + H_2O \rightarrow NaOH + NaSH$$

The total initial concentration of the cooking liquors, including the moisture in the chips, was 50 grams per liter. The volume of the cooking liquors was 48 gallons per hundred pounds of oven-dry wood.

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A linear temperature-pressure schedule was followed in the digestions, allowing 1-1/2 hours to raise the temperature of the autoclave from that of the room to the maximum 170° C. Previous penetration experiments have shown that this length of time is adequate to secure sufficient penetration of the cooking liquors to effect even cooking throughout.

After the chips had been held in the three digesters at the maximum temperature for an equal period (1-1/2 hours), the jacket-steam pressure was released and the autoclaves rapidly cooled to atmospheric pressure. The pulped chips were dumped on wire screens, washed to remove superficial spent liquors, and later broken up in a small vat by rapid agitation. The pulps were washed to remove the remaining spent liquors and then screened through a 12-cut flat screen. The screened pulps were pressed to approximately 33 percent oven dryness, sampled for moisture, and weighed in closed vessels for the determination of yield. So as to have ample material for duplicate physical tests, the three similar digestions were combined by again agitating in water suspension. The combined mass was again pressed to 33 percent oven dryness and stored in airtight cans until tested.

All calculations of chemicals and yields are based on the calculated weight of the oven-dry chips charged. Bleach consumption and the results of chemical analysis, on the other hand, are calculated on the weight of the oven-dry pulp tested.

Method of Testing the Pulps

The strength properties of the pulps were determined on test sheets prepared after processing 360-gram (oven-dry basis) portions in 23 liters of water for definite intervals in a 1-1/2 pound beater provided with a 6,500-gram weight on the bedplate. Bleaching tests were made on the pulps by treating 15-gram samples by a single-stage method with known amounts of calcium hypochlorite solutions. The color of the bleached and unbleached pulps was determined by comparison with a standard white magnesia block in an Ives tint photometer.

Samples of the unbleached pulps were analyzed for their total cellulose, alpha-cellulose, lignin, pentosan contents, and their chlorine absorbing power according to standard methods adopted by the Forest Products Laboratory.

The yield and physical and chemical data resulting from these pulping experiments are recorded in table 2.

Results and Discussion

Yield of Pulp

The average yield of screened pulp on a weight basis for the three trees examined was 77.7 percent, which indicates about the same degree of cooking as that employed in the previously published experiments. The yield of screened pulp from the two loblolly pine trees averaged 48.3 percent for the entire trunk of the codominant tree (A) and 48.1 percent for the suppressed tree (E),
while that of the codominant shortleaf pine tree (B) was 1.4 percent lower than the average of the two shortleaf pine trees or 46.8 percent. The yield for the top log of the dominant shortleaf pine tree D-1 was 1.7 percent higher than that of the top log of the codominant tree (B) of the same species, while the top log of the dominant loblolly pine tree (C-1) was 1.4 percent lower than that of the top log of the codominant tree (A). In general, the average pulp yields parallel fairly closely the total cellulose content of the individual trees.

As previously reported, the butt logs of all three trees gave the highest percentage of screenings. Considering yields of pulp on a volume basis, which takes into account the increase in density of the wood in progressing from the top downward, the butt logs gave from 14.3 to 38.9 percent higher yields of screened pulp than was obtained from the top logs. For example, the top logs of tree B gave 10.8 pounds, while the butt logs gave 15.0 pounds of screened pulp per cubic foot of solid peeled wood. A point of further interest is that the highest yield of screened pulp per cubic foot of solid barked wood was obtained from the suppressed tree (E) of loblolly pine. This tree also showed the least variation in volume yield of screened pulp at different locations in its trunk.

Pulp Quality -- Chemical

Under the cooking conditions employed, the cellulose content of the pulps ranged between 91.3 and 93.3 percent, averaging approximately 92.2 percent, which again indicates the same degree of pulping as that in the previous report covering the four major species of southern yellow pine. The percentage of cellulose in the individual pulps showed but little variation from this mean value. The alpha-cellulose content of the pulps averaged 76.8 percent and also showed small deviations from this average figure. The pulps from the top bolts contained a slightly lower percentage of total and alpha cellulose than those from the lower portions of the trees. The lignin and pentosan content and their chlorine numbers were about the same for all the pulps irrespective of location of wood from which they were made.

The bleach requirement of the pulps obtained both by chlorine absorption and by a single-stage bleaching test and Ives tint-photometer readings indicate approximately the same degree of cooking as that employed previously for comparison of the four major species of southern yellow pine.

The Roe chlorine absorption of the pulps averaged 5.3 grams of chlorine per hundred grams of oven-dry pulp and their whiteness (Ives blue reading) when bleached in a single-stage operation with 25 percent of their oven-dry weight of calcium hypochlorite in solution averaged approximately 51 parts blue. Although individual pulps deviated from these values by 1 gram of chlorine and 5 parts in the Ives blue reading, no particular relation with springwood content could be noticed.
The strength development curves for the sulphate pulps made from top, middle, and butt portions of the several trees and also those from the top logs only of the two additional saw-log trees are recorded in figures 1 and 2. These curves, illustrating the change in the strength properties of the pulps as their freenesses (Schopper-Riegler) decreased with time of beating in a standard test beater, show the same trends as previously reported, in that considerable differences were found in the strength properties of the pulps prepared from the top, middle, and butt portions of a single tree. As the ratio of springwood to summerwood increases in proceeding from the butt to the top of a tree, the bursting and tensile strength of the sulphate pulps show considerable tendency to increase. On the other hand, as the percentage of summerwood increases in going downward in the tree trunk the tearing strength and the bulkiness of the pulps show a tendency to increase. Although the causes of these phenomena were explained, in general, in the previous article it should be emphasized that the thin-walled, cylinder-like fibers collapse and flatten out into ribbons upon drying, thus making a greater surface of contact between the fibers in a sheet of paper than do the thick-walled needle-like summerwood fibers. Since bursting and tensile strength properties of paper are to a greater extent proportional to surface adhesion than to the ultimate strength of the individual fibers, these particular properties show an increase as the percentage of thin-walled collapsible springwood fibers is increased.

On the other hand, in measuring the resistance of paper to tear, should many of the individual fibers be broken across their longitudinal axis, wall thickness of individual fibers would be presumed to play a more important role in this particular strength property of paper than surface contact. Consequently, with an increase in the percentage of the thick-walled summerwood component, resistance to tear would be expected to show an increase.

However, the springwood-strength relationship does not always conform to the general rule. Among certain trees examined exceptions are sometimes found, in which cases the bursting strength of a pulp does not show the predicted rate of increase with increase in the springwood content. In other cases, the reverse is true, that is, in spite of the fact that the percentage of springwood does not show a normal increase in proceeding upward in a tree, the bursting strength, nevertheless, shows an increase such as might be expected only if a normal increase in springwood in that particular location in the tree. Specific examples of the first type may be found in graphs of the previously published article and also in examining the data and the strength curves for the top log of shortleaf pine tree D-1 and particularly for loblolly pine tree C-1.

For the case in point, it will be seen that the bursting strength values reached a maximum of 1.44 for the former and only 1.30 points for the latter which had a springwood content of 82 percent, whereas the top logs of other trees of high springwood content give bursting strength values of at least 1.50 points.

An example of the other phenomenon will be found in examining the curves for the suppressed loblolly pine tree, E. This particular tree showed no increase
in springwood above the middle of its trunk. Nevertheless, the bursting strength of the sulphate pulp prepared from the top logs showed a normal increase equivalent to, at least, a 20 percent increase in springwood. The tearing and tensile strength values for the pulps made from the several sections of this particular tree remained static even though there was a difference in springwood content between the butt and middle logs. According to the evaluation data, the top log of this tree should produce pulps similar to middle- and butt-cut material and, except for an abnormal increase in bursting strength in proportion to springwood content, other strength properties are in accord with the conclusions drawn from the physical data for this tree.

At the present writing many of these anomalies are not easily explained. They are probably attributable to certain chemical, physical, or structural properties of the wood other than to its content of springwood or summerwood.

Both decay\(^2\) in wood and compression wood\(^2,4,5,6\) with its abnormally large fibril angle in the secondary wall of the tracheids have been shown to be the cause of low strength properties in both sulphate and sulphite pulps. In addition, the rate at which wood, particularly springwood, is laid down during the growth of the tree may also have a bearing on pulp quality. Spiral graining of the tree trunk may produce in the internal structure of the fibers that peculiar twist so common to compression wood and thus cause weakness in the pulps manufactured therefrom.

In further examining the top logs of shortleaf pine tree D-1 and of loblolly pine tree C-1, and comparing them with the others tested, it was found that both logs contained wood-destroying fungi, the latter being in more of an advanced stage of decay than the former, which would at least partially account for the low strength properties of the resulting kraft pulps. In addition, both of these logs were found to be spiral grained and log C-1 was of very rapid growth.

While at present there is no evidence to show that spiral graining of wood affects the strength properties of pulps, figure 2 (center) indicates that certain very rapid growth-rate material may show a decrease in pulp strength. In this figure a similar strength trend to that of log C-1 was found for very rapid growth slash pine. The bursting and tensile strength properties of the slash pine sulphate pulps showed an increase proportional to the increase in springwood in comparing the butt and middle sections of the tree. However, in proceeding further up the tree these properties showed a decrease, even

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\(^2\)USDA Bulletin 1298 (1925).
\(^5\)C. E. Curran. Paper Trade Journal 103(11),36 (September 10, 1936); ibid 106(23),40 (June 9, 1938).
though the top logs contained an even higher percentage of springwood than did the lower portions of the tree. Unfortunately, the lower logs of tree C-1 were not shipped to the Laboratory for comparison. Perhaps the strength properties of the pulps from the other parts of the tree would also have been abnormally low.

The curves in figure 2 (right) summarize the effect of springwood content on the strength properties of sulphate pulps at 700 cc. Schopper-Riegler freeness. Except for the factors already noted, the points fall fairly close to curves indicating the trend in pulp strength with change in the ratio of springwood to summerwood. It will be noticed that as springwood increases the bursting and tensile strengths of the pulps increase and resistance to tear decreases.

Conclusions

The results of the experiments herein reported confirm those previously published, namely, that provided other physical, chemical, or structural properties of the wood do not interfere, the springwood-summerwood ratio in the raw material is a criterion in predicting the physical characteristics of pulps prepared from the southern yellow pines. Again greater differences were found in the strength properties of the pulps prepared from different parts of the same tree than were obtained from the wood of different species of southern yellow pine of similar growth rate and physical properties.

Decay, compression wood, and possibly wood of spiral graining and very rapid growth material appear to give abnormal strength values in the pulps obtained therefrom.

The proper selection of the raw material is, therefore, as essential to the production of uniform grades of pulp for utilization in specific products as cooking and subsequent processing of the fiber.

When pulped under identical conditions, butt logs again appeared to give the highest percentage of screenings.

Top-cut bolts obtained from above the merchantable length of dominant saw-log trees gave medium strength values when not influenced by decay.
<table>
<thead>
<tr>
<th>Tree data</th>
<th>Physical data</th>
<th>Chemical data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tree 1</td>
<td>Total length: 75 ft; Height: 50 ft; Age: 80 yrs.</td>
<td>Log: 6 ft; Stem: 4 ft; bark: 1 in.</td>
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<tr>
<td>Height = 50 ft; trunk = 4 ft; wide = 6 ft; stem = 8 in.</td>
<td>Growth: 0.5 in. per year; Length: 75 ft; Bark: 1 in.</td>
<td>Wood: 40 ft; Bark: 10 ft; Length: 50 ft; Bark: 1 in.</td>
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<td>Crown: 40 ft; Log: 6 ft; Trunk: 4 ft</td>
<td>Biennial; Companion: 10 ft; Heartwood: 40 ft; Mil: 20 ft</td>
<td>Length: 80 yrs; Bark: 1 in; Length: 50 ft; Bark: 1 in</td>
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<table>
<thead>
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<th>Pine</th>
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<tr>
<td>B</td>
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<td>59.4</td>
<td>74.4</td>
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<td>69.1</td>
<td>59.1</td>
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<td>D</td>
<td>59.2</td>
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<tr>
<td>D</td>
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1. Data supplied by cooperators.
2. Physical data were obtained on clear wood cut from the center of each log.
3. Considerable bark was lost in transit.
4. Three logs were taken from the base before cutting into pulpwood.
5. Two logs were taken at the hole before cutting into pulpwood.
Table 2: Effect of crop protection as influenced by position of the location on yield and quality of cotton fiber from selected species of cottons in Iowa

(Continued)
<table>
<thead>
<tr>
<th>No.</th>
<th>Crop</th>
<th>Treatment</th>
<th>Growth stage</th>
<th>Nitrogen</th>
<th>Phosphorus</th>
<th>Potassium</th>
<th>Zinc</th>
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<th>Magnesium</th>
<th>Manganese</th>
<th>Boron</th>
<th>Molybdenum</th>
<th>Silicon</th>
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**Growth and yield data**
- Height (cm)
- Number of Tillers
- Fresh weight (kg)
- Dry weight (kg)
- Grain yield (kg)
- Total yield (kg)

**Test data**
- Sulfate (ppm)
- Nitrate (ppm)
- Phosphate (ppm)
- Potassium (ppm)

**Efficacy**
- Yield response
- Nitrate accumulation
- Phosphate uptake
- Potassium use

**Chemical analysis**
- Nitrogen
- Phosphorus
- Potassium
- Zinc
- Copper
- Magnesium
- Manganese
- Boron
- Molybdenum
- Silicon
- Total sulfur

*Note: All values are in ppm.*

**Additional notes:**
- All crops were grown in 3-gallon, soil-banked, square, pot containers with identical soils.
- The crops were watered with a solution of 200 ppm of each nutrient.
- The experiment was conducted under greenhouse conditions.

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**Table 2—Effect of soil properties on yield and quality of selected crops**

(Continued)

**Chemical analysis**
- Nitrogen
- Phosphorus
- Potassium
- Zinc
- Copper
- Magnesium
- Manganese
- Boron
- Molybdenum
- Silicon
- Total sulfur

**Additional notes:**
- All crops were grown in 3-gallon, soil-banked, square, pot containers with identical soils.
- The crops were watered with a solution of 200 ppm of each nutrient.
- The experiment was conducted under greenhouse conditions.

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**Table 3—Effect of soil properties on yield and quality of selected crops**

(Continued)

**Chemical analysis**
- Nitrogen
- Phosphorus
- Potassium
- Zinc
- Copper
- Magnesium
- Manganese
- Boron
- Molybdenum
- Silicon
- Total sulfur

**Additional notes:**
- All crops were grown in 3-gallon, soil-banked, square, pot containers with identical soils.
- The crops were watered with a solution of 200 ppm of each nutrient.
- The experiment was conducted under greenhouse conditions.

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**Table 4—Effect of soil properties on yield and quality of selected crops**

(Continued)

**Chemical analysis**
- Nitrogen
- Phosphorus
- Potassium
- Zinc
- Copper
- Magnesium
- Manganese
- Boron
- Molybdenum
- Silicon
- Total sulfur

**Additional notes:**
- All crops were grown in 3-gallon, soil-banked, square, pot containers with identical soils.
- The crops were watered with a solution of 200 ppm of each nutrient.
- The experiment was conducted under greenhouse conditions.

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**Table 5—Effect of soil properties on yield and quality of selected crops**

(Continued)

**Chemical analysis**
- Nitrogen
- Phosphorus
- Potassium
- Zinc
- Copper
- Magnesium
- Manganese
- Boron
- Molybdenum
- Silicon
- Total sulfur

**Additional notes:**
- All crops were grown in 3-gallon, soil-banked, square, pot containers with identical soils.
- The crops were watered with a solution of 200 ppm of each nutrient.
- The experiment was conducted under greenhouse conditions.
Figure 1.—Effect of springwood-summerwood ratio on strength properties of southern yellow pine sulphate pulps.
Figure 2.—Effect of springwood-summerwood ratio on strength properties of southern yellow pine sulphate pulps.