

**SEMICHEMICAL AND SULFITE PULPING EXPERIMENTS AND  
PRODUCTION OF CORRUGATING BOARDS  
FROM BLACKJACK OAK**

**May 1939**



**SCHOOL OF FORESTRY  
OREGON STATE COLLEGE  
CORY**



(Report)

**UNITED STATES DEPARTMENT OF AGRICULTURE  
FOREST SERVICE  
FOREST PRODUCTS LABORATORY  
Madison, Wisconsin**

**In Cooperation with the University of Wisconsin**

SEMICHEMICAL AND SULFITE PULPING EXPERIMENTS, AND PRODUCTION OF  
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Blackjack oak is an abundant and widely distributed southern hardwood which at present is little used as lumber and finds very little outlet in the pulp and paper industry. Semichemical pulping experiments have therefore been carried out on this wood with corrugating board in view as the final product. In addition, several preliminary sulfite digestions have been made in order to evaluate the wood according to this process.

The Wood

Blackjack oak (Quercus marilandica) is a ring-porous hardwood, in which class the only commercially used hardwood pulpwood is chestnut. Properties reported<sup>1</sup> for the wood used in the present study are presented in table 1.

The high density of the wood (39.6 pounds per cubic foot) is offset to some extent by a high barking loss (18.4 percent) and, particularly in this shipment, by extensive heart rot. The chemical analysis of the wood showed the high pentosan content characteristic of hardwoods, but the cellulose content was relatively low. The alkali solubility was possibly lower than would be expected from the amount of decay present. The fiber dimensions have been reported<sup>2</sup> to be on the

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<sup>1</sup>Physical and chemical properties of blackjack oak (Quercus marilandica) pulpwood shipment 1508. E. R. Schafer and J. C. Pew. Project L-168-5; Problem D-171.

<sup>2</sup>Identification of important North American Oaks. G. B. Sudworth and C. D. Mell. U. S. D. A. Forest Service Bull. 102. Wash. 1911.

Table 1.--Physical and chemical properties of blackjack oak

|      |             |                                  |            |              |                  |                |       |     |
|------|-------------|----------------------------------|------------|--------------|------------------|----------------|-------|-----|
| Age  | Growth rate | Density (O.D. weight: green vol. | Heart-wood | Barking loss | Average diameter | Color analysis | Heart | Sap |
| 47.8 | 17.1        | 39.6                             | 45.8       | 18.4         | 5.6              | 18             | 37    |     |

|           |                 |        |                 |                    |           |                               |       |
|-----------|-----------------|--------|-----------------|--------------------|-----------|-------------------------------|-------|
| Cellulose | Alpha cellulose | Lignin | Total pentosans | Hot 1 percent NaOH | Hot water | Solubility in Alcohol-benzene | Ether |
| 56.6      | 43.9            | 26.3   | 20.1            | 15.0               | 4.7       | 3.5                           | 0.6   |



average 1.38 mm. in length and 0.027 mm. in diameter, which again fall in the hardwood range. The dark color of the wood, especially the heartwood, would be reflected in the color of any semichemical pulp prepared from it.

### Semichemical Pulping

The objects of the blackjack oak semichemical digestions were a brief examination of some variables of semichemical pulping for the purpose of developing a suitable procedure for this wood, the production of pulp covering the usual range of yields for this type of pulp, and the evaluation of these pulps, especially for corrugating board.

The first five semichemical digestions were made in a 1.5 cubic foot, steam-jacketed autoclave lined with a stainless alloy, and the last three digestions in a steel rotary digester heated by direct steam and having a capacity of 69 cubic feet. The smaller digester was heated by both direct and indirect steam, while only direct steam was used for the larger digester; in the latter, then, there was more dilution of the cooking liquor by steam condensation. The liquor for each autoclave digestion was made up from fresh sodium sulfite and sodium bicarbonate. The liquor for the first digestion in the rotary digester was made up from fresh chemicals, and that for the succeeding digestions from the blowback from the preceding digestion fortified to the desired concentration. Five-eighths-inch chips were used in all instance. The amounts of wood and liquor used, the liquor concentration, the impregnation and digestion conditions, and the yield of pulp and its properties are all summarized in table 2.

In the first autoclave digestion (No. S-210) conditions were used which had in previous digestions of other species resulted in high yield (about 80 percent) and which included a relatively low impregnation liquor concentration. The extremely high yield of 96.1 percent which was obtained under these conditions signified their inadequacy and indicated a refractory nature for the blackjack oak as far as this pulping treatment is concerned.

In the second autoclave digestion (No. S-211) the chemical concentration of the impregnation liquor was approximately doubled and the impregnation time increased from 2.0 to 2.5 hours, the other conditions remaining closely the same (table 2). These changes in impregnation liquor and time effected an increase in the amount of chemical retained by the chips from 4.1 to 12.6 pounds of sulfite and from 2.3 to 4.8 pounds of bicarbonate (sodium carbonate basis) per 100 pounds of wood, which was sufficient to cause solution of wood material to the extent that the yield was reduced to 77.8 percent. These conditions, then, are probably satisfactory for producing a pulp in the yield range of 75 to 80 percent.

The blowback liquor, liquor samples taken during the course of the digestion, and the final liquor sample from all the semichemical digestions were extremely dark colored, with a reddish-brown tinge. The presence of some dye material made analysis of these liquors very difficult, especially for samples taken in the later stages of the digestion. These latter analytical results were therefore somewhat erratic.

Determinations of pH values were made during the course of several cooks. The impregnation liquor values varied from 8.5 to 9.0, the blowback liquors covered a similar range, and the final values varied from 7.5 to 8.5. The general trend during the course of a cook was for a small decrease in the pH value. These data, however, appeared to have no value from a control standpoint.

In an endeavor to obtain a pulp yield in the range of 65 to 70 percent, the conditions used in the previous digestion (No. S-211) were modified so that longer impregnation and digestion times were allowed; the former was increased 0.7 and the latter 1.0 hour for No. S-212. The impregnation liquor had approximately the same concentration as before. The lengthened impregnation time did not affect the total chemical retention but did lead to a somewhat different distribution, more of the bicarbonate and less of the sulfite being present for digestion purposes. The decrease in amount of sodium sulfite in the digestion liquor was not quite balanced by the increased digestion time, so the pulp yield was slightly higher, 79.7 as compared to 77.8 percent.

The relatively large consumption of sodium bicarbonate in the two previous digestions suggested that larger amounts of this chemical were needed in pulping blackjack oak as compared to other woods in order to maintain optimum semichemical-pulping conditions. The effect of greatly increased bicarbonate content of the impregnation liquor was then determined in a digestion in which the bicarbonate content of this liquor comprised half the chemical by weight (No. S-213, table 2). Applying the same impregnation conditions as used in No. S-212, a large increase in retention of both sulfite and bicarbonate resulted from the increased bicarbonate content of the impregnation liquor, although the retention ratio of sulfite to bicarbonate was the same as found in No. S-212. The increased chemical present in the cooking liquor necessitated a higher digestion temperature in order to complete the pulping in a reasonable time, so the maximum pulping temperature was increased from 172 to 181° C. The presence of a larger amount of chemical and the increased pulping temperature effected more complete pulping than before, the yield being reduced to 69.9 percent.

The chemical consumption data for the previous digestions indicated that pulp yield was quite dependent on the amount of sodium sulfite present during the pulping operation with sufficient sodium bicarbonate at hand and suitable pulping conditions. These data also showed that sodium sulfite retention in turn was dependent on the bicarbonate as well as sulfite concentration in the impregnation liquor and the impregnation conditions.

In determining the effect of a higher sodium sulfite concentration, and at the same time attempting to produce a pulp yield of about 60 percent, a digestion (No. S-214) was made using an impregnation liquor with materially increased sulfite and somewhat augmented bicarbonate contents (table 2). With unchanged impregnation conditions the strengthened impregnation liquor led to increased sulfite retention but decreased bicarbonate retention as compared to the previous digestion (No. S-213) again bringing out the dependence of the relative amounts of sulfite and bicarbonate absorbed on their relative concentrations in the impregnation liquor. The enhanced sulfite content of the digestion liquor made necessary a considerably longer digestion at the pulping temperature of 183° C. in order to reduce the content of the residual liquor to an amount which indicated a desired degree of pulping. This time was 7.8 hours in comparison with 3.6 hours for the previous digestion. This additional pulping enabled the yield to be reduced to 61.2 percent, which is close to the minimum yield for semi-chemical pulping.

The larger scale semichemical digestions (Nos. 2493-N to 2495-N), which had as their main purpose the production of pulp in quantities sufficient for the corrugating board studies, were carried out under conditions falling in the same range as those used for the autoclave digestions.

In the first digestion made in the rotary digester (No. 2493-N) the high bicarbonate concentration of the cooking liquor and the long impregnation time (4 hours), as expected, led to about equal retention on a weight basis of sulfite and bicarbonate. With the pulping conditions used (table 2) this liquor resulted in a pulp yield of 71.1 percent, which is in line with the previous yield data when the factors affecting yield are considered.

In the next digestion (No. 2494-N) the impregnation liquor concentration was reduced somewhat, more so in bicarbonate than sulfite content, as was the impregnation time; the outcome was a slight increase in sulfite available for pulping but about the same total chemical retention. The increase in sulfite content of the digesting liquor, however, was more than offset by a shorter pulping time, the net result being a pulp yield of 76.6 percent.

The final digestion (No. 3495-N) differed from all the others in that chips from unbarked wood were used. In this digestion the impregnation liquor had a relatively low bicarbonate content (21.1 as compared to 34.2 grams per liter in No. 3494-N) and the impregnation temperature was higher than before, the consequence being that a large amount of sulfite and an average amount of bicarbonate were available for pulping action. In order to obtain a desired yield of around 75 percent, the pulping time was shortened to 1.2 hours. The yield obtained was 76.3 percent.

Four of the pulps from the autoclave digestions (Nos. S-211 to S-214) were broken up in a small beater and their strength characteristics determined in a test beater. Bursting and tearing strength figures for two freeness values, 800 and 550 cc., showed no great differences between these pulps which cover a yield range from 61.2 to 79.7 percent. Although all strength values were low, they appeared to fall in a satisfactory range for material of this type. The pulp produced in the lowest yield had somewhat higher strength values than the others.

#### Corrugating Board

The semichemical pulp chips from the large scale digestions were defibered and processed by beating and subsequent jordan treatment or by rod mill treatment. The resulting pulps were then run into corrugating board on the Forest Products Laboratory paper machine. No systematic study was made in processing the pulps. Rather, the objective was to make a board having properties within the wide range of commercial boards suitable for corrugating, with a minimum amount of processing. Data on the paper-machine runs are presented in table 3. In addition, the properties of four commercial corrugating board samples are included for comparison.

The pulp for the first paper-machine run (No. 1323) still contained, after the beater processing, numerous small, woody fiber bundles which were not cleared up by the jordan treatment; this indicated inadequate processing which resulted in low strength for this particular board. In the next trial (machine run No. 1324) less drastic treatment was given over a longer beating period, which resulted in improved board properties.

Rod mill treatment appeared to be possibly more suitable than beater treatment for processing these semichemical-pulp chips, particularly with regard to strength development and reduction of shives. The properties of the boards from paper-machine runs Nos. 1326 and 1327 were superior to those of three commercial boards, the groundwood and the chestnut chip, with which the blackjack oak boards would ordinarily be classed. The Kraft board had the highest bursting and tearing strength values.

Only one machine run was made on each of the higher yield pulps, No. 2494-N from barked wood, and No. 2495-N from unbarked wood after beater and jordan treatment. The strength of the boards made from both was below that obtained in some of the previous runs, and the unbarked wood (No. 2495-N) gave lower strength values than the barked wood (No. 2494-N). The strength could undoubtedly be improved, and the slivers reduced by suitable processing. However, both gave boards having strength within the range of that obtained from the commercial board samples, and the board from the unbarked wood (No. 1330) was successfully corrugated. Machine runs Nos. 1325, 1326, 1327 were likewise corrugated without difficulty.



Table 3.---Properties of blackjack oak and commercial boards

| Material                      | Cook No. | Machine : run No. | Kind of processing | Properties of boards |                    |                    |               |         |
|-------------------------------|----------|-------------------|--------------------|----------------------|--------------------|--------------------|---------------|---------|
|                               |          |                   |                    | Caliper : strength   | Tearing : strength | Tensile : strength | Stretch       |         |
|                               |          |                   |                    | Points               | Pt./lb./ream       | Gm./lb./ream       | Lbs./sq. inch | Percent |
| Blackjack oak                 | 2493-N   | 1323              | Beater and jordan  | 12.2                 | 0.14               | 0.64               | 1588          | 2.7     |
| Blackjack oak                 | 2493-N   | 1324              | Beater and jordan  | 10.6                 | .36                | .90                | 3370          | 4.2     |
| Blackjack oak                 | 2493-N   | 1325              | Beater and jordan  | 10.4                 | .32                | .63                | 3045          | 3.5     |
| Blackjack oak                 | 2493-N   | 1326              | Rod mill           | 9.2                  | .46                | .99                | 4000          | 5.0     |
| Blackjack oak                 | 2493-N   | 1327              | Rod mill           | 9.0                  | .47                | .87                | 4420          | 5.1     |
| Blackjack oak                 | 2493-N   | 1328              | Rod mill           | 15.7                 | .42                | 1.01               | 3450          | 4.6     |
| Blackjack oak                 | 2494-N   | 1329              | Beater and jordan  | 8.9                  | .31                | ---                | 2982          | 4.2     |
| Blackjack oak                 | 2495-N   | 1330              | Beater and jordan  | 9.6                  | .22                | .58                | 2360          | 2.9     |
| Commercial corrugating boards |          |                   |                    |                      |                    |                    |               |         |
| Kraft                         | ---      | ---               | ---                | 9.6                  | .73                | 2.80               | 3945          | 5.5     |
| Pine                          | ---      | ---               | ---                | 10.4                 | .31                | .74                | 2987          | 2.9     |
| groundwood                    | ---      | ---               | ---                | 10.9                 | .23                | .72                | 2440          | 2.4     |
| Chestnut chip                 | ---      | ---               | ---                | 10.5                 | .17                | .64                | 1950          | 1.6     |
| Straw                         | ---      | ---               | ---                |                      |                    |                    |               |         |

<sup>1</sup>Machine run 1328 was a liner board type, the others being designated as corrugating board.

Table 4.—Blackjack oak sulphite pulping<sup>1</sup> results

| Cook No. | Time from 110° C. to maximum temperature: | Maximum tempera- ture | Total: time | Pulp yield |           |              | Bleach used                    |
|----------|---|-----------------------|-------------|------------|-----------|--------------|--------------------------------|
|          |   |                       |             | Total:     | Screened: | Screen- ings |                                |
|          | Hours                                     | °C.                   | Hours:      | Percent    |           |              | Percent standard bleach powder |
| 3803-I   | 1.5                                       | 140                   | 8.3         | 43.4       | 39.8      | 3.6          | 40                             |
| 3848-I   | 2.0                                       | 135                   | 10.8        | 42.1       | 41.5      | .6           | 35                             |

| Color of bleached pulp | Beater strength tests |                  |         | Pulp chemical tests |             |                    |
|------------------------|-----------------------|------------------|---------|---------------------|-------------|--------------------|
|                        | Bursting strength     | Tearing strength |         |                     |             |                    |
|                        | Freeness-S. R.        | Freeness-S. R.   |         |                     |             |                    |
|                        | 800 cc.               | 550 cc.          | 800 cc. | 550 cc.             | Cellu- lose | Alpha cellul- lose |
|                        |                       |                  |         |                     |             |                    |
|                        |                       |                  |         |                     |             |                    |
| Ives parts blue        | Pt./lb./ream          | Gm./lb./ream     | Percent |                     |             |                    |
| 82                     | 0.23                  | 0.30             | 0.49    | 0.49                | —           | —                  |
| 82                     | .20                   | .28              | .48     | .45                 | 92.8        | 83.2 2.1           |

<sup>1</sup>Acid:—5.5 percent total and 1.3 percent combined SO<sub>2</sub>; penetration:— 1.5 hours to 110° C. and 1 hour at 110° C. ; pressure— 80 pounds per square inch maximum.

Other physical tests on the blackjack oak boards giving the stress at maximum load and modulus of elasticity compared favorably with those obtained from commercial corrugating boards made from straw, pine wood, kraft pulp, and chestnut chips.

#### Sulfite Pulping

Two preliminary sulfite digestions of blackjack oak were made in the 50-pound digester using moderate pulping conditions and the customary 5/8-inch chips. The actual conditions used and the results obtained are tabulated in table 4.

Liquor samples taken during and at the end of digestions were extremely dark colored, but not cloudy. Analysis by the Sander method was virtually impossible.

The high screenings and high bleach requirement obtained from the first digestion (No. 3803-I) indicated incomplete pulping in spite of the low yield (43.4 percent total yield). The second digestion (No. 3848-I) made under milder conditions (a longer time at lower temperatures) gave a low percentage of screenings indicating satisfactory pulping, although the bleach requirement was again relatively high and the yield was low (41.5 percent total). The blow liquor, although very dark, was clear in both instances, signifying the absence of any "burning" effect.

The pulps were shivey, dark-colored, and had very little strength. The bursting strength was around 0.30 point and tearing strength less than 0.50 gram per pound per ream at a freeness value of 550 cc. The strength values were much lower than had been found for other hardwood sulfite pulps.

Although it is apparent that blackjack oak chips can be readily reduced by the sulfite process with a low amount of screenings, the uncooked slivers, the high bleach requirement, and especially the inferior strength of the pulp would very likely limit any extensive use of the wood for this purpose unless some improvement in these properties can be effected.

#### Summary

Blackjack oak, a little used, dark colored, dense hardwood with physical and chemical properties quite typical of hardwoods, has been successfully pulped by the semichemical process. High sodium sulfite and bicarbonate concentrations appeared to be necessary in the impregnation liquors in order to enable sufficient chemical retention to bring about the desired pulping, especially if low yield pulps are to be obtained.

Board for corrugating purposes made from the blackjack oak (both barked and unbarked) semichemical pulp was equal or superior to comparative commercial boards. This board was also satisfactorily corrugated.

Sulfite pulp from blackjack oak was produced in low yields with small amounts of screenings, but was dark colored, difficult to bleach, and very low in strength.

TABLE