INSTITUTE OF
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*Appleton Wisconsin*

# BARK AND WOOD PROPERTIES OF PULPWOOD SPECIES AS RELATED TO SEPARATION AND SEGREGATION OF CHIP/BARK MIXTURES

Project 3212

Report Four

A Progress Report

to

MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY

September 15, 1975

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## SUMMARY

Northern white oak has a wood specific gravity of 0.64 and an average bark specific gravity of 0.58. Bark extractives levels averaged 7.2%. Morphologically, the bark contains fiber and some sclereids and sieve tubes. Pulping northern white oak bark gave a solids yield of 31-38%. Screening of the bark pulp resulted in approximately 86% of the solids passing through the 100-mesh screen. The fraction retained on the 60- and 100-mesh screens contained 3 grams of fiber and 0.2 gram of other material per 100 grams of bark pulped. It appears that reasonable separation and segregation of northern white oak wood and bark chip mixtures could be accomplished through screening, hammermilling the fractions high in bark and rescreening. Hammermilling tests resulted in a 37% reduction in bark and a 5% wood loss based upon material retained on the 14-mesh screen and a 46% reduction in bark and 7% wood loss based upon material retained on the 10-mesh screen. Segregation through water flotation is possible but the small range of moisture contents at which it could be achieved do not make it a feasible method.

Southern white oak, based upon values in the literature and measurement data from trees sampled as part of the project, has an average wood specific gravity of 0.67 and a bark specific gravity of 0.56. Extractives levels for bark averaged 8.6%. Pulping southern white oak bark produced a solids yield of approximately 37%. Of this, approximately 3% were usable fibers and 0.3% were sieve tubes. Southern white oak reacted very much like northern white oak to various tests designed to separate and segregate wood/bark chip mixtures. As with northern

white oak, screening, hammermilling fractions high in bark and rescreening should result in good bark reduction and a small wood loss. Hammermilling tests resulted in a 3% wood loss and a 38% bark reduction, based upon material retained on a 14-mesh screen. If the size of the material retained is increased to 10 mesh, the result is a 58% reduction in bark levels and a 6% wood loss. Segregation through water flotation is not a feasible method due to the narrow range of moisture contents at which segregation would take place.

Southern red oak had a wood specific gravity of 0.60 and a bark specific gravity of 0.70. Extractives levels were 4.8 and 11.6%, respectively, for the wood and bark. Morphologically, the bark contains mainly fibers, sieve tubes and sclereids. Pulping southern red oak bark gave a solids yield of approximately 31%. Screening the pulp resulted in 4% fibers and 0.2% of other material being retained on the 60- and 100-mesh screens. This species also responded well to the hammermilling technique with a 6% wood loss and 46% reduction in bark, based on the material retained on the 14-mesh screen. Based on these results, the screening-hammermilling-rescreening technique has some merit and it is possible improvements in screening would result in even better segregation. As with the other oaks, the narrow range of moisture contents at which segregation could be achieved make water flotation an impractical method.

Sweetgum was found to have a wood specific gravity of 0.44 and a bark specific gravity of 0.42. Extractives levels were 2.6 and 10.2%, respectively, for wood and bark. Morphologically, sweetgum bark contains fiber and sieve tubes but very few sclereids. Sieve tubes act mainly as filler and possibly as a bonding material. The bark, when pulped, had a solids yield of 32-37%. Screening resulted in 5% fibers and 3% sieve tubes being retained on the 60- and 100-mesh screens.

Pulping of sweetgum bark is more attractive than for many other species because of the fiber and sieve tube yield and lack of sclereids. Water flotation would not be a good method to use on sweetgum to effect segregation of wood and bark because both fractions float, even at very high moisture contents. Hammermilling and screening showed moderate results with a 7% wood loss and 32% bark removal when the material on the 14-mesh and larger screens was retained. However, most of the bark remaining was inner bark which is high in fiber.

## INTRODUCTION

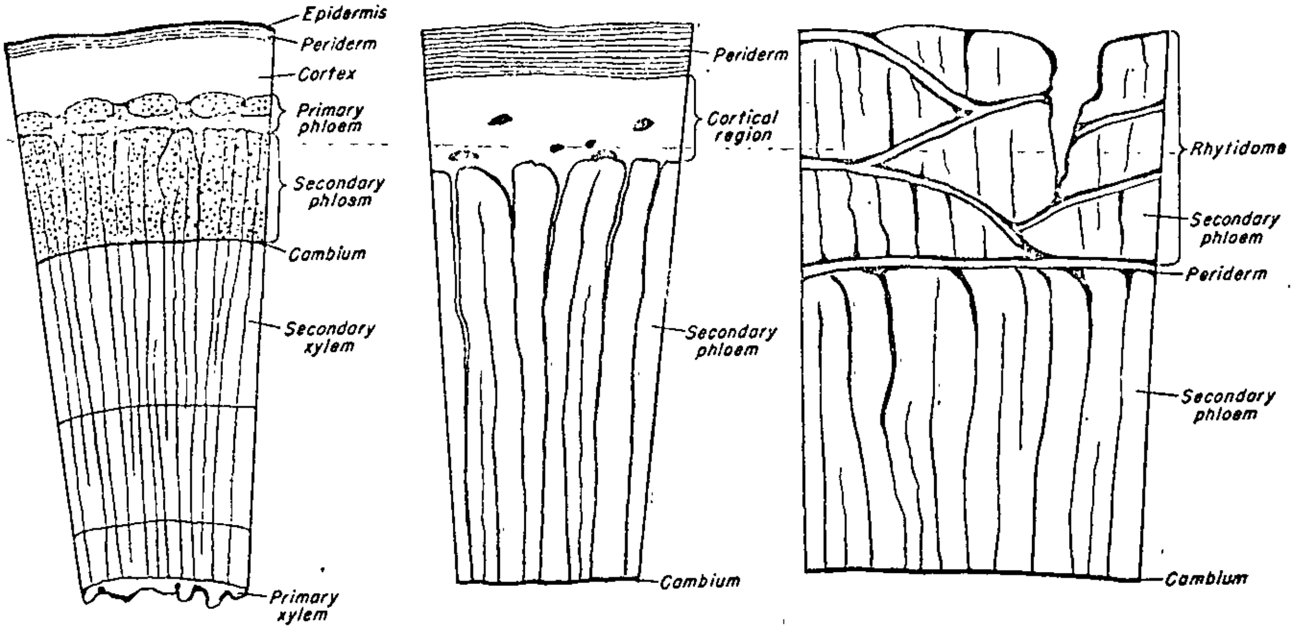
Future wood fiber supply continues to be an item of major concern for the pulp and paper industry. Energy used in harvesting and processing wood has recently become an additional item of concern. Whole-tree utilization appears to be one important way to increase existing raw material supplies immediately. Use of short-rotation intensive forest management appears to be an additional procedure for increasing our overall wood supply. Under most forest management schemes, both approaches require the utilization of a certain portion of unbarked wood. How best to handle a specific bark problem requires an adequate knowledge of bark of the tree species involved. Items that should be considered in evaluating a particular situation are numerous. Such factors as the species mix employed, end product requirements and mill digester, cleaner and recovery furnace capacity are items of major concern when the pulping of bark is considered. Fiber yield, extractive levels and ash content, along with equipment wear problems, must also be considered in the pulping of bark. When bark removal is required, the best method to use will depend upon such factors as specific gravity, strength, toughness, wood/bark adhesion and the fuel value of the wood and bark of the species employed.

Project 3212 is attempting to properly identify the industry's bark problem by adequately characterizing the barks of the major pulpwood species of the United States. Adequate bark characterization will make it possible to develop the best solution for specific mill situations. Early bark measurement information included most of the above-described information with the exception of ash content and fuel value. Ash content, because of recovery furnace scaling problems, and fuel value, because of energy needs, have been added to the list of required information.

Prior to July 1, 1975, Project 3212 was a group project supported by six interested companies. On July 1, 1975 the program was switched to a dues-funded project. Three reports were issued prior to the change to a dues-funded status and the original members of the group project have generously agreed to release the information in these reports to all Institute member companies. Progress Report One described in detail the experimental methods employed and provided bark information for quaking aspen, sugar maple, white birch, and northern red oak. Progress Report Two covered loblolly pine, slash pine, Douglas-fir and western hemlock while Progress Report Three summarized the bark information for white spruce, balsam fir, jack pine and eastern cottonwood. The report that follows presents a comprehensive description of the bark of northern white oak, southern white oak, southern red oak, and sweetgum.

## TREE GROWTH AND BARK DEVELOPMENT

Tree growth and bark development were covered in Project 3212, Progress Report One. To briefly summarize, a tree grows through elongation and enlargement of the bole and crown (primary growth) and thickening of the bole (secondary growth). The bark consists of the inner bark (secondary phloem), which is partly physiologically active, and the outer bark, which is mainly functionless. Tissues in the inner bark are constantly being developed and the first-formed layers of periderm may be cut off from the vital processes of the tree. This can result in roughened bark which may either be cast off or retained as in the case of deeply fissured trees. In smooth-barked trees the first-formed periderm may persist for many years. Figure 1, taken from Chang (1) illustrates the tissues found in different kinds of bark and is provided, along with the Glossary, to help the reader better understand the bark descriptions that follow.



*1. Young stem*

*2. Mature bark without rhytidome formation*

*3. Mature bark with rhytidome*

Figure 1. Diagrammatic Drawings Showing the Main Tissue in Different Types of Bark. (1) Cross Section of Young Branch of Stem. (2) Cross Section of Bark Having Persistent Cortex, such as that in the Middle-Aged Balsam Fir and Quaking Aspen. (3) Mature Bark with Rhytidome Formation

# EXPERIMENTAL PROCEDURES

The experimental procedures employed have, as much as possible, been standardized and the same methods used for each tree species. Progress Report One should be referred to for complete descriptions of the experimental procedures used.

Tree size and sample location were standardized and utilized trees 7 to 9 inches in diameter at breast height (4-1/2 feet). All measurements were made on samples from the breast high location or from 12 to 18-inch bolts obtained from the area just below the breast high sample.

Specific gravity was determined using a water displacement technique that is a modification of the TAPPI Standard Method, T 18 m-53, and results are expressed in terms of ovendry weight/green volume. The bark micropulping procedure was that of Thode, et al. (2). After micropulping, the bark was rinsed, fiberized in a Waring Blendor and decanted on a sintered glass funnel. It was then put through a series of screens and the material on each screen examined for the type of cellular material it contained.

The wood/bark adhesion method measured shear parallel to the grain on a small, specially prepared sample using the Instron tester. Representative growing and dormant season adhesion samples were immersed in ethyl alcohol immediately after testing for later morphological examination.

Bark strength measurements were made using essentially the same procedure as used in measuring wood/bark adhesion (shear parallel to the grain). Bark toughness measured the energy required to rupture a small bark or wood sample by bending with a force parallel to the diameter of the tree. A "Micro Pulverizer" was modified to provide a hammermilling test on standard bark and wood chips. After the chips were fed through the pulverizer, they were separated on a series of soil screens and the percentage on each screen calculated.

Basic density of standard wood and bark chips at various moisture contents was determined using a pycnometer and the chemical, heptane, as the displacement medium. Moisture content was calculated as (wet wt.-o.d. wt.)/ o.d. wt. Density was calculated as $(c \cdot d)/[c-(b-a)]$ where:

$a$ = weight of pycnometer + heptane

$b$ = weight of pycnometer + heptane + chip

$c$ = weight of chip (wet — before being placed in heptane)

$d$ = density of heptane.

# BARK AND WOOD PROPERTIES OF WHITE OAK (Quercus alba L.)*

## SILVICULTURAL CHARACTERISTICS AND GEOGRAPHIC RANGE

White oak is widespread throughout the eastern and central United States. Its range extends from southern Maine, south to northern Florida, west to eastern Texas, and northward to southeastern Minnesota. It is generally absent in the Delta region of the lower Mississippi and the Gulf coastal areas. With a wide variety of climatic conditions and soil types in this extensive range, the west slopes of the Appalachian Mountains and the Ohio and central Mississippi River valleys have the optimum conditions for the growth of white oak. Development is best on deep, well-drained loamy soils; however, growth is good on all except the driest, shallow soils. White oak, a large tree with great longevity, normally reaches heights of 100 feet with diameters of 4 feet on good sites.

## WOOD AND BARK MORPHOLOGY

### Wood

White oak, with a white to light-brown sapwood and dark-brown heartwood, is a usually straight-grained, hard, and heavy to very heavy, ring-porous wood. Except in slow-grown stock, growth rings are very distinct with large earlywood pores often occluded with tyloses in the heartwood, forming a band 1-3 pores in width. The transition from earlywood to latewood is generally abrupt. Latewood pores, small, thin-walled and more numerous, are scattered in radially aligned flame-shaped tracts of light-colored parenchymatous tissue. Conspicuous compound rays, averaging 0.5-1.25 inches high, are separated by numerous, indistinct, narrow rays.

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*Although white oak is referred to as northern and southern white oak in this report, both are Quercus alba and are just from different sources.

Composition and structure of the xylem of the various oaks in the white oak group are generally indistinguishable. Latewood vessels number 20-120 per sq mm and the diameter of the largest earlywood vessels range from 180-380 µm. The average length of Q. alba L. vessels is 0.4 mm. Vasicentric tracheids, short, irregularly shaped cells, are present, forming part of the flame-shaped tracts in the latewood vessel area and the conjunctive tissue between the earlywood vessels and the rays. Longitudinal parenchyma are abundant, paratracheal, and intermingled with the tracheids. Xylem fibers, medium thick to thick-walled and frequently gelatinous, have diameters of 14-22 µm and an average length of 1.39 mm. Rays, averaging 27.9% of the total wood volume, are unstoried and homocellular. The broad compound rays are 12-30+ seriate, 150-400+ µm wide in the middle, and hundreds of cells high in height. The narrow rays, very numerous and usually uniseriate, vary greatly in height, 1-20+ cells.

## Bark

On the outer surface, white oak bark is ash-grey and characterized by long fissures formed by flatly overlapping ridges of rather thin scales. The outer bark is composed of many layers of rhytidome formed by the yellowish-brown periderm or cork alternating with the isolated brown phloem tissues. Rhytidome of the white oaks is usually soft, rather loose, and on cross section, often wider than the creamy light-colored inner bark, usually about 1 inch wide. Broad rays and sclereid groups are visible. In the trees used in this study, the outer bark accounted for 50-60% of the total bark thickness by weight for northern white oak and 28-45% for southern white oak. Figure 2 illustrates a cross section of northern and southern white oak wood and bark. Appendix Table XXVII describes the trees used in this study.

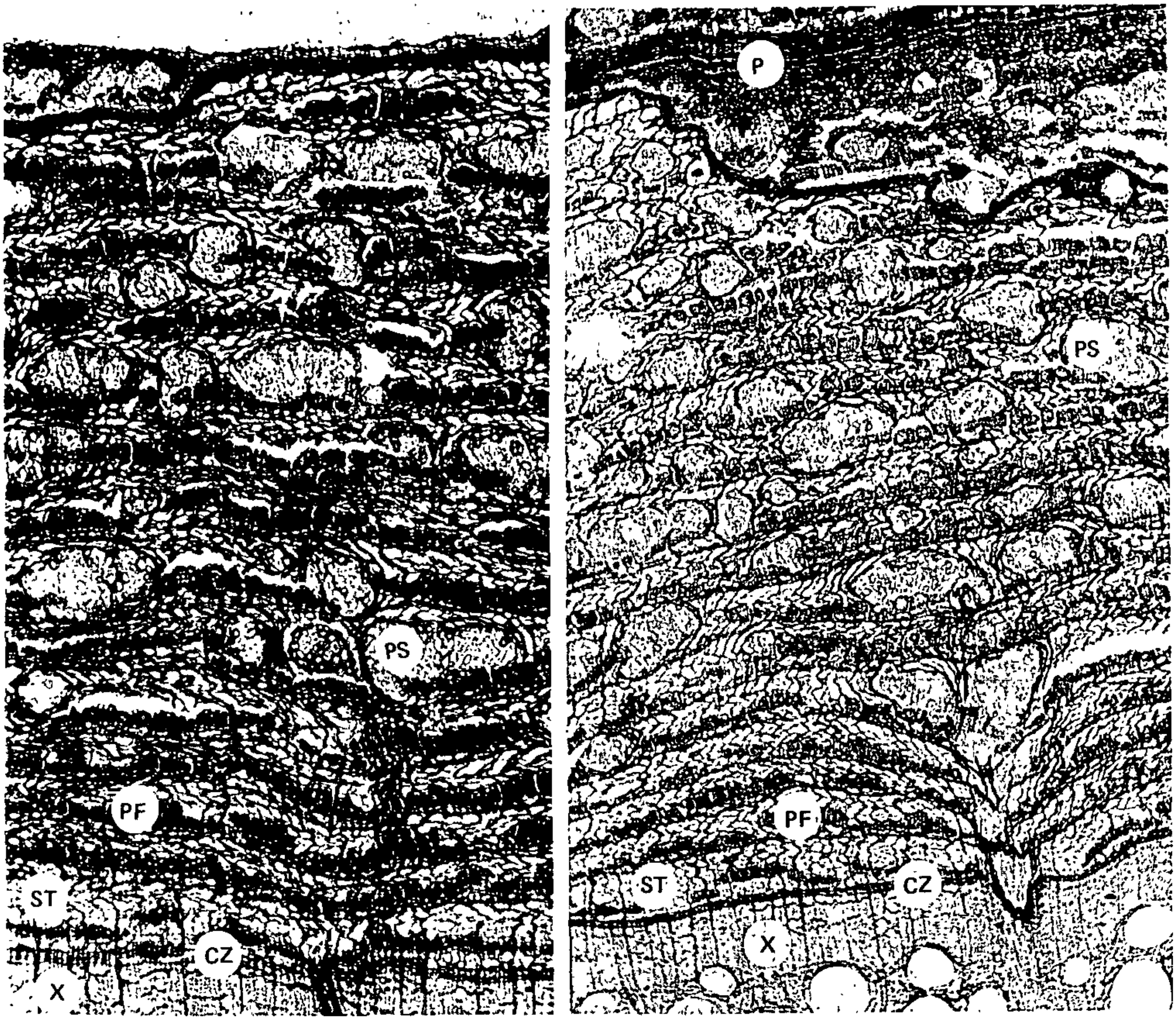

Figure 2. Cross Section of Northern White Oak (Left) and Southern White Oak (Right). Illustrated Is the Xylem (X), Cambium Zone (CZ), Phloem Fibers (PF), Phloem Sclereids (PS), Sieve Tubes (ST), and Periderm (P). Magnification — 50X Left, 35X Right

## Anatomical Structure of Mature Bark

Bark of a young tree starting from the outer surface of the bark, is composed of a layer of compactly arranged epidermal cells, thin-walled phellem cells, parenchymatous cortex cells with a few layers of collenchymalike cells near the periderm and 4-6 layers of well-developed primary phloem fibers with some sclereids between the archlike bands. Sieve tubes, parenchyma and phloem rays form the inner bark (secondary phloem) in the young stem.

In mature white oak bark, periderm layers are conspicuously aligned in tangential bands, but broken regularly along the radius of the extending phloem rays. The last-formed periderm is composed of 1-2 layers of phelloderm, a layer of phellogen and 2-5 layers of phellem. Zones of the regular thin-walled phellem cells often alternate with 1-2 layers of thick-walled cells. Regularly aligned, peridermal cells are rectangular on cross and radial sections with radial diameters from 8-15 μm, tangential diameters from 20-35 μm, and heights, about 30 μm.

Sieve tubes in the secondary phloem are usually in groups of 4-10 and the space (or area) confined by the phloem rays, parenchyma and sclerenchyma cells. Except in the last-formed bands next to the cambium, sieve tubes are crushed to varying degrees. Sieve tube elements vary from 220-590 μm in length. Often, short companion cells, about 50 μm high and in strands of up to 6 cells, are associated with them at their narrow dimension. Parenchyma cells, frequently aligned in tangential bands of 3-5 layers, are more or less rectangular in cross section, about 20-30 μm in tangential diameter and 50-100 μm in height. Forming strands about the same length as phloem fibers, crystalliferous parenchyma strands [probably containing calcium oxalate (3,4)], are found at the margin of phloem fiber bands.

Sclerenchyma cells appear in the form of phloem fibers and sclereids. Phloem fibers, in mostly 2-3 layers, are aligned in discontinuous tangential bands. The fibers are polygonal on cross section with diameters of 15-20 μm and a very narrow lumen. Cell walls are about 6 μm thick with the secondary walls sometimes separated into 2 layers. Tapering gradually to pointed ends, phloem fibers vary from 0.53-1.39 mm in length. Maturation often starting at the current season's growth, phloem fibers usually form pure bands with the crystalliferous parenchyma strands. At an old growth region, fiber bands may connect with a sclereid band. Sclereids are transformed parenchyma and ray cells with irregularly expanded thick walls. Sclereids form small groups that are initiated and often mature very near to the cambial region. The groups may be tangentially elongated and form short bands, or scattered within the broad phloem rays.

White oak phloem rays are of two types. The narrow, homocellular, usually uniseriate rays are usually about 22 cells or 150-200 μm high. The homocellular broad rays may be nearly 30 seriate. Cells within the broad rays often become lignified, beginning in the middle portion of the ray. Cells become very thick, usually retaining their original shape and size, but at times expanded and irregular in shape, and often contain crystals.

The transformed secondary phloem tissues in the rhytidome outside of the last-formed periderm are mostly lignified and expanded cells mixed with sclereid groups. Sieve tubes are usually crushed and phloem fibers are not as abundant as in the inner bark.

## SPECIFIC GRAVITY, EXTRACTIVES AND FIBROUS YIELD

Basic information on such bark properties as specific gravity, level of extractives, fiber yield and the presence of such morphological elements as phloem fibers and sclereids are expected to be useful in determining the need and possible methods of separating and segregating wood/bark chip mixtures*. Whenever possible, data on bark have been compared with similar information on wood.

## Specific Gravity

Table I summarizes the information available on wood and bark of northern white oak. Specific gravity is most often expressed in terms of ovendry weight divided by green volume. It should be noted that several of the values in Table I are ovendry weights divided by ovendry volumes. Information expressed in terms of green weight divided by green volume is useful when examining the possibilities of liquid flotation as a means of segregating wood/bark chip mixtures. Information in this report under the section Water Flotation Behavior compares the basic density (green weight divided by green volume) of northern white oak at several moisture contents.

An average specific gravity (ovendry weight/green volume) of approximately 0.64 appears appropriate for the wood of northern white oak. Our limited data do not show a large difference between heartwood and sapwood although, for the two trees sampled, the heartwood was higher in specific gravity.

The specific gravity of the total (inner + outer) bark of northern white oak appears somewhat lower than that of the wood. The inner bark of the two trees sampled as part of the project was higher in specific gravity than the outer bark. Overall values suggested for use in species comparisons are 0.64 for wood and 0.65, 0.52 and 0.58 for inner, outer and total bark.

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*Throughout this report the term separation has been used to designate separation or detachment of wood from bark while segregation has been used to indicate removal of either the bark or wood fraction from wood/bark chip mixtures.

### TABLE I

### NORTHERN WHITE OAK SPECIFIC GRAVITY INFORMATION

(Ovendry weight/green volume)

| Wood | Bark | | | |
|---|---|---|---|---|
| Average | Inner | Outer | Total | Reference & Remarks |
| 0.60 | | | | IUFRO (5) |
| 0.60 | | | | Besley (U.S.) (6) |
| 0.65 | | | | Besley (Canada) (6) |
| | 0.76 | 0.63 | | Fournier & Goulet (7) |
| 0.56 (sapwood)<br>0.68 (heartwood) | 0.62 | 0.48 | 0.53 | IPC 3212-17 |
| 0.64 (sapwood)<br>0.72 (heartwood) | 0.58 | 0.46 | 0.53 | IPC 3212-18 |
| 0.65 | | | | Project 2977 (bur oak) |
| 0.59 | | | | Isenberg (8) |
| | | | 0.68[a] | Harkin & Rowe (9) |
| 0.70[a] | | | | Isenberg (8) |

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[a] Ovendry weight/ovendry volume.

## Extractives

Extractives in wood and bark are important because, when present in large amounts, they not only result in reduced yield of fibrous material but ultimately can be expected to result in paper machine "pitch problems." Recent needs to reduce total water use through closed white water systems are expected to accentuate problems in this area. No attempt has been made in this report to go beyond determining the total alcohol-benzene extractives. Such extractives information is expected to provide an appropriate indication regarding possible pitch problems when large amounts of bark are pulped. Further detailed examination of the types of extractives involved is recommended using specific bark sources if preliminary comparisons suggest pitch and yield problems may develop.

A range in levels of extractives in wood from 2.2 to 2.6% was found in the two trees investigated as part of this project (see Table II). Based upon information obtained from the two trees investigated plus information from the literature, the bark of northern white oak can be expected to have an extractives level of 7.2%. This is a relatively low level and should not cause much of a problem except possibly in those instances where high percentages of bark have been concentrated in a particular chip fraction by screening or other mechanical treatments. Levels of extractives in the bark of both northern and southern white oak were comparable, 7.2 and 8.6% respectively.

TABLE II

NORTHERN WHITE OAK ALCOHOL-BENZENE EXTRACTIVES

| Type of Material | Extractives, % | Source |
|---|---|---|
| Wood | 2.6 | IPC 3212-17 |
| Wood | 2.2 | IPC 3212-18 |
| Bark | 4.6 | IPC 3212-17 |
| Bark | 7.4 | IPC 3212-4 |
| Bark | 9.8 | Murphey, _et al_. (_10_) |
| Bark | 7.1 | Harkin & Rowe (_9_) |

## Fibrous Yield

Increasing emphasis is being placed on pulping bark rather than debarking bolts or segregating wood/bark chip mixtures. Important to determining the usefulness of this approach with a particular species is determining the proportion of lignified cells that exist in the bark and that will survive normal cooking procedures. Also, it is important to determine what percentage of these cells will contribute in a favorable way to the resulting paper product.

Sclereids are short, thick-walled, heavily lignified cells. When not fully cooked, as could occur in high-yield pulping, clumps of sclereids may cause so-called "fish eyes" in certain grades (calendered) of paper. According to Chang

(1) 23.5% of the tissue elements in the inner bark of white oak are sclereids. However, most of these are lost in pulping the bark.

In the inner bark of some species there occur bands of heavily lignified fibers described in the literature as phloem fibers or sclerenchyma fibers. These fibers are the principal bark elements to survive chemical pulping and contribute to overall pulp yield and sheet strength. Chang (1) estimated that 18.7% of the inner bark of white oak was composed of phloem fibers based upon examination of cross sections. As a further check on pulp yield and the nature of fibrous material produced from northern white oak, 20 to 30-gram samples were pulped using the IPC Standard Kraft Micropulping Procedure. For a complete description of the procedure see the section on Experimental Procedures. Table III summarizes the results of this investigation.

Micropulping northern white oak bark resulted in a 31 to 38% yield of solids, based on three cooks. When screened, most (80-90%) of the material went through the 200-mesh screen. The elements retained on the coarse screens (60 and 100-mesh) were primarily fibers and sieve tubes. The "on 150 mesh" and "on 200 mesh" material was mainly sieve tubes with some fibers and sclereids. The "through 200 mesh" contained 90-95% peridermal and parenchymatous cells. Figure 3 illustrates the type of material retained on the 60 and 150-mesh screens.

Based upon very limited numbers of bark sample observations, it appears that, for every 100 grams of bark that is pulped, about 35 grams of solids will result. Of this 35 grams, about 3 grams (3%) of fibers and 0.2 gram (0.2%) of other material will be produced. This assumes that only the material on the 60 and 100-mesh screens would end up in and contribute in any significant way to the final product. The remaining material would be lost in washing and cleaning

## TABLE III

## NORTHERN WHITE OAK MICROPULPING INVESTIGATIONS

| Data | Sample No. 3212-17 | Sample No. 3212-18 | Remarks[a] |
|---|---|---|---|
| Yield, % solids | 37.8 | 37.0 | |
| Fraction | | | |
| on 60 mesh, % | 6.2 | 8.4 | Fraction contained 95+% phloem fibers, <5% sclereids and traces of sieve tubes (<1%), peridermal and parenchymatous cells (<1%), and crystalliferous parenchyma (<1%). The arithmetic average length of the phloem fibers in this fraction was 1.09 mm |
| on 100 mesh, % | 2.3 | 2.6 | Fraction contained principally phloem fibers (90-95%) with small percentages of sclereids (<5%), peridermal and parenchymatous cells (<5%) and traces of sieve tubes (<1%) and crystalliferous parenchyma (<1%) |
| on 150 mesh, % | 1.4 | 1.6 | Fraction contained fairly high percentages of sieve tubes (40-50%) and phloem fibers (30-40%) with small percentages of sclereids (10-20%), peridermal and parenchymatous cells (5-10%) and a trace of crystalliferous parenchyma (<1%). The arithmetic average length of sieve tubes in this fraction was 0.44 mm |
| on 200 mesh, % | 2.6 | 2.4 | Fraction contained sieve tubes (30-40%), sclereids (20-30%), peridermal and parencymatous cells (20-30%), phloem fibers (50-60%) and crystalliferous parenchyma (<5%) |
| through 200 mesh, % | 87.5 | 85.0 | Fraction contained fairly large percentages of peridermal and parenchymatous cells (40-50%), sclereids (20-30%) and crystalliferous parenchyma (20-30%) with a relatively small percentage of sieve tubes (<5%) and a trace of phloem fibers |

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[a] Percentages in each fraction on a weight basis.

Figure 3. The 60-Mesh Screen (Top) Contained by Weight Principally Phloem Fibers (95+%). The 150-Mesh Screen (Bottom) Contained High Percentages of Sieve Tubes (40-50%), Some Phloem Fibers (30-40%) with Smaller Percentages of Sclereids (10-20%). Magnification 75X. Symbols Include Sclereids (PS), Phloem Fibers (PF) and Sieve Tubes (ST)

operations. Results for both northern and southern white oak were similar with 3% fibers and 0.3% sieve tubes produced from pulping southern white oak.

## WOOD/BARK ADHESION

Wood/bark adhesion differences have been suggested as one of the reasons for the differences encountered in the ease of debarking pulpwood species. The same factors influencing debarking of pulpwood are expected to influence debarking of wood chips. The approach taken in the study has been to obtain growing season and dormant season information on (1) magnitude of wood/bark adhesion, (2) morphological structures associated with wood/bark adhesion, and (3) reasons for differences between species in adhesion.

Wood/bark adhesion values were measured for northern white oak samples collected July 24 (growing season) and October 22 (dormant season). Using the sampling and testing procedures described in the section on Experimental Procedures in Report One, shear parallel to the grain was measured. After testing, the samples were examined to determine the location of the zone of failure. Figure 4 illustrates the zone of failure for nothern white oak during both the growing and dormant seasons. During the growing season, wood/bark adhesion was low (4.8 $kg/cm^2$) and the failure zone was located between immature xylem cells in the region of the cambium zone. Also included in Fig. 4 is a large ray "pull-out" area. During the dormant season, wood/bark adhesion increased to 7.8 $kg/cm^2$ and the failure zone occurred in the inner bark, primarily between phloem sieve tubes and parenchyma cells and tangential bands of phloem fibers close to the cambium. The ray "pull-out" area extends into the xylem.

Bur oak, a member of the white oak family, was sampled for wood/bark adhesion measurements as part of an earlier project (Project 2929). The bark

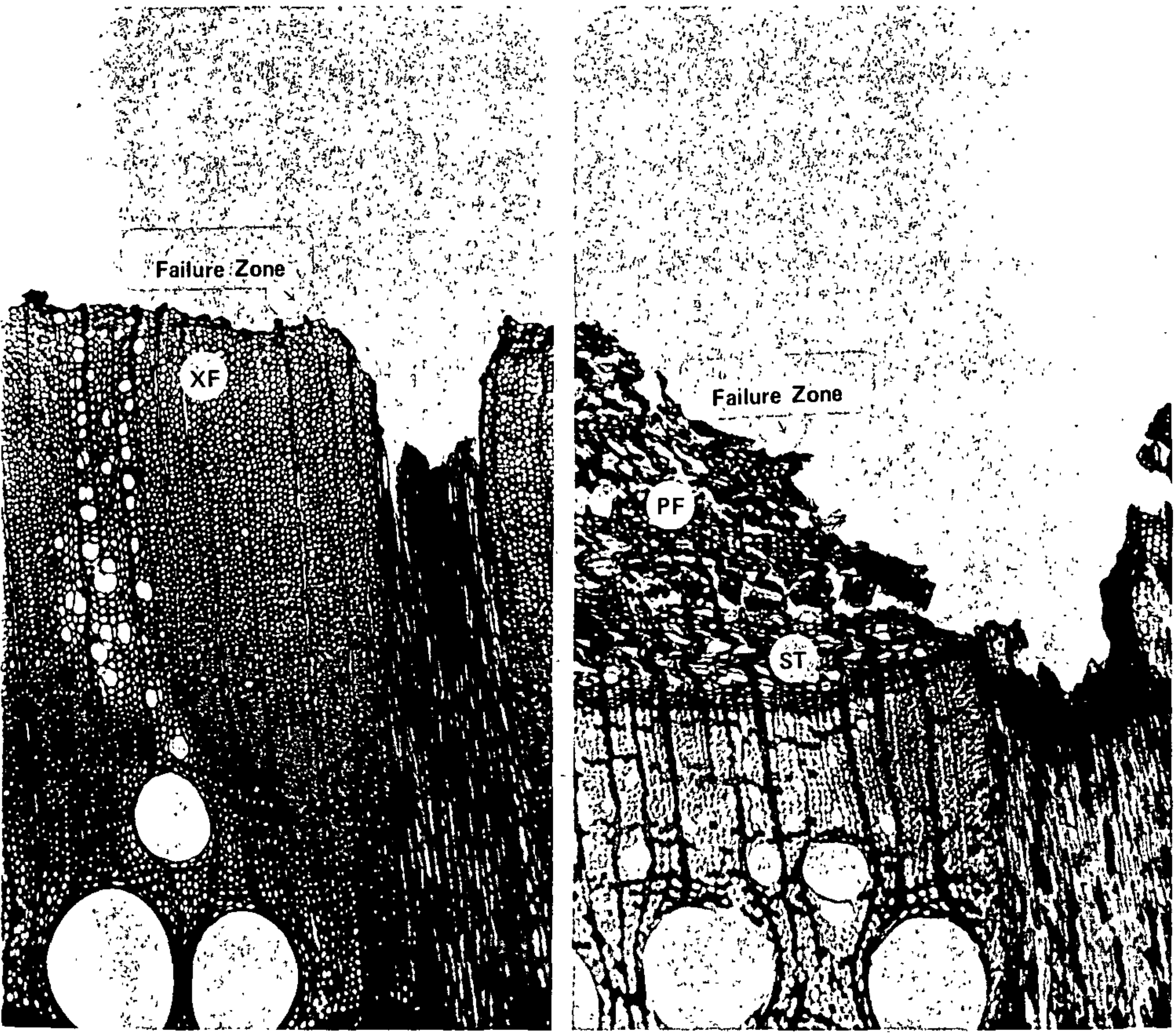

Figure 4. Illustrated Is the Northern White Oak Failure Zone for Both the Growing Season (Left) and Dormant Season (Right). The Growing Season Failure Zone Was Located Between Immature Newly-Formed Xylem Fibers (XF) in the Region of the Cambium Zone. During the Dormant Season, the Failure Zone Was Located in the Inner Bark, Primarily Between Phloem Sieve Tubes (ST), Parenchyma Cells and Bands of Phloem Fibers (PF) Close to the Cambium. Magnification 75X

peeling season extended from approximately April 28 to August 17. However, the peeling season may not normally last that long as the trees sampled in that project were growing on a moist site and benefited from favorable climatic conditions.

Failure during the growing season occurred in the cambium zone and moved to the inner bark during the dormant season, in the immature phloem sieve tube and parenchyma area. Wood/bark adhesion values for the growing and dormant seasons were 5.8 and 9.6 kg/cm$^2$, respectively. These values are somewhat higher than those obtained for northern white oak but still fairly comparable. Photomicrographs taken of both northern white oak and bur oak suggest that the large rays found in oak may "lock" the bark to the wood and make removal difficult during the dormant season.

As a result of measurement data taken on the species included in Appendix Table XXVIII and the measurement data reported in the previous reports for this project, it is clear that dormant season wood/bark adhesion is related to inner bark strength and inner bark strength is in turn related to inner bark morphology. The presence of phloem fibers in the inner bark of hardwoods appears to be associated with high dormant season wood/bark adhesion. High numbers of sclereids and a lack of phloem fibers seem to be associated with low dormant season wood/bark adhesion and low bark strength.

Separation (breaking the bond between bark and wood in a chip) is an important first step in segregation (removal of bark particles from wood chips). Separation during the growing season, when wood/bark adhesion is low, can usually be accomplished by the action of the chipper. During the dormant season, adhesion is greater and separation by chipper action is less successful. Use of a ring debarker (<u>11</u>) showed poor results for white oak in early spring to late fall with a ranking of 8-9* and moderate results in the winter using steam or a hot pond (ranking of 5-6*). However, bur oak bolts, collected in mid-November and chipped unfrozen in December, showed good separation of wood and bark through the chipper action. An estimated 10% or less of the bark chips had wood attached.

Several approaches were tried with hardwoods and two softwoods in Project 2929 to reduce adhesion that might have some promise. These methods included chemical, thermal, and biological methods. These methods have not been tried with northern white oak but are worthy of further consideration and are discussed in the section on Between-Species Comparisons.

## BARK STRENGTH, TOUGHNESS AND REACTION TO HAMMERMILLING

Bark strength and toughness measurements are included as part of the characterization of bark because it was felt that when these measurements are compared with the results obtained in wood/bark adhesion tests, with the difficulty encountered in conventional debarking and with bark morphology, the "why" of bark separation and segregation would eventually emerge.

Hammermilling has been widely used in bark utilization to prepare fractions for use as horticultural mulch, soil conditioners, and as additives to a number of types of products. A simulated hammermilling test was developed in an effort to relate the hammermilling of bark (and wood) to bark strength, toughness and morphology.

As discussed in the section on Experimental Procedures, bark strength measures shear parallel to the grain while bark toughness measures the energy

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*Ranking system of 1 — easy to debark to 10 — hard to debark.

required to rupture a thin specimen by a bending force perpendicular to the grain (parallel to the tree diameter). Table IV summarizes the bark strength and toughness tests made on the wood and bark of northern white oak. Also included for comparison purposes (Appendix Table XXIX) are the bark strength values for a number of pulpwood species of interest.

TABLE IV

SUMMARY OF STRENGTH AND TOUGHNESS MEASUREMENTS MADE ON WOOD AND BARK OF NORTHERN WHITE OAK[a]

| Material | Strength | Toughness |
|---|---|---|
| Wood | -- | 0.62 |
| Inner bark | 4.6 | 0.16 |
| Outer bark | 3.2 | 0.10 |

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[a] Determinations average of two different trees.

The inner bark strength of northern white oak is very comparable to that of bur oak (4.6 vs. 4.5 kg/cm$^2$) which is also a member of the white oak family. The strength of the inner bark of northern white oak is less than that of quaking aspen and eastern cottonwood, for example, but more than white birch and sugar maple. This is probably due to the presence of some phloem fibers in the inner bark of northern white oak. White birch and sugar maple have essentially no fiber in the inner bark while aspen and cottonwood have greater amounts of fiber than oak.

Hammermilling, followed by screening, resulted in a moderate reduction in levels of bark. When the half-sized chips for the two trees investigated were hammermilled, and the material on the 14-mesh screen retained, the result was a 5% loss in wood and a 37% reduction in bark. The bark removed appeared to be mostly outer bark. Since the inner bark has a specific gravity near that of the wood, hammermilling, followed by water flotation, would not, in all probability, result in effective segregation. However, the fiber contained in the inner bark could be of some value. If the material on the 5 and 10-mesh screens only were retained, the results would be a 46% reduction in levels of bark and a 7% loss in wood. This additional 2% wood loss might be acceptable in view of the increased amount of bark removed. Summarized in Table V are the results of the hammermilling tests run on northern white oak wood and bark. Figure 5 illustrates the effect of hammermilling on the wood and bark of northern white oak. The wood loss for northern white oak was considerably less than that for northern red oak (see Project 3212, Progress Report One). It is possible that improvements could be made in screening by taking advantage of the differences in configuration of wood and bark chips evident in Fig. 5 (12,13). Summary Table XXVI compares bark strength, toughness and reaction to hammermilling of northern white oak to other species tested thus far.

## WATER FLOTATION BEHAVIOR

One possible method of segregating wood/bark chip mixtures is by water flotation. Knowledge of the flotation characteristics of wood and bark is also expected to be important when certain types of chip washing procedures are employed. Earlier investigations into water flotation segregation (Project 2977) revealed that chip size, specific gravity, moisture content and rate of moisture uptake were factors in the flotation behavior of bark and wood chips. Budget limitations do not permit examination of all factors involved and, as a result, the influence of chip size has been eliminated from the variables considered.

## TABLE V

## SUMMARY OF HAMMERMILLING TEST ON NORTHERN WHITE OAK

| | | Fraction Retained on Standard Screen[a], % | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Tree No. | Type Material | 5 Mesh | 10 Mesh | 14 Mesh | 20 Mesh | 28 Mesh | <28 Mesh | Remarks |
| 3212-17 | Bark | 20 | 29 | 11 | 7 | 8 | 25 | Larger mesh screens contained equal amounts of inner & outer bark. More outer bark on smaller screens. Inner bark stringy |
| | Sapwood | 57 | 30 | 6 | 2 | 1 | 2 | |
| | Heartwood | 87 | 7 | 2 | 1 | 1 | 2 | |
| 3212-18 | Bark | 38 | 22 | 7 | 5 | 6 | 22 | More inner than outer bark on larger mesh screens. Increasing amount of outer bark on smaller mesh screens. Inner bark stringy |
| | Sapwood | 82 | 11 | 2 | 1 | 1 | 2 | |
| | Heartwood | 84 | 10 | 1 | 1 | 1 | 3 | |

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[a] Standard soil screen sizes; 5 mesh has 5 wires per inch and an opening of 4.00 mm, 10 mesh has 10 wires per inch and an opening of 2.0 mm, 14 mesh has 14 wires per inch and an opening of 1.168 mm, 20 mesh has 20 wires per inch and an opening of 1.00 mm, and the 28 mesh screen has 28 wires per inch and an opening of 0.589 mm.

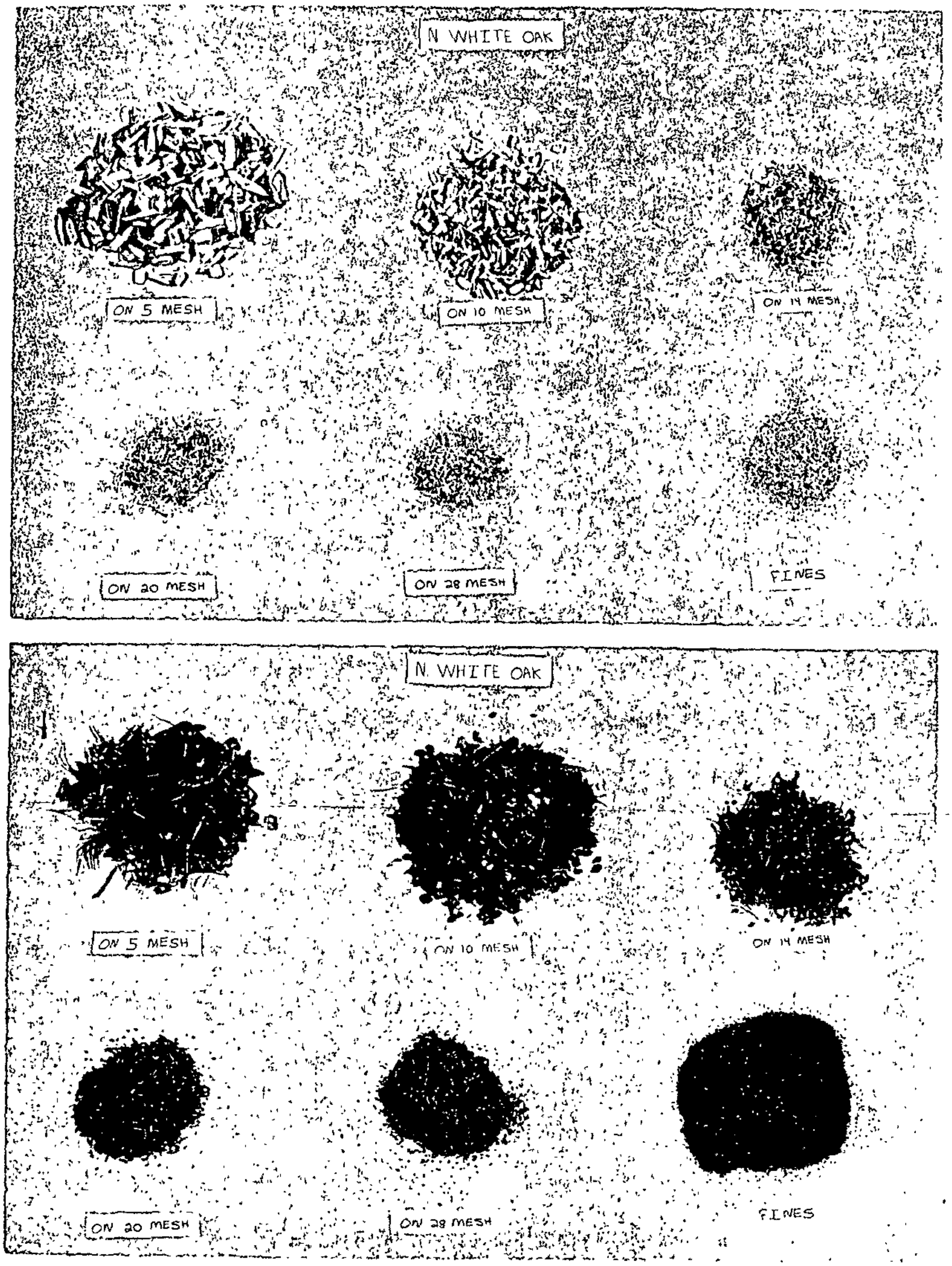

Figure 5. Illustrated Is the Effect of Hammermilling on Northern White Oak Wood (Top) and Bark (Bottom)

Two procedures were used to examine the water flotation behavior of wood and bark. One procedure involved measuring the density* (green weight divided by green volume) of simulated chips at a number of different moisture contents. The second technique involved measuring the rate of moisture uptake and sinking of wood and bark chips in what have been designated as "dwell time" studies.

## Density Determinations

Simulated chips were used in determining the relationship between moisture content and the density of bark and wood. Wood and bark from two northern white oak trees (IPC 3212-17 and IPC 3212-18) were used in making the determinations. The moisture content of the chip samples was adjusted by equilibrating in small jars to which had been added appropriate amounts of water. The extremely accurate pycnometer method described in the Experimental Procedures in Report One was used in determining density. Bark samples used were "whole bark" samples, a combination of both inner and outer bark. Small chips of inner and outer barks were also tested. Our limited data showed the inner bark to be higher in density than whole bark and the outer bark slightly lower.

Figure 6 illustrates the relationship that was found between moisture content and density. The linear relationship shown was obtained by fitting the least squares regression line through the data. The dashed lines are two

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*The term density is used in this report to indicate the weight of wood and bark samples and is expressed in terms of green weight divided by green volume. This is in contrast to the term specific gravity, which also is an expression of the weight of a sample, but in this case it is in terms of dry weight divided by green volume.

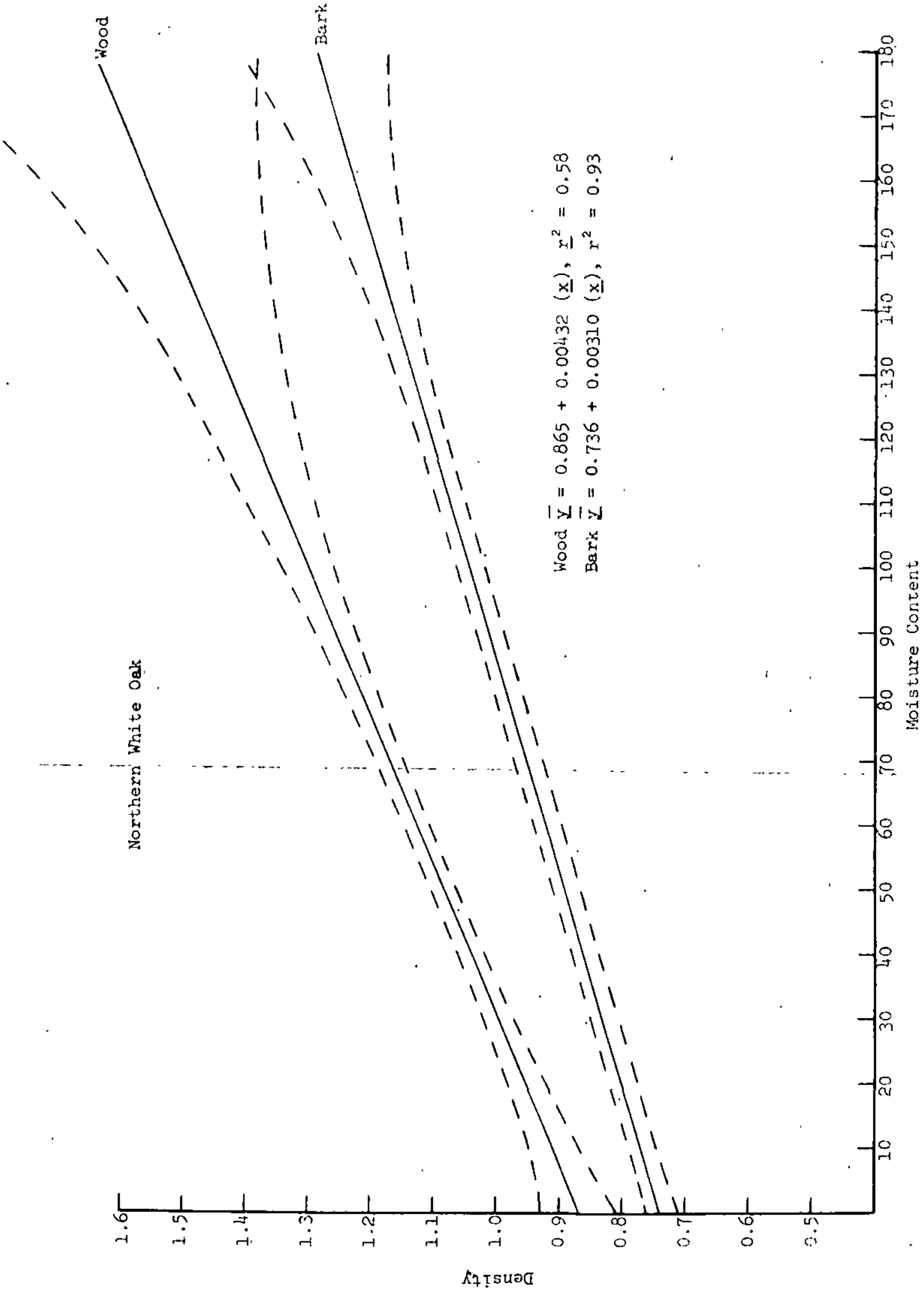

Figure 6. Illustrated Is the Relationship Between Basic Density and Moisture Content for Northern White Oak. The Dashed Lines are Two Standard Deviations Above and Below the Mean

standard deviations above and below the average values. The standard deviation of the regression line is considerably less than would have been obtained if conventional mill-run chips had been used for the water flotation studies, because the simulated chips were uniform in size and shape, had a uniform level of moisture and were relatively free of knots, reaction wood, etc. Water segregation is believed to be possible when one fraction has a density of less than one and the other greater than one at a specific moisture content.

The data indicate that at moisture contents of between 45 and 70%, most wood chips could be expected to sink (density greater than 1). Northern white oak bark, on the other hand, when at the same moisture level, could be expected to float. However, this is a rather narrow range of moisture contents. In previous work done at the Institute (Project 2977), the bark of bur oak, a member of the white oak family, also showed a strong tendency to float. In these tests, no effective segregation took place at 20% moisture content. At 45% moisture the results were more satisfactory with 95% of the wood recovered and 0.5% bark contamination in the "on 3/4-inch" chips. Bark contamination in the "on 1/4-inch" chips was 12.7%. These results are in agreement with the density-moisture content curves for northern white oak wood and bark.

## Dwell Time Investigations

An investigation of dwell time involves nothing more than taking wood and bark chips at some standard moisture content, placing them on a water surface and observing the time it takes the material to pick up enough water to sink. Information on dwell time is useful because moisture uptake rates could have a considerable influence on the success of a segregation procedure (or chip-washing procedure) and would provide information on the rate at which segregation could be expected. A species in which either the bark or the wood takes up moisture rapidly could be expected to have a relatively short segregation time. For other species, where specific gravity and density of the wood and bark are similar, and moisture uptake is similar, considerable difficulty in segregation can be anticipated. Time required for chip samples to sink decreases as the sample moisture content at the start of the trial increases.

Half-sized simulated chips (1 x 0.3 x 0.2 inch) were used in the dwell time tests. Prior to testing, the samples were equilibrated in 50% RH and had a moisture content of approximately 20% (ovendry basis). Table VI summarizes the results for northern white oak. These results agree fairly closely with the results obtained in the density determinations and indicate that segregation of wood/bark chip mixtures through water flotation is not a very useful method due to the narrow range of moisture contents at which segregation could be achieved.

## DATA INTERPRETATION

The bark of northern white oak contains fiber and some sclereids. However, when pulped, most of the sclereids are lost. For every 100 grams of bark that are pulped, about 3 grams of fiber and 0.2 gram of other material will be produced. With the fiber yield, lack of sclereids in the pulp and low bark extractives (7%), this species may be a suitable candidate in some instances for pulping with the bark.

Segregation of wood/bark chip mixtures through water flotation is possible but the small range of moisture contents at which it could be achieved do not make it a feasible method.

Separation of wood and bark through chipper action and hammermilling appears to have some possibility. Bur oak, a member of the white oak family,

## TABLE VI

## SUMMARY OF DWELL TIME RESULTS FOR NORTHERN WHITE OAK[a]

| Sample No. | Time Interval, min | Sinkers, % | Floaters, % |
|---|---|---|---|
| IPC 3212-17 | after 5 | 0 | 100 |
| Bark | 15 | 0 | 100 |
| | 60 | 1.2 | 98.8 |
| | 240 | 2.6 | 97.4 |
| IPC 3212-17 | after 5 | 0 | 100 |
| Sapwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-17 | after 5 | 0 | 100 |
| Heartwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0.7 | 99.3 |
| IPC 3212-18 | after 5 | 0 | 100 |
| Bark | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-18 | after 5 | 0 | 100 |
| Sapwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 6.7 | 93.3 |
| IPC 3212-18 | after 5 | 0 | 100 |
| Heartwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 6.6 | 93.4 |

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[a] Starting moisture content 20%.

when chipped during the dormant season had 10% or less of the bark chips with wood attached. Hammermilling white oak wood and bark chips resulted in a 5% wood loss and 37% bark reduction based upon material retained on the 14-mesh screen. When only material on the 10-mesh and larger screens was retained, the result was a 46% reduction in bark and a 7% wood loss. It is possible that a quick separation could be made by screening, hammermilling the fractions high in bark and rescreening.

## RELATED LITERATURE

There are a number of papers on the economics and mechanics of segregating wood/bark chip mixtures. They include papers by Auchter and Horn (14), Hooper (15) and Biltonen, *et al*. (16).

# BARK AND WOOD PROPERTIES OF SOUTHERN WHITE OAK (Quercus alba L.)

## SILVICULTURAL CHARACTERISTICS AND GEOGRAPHIC RANGE

White oak is widespread throughout the eastern and central United States, occupying a wide variety of soil types. See the description in the northern white oak section for more details on silvicultural characteristics and geographic range.

## WOOD AND BARK MORPHOLOGY

The morphology of wood and bark is the same for both northern and southern sources of white oak. The morphology section for northern white oak should be reviewed for details on the structure of wood and bark.

## SPECIFIC GRAVITY, EXTRACTIVES AND FIBROUS YIELD

Basic information on such bark properties as specific gravity, level of extractives, fiber yield and the presence of such morphological elements as phloem fibers and sclereids are expected to be useful in determining the need and possible methods of separating and segregating wood/bark chip mixtures.* Whenever possible, data on bark have been compared with similar information on wood.

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*Throughout this report the term separation has been used to designate separation or detachment of wood from bark while segregation has been used to indicate removal of either the bark or wood fraction from wood/bark chip mixtures.

## Specific Gravity

Table VII summarizes the information available on wood and bark of southern white oak. Specific gravity is most often expressed in terms of ovendry weight divided by green volume. It should be noted that one of the values in Table VII is ovendry weight divided by ovendry volume. Information expressed in terms of green weight divided by green volume is useful when examining the possibilities of liquid flotation as a means of segregating wood/bark chip mixtures. Information in this report under the section Water Flotation Behavior compares the basic density (green weight divided by green volume) of southern white oak at several moisture contents.

TABLE VII

SOUTHERN WHITE OAK SPECIFIC GRAVITY INFORMATION

(Ovendry weight/green volume)

| Wood | Bark | | | |
|---|---|---|---|---|
| Average | Inner | Outer | Total | References & Remarks |
| 0.60 | | | | IUFRO (5) |
| 0.59 | | | | Isenberg (8) |
| 0.68 (sapwood) | 0.71 | 0.45 | 0.57 | IPC 3212-62 |
| 0.69 (heartwood) | | | | |
| 0.66 (sapwood | 0.68 | 0.43 | 0.56 | IPC 3212-63 |
| 0.71 (heartwood) | | | | |
| 0.67 (stem wood). | | | 0.54 (stem bark) | Manwiller (17) |
| 0.64 (branch wood) | | | 0.49 (branch bark) | Manwiller (17) |
| | | | 0.61[a] | Harkin & Rowe (post oak) (9) |

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[a] Ovendry weight/ovendry volume.

An average specific gravity (ovendry weight/green volume) of approximately 0.67 appears appropriate for the wood of southern white oak. Our limited data do not show a large difference between heartwood and sapwood although, for the two trees sampled, the heartwood was higher in specific gravity.

The specific gravity of the total (inner + outer) bark of southern white oak appears somewhat lower than that of the wood. The inner bark of the two trees sampled as part of the project was higher in specific gravity than the outer bark. These are the same trends as reported for northern white oak. Overall values suggested for use in species comparisons are 0.67 for wood and 0.70, 0.44 and 0.56 for inner, outer, and total bark. For additional information on the specific gravity of white oak wood and bark see the table for northern white oak (Table I).

## Extractives

Extractives in wood and bark are important because, when present in large amounts, they not only result in reduced yield of fibrous material but ultimately can be expected to result in paper machine "pitch problems." Recent needs to reduce total water use through closed white water systems are expected to accentuate problems in this area. No attempt has been made in this report to go beyond determining the total alcohol-benzene extractives. Such extractives information is expected to provide an appropriate indication regarding possible pitch problems when large amounts of bark are pulped. Further detailed examination of the types of extractives involved is recommended using specific bark sources if preliminary comparisons suggest pitch and yield problems may develop.

A level of extractives in wood of 5.0 was reported in one study (see Table VIII). This is just slightly higher than the extractives level reported for northern white oak wood. Based upon information obtained from the two trees investigated plus information from the literature, the bark of southern white oak can be expected to have an extractives level of 8.6%. Investigations on chestnut oak bark (<u>18</u>) indicate inner bark has higher levels of extractives than outer bark and dormant season levels of extractives are higher than growing season levels. This may, in part, account for the variation encountered. This is a relatively low level and should not cause much of a problem except possibly in those instances where high percentages of bark have been concentrated in a particular chip fraction by screening or other mechanical treatments. Extractives levels for northern white oak averaged 7.2%.

TABLE VIII

SOUTHERN WHITE OAK ALCOHOL-BENZENE EXTRACTIVES

| Type of Material | Extractives, % | Source |
|---|---|---|
| Stemwood | 5.0 | Manwiller (17) |
| Branchwood | 5.9 | Manwiller (17) |
| Wood | 4.1 | Isenberg (post oak) (8) |
| Stem bark | 12.9 | Manwiller (17) |
| Branch bark | 16.3 | Manwiller (17) |
| Bark | 6.4 | IPC 3212-62 |
| Bark | 6.4 | IPC 3212-63 |

## Fibrous Yield

Increasing emphasis is being placed on pulping bark rather than debarking bolts or segregating wood/bark chip mixtures. Important to determining the usefulness of this approach with a particular species is determining the proportion of lignified cells that exist in the bark and that will survive normal cooking procedures. Also, it is important to determine what percentage of these cells will contribute in a favorable way to the resulting paper product.

Sclereids are short, thick-walled, heavily lignified cells. When not fully cooked, as could occur in high-yield pulping, clumps of sclereids may cause so-called "fish eyes" in certain grades (calendered) of paper. As stated in the section on northern white oak, Chang (1) found that 23.5% of the tissue elements in the inner bark of white oak are sclereids. However, most of these are lost in pulping the bark.

In the inner bark of some species there occurs bands of heavily lignified fibers described in the literature as phloem fibers or sclerenchyma fibers. These fibers are the principal bark elements to survive chemical pulping and contribute to overall pulp yield and sheet strength. Chang (1) estimated that 18.7% of the inner bark of white oak was composed of phloem fibers based upon examination of cross sections. As a further check on pulp yield and the nature of fibrous material produced from southern white oak, 20 to 30-gram samples were pulped using the IPC Standard Kraft Micropulping Procedure. For a complete description of the procedure see the section on Experimental Procedures. Table IX summarizes the results of this investigation.

Micropulping southern white oak bark resulted in approximately a 37% yield of solids. When screened, most (75-85%) of the material went through the 200-mesh screen. The elements retained on the coarse screens (60 and 100 mesh) were primarily fibers. The "on 150 mesh" and "on 200 mesh" material was mainly sieve tubes, fibers and sclereids. The "through 200 mesh" contained 70-80% schlereids. Figure 7 illustrates the type of material retained on the 60 and 150-mesh screens.

Based upon very limited numbers of bark sample observations, it appears that, for every 100 grams of bark that are pulped, about 37 grams of solids will result. Of this 37 grams, about 3 grams (3%) of fibers and 0.3 gram (0.3%) of sieve tubes will be produced. This assumes that only the material on the 60 and 100-mesh screens would end up in and contribute in any significant way to the final product. The remaining material would be lost in washing and cleaning operations. These results are very similar to those obtained for northern white oak.

## TABLE IX

## SOUTHERN WHITE OAK MICROPULPING INVESTIGATIONS

| Data | Sample No. 3212-62 | Sample No. 3212-63 | Remarks[a] |
|---|---|---|---|
| Yield, % solids | 36.5 | 36.7 | |
| Fraction | | | |
| on 60 mesh, % | 6.4 | 10.8 | Fraction contained 95+% phloem fibers with a small percentage of sieve tubes (<5%), traces of sclereids (<1%), peridermal and parenchymatous cells (<1%) and crystalliferous parenchyma (<1%). The arithmetic average length of the phloem fibers in this fraction was 1.15 mm |
| on 100 mesh, % | 2.6 | 4.0 | Fraction contained a large percentage of phloem fibers (70-80%) with small percentages of sieve tubes (10-20%), peridermal and parenchymatous cells (5-10%), sclereids (<5%) and crystalliferous parenchyma (<5%). The arithmetic average length of the sieve tubes in this fraction was 0.54 mm |
| on 150 mesh, % | 3.2 | 5.6 | Fraction contained sieve tubes (40-50%), phloem fibers (20-30%), peridermal and parenchymatous cells (10-20%), sclereids (5-10%), and crystalliferous parenchyma (<5%). |
| on 200 mesh, % | 4.0 | 4.6 | Fraction contained relatively high percentages of sclereids (40-50%) and sieve tubes (30-40%), with small percentages of peridermal and parenchymatous cells (5-10%), phloem fibers (<5%) and crystalliferous parenchyma (<5%) |
| through 200 mesh, % | 83.8 | 75.0 | Fraction contained a large percentage of sclereids (70-80%) with smaller percentages of peridermal and parenchymatous cells (10-20%) and a trace of sieve tubes (<1%) |

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[a] Percentages in each fraction on a weight basis.

Figure 7. The 60-Mesh Screen (Top) Contained by Weight Principally Phloem Fibers (95+%). The 150-Mesh Screen (Bottom) Contained Sieve Tubes (40-50%), Phloem Fibers (20-30%), Peridermal and Parenchymatous Cells (10-20%) and Sclereids (5-10%). Magnification 75X. Symbols Illustrate Phloem Fibers (PF), Sieve Tubes (ST) and Sclereids (PS)

## WOOD/BARK ADHESION

Wood/bark adhesion differences have been suggested as one of the reasons for the differences encountered in the ease of debarking pulpwood species. The same factors influencing debarking of pulpwood are expected to influence debarking of wood chips. The approach taken in the study has been to obtain growing season and dormant season information on (1) magnitude of wood/bark adhesion, (2) morphological structures associated with wood/bark adhesion, and (3) reasons for differences between species in adhesion.

Wood/bark adhesion values were measured for southern white oak samples collected only during the dormant season (February 6). Using the sampling and testing procedures described in the section on Experimental Procedures in Report One, shear parallel to the grain was measured. After testing, the samples were examined to determine the location of the zone of failure. Figure 8 illustrates the zone of failure for southern white oak during the dormant season. The failure zone during the dormant season occurred in the inner bark between phloem parenchyma cells, sieve tubes, and tangential bands of phloem fibers close to the cambium. Wood/bark adhesion measured 7.2 kg/cm$^2$, very close to the value obtained for northern white oak (7.8 kg/cm$^2$). It is likely that growing season adhesion values would also be close to those obtained for northern white oak (4.8 kg/cm$^2$).

As a result of measurement data taken on the species included in Appendix Table XXVIII and the measurement data reported in the previous reports for this project, it is clear that dormant season wood/bark adhesion is related to inner bark strength and inner bark strength is, in turn, related to inner bark morphology. The presence of phloem fibers in the inner bark of hardwoods appears to be associated with high dormant season wood/bark adhesion. High numbers of sclereids and a lack of phloem fibers seem to be associated with low dormant season wood/bark adhesion and low bark strength.

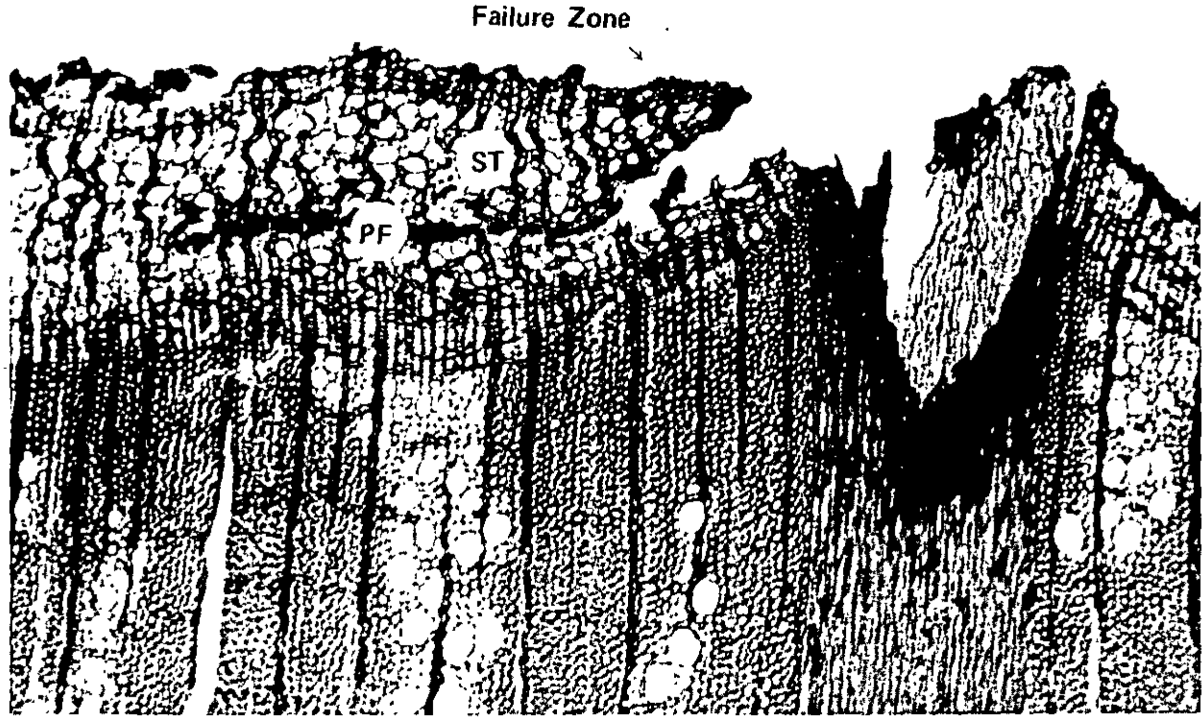

Figure 8. Illustrated Is the Southern White Oak Dominant Season Failure Zone. The Dormant Season Failure Zone Was Located in the Inner Bark Between Phloem Parenchyma Cells, Sieve Tubes (ST) and Tangential Bands of Phloem Fibers (PF) Close to the Cambium. Magnification 75X

Separation (breaking the bond between bark and wood in a chip) is an important first step in segregation (removal of bark particles from wood chips). Separation during the growing season, when wood/bark adhesion is low, can usually be accomplished by the action of the chipper. During the dormant season, adhesion is greater and separation by chipper action is less successful.

As mentioned previously, several approaches were tried with hardwoods and two softwoods in Project 2929 to reduce adhesion that might have some promise. These methods included thermal, chemical, and biological methods. These methods have not been tried with southern white oak but are worthy of further consideration and are discussed in the section on Between-Species Comparisons.

## BARK STRENGTH, TOUGHNESS AND REACTION TO HAMMERMILLING

Bark strength and toughness measurements are included as part of the characterization of bark because it was felt that when these measurements are compared with the results obtained in wood/bark adhesion tests, with the difficulty encountered in conventional debarking and with bark morphology, the "why" of bark separation and segregation would eventually emerge.

Hammermilling has been widely used in bark utilization to prepare fractions for use as horticultural mulch, soil conditioners, and as additives to a number of types of products. A simulated hammermilling test was developed in an effort to relate the hammermilling of bark (and wood) to bark strength, toughness and morphology.

As discussed in the section on Experimental Procedures, bark strength measures shear parallel to the grain while bark toughness measures the energy required to rupture a thin specimen by a bending force perpendicular to the grain (parallel to the tree diameter). Table X summarizes the bark strength and toughness tests made on the wood and bark of southern white oak. Also included for comparison purposes (Table XXIX, Appendix) are the bark strength values for a number of pulpwood species of interest.

Unfortunately, bark strength was run on the total bark for southern white oak rather than separately on the inner and outer bark. However, the value obtained, 4.7 kg/cm$^2$, is similar to the values obtained for the other oaks investigated. Again, the value is greater than that obtained for species with little or no fiber in the inner bark. The higher value is probably associated with the presence of some phloem fiber in the inner bark of southern white oak.

TABLE X

SUMMARY OF STRENGTH AND TOUGHNESS MEASUREMENTS
MADE ON WOOD AND BARK OF SOUTHERN WHITE OAK[a]

| Material | Strength | Toughness |
|---|---|---|
| Wood | -- | 0.98 |
| Inner bark | 4.7[b] | 0.12 |
| Outer bark | | 0.09 |

---

[a] Determinations average of two different trees.

[b] Test mistakenly performed on total bark.

Toughness tests have been run on a number of species and results given in Wood Handbook (19). The tests were done on both tangential and radial sections and represented the energy required to rapidly cause complete failure in a centrally loaded bending specimen. Our toughness test is done on a tangential section at 20% moisture content and is a measure of the energy required to rupture a thin specimen by a bending force perpendicular to the grain. At 13-14% moisture content Wood Handbook results show overcup oak (a southern white oak) with a tangential value of 310 and sugar maple with 360. Our results also indicate that sugar maple wood is somewhat tougher than southern white oak with values of 0.98 for southern white oak and 1.20 for sugar maple. When enough data are obtained, correlations will be run between IPC values and hammermilling results.

Hammermilling, followed by screening, resulted in a moderate reduction in levels of bark. When the half-sized chips for the two trees investigated were hammermilled, and the material on the 14-mesh screen retained, the result was a 3% loss in wood and a 38% reduction in bark. The bark removed appeared to be mostly outer bark. These results compare favorably with those obtained for northern white oak. Again, since the inner bark has a specific gravity near that of the wood, hammermilling, followed by water flotation would not, in all probability, results in effective segregation. However, the fiber contained in the inner bark could be of some value. If the material on the 5 and 10-mesh screens only were retained, the results would be a 58% reduction in levels of bark and a 6% wood loss. This additional 3% loss in wood might be acceptable in view of the increased amount of bark removed. Summarized in Table XI are the results of the hammermilling tests run on southern white oak wood and bark. Figure 9 illustrates the effect of hammermilling on wood and bark of southern white oak. It is possible that improvements could be made in screening by taking advantage of the differences in configuration of wood and bark chips evident in Fig. 9 (12,13). Summary Table XXVI compares bark strength, toughness and reaction to hammermilling of southern white oak to other species tested thus far.

## WATER FLOTATION BEHAVIOR

One possible method of segregating wood/bark chip mixtures is by water flotation procedures. Knowledge of the flotation characteristics of wood and bark is also expected to be important when certain types of chip washing procedures are employed. Earlier investigations into water flotation segregation (Project 2977) revealed that chip size, specific gravity, moisture content and rate of moisture uptake were factors in the flotation behavior of bark and wood chips. Budget limitations do not permit examination of all factors involved and, as a result, the influence of chip size has been eliminated from the variables considered.

## TABLE XI

## SUMMARY OF HAMMERMILLING TEST ON SOUTHERN WHITE OAK

| Tree No. | Type Material | Fraction Retained on Standard Screen[a], % 5 Mesh | 10 Mesh | 14 Mesh | 20 Mesh | 28 Mesh | <28 Mesh | Remarks |
|---|---|---|---|---|---|---|---|---|
| 3212-62 | Bark | 24 | 20 | 17 | 9 | 10 | 19 | More inner than outer bark on larger mesh screens. Increasing amounts of outer bark on smaller mesh screens. Inner bark stringy |
| | Sapwood | 86 | 9 | 1 | 1 | 1 | 2 | |
| | Heartwood | 89 | 7 | 1 | 1 | <1 | 2 | |
| 3212-63 | Bark | 16 | 25 | 21 | 11 | 10 | 18 | Same as above |
| | Sapwood | 84 | 9 | 2 | 1 | 1 | 3 | |
| | Heartwood | 90 | 6 | 1 | 1 | <1 | 2 | |

---

[a] Standard soil screen sizes; 5 mesh has 5 wires per inch and an opening of 4.00 mm, 10 mesh has 10 wires per inch and an opening of 2.0 mm, 14 mesh has 14 wires per inch and an opening of 1.168 mm, 20 mesh has 20 wires per inch and an opening of 1.00 mm, and the 28 mesh screen has 28 wires per inch and an opening of 0.589 mm.

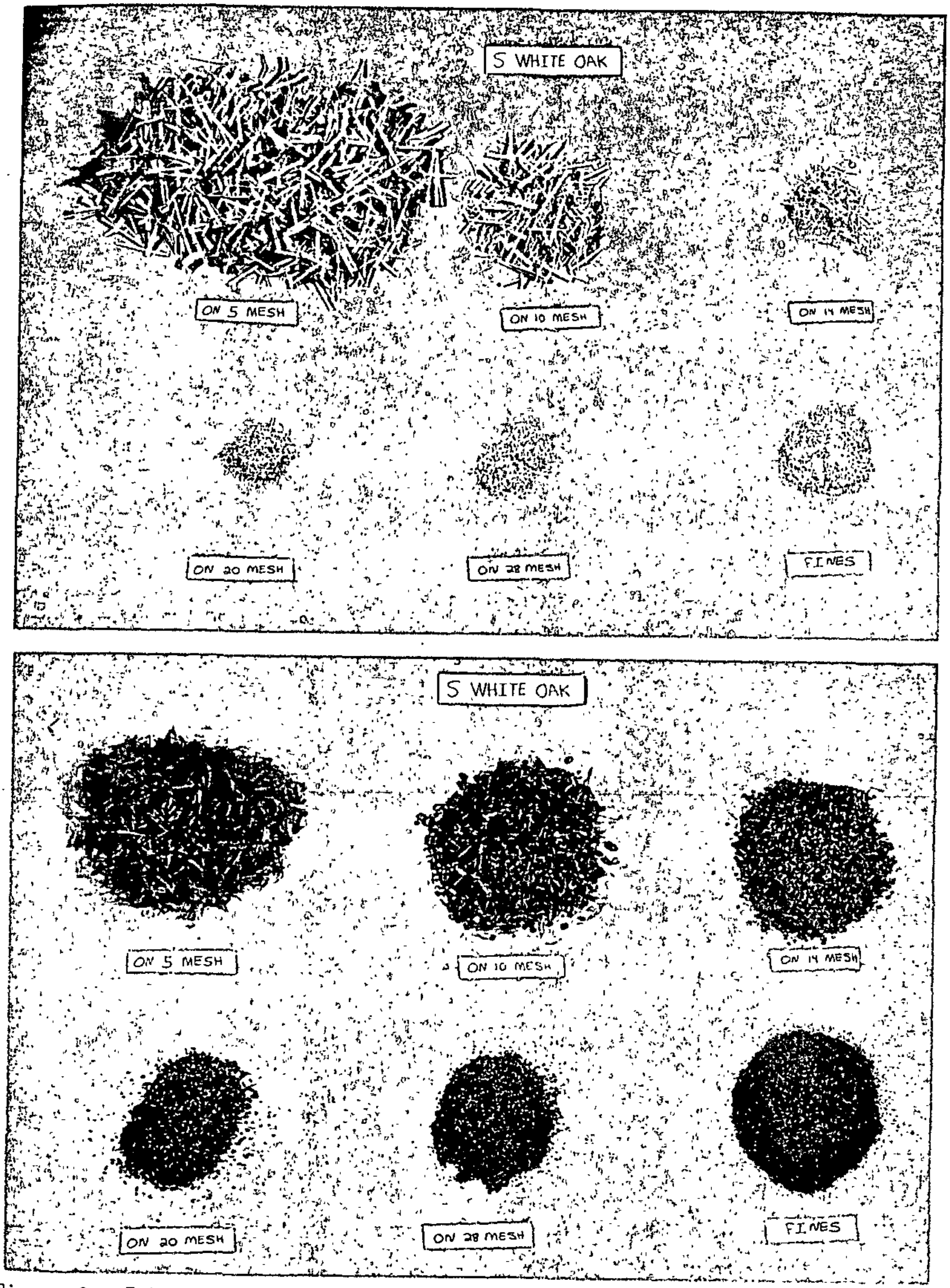

Figure 9. Illustrated Is the Effect of Hammermilling on Southern White Oak Wood (Top) and Bark (Bottom)

Two procedures were used to examine the water flotation behavior of wood and bark. One procedure involved measuring the density* (green weight divided by green volume) of simulated chips at a number of different moisture contents. The second technique involved measuring the rate of moisture uptake and sinking of wood and bark chips in what have been designated as "dwell time" studies.

## Density Determinations

Simulated chips were used in determining the relationship between moisture content and the density of bark and wood. Wood and bark from two southern white oak trees (IPC 3212-62 and IPC 3212-63) were used in making the determinations. The moisture content of the chip samples was adjusted by equilibrating in small jars to which had been added appropriate amounts of water. The extremely accurate pycnometer method described in the Experimental Procedures in Report One was used in determining density. Bark samples used were "whole bark" samples, a combination of both inner and outer bark. Small chips of inner and outer bark were also tested. As with northern white oak, our limited data showed the inner bark to be higher in density than whole bark and the outer bark slightly lower.

Figure 10 illustrates the relationship that was found between moisture content and density. The linear relationship shown was obtained by fitting the least squares regression line through the data. The dashed lines are two standard deviations above and below the average values. The standard deviation of the regression line is considerably less than would have been obtained if conventional

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*The term density is used in this report to indicate the weight of wood and bark samples and is expressed in terms of green weight divided by green volume. This is in contrast to the term specific gravity, which also is an expression of the weight of a sample, but in this case it is in terms of dry weight divided by green volume.

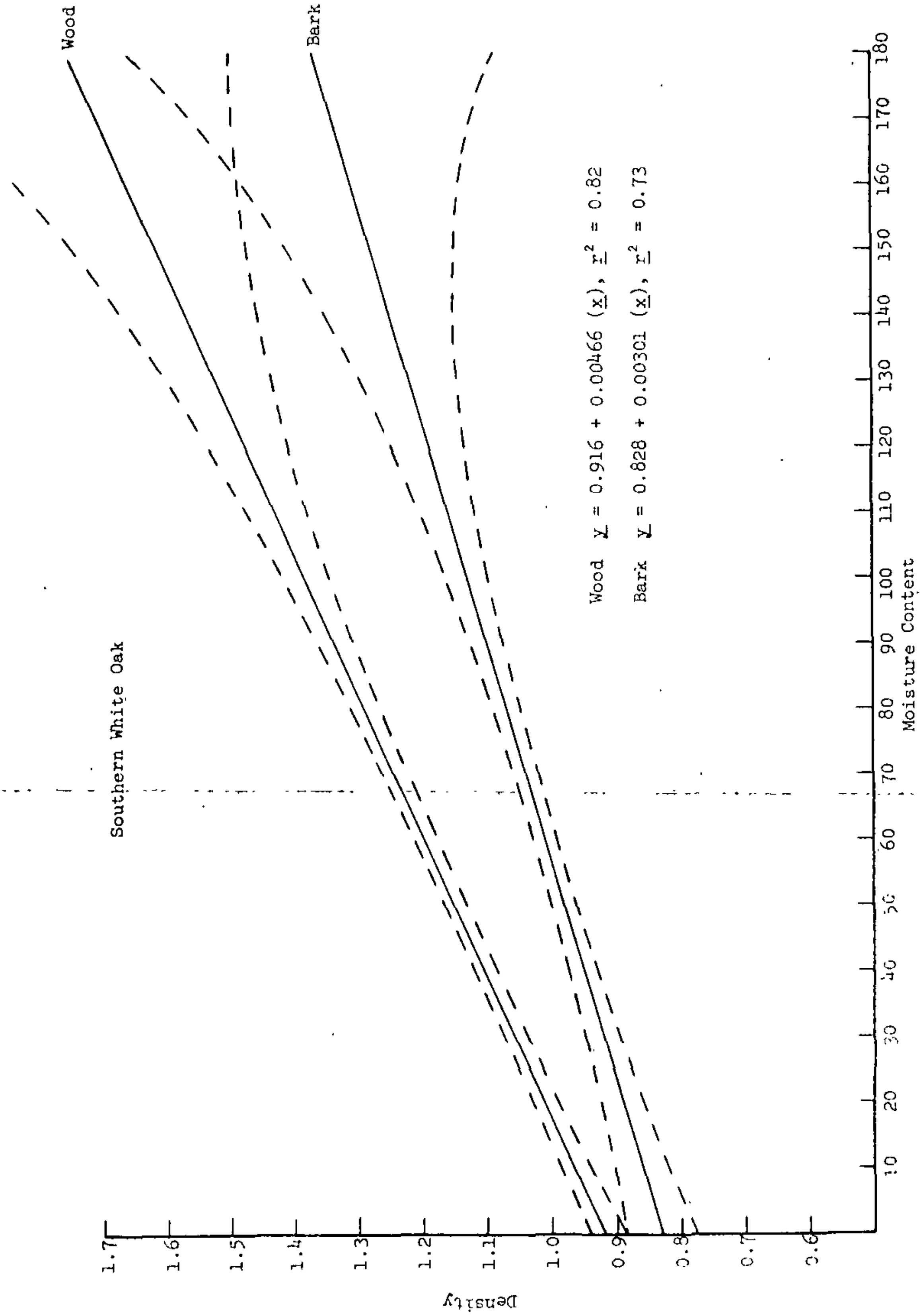

Figure 10. Illustrated Is the Relationship Between Basic Density and Moisture Content for Southern White Oak. The Dashed Lines are Two Standard Deviations Above and Below the Mean

mill-run chips had been used for the water flotation studies, because the simulated chips were uniform in size and shape, had a uniform level of moisture and were relatively free of knots, reaction wood, etc. Water segregation is believed to be possible when one fraction has a density of less than one and the other greater than one at a specific moisture content.

The data indicate that at moisture contents of between 25 and 50%, most wood chips could be expected to sink (density greater than 1). Southern white oak bark, on the other hand, when at the same moisture level, could be expected to float. Again, as with northern white oak, this is a rather narrow range of moisture contents. Although they are the same species, southern white oak had a slightly lower range of moisture contents to effect segregation than did northern white oak.

## Dwell Time Investigations

An investigation of dwell time involves nothing more than taking wood and bark chips at some standard moisture content, placing them on a water surface and observing the time it takes the material to pick up enough water to sink. Information on dwell time is useful because moisture uptake rates could have a considerable influence on the success of a segregation procedure (or chip-washing procedure) and would provide information on the rate at which segregation could be expected. A species in which either the bark or the wood takes up moisture rapidly could be expected to have a relatively short segregation time. For other species, where specific gravity and density of the wood and bark are similar, and moisture uptake is similar, considerable difficulty in segregation can be anticipated. Time required for chip samples to sink decreases as the sample moisture content at the start of the trial increases.

Half-sized simulated chips (1 x 0.3 x 0.2 inch) were used in the dwell time tests. Prior to testing, the samples were equilibrated in 50% RH and had a moisture content of approximately 20% (ovendry basis). Table XII summarizes the results for southern white oak. These results show a strong tendency for both the wood and bark to float with the exception of the sapwood of 3212-62 in which almost half the sapwood sank after 4 hours. It does not appear, however, that this would be a consistent trend. As with northern white oak, segregation of wood/bark chip mixtures through water flotation would not be a very useful method due to the narrow range of moisture contents at which segregation could be achieved.

## DATA INTERPRETATION

The results of the various tests run on southern white oak were very similar to those obtained for northern white oak. The bark of both contains some fiber and sclereids with most of the sclereids lost in pulping. For every 100 grams of southern white oak bark that are pulped, about 3 grams of fiber and 0.3 gram of sieve tubes would be produced. The sieve tubes could be used as filler material in paper but probably would not contribute in any useful way to paper properties. Because of the fiber yield, lack of sclereids in the pulp and low bark extractives (7%), southern white oak also may be suitable for pulping with the bark in some instances.

As with northern white oak, segregation of wood/bark chip mixtures through water flotation is possible but the small range of moisture contents at which it could be achieved do not make it a feasible method. Southern white oak had a slightly lower range of moisture contents at which segregation of wood/bark chip mixtures could be expected than did northern white oak.

## TABLE XII

## SUMMARY OF DWELL TIME RESULTS FOR SOUTHERN WHITE OAK

| Sample No. | Time Interval, min | Sinkers, % | Floaters, % |
|---|---|---|---|
| IPC 3212-62 Bark | after 5 | 0 | 100 |
| | 15 | 1.0 | 99 |
| | 60 | 2.6 | 97.4 |
| | 240 | 3.4 | 96.6 |
| IPC 3212-62 Sapwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 41.2 | 58.8 |
| IPC 3212-62 Heartwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-63 Bark | after 5 | 0.1 | 99.9 |
| | 15 | 0.1 | 99.9 |
| | 60 | 0.1 | 99.9 |
| | 240 | 1.3 | 98.7 |
| IPC 3212-63 Sapwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 3.4 | 96.6 |
| IPC 3212-63 Heartwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 22.2 | 77.8 |

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[a] Starting moisture content 20%.

Again, like northern white oak, separation and segregation of wood/bark chip mixtures through screening, hammermilling the fractions high in bark and rescreening appear to have some merit. Hammermilling southern white oak wood and bark chips resulted in a 3% wood loss and a 38% reduction in bark levels based upon material retained on a 14-mesh screen. If material on only the 10-mesh and larger screens is retained, the result is a 58% reduction in levels of bark and a 6% wood loss.

## RELATED LITERATURE

As cited previously, there are a number of papers on the economics and mechanics of segregating wood/bark chip mixtures. They include papers by Auchter and Horn (14), Hooper (15), and Biltonen, *et al*. (16).

# BARK AND WOOD PROPERTIES OF SOUTHERN RED OAK
# (*Quercus falcata* Michx.)

## SILVICULTURAL CHARACTERISTICS AND GEOGRAPHIC RANGE

Southern red oak is found throughout most of the southeastern United States. Its range extends south from Long Island, New York to northern Florida, west to the Brazos River Valley in Texas and northeastward through eastern Oklahoma and southern Missouri, to southern Illinois and Ohio. Within this range, this species is very rare only in the bottom lands of the lower Mississippi Delta. One of the commonest of the upland southern oaks, southern red oak characteristically occurs on dry, sandy or clay soils and most frequently at elevations up to 2000 ft. It is usually found on dry ridgetops and slopes facing south and west. Hot summers, short, mild winters and no distinct dry season characterize the climatic conditions in this range. A medium-sized tree, southern red oak is usually 70-80 ft in height and 2-3 ft in diameter at maturity.

## WOOD AND BARK MORPHOLOGY

### Wood

Wood features of the *Quercus falcata* Michx. are similar to those of other red oaks, subgenus *Erythrobalanus* Spach. Sapwood is nearly white, 1-2 inches thick, and the heartwood, a pale brown tinged with red, has large usually open earlywood pores forming a conspicuous band 1-4 pores in width. The transition from earlywood to latewood is gradual to more or less abrupt with smaller, thick-walled and more numerous pores in the latewood (10-30 vessels per mm$^2$). Conspicuous rays are unstoried and homocellular. Broad compound rays, approximately 12-30 seriate, 150-400 μm in diameter and hundreds of cells in length, are separated on a tangential surface by numerous narrow rays, usually uniseriate and very variable in length, 1-20+ cells. Tracheids intermingle with paratracheal parenchyma and form part of the conjunctive tissue between the earlywood pores and the rays, and most of the light-colored tissue in the latewood vessel area. Parenchyma, very abundant, are usually metatracheal or metatracheal-diffuse and zonate in fine, more or less regular, tangential lines in the outer portion on the ring. Fibers, medium thick to thick-walled, measure 14-22 μm in diameter and average 1.4 mm in length.

## Bark

Bark of the southern red oak is dark brown to nearly black with thick rough ridges separated by deep, narrow fissures. The rhytidome is usually hard and firm and about the same width or narrower than the slightly yellow inner bark (secondary phloem). Broad phloem rays, often dilated early in the inner bark and spreading widely at the terminal regions, and more or less curved sclerenchyma lines are visible to the naked eye. In the trees used in this study, the inner bark accounted for approximately 53% of the total bark thickness-by-weight. Figure 11 illustrates cross sections of southern red oak wood and bark. Appendix Table XXVII describes the trees used in this study.

## Anatomical Structure of Bark

Southern red oak bark has the basic pattern of the subgenus *Erythrobalanus* Spach. (red oak group). Bark of young trees is basically similar in cell type and tissue arrangement to the young white oak. In the mature bark, the composition, cell size and shape of the periderm are also fundamentally alike. However, the periderm formation is less frequent and irregularly spaced, and, as the phloem rays seldom extend through the periderm, it is tangentially longer. As a result, the rhytidome is compact and comparatively narrower with

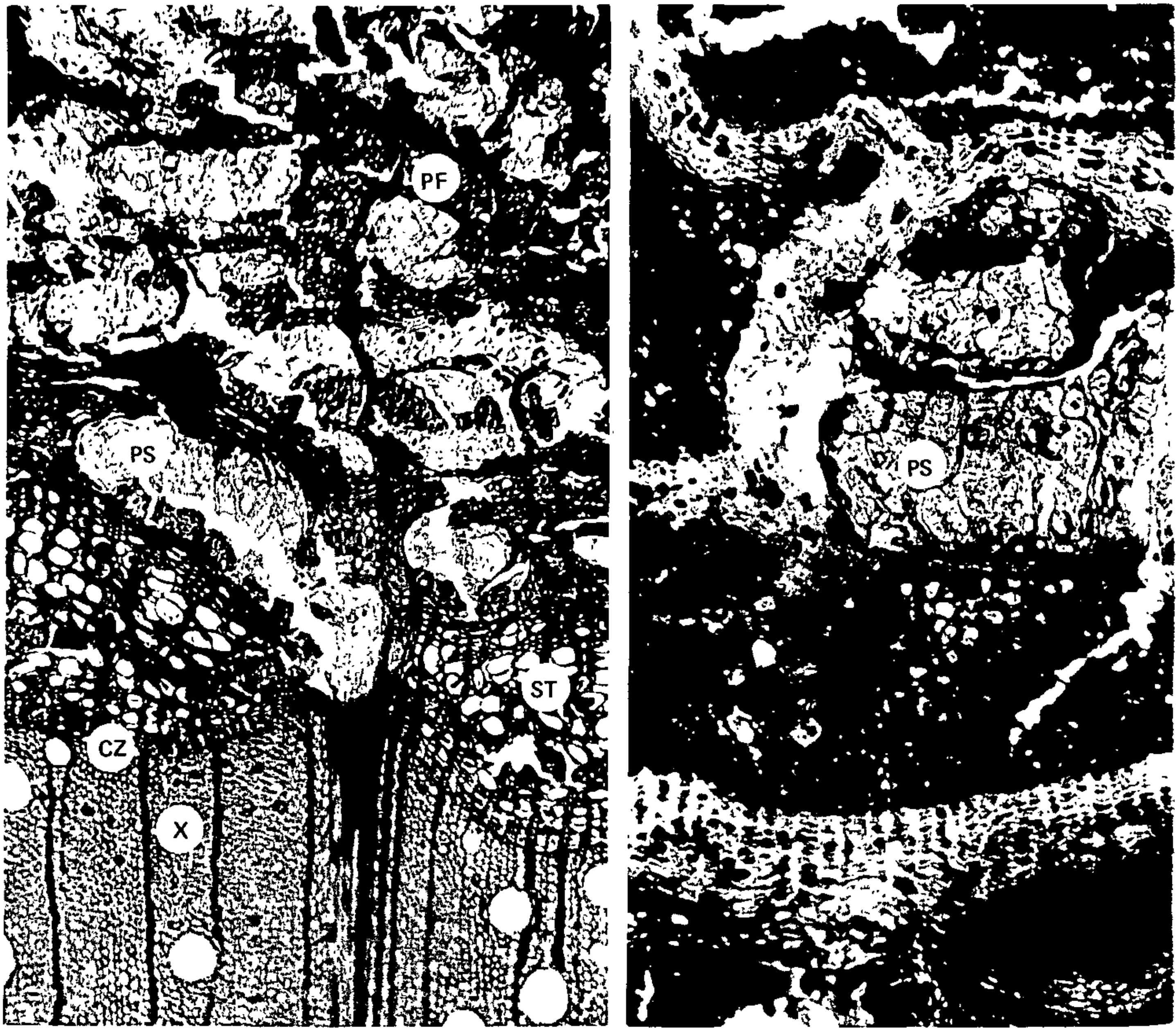

Figure 11. Cross Section of Southern Red Oak. Photomicrograph on Left Shows Xylem (X), Cambium Zone (CZ), Phloem Fibers (PF), Phloem Sclereids (PS), and Sieve Tubes (ST). Photomicrograph on the Right Is a Cross Section of the Outer Bark Showing Large Groups of Sclereids (PS). Magnification — 75X Left, 110X Right

fewer periderm layers. In red oaks, the well-developed suberized phellem cells are often twice as thick as those of the white oak.

The inner bark (secondary phloem) is composed of sieve tubes, parenchyma and sclerenchyma cells, and phloem rays. Sieve tubes, confined by the rays, parenchyma and sclerenchyma cells, are in groups of 4 to 10 and vary in length from 240-655 µm. Except in the last-formed bands close to the cambium, the sieve tubes are crushed to varying degrees. More or less rectangular parenchyma cells are aligned in tangential rows of 3-5 layers in a basic tissue pattern similar to the white oak. Transformed parenchyma strands form sclereid groups.

Sclerenchyma appear as both sclereids and phloem fibers and often tend to be rather loosely arranged. Sclereids usually develop later than the fibers and generally do not appear near the cambium except within the broad rays. Mature groups may be tangentially elongated and form short bands. Fibers develop early and mature very close to the cambium, often in the current growth season. Typical red oak phloem fibers, often in up to 6 layers, are aligned in short tangential bands with crystalliferous parenchyma cells associated at the margins. The tapered fibers are polygonal in cross section and vary from 0.57-1.47 mm in length and 15-25 µm in diameter at the broadest portion with a narrow lumen of about 2 µm. Between two dilated broad rays, sclerenchyma is composed mostly of fibers with only scattered sclereids, and due to the wide dilation of these rays, the sclereid groups at the outer portion of the inner bark and rhytidome appear in a more sporadic fashion than in white oak.

Red oak rays are homocellular and of two types. The narrow rays are usually uniseriate and about 12 cells or 150-200 µm high. Broad rays may be 30 seriate and become dilated rather early, spreading open quite wide at the outer part of the secondary phloem and often causing an archlike pattern in the other tissues. About the middle portion of the broad rays, cells often become "lignified" and very thick, usually retaining their original size and shape, but at times expanding and becoming irregular. Sclereid groups appear at regular intervals along the radius of the broad rays.

## SPECIFIC GRAVITY, EXTRACTIVES AND FIBROUS YIELD

Basic information on such bark properties as specific gravity, level of extractives, fiber yield and the presence of such morphological elements as phloem fibers and sclereids are expected to be useful in determining the need and possible methods of separating and segregating wood/bark chip mixtures*. Whenever possible, data on bark have been compared with similar information on wood.

### Specific Gravity

Table XIII summarizes the information available on wood and bark of southern red oak. Specific gravity is most often expressed in terms of ovendry weight divided by green volume. It should be noted that several of the values in the table are ovendry weights divided by ovendry volumes. Information expressed in terms of green weight divided by green volume is useful when examining the possibilities of liquid flotation as a means of segregating wood/bark chip mixtures. Information in this report under the section Water Flotation Behavior compares the basic density (green weight divided by green volume) of southern red oak at several moisture contents.

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*Throughout this report the term separation has been used to designate separation or detachment of wood from bark while segregation has been used to indicate removal of either the bark or wood fraction from wood/bark chip mixtures.

## TABLE XIII

## SOUTHERN RED OAK SPECIFIC GRAVITY INFORMATION

(Ovendry weight/green volume)

| Wood | Bark | | | |
|---|---|---|---|---|
| Average | Inner | Outer | Total | Reference & Remarks |
| 0.52 | | | | IUFRO (5) |
| 0.57 (young willow oak) | | | | Barker (20) |
| 0.58 (mature willow oak) | | | | |
| 0.61 (stem wood) | | | 0.60 (stem bark) | Manwiller (17) |
| 0.62 (branch wood) | | | 0.50 (branch bark) | Manwiller (17) |
| 0.57 | | | | Isenberg (8) |
| 0.60 (sapwood) | 0.70 | 0.73 | 0.73 | IPC 3212-29 |
| 0.62 (heartwood) | | | | |
| 0.58 (sapwood) | 0.74 | 0.76 | 0.75 | IPC 3212-30 |
| 0.60 (heartwood) | | | | |
| 0.67[a] | | | | Isenberg (8) |
| | | | 0.81[a] | Harkin & Rowe (9) |

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[a] Ovendry weight/ovendry volume.

An average specific gravity (ovendry weight/green volume) of approximately 0.60 appears appropriate for the wood of southern red oak. Our limited data do not show a large difference between heartwood and sapwood although, again, for the two trees sampled, the heartwood was higher in specific gravity. This was also true for northern red oak.

The specific gravity of the total (inner + outer) bark of southern red oak appears somewhat higher than that of the wood. This is the reverse of the trends found for northern and southern white oak but similar to that of northern red oak. Overall values suggested for use in species comparisons are 0.60 for wood and 0.68, 0.70 and 0.70 for inner, outer and total bark, respectively.

## Extractives

Extractives in wood and bark are important because, when present in large amounts, they not only result in reduced yield of fibrous material but ultimately can be expected to result in paper machine "pitch problems." Recent needs to reduce total water use through closed white water systems are expected to accentuate problems in this area. No attempt has been made in this report to go beyond determining the total alcohol-benzene extractives. Such extractives information is expected to provide an appropriate indication regarding possible pitch problems when large amounts of bark are pulped. Further detailed examination of the types of extractives involved is recommended using specific bark sources if preliminary comparisons suggest pitch and yield problems may develop.

A range in levels of extractives from 2.1 to 6.1% has been reported for southern red oak (see Table XIV). For between-species comparisons, an extractives level of 4.8% is suggested for the wood of southern red oak. Based upon information obtained from the two trees sampled as part of this project, the bark of southern red oak can be expected to have an extractives level of 11.6%. This is an intermediate level of extractives and similar to that of northern red oak. This level of extractives is not expected to be a serious problem except in those instances where high percentages of bark have been concentrated in a particular chip fraction by screening or other mechanical techniques.

## Fibrous Yield

Increasing emphasis is being placed on pulping bark rather then debarking bolts or segregating wood/bark chip mixtures. Important to determining the usefulness of this approach with a particular species is determining the proportion of lignified cells that exist in the bark and that will survive normal cooking procedures. Also, it is important to determine what percentage of these cells will contribute in a favorable way to the resulting paper product.

TABLE XIV

SOUTHERN RED OAK ALCOHOL-BENZENE EXTRACTIVES

| Type of Material | Extractives, % | Source |
|---|---|---|
| Young willow oak wood | 2.1 | Barker (20) |
| Mature willow oak wood | 4.6 | Barker (20) |
| Stemwood | 5.0 | Manwiller (17) |
| Branchwood | 6.1 | Manwiller (17) |
| Stem bark | 14.7 | Manwiller (17) |
| Branch bark | 15.8 | Manwiller (17) |
| Bark | 8.0 | IPC 3212-29 |
| Bark | 12.2 | IPC 3212-30 |

Sclereids are short, thick-walled, heavily lignified cells. When not fully cooked, as could occur in high-yield pulping, clumps of sclereids may cause so-called "fish eyes" in certain grades (calendered) of paper. According to Chang (1), 31.6% of the tissue elements in the inner bark of red oak are sclereids. However, a large proportion of these sclereids are lost when the bark is pulped.

In the inner bark of some species there occurs bands of heavily lignified fibers described in the literature as phloem fibers or sclerenchyma fibers. These fibers are the principal bark elements to survive chemical pulping and contribute to overall pulp yield and sheet strength. Chang (1) estimated that 12.4% of the inner bark of red oak was composed of phloem fibers based upon examination of cross sections. As a further check on pulp yield and the nature of fibrous material produced, 20 to 30-gram samples were pulped using the IPC Standard Kraft Micropulping Procedure. For a complete description of the procedure see the section on Experimental Procedures. Table XV summarizes the results of this investigation.

Micropulping southern red oak bark resulted in approximately a 31% yield of solids. When screened, most (75-85%) of the material went through the 200-mesh screen. The elements retained on the coarse screens (60 and 100 mesh) were primarily fibers. The "on 150 mesh" and "on 200 mesh" material was mainly sieve tubes, sclereids and fibers. The "through 200 mesh" contained 60-70% sclereids with smaller percentages of peridermal and parenchymatous cells. Figure 12 illustrates the type of material retained on the 60 and 150-mesh screens.

Based upon very limited numbers of bark sample observations, it appears that, for every 100 grams of bark that are pulped, about 31 grams of solids will result. Of this 31 grams, about 4 grams (4%) of fibers and 0.2 gram (0.2%) of other material will be produced. This assumes that only the material on the 60 and 100-mesh screens would end up in and contribute in any significant way to the final product. The remaining material would be lost in washing and cleaning operations.

## WOOD/BARK ADHESION

Wood/bark adhesion differences have been suggested as one of the reasons for the differences encountered in the ease of debarking pulpwood species. The same factors influencing debarking of pulpwood are expected to influence debarking of wood chips. The approach taken in the study has been to obtain growing season and dormant season information on (1) magnitude of wood/bark adhesion, (2) morphological structures associated with wood/bark adhesion, and (3) reasons for differences between species in adhesion.

## TABLE XV

## SOUTHERN RED OAK MICROPULPING INVESTIGATIONS

| Data | Sample No. | | Remarks[a] |
|---|---|---|---|
| | 3212-29 | 3212-30 | |
| Yield, % solids | 31.2 | 30.2 | |
| Fraction | | | |
| on 60 mesh, % | 10.0 | 7.4 | Fraction contained principally phloem fibers (95+%) with traces of sieve tubes (<1%), sclereids (<1%), peridermal and parenchymatous cells (<1%) and crystalliferous parenchyma (<1%). The arithmetic average length of the phloem fibers in this fraction was 1.14 mm |
| on 100 mesh, % | 3.0 | 3.5 | Fraction contained principally phloem fibers (80-90%) with small percentages of sieve tubes (5-10%), sclereids (5-10%), peridermal and parenchymatous cells (<5%) and a trace of crystalliferous parenchyma (<1%). The arithmetic average length of the sieve tubes in this fraction was 0.51 mm |
| on 150 mesh, % | 2.4 | 1.7 | Fraction contained principally sieve tubes (80-90%) with small percentages of phloem fibers (10-20%) and sclereids (<5%) and traces of peridermal and parenchymatous cells (<1%) and crystalliferous parenchyma (<1%) |
| on 200 mesh, % | 5.5 | 1.9 | Fraction contained a large percentage of sclereids (50-60%) with small percentages of sieve tubes (20-30%), phloem fibers (10-20%), peridermal and parenchymatous cells (<5%) and crystalliferous parenchyma (<5%) |
| through 200 mesh, % | 79.1 | 85.5 | Fraction contained a large percentage of sclereids (60-70%) with small percentages of peridermal and parenchymatous cells (20-30%), sieve tubes (<5%), crystalliferous parenchyma (<5%) and a trace of phloem fibers (<1%) |

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[a] Percentages in each fraction on a weight basis.

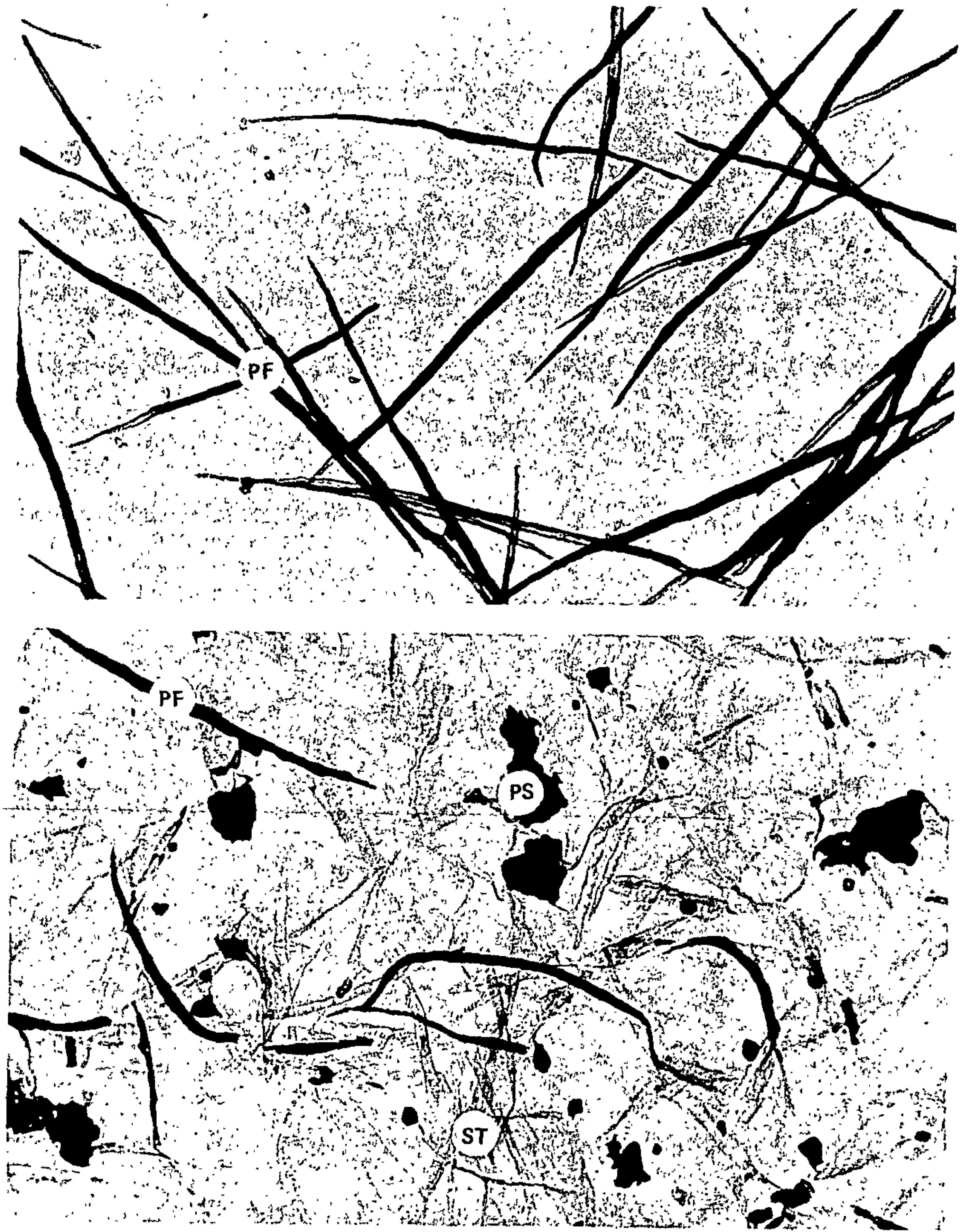

Figure 12. The 60-Mesh Screen (Top) Contained by Weight *Principally Phloem Fibers* (95+%). The 150-Mesh Screen (Bottom) Contained Primarily Sieve Tubes (80-90%) with Smaller Percentages of Phloem Fibers (10-20%) and Sclereids (<5%). Magnification 75X. Symbols Illustrate Phloem Fibers (PF), Sieve Tubes (ST) and Sclereids (PS)

Wood/bark adhesion values were measured for southern red oak samples collected August 1 (growing season) and September 30 and October 7 (dormant season). Using the sampling and testing procedures described in the section on Experimental Procedures in Report One, shear parallel to the grain was measured. After testing, the samples were examined to determine the location of the zone of failure. Figure 13 illustrates the zone of failure for southern red oak during both the growing and dormant seasons. During the growing season, wood/bark adhesion was low (5.4 $kg/cm^2$) and the failure zone was located between immature xylem cells in the region of the cambium zone. Also included in Fig. 13 is a large ray "pull-out" area. This is a common occurrence in oak species. During the dormant season, wood/bark adhesion increased to 8.2 $kg/cm^2$ and the failure zone occurred in the inner bark, primarily between collapsed phloem sieve tubes and parenchyma cells adjacent to tangential bands of phloem fibers. Failure occurred fairly close to the cambium. Adhesion values obtained for southern red oak are comparable to those obtained for northern red oak during the growing and dormant seasons.

As a result of measurement data taken on the species included in Appendix Table XXVIII and the measurement data reported in the previous reports for this project, it is clear that dormant season wood/bark adhesion is related to inner bark strength and inner bark strength is, in turn, related to inner bark morphology. The presence of phloem fibers in the inner bark of hardwoods appears to be associated with high dormant season wood/bark adhesion. High numbers of sclereids and a lack of phloem fibers seem to be associated with low dormant season wood/bark adhesion and low bark strength.

Separation (breaking the bond between bark and wood in a chip) is an important first step in segregation (removal of bark particles from wood chips).

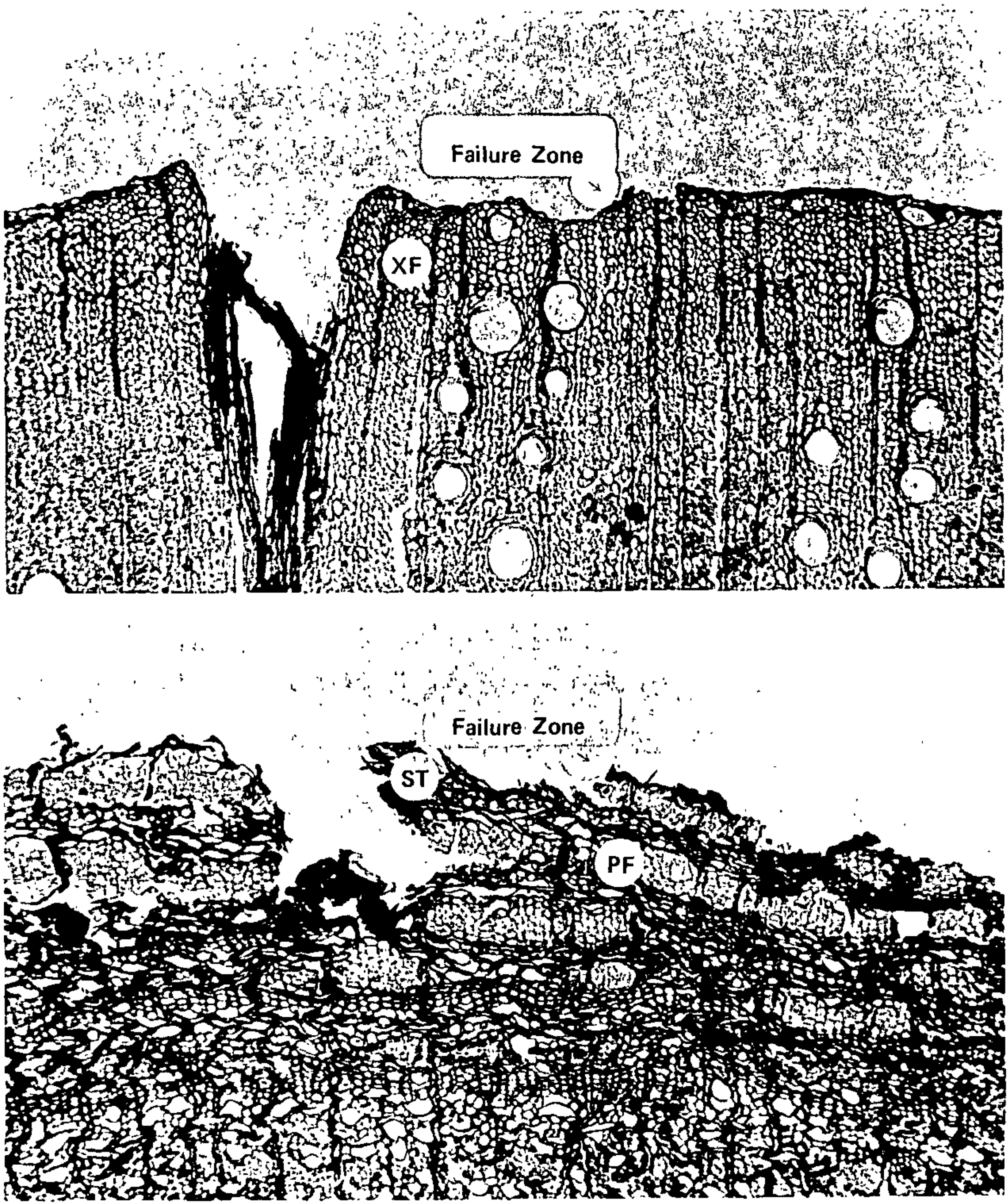

Figure 13. Illustrated Is the Southern Red Oak Failure Zone for Both the Growing Season (Top) and Dormant Season (Bottom). The Growing Season Failure Zone Was Located Between Immature Newly-Formed Xylem Fibers (XF) in the Region of the Cambium Zone. Large Ray "Pull-Out Area" Is Evident. During the Dormant Season, the Failure Zone Was Located in the Inner Bark Primarily Between Collapsed Sieve Tubes (ST) and Parenchyma Cells Adjacent to Tangential Bands of Phloem Fibers (PF). Magnification 75X

Separation during the growing season, when wood/bark adhesion is low, can usually be accomplished by the action of the chipper. During the dormant season, adhesion is greater and separation by chipper action is less successful.

Another method to effect wood/bark separation that is worthy of consideration is compression debarking. Arola (21), working with northern red oak, obtained 87.1% bark removal and 5.2% wood loss using compression debarking in combination with a steaming pretreatment and drubbing after compression debarking. It is expected that similar results could be obtained with southern red oak.

As mentioned previously, several promising approaches were tried with hardwoods and two softwoods in Project 2929 to reduce adhesion. These methods included chemical, thermal, and biological methods. These methods have not been tried with southern red oak but are worthy of further consideration and are discussed in the section on Between-Species Comparisons.

## BARK STRENGTH, TOUGHNESS AND REACTION TO HAMMERMILLING

Bark strength and toughness measurements are included as part of the characterization of bark because it was felt that when these measurements are compared with the results obtained in wood/bark adhesion tests, with the difficulty encountered in conventional debarking and with bark morphology, the "why" of bark separation and segregation would eventually emerge.

Hammermilling has been widely used in bark utilization to prepare fractions for use as horticultural mulch, soil conditioners, and as additives to a number of types of products. A simulated hammermilling test was developed in an effort to relate the hammermilling of bark (and wood) to bark strength, toughness and morphology.

As discussed in the section on Experimental Procedures, bark strength measures shear parallel to the grain while bark toughness measures the energy required to rupture a thin specimen by a bending force perpendicular to the grain (parallel to the tree diameter). Table XVI summarizes the bark strength and toughness tests made on the wood and bark of southern red oak. Also included for comparison purposes (Appendix Table XXIX) are the bark strength values for a number of pulpwood species of interest.

TABLE XVI

SUMMARY OF STRENGTH AND TOUGHNESS MEASUREMENTS MADE ON WOOD AND BARK OF SOUTHERN RED OAK[a]

| Material | Strength | Toughness |
|---|---|---|
| Wood | -- | 0.55 |
| Inner bark | 3.6 | 0.11 |
| Outer bark | 3.4 | 0.14 |

---

[a] Determinations average of two different trees.

The inner bark strength of southern red oak is more than that of northern red oak but still within the intermediate range. Because it does have some fiber in the inner bark, the value is higher than that obtained for white birch and sugar maple which have essentially no inner bark fiber.

Hammermilling, followed by screening, resulted in the highest levels of bark removed of any species investigated thus far. When the half-sized chips for the two trees investigated were hammermilled, and the material on the 14-mesh screen retained, the result was a 6% loss in wood and a 46% reduction in bark. The bark removed appeared to be mostly outer bark. Since the inner bark has a specific gravity which is somewhat higher than that of the wood, it is possible that hammermilling could be used together with water flotation to effect segregation of wood/bark chip mixtures. However, the fiber contained in the inner bark could be of some value. If the material on the 5 and 10-mesh screens only were retained, the results would be a 62% reduction in levels of bark and an 11% wood loss. This wood loss is fairly high and might not be justified by the increased bark removal. Summarized in Table XVII are the results of the hammermilling tests run on southern red oak wood and bark. Figure 14 illustrates the effect of hammermilling on the wood and bark of southern red oak. The wood loss was less and bark removed was greater for southern red oak than for northern red oak (see Project 3212, Progress Report One). It is possible that improvements could be made in screening by taking advantage of the differences in configuration of wood and bark chips evident in Fig. 14 (<u>12</u>,<u>13</u>). Summary Table XXVIII (see Between-Species Comparisons) compares bark strength, toughness and reaction to hammermilling of southern red oak with other species tested thus far.

## WATER FLOTATION BEHAVIOR

One possible method of segregating wood/bark chip mixtures is by water flotation procedures. Knowledge of the flotation characteristics of wood and bark is also expected to be important when certain types of chip-washing procedures are employed. Earlier investigations into water flotation segregation (Project 2977) revealed that chip size, specific gravity, moisture content and rate of moisture uptake were factors in the flotation behavior of bark and wood chips. Budget limitations do not permit examination of all factors involved and, as a result, the influence of chip size has been eliminated from the variables considered.

## TABLE XVII

## SUMMARY OF HAMMERMILLING TEST ON SOUTHERN RED OAK

| Tree No. | Type Material | Fraction Retained on Standard Screen[a], % 5 Mesh | 10 Mesh | 14 Mesh | 20 Mesh | 28 Mesh | <28 Mesh | Remarks |
|---|---|---|---|---|---|---|---|---|
| 3212-29 | Bark | 19 | 26 | 13 | 9 | 11 | 22 | Mostly inner bark on largest mesh screen. Increasing amounts of outer bark on smaller mesh screens |
| | Sapwood | 74 | 16 | 4 | 2 | 1 | 3 | |
| | Heartwood | 81 | 12 | 3 | 1 | 1 | 3 | |
| 3212-30 | Bark | 9 | 23 | 18 | 13 | 15 | 22 | Equal amounts of inner & outer bark on largest mesh screen. Increasing amounts of outer bark on smaller mesh screens |
| | Sapwood | 51 | 31 | 8 | 3 | 2 | 4 | |
| | Heartwood | 84 | 8 | 3 | 1 | 1 | 3 | |

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[a] Standard soil screen sizes; 5 mesh has 5 wires per inch and an opening of 4.00 mm, 10 mesh has 10 wires per inch and an opening of 2.0 mm, 14 mesh has 14 wires per inch and an opening of 1.168 mm, 20 mesh has 20 wires per inch and an opening of 1.00 mm, and the 28 mesh screen has 28 wires per inch and an opening of 0.589 mm.

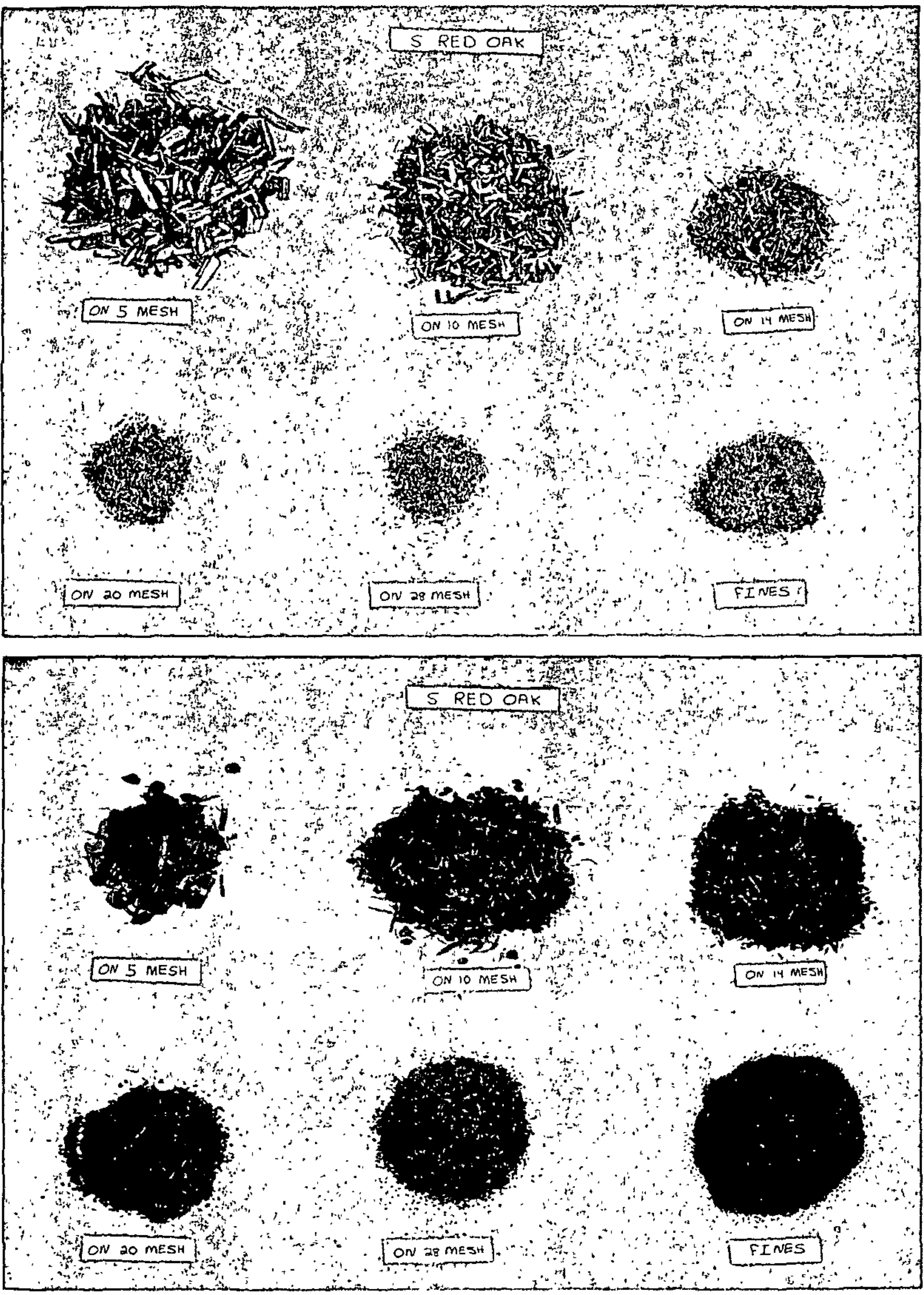

Figure 14. Illustrated Is the Effect of Hammermilling on Southern Red Oak Wood (Top) and Bark (Bottom)

Two procedures were used to examine the water flotation behavior of wood and bark. One procedure involved measuring the density* (green weight divided by green volume) of simulated chips at a number of different moisture contents. The second technique involved measuring the rate of moisture uptake and sinking of wood and bark chips in what have been designated as "dwell time" studies.

## Density Determinations

Simulated chips were used in determining the relationship between moisture content and the density of bark and wood. Wood and bark from two southern red oak trees (IPC 3212-29 and IPC 3212-30) were used in making the determinations. The moisture content of the chip samples was adjusted by equilibrating in small jars to which had been added appropriate amounts of water. The extremely accurate pycnometer method described in the Experimental Procedures in Report One was used in determining density. Bark samples used were "whole bark" samples, a combination of both inner and outer bark. Small chips of inner and outer bark were also tested. The densities of inner, outer and whole bark appeared very similar for southern red oak and somewhat higher than either heartwood or sapwood.

Figure 15 illustrates the relationship that was found between moisture content and density. The linear relationship shown was obtained by fitting the least squares regression line through the data. The dashed lines are two standard

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*The term density is used in this report to indicate the weight of wood and bark samples and is expressed in terms of green weight divided by green volume. This is in contrast to the term specific gravity, which also is an expression of the weight of a sample, but in this case it is in terms of dry weight divided by green volume.

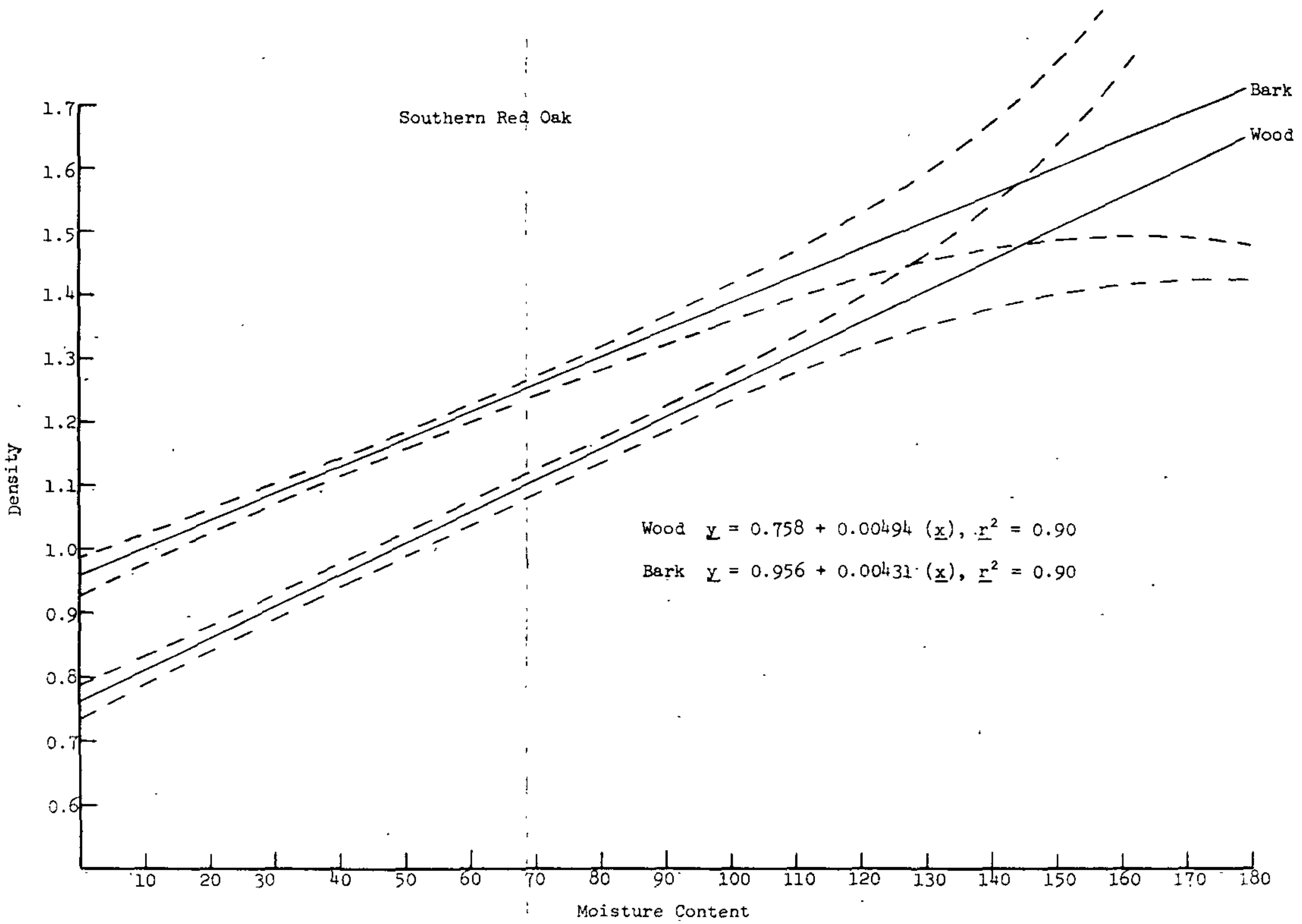

Figure 15. Illustrated Is the Relationship Between Basic Density and Moisture Content for Southern Red Oak. The Dashed Lines are Two Standard Deviations Above and Below the Mean

deviations above and below the average values. The standard deviation of the regression line is considerably less than would have been obtained if conventional mill-run chips had been used for the water flotation studies, because the simulated chips were uniform in size and shape, had a uniform level of moisture and were relatively free of knots, reaction wood, etc. Water segregation is believed to be possible when one fraction has a density of less than one and the other greater than one at a specific moisture content.

The data indicate that at moisture contents of between 20 and 40%, most bark chips could be expected to sink (density greater than 1). Southern red oak wood, on the other hand, at the same moisture contents, could be expected to float. Northern red oak bark also sank while the wood floated although the levels of moisture required were higher (50-70%). Results for both red oaks are the opposite of those obtained for northern and southern white oak, in which the bark floated and wood sank at appropriate moisture contents.

## Dwell Time Investigations

An investigation of dwell time involves nothing more than taking wood and bark chips at some standard moisture content, placing them on a water surface and observing the time it takes the material to pick up enough water to sink. Information on dwell time is useful because moisture uptake rates could have a considerable influence on the success of a segregation procedure (or chip-washing procedure) and would provide information on the rate at which segregation could be expected. A species in which either the bark or the wood takes up moisture rapidly could be expected to have a relatively short segregation time. For other species, where specific gravity and density of the wood and bark are similar, and moisture uptake is similar, considerable difficulty in segregation can be anticipated. Time required for chip samples to sink decreases as the sample moisture content at the start of the trial increases.

Half-sized simulated chips (1 x 0.3 x 0.2 inch) were used in the dwell time tests. Prior to testing, the samples were equilibrated in 50% RH and had a moisture content of approximately 20% (ovendry basis). Table XVIII summarizes the results for southern red oak. These results agree with density determinations obtained for the wood of southern red oak. However, more of the bark should have sunk, particularly for 3212-30. It is possible that the moisture content required might have to be increased, resulting in an even narrower range of moisture contents at which segregation can be achieved. Apparently, the bark does not take up water readily.

## DATA INTERPRETATION

There appear to be several possibilities for upgrading the quality of southern red oak chip mixtures. Data are not available on the effectiveness of chipper action on separation of bark from wood during the dormant season but IPC experience suggests good separation is obtained from the impact of the chipper knives when thick-barked high specific gravity species such as oak are involved. Compression debarking, combined with a steaming and drubbing treatment (21), worked well on northern red oak (87.1% bark removal and 5.2% wood loss) and the results could be expected to be similar for southern red oak.

Specific gravity, dwell time and density *vs*. moisture content information make the use of water flotation a rather poor approach. The range of moisture contents at which segregation of wood and bark chips could be achieved is very narrow (20-40%). At suitable moisture contents, the bark sank and the wood floated. If this moisture content range could be achieved, it is possible

## TABLE XVIII

## SUMMARY OF DWELL TIME RESULTS FOR SOUTHERN RED OAK

| Sample No. | Time Interval, min | Sinkers, % | Floaters, % |
|---|---|---|---|
| IPC 3212-29 | after 5 | 0 | 100 |
| Bark | 15 | 6.2 | 93.8 |
| | 60 | 28.0 | 72.0 |
| | 240 | 87.6 | 12.4 |
| IPC 3212-29 | after 5 | 0 | 100 |
| Sapwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-29 | after 5 | 0 | 100 |
| Heartwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-30 | after 5 | 3.3 | 96.7 |
| Bark | 15 | 3.3 | 96.7 |
| | 60 | 13.4 | 86.6 |
| | 240 | 40.6 | 59.4 |
| IPC 3212-30 | after 5 | 0 | 100 |
| Sapwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-30 | after 5 | 0 | 100 |
| Heartwood | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |

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[a] Starting moisture content 20%.

that, besides the bark, much of the undesirable wood (reaction wood, knots, etc.) would also be eliminated by sinking with the bark.

Hammermilling wood and bark chips resulted in a 6% wood loss and a 46% reduction in levels of bark if the material on the 14-mesh and larger screens was retained. If only the material on the 10-mesh and larger screens was retained, the result would be an 11% wood loss and a 62% reduction in bark levels. This rather large wood loss may not be justified by increased bark removal unless the material removed has considerable value as fuel. However, it appears that the bark could be concentrated into one or two small-sized chip fractions by screening and those fractions hammermilled and rescreened.

Another alternative would be to pulp the bark. Bark micropulping results indicate screening the pulp would remove most of the sclereids and result in 4% usable fiber and 0.2% of other material.

## RELATED LITERATURE

Again, there are a number of papers on the economics and mechanics of segregating wood/bark chip mixtures. They include papers by Auchter and Horn (14), Hooper (15), and Biltonen, et al. (16).

# BARK AND WOOD PROPERTIES OF SWEETGUM
# (Liquidambar styraciflua L.)

## SILVICULTURAL CHARACTERISTICS AND GEOGRAPHIC RANGE

Sweetgum grows naturally throughout most of the southeastern United States. It ranges from Connecticut south to central Florida, west to Texas, Oklahoma, and Missouri, and north to southern Illinois. It is also found sporadically throughout Mexico and Central America.

Growth of sweetgum varies greatly with environmental conditions. The species tolerates a wide variety of soils, elevations in the U.S. from sea level to 2000 ft, rainfall of 40-80 inches per year, and average annual minimum temperatures ranging from -10 to 60°F. Best growth occurs on rich, moist alluvial clay and loam soils of river bottoms. Depending upon the site, mature trees vary from 80-120 ft in height and 1.5-3 ft in diameter. Where conditions are suitable, natural regeneration is abundant from both seeds and sprouts.

## WOOD AND BARK MORPHOLOGY

### Wood

Sweetgum wood is moderately hard and heavy, diffuse porous, and uniform in texture. The sapwood (sap gum) is wide and white with frequently a pinkish hue. Heartwood (red gum) varies from shades of red to brown in color. Growth rings are inconspicuous with small, uniform and numerous pores. Rays are very close and not distinct. Longitudinal traumatic (wound) gum ducts are sometimes present in tangential rows appearing at wide intervals and are often occluded with white deposits.

Vessel elements, fiber tracheids, longitudinal parenchyma and heterocellular rays are the main components of sweetgum xylem. Vessels account for approximately 55% of the wood volume (22). On transverse sections, the pores of the longitudinal vessels number 124-245 per $mm^2$. More or less angled and with relatively thin walls, approximately 3 μm, the vessel elements have scalariform perforation plates and spiral thickenings on the tapering ends. Longer than average for hardwoods, vessel elements of sweetgum are approximately 1.32 mm in length. Fiber tracheids, strongly angled on transverse sections, have moderately thick walls, 7 ± 1.2 μm, and measure 1.82 ± 0.16 mm in length (22). Distributed irregularly among the fiber tracheids, the longitudinal parenchyma have the same angular shape and dimensions of the tracheids but with thinner walls, approximately 2.5 μm, and form strands consisting of a number of cells. Rays, numbering 6-9 per mm tangentially on transverse section, are unstoried and both 1-seriate and 2-3 seriate. When present, gum canals are arranged in a uniseriate tangential row with angled orifice.

## Bark

Generally the bark of the mature sweetgum is grayish-brown in color and deeply furrowed into narrow, flaky ridges. The outer bark is soft and rather loose and the inner bark somewhat more fibrous and lighter in color, turning dark after exposure. Bark of the more vigorous trees appears lighter, thick, and more deeply fissured with pronounced, rounded ridges. That of trees of low vigor, is darker, thinner and flatter. In the trees used in this study, the outer bark accounted for approximately 70% of the total width of the bark. Figure 16 illustrates cross sections of sweetgum wood and bark. Appendix Table XXVII describes the trees used in this study.

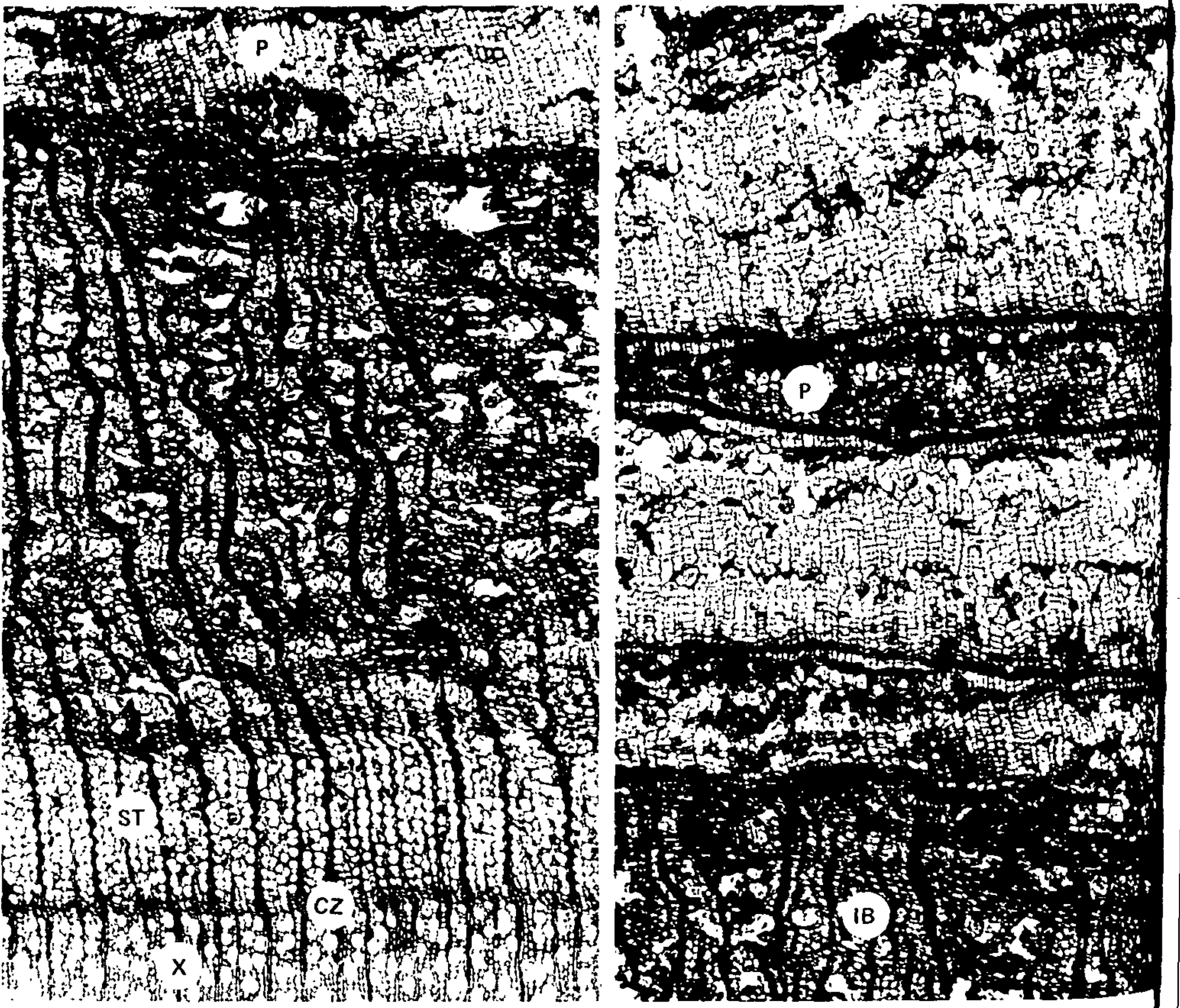

Figure 16. Cross Section of Sweetgum. Photomicrograph on Left Shows Xylem (X), Cambium Zone (CZ), Sieve Tubes (ST), and Last-Formed Periderm (P). Photomicrograph on the Right Is a Cross Section of Part of the Inner Bark (IB) and Rhytidome Showing Periderm Layers (P). Magnification 50X

Anatomical Structure of Bark

Generally broader than the inner bark, rhytidome formation begins very early in young trees and has conspicuous and sometimes broader periderm layers. In the mature tree, a periderm band is composed of 2-4 layers of phelloderm, a layer of phellogen and a number of layers of phellem. The first developed phellem cells are slightly suberized, rectangular in shape and thin-walled. Occasionally, a few of these cells become lignified. The regular phellem cells, typically suberized, are usually rectangular in shape, about 30 μm in tangential diameter and 8 μm in radial diameter. Phelloderm cells are parenchymatous in nature but sometimes become lignified. In 8-year-old bark, rhytidome formation and sclerenchyma in the secondary phloem are developed and a basic pattern of mature bark is established (1).

Sieve tubes, parenchyma cells, sclerenchyma and phloem rays make up the secondary phloem (inner bark) of the mature sweetgum. Rather long with compound sieve plates at the ends, sieve tubes are aligned in radial rows of 1-6. Usually about 30 μm, although up to 50 μm, in diameter, the sieve tube elements are variable in length from 545-1240 μm. The mean length is 840 with a standard deviation of 133. Associated with the sieve tube elements are companion cells, 6-8 in a strand.

Parenchyma cells appear both sporadically distributed throughout the secondary phloem and as a narrow band aligned more or less continuously along the tangential plane. The bands consist of usually 1-3 layers of parenchyma cells which may eventually originate a new periderm. Cells in the scattered parenchyma strands often enlarge or become lignified, and with the phloem ray cells, are the origin of sclereid formation in the rhytidome.

Sclerenchyma in the secondary phloem of sweetgum consists of fibers and sclereids. Appearing sporadically very close to the cambium, and often 20 cells away from this region, small groups or bands composed of 6-8 layers of fibers mixed with the sclereids, are aligned in more or less tangential rows. The fibers, often slightly twisted and with rounded ends, are variable in length from 0.51-1.56 mm. The mean length is 1.05 with a standard deviation of 0.28. With very thick cell walls showing lamellate layers, the diameter of the fiber is usually 20-30 µm, and up to 50 µm or more. The short sclereids originate and are transformed from the parenchyma strands and a portion of the adjacent ray cells. They may be simply "lignified," retaining their original size and shape, or become irregular in size and shape and twisted.

## SPECIFIC GRAVITY, EXTRACTIVES AND FIBROUS YIELD

Basic information on such bark properties as specific gravity, level of extractives, fiber yield and the presence of such morphological elements as phloem fibers and sclereids are expected to be useful in determining the need and possible methods of separating and segregating wood/bark chip mixtures*. Whenever possible, data on bark have been compared with similar information on wood.

### Specific Gravity

Table XIX summarizes the information available on wood and bark of sweetgum. Specific gravity is most often expressed in terms of ovendry weight divided by green volume. It should be noted that several of the values in the table are ovendry weights divided by ovendry volumes. Information expressed in

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*Throughout this report the term separation has been used to designate separation or detachment of wood from bark while segregation has been used to indicate removal of either the bark or wood fraction from wood/bark chip mixtures.

terms of green weight divided by green volume is useful when examining the possibilities of liquid flotation as a means of segregating wood/bark chip mixtures. Information in this report, under the section Water Flotation Behavior, compares the basic density (green weight divided by green volume) of sweetgum at several moisture contents.

TABLE XIX

SWEETGUM SPECIFIC GRAVITY INFORMATION

(Ovendry weight/green volume)

| Wood | Bark | | | |
|---|---|---|---|---|
| Average | Inner | Outer | Total | Reference & Remarks |
| 0.46 | | | | IUFRO (5) |
| 0.47 (young gum) | | | | Barker (20) |
| 0.48 (mature gum) | | | | |
| 0.45 (stem wood) | | | 0.37 (stem bark) | Manwiller (17) |
| 0.45 (branch wood) | | | 0.42 (branch bark) | Manwiller (17) |
| | | 0.36 | | Fournier & Goulet (7) |
| 0.44 | | | | Isenberg (8) |
| 0.43 (sapwood) | 0.57 | 0.36 | 0.48 | IPC 3212-25 |
| 0.44 (heartwood) | | | | |
| 0.40 (sapwood) | 0.45 | 0.37 | 0.41 | IPC 3212-26 |
| 0.40 (heartwood) | | | | |
| | | | 0.58[a] | Harkin & Rowe (9) |
| 0.52[a] | | | | Isenberg (8) |

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[a] Ovendry weight/ovendry volume.

An average specific gravity (ovendry weight/green volume) of approximately 0.44 appears appropriate for the wood of sweetgum. Our limited data show the heartwood and sapwood to be very close in specific gravity.

The specific gravity of the total (inner + outer) bark of sweetgum is also very close in specific gravity to that of the wood. The inner bark of the two trees sampled as part of the project was higher in specific gravity than the outer bark. Overall values suggested for use in species comparisons are 0.44 for wood and 0.51, 0.36 and 0.42 for inner, outer, and total bark.

## Extractives

Extractives in wood and bark are important because, when present in large amounts, they not only result in reduced yield of fibrous material but ultimately can be expected to result in paper machine "pitch problems." Recent needs to reduce total water use through closed white water systems are expected to accentuate problems in this area. No attempt has been made in this report to go beyond determining the total alcohol-benzene extractives. Such extractives information is expected to provide an appropriate indication regarding possible pitch problems when large amounts of bark are pulped. Further detailed examination of the types of extractives involved is recommended using specific bark sources if preliminary comparisons suggest pitch and yield problems may develop.

A range in levels of extractives from 1.5 to 4.2% has been reported for sweetgum (see Table XX). For between-species comparisons, an extractives level of 2.6% is suggested for the wood of sweetgum. The extractives content of sweetgum bark apparently varies greatly from source to source (and very likely seasonally and with proportion of inner bark). IPC samples (No. 25 and 26) had only 4.3 and 4.4% extractives while stem bark values of 12.9% for Manwiller and 11.0% for a third IPC sample from Georgia suggest an average value of 10.4% should be used. This is an intermediate level of extractives and should not be a serious problem except in those instances where high percentages of bark have been concentrated in a particular chip fraction by screening or other mechanical techniques.

## Fibrous Material

Increasing emphasis is being placed on pulping bark rather than debarking bolts or segregating wood/bark chip mixtures. Important to determining the usefulness of this approach with a particular species is determining the proportion of lignified cells that exist in the bark and that will survive normal cooking procedures. Also, it is important to determine what percentage of these cells will contribute in a favorable way to the resulting paper product.

TABLE XX

SWEETGUM ALCOHOL-BENZENE EXTRACTIVES

| Type of Material | Extractives, % | Sources |
|---|---|---|
| Young wood | 1.5 | Barker (20) |
| Mature wood | 2.0 | Barker (20) |
| Stemwood | 2.3 | Manwiller (17) |
| Branchwood | 3.1 | Manwiller (17) |
| Wood | 2.0 | Isenberg (8) |
| Wood | 4.2 | Isenberg (8) |
| Stem bark | 12.9 | Manwiller (17) |
| Branch bark | 15.8 | Manwiller (17) |
| Bark | 19.2 | Harkin & Rowe (9) |
| Bark | 4.4 | IPC 3212-25 |
| Bark | 4.3 | IPC 3212-26 |
| Bark | 11.0 | IPC 3212-80 |

Sclereids are short, thick-walled, heavily lignified cells. When not fully cooked, as could occur in high-yield pulping, clumps of sclereids may cause so-called "fish eyes" in certain grades (calendered) of paper. According to Chang (1), only 2.8% of the tissue elements in the inner bark of sweetgum are sclereids. Sclereids also were found to be a very minor part of our pulped barks.

In the inner bark of some species there occurs bands of heavily lignified fibers described in the literature as phloem fibers or sclerenchyma fibers. These fibers are the principal bark elements to survive chemical pulping and contribute to overall pulp yield and sheet strength. Chang (1) estimated that 16.3% of the inner bark of sweetgum was composed of phloem fibers based upon examination of cross sections. As a further check on pulp yield and the nature of fibrous material produced from sweetgum, 20 to 30-gram samples were pulped using the IPC Standard Kraft Micropulping Procedure. For a complete description of the procedure see the section on Experimental Procedures. Table XXI summarizes the results of this investigation.

The other elements in the bark of sweetgum which have an effect on the pulp are sieve tubes. The short, thin-walled sieve tubes could be used as filler material in paper. However, it is questionable, other than an increase in pulp yield, whether they would contribute in any useful way to paper properties. When subjected to beating, they probably would not fibrillate to any appreciable extent. A sheet of paper, made entirely of sieve tubes, would probably be extremely brittle and low in strength.

Micropulping sweetgum bark resulted in a 32 to 38% yield of solids. When screened, a large part (48-65%) of the material went through the 200-mesh screen. Retained on the coarse screens (60 and 100 mesh) were primarily fibers with a smaller percentage of sieve tubes. The "on 150 mesh" and "on 200 mesh" material was mainly sieve tubes with some sclereids and fibers. The "through 200 mesh" contained 90-95% peridermal and parenchymatous cells. Figure 17 illustrates the type of material retained on the 60 and 150-mesh screens.

Based upon very limited numbers of bark sample observations, it appears that, for every 100 grams of bark that are pulped, about 35 grams of solids will result. Of this 35 grams, about 5 grams (5%) of fibers and 3 grams (3%) of sieve tubes will be produced. This assumes that only the material on the 60 and 100-mesh screens would end up in and contribute in any significant way to the final product. The remaining material would be lost in washing and cleaning operations. The amount of fiber and sieve tubes recovered from pulping

## TABLE XXI

## SWEETGUM MICROPULPING INVESTIGATIONS

| Data | Sample No. 3212-25 | Sample No. 3212-26 | Remarks[a] |
|---|---|---|---|
| Yield, % solids | 32.4 | 37.4 | |
| Fraction | | | |
| on 60 mesh, % | 10.7 | 17.2 | Fraction contained principally phloem fibers (80-90%) with small percentages of sieve tubes (10-20%), sclereids (<5%), longitudinal parenchyma (<5%) and peridermal and parenchymatous cells (<1%). The arithmetic average length of phloem fibers in this fraction was 0.95 mm |
| on 100 mesh, % | 12.7 | 18.9 | Fraction contained large percentages of sieve tubes (50-60%) and phloem fibers (40-50%) with small percentages of longitudinal parenchyma (<5%), sclereids (<5%) and peridermal and parenchymatous cells (<1%). The arithmetic average length of the sieve tubes in this fraction was 0.80 mm |
| on 150 mesh, % | 8.7 | 10.2 | Fraction contained a large percentage of sieve tubes (60-70%), with small percentages of phloem fibers (10-20%), sclereids (5-10%), longitudinal parenchyma (<5%) and peridermal and parenchymatous cells (<1%) |
| on 200 mesh, % | 3.9 | 5.3 | Fraction contained sieve tubes (30-40%), sclereids (20-30%), peridermal and parenchymatous cells (20-30%), longitudinal parenchyma (10-20%), phloem fibers (<1%) |
| through 200 mesh, % | 64.0 | 48.4 | Fraction contained principally peridermal and parenchymatous cells (90-95%) with a small percentage of sclereids (5-10%) |

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[a] Percentages in each fraction on a weight basis.

Figure 17. The 60-Mesh Screen (Top) Contained by Weight Principally Phloem Fibers (80-90%) with Smaller Percentages of Sieve Tubes (10-20%). The 150-Mesh Screen (Bottom) Contained a Large Percentage of Sieve Tubes (60-70%) with Smaller Percentages of Phloem Fibers (10-20%) and Sclereids (5-10%). Magnification 75X. Symbols Include Phloem Fibers (PF), Sieve Tubes (ST) and Sclereids (PS)

of the bark, combined with an intermediate level of extractives, make pulping of sweetgum bark an attractive possibility.

## WOOD/BARK ADHESION

Wood/bark adhesion differences have been suggested as one of the reasons for the differences encountered in the ease of debarking pulpwood species. The same factors influencing debarking of pulpwood are expected to influence debarking of wood chips. The approach taken in the study has been to obtain growing season and dormant season information on (1) magnitude of wood/bark adhesion, (2) morphological structures associated with wood/bark adhesion, and (3) reasons for differences between species in adhesion.

Wood/bark adhesion values were measured for sweetgum samples collected July 25 (growing season) and September 30 and October 7 (dormant season). Using the sampling and testing procedures described in the section on Experimental Procedures in Report One, shear parallel to the grain was measured. After testing, the samples were examined to determine the location of the zone of failure. Figure 18 illustrates the zone of failure for sweetgum during both the growing and dormant seasons. During the growing season, wood/bark adhesion was unusually high (10.2 kg/cm$^2$), considerably higher than any species tested thus far. The failure zone occurred in the xylem between immature xylem cells and cambial initials. During the dormant season, wood/bark adhesion increased to 15.3, also a relatively high value, and the failure zone occurred in the inner bark, primarily between the sieve tubes located immediately adjacent to the cambium zone.

As a result of measurement data taken on the species included in Appendix Table XXVIII and the measurement data reported in the previous reports for this project, it is clear that dormant season wood/bark adhesion is related

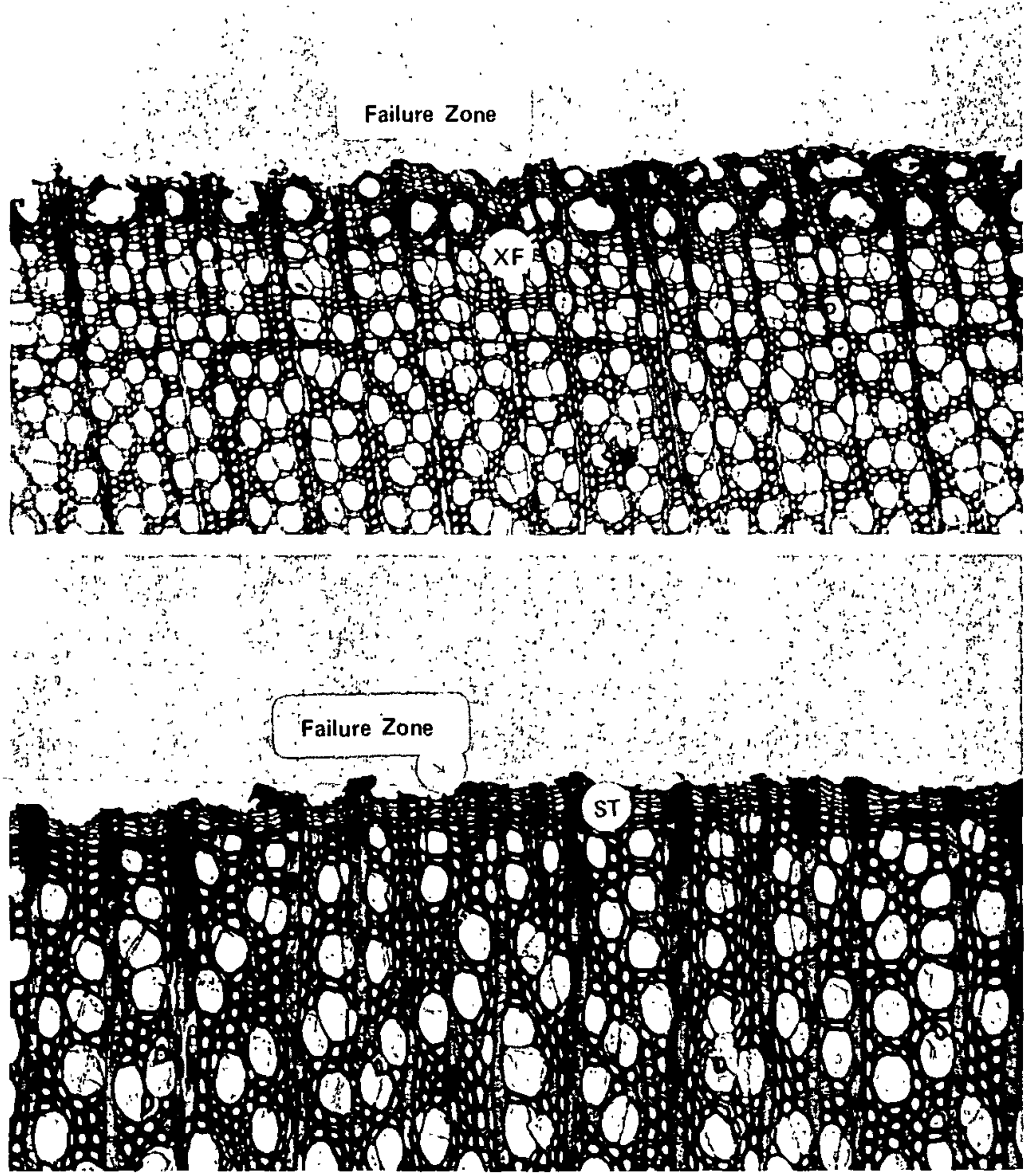

Figure 18. Illustrated Is the Sweetgum Failure Zone for Both the Growing Season (Top) and Dormant Season (Bottom). The Growing Season Failure Zone Was Located Between Immature Xylem Fibers (XF) in the Region of the Cambium Zone. During the Dormant Season, the Failure Zone Was Located in the Inner Bark, Primarily Between Sieve Tubes (ST) Located Immediately Adjacent to the Cambium Zone. Magnification — 75X Top, 125X Bottom

to inner bark strength and inner bark strength is, in turn, related to inner bark morphology. The presence of phloem fibers in the inner bark of hardwoods appears to be associated with high dormant season wood/bark adhesion. High numbers of sclereids and a lack of phloem fibers seem to be associated with low dormant season wood/bark adhesion and low bark strength. Sweetgum bark has a fair amount of fiber and relatively few sclereids and this could be the reason for the high wood/bark adhesion values obtained.

Separation (breaking the bond between bark and wood in a chip) is an important first step in segregation (removal of bark particles from wood chips). Separation during the growing season, when wood/bark adhesion is low, can usually be accomplished by the action of the chipper. During the dormant season, adhesion is greater and separation by chipper action is less successful.

Several approaches were tried with hardwoods and two softwoods in Project 2929 to reduce adhesion that might have some promise. These methods included chemical, thermal and biological methods. These methods have not been tried with sweetgum but are worthy of further consideration and are discussed in the section on Between-Species Comparisons.

## BARK STRENGTH, TOUGHNESS AND REACTION TO HAMMERMILLING

Bark strength and toughness measurements are included as part of the characterization of bark because it was felt that when these measurements are compared with the results obtained in wood/bark adhesion tests, with the difficulty encountered in conventional debarking and with bark morphology, the "why" of bark separation and segregation would eventually emerge.

Hammermilling has been widely used in bark utilization to prepare fractions for use as horticultural mulch, soil conditioners, and as additives to a number of types of products. A simulated hammermilling test was developed in an effort to relate the hammermilling of bark (and wood) to bark strength, toughness and morphology.

As discussed in the section on Experimental Procedures, bark strength measures shear parallel to the grain while bark toughness measures the energy required to rupture a thin specimen by a bending force perpendicular to the grain (parallel to the tree diameter). Table XXII summarizes the bark strength and toughness tests made on the wood and bark of sweetgum. Also included for comparison purposes (Appendix Table XXIX) are the bark strength values for a number of pulpwood species of interest.

TABLE XXII

SUMMARY OF STRENGTH AND TOUGHNESS MEASUREMENTS
MADE ON WOOD AND BARK OF SWEETGUM[a]

| Material | Strength | Toughness |
|---|---|---|
| Wood | -- | 0.28 |
| Inner bark | 8.1 | 0.20 |
| Outer bark | 5.2 | 0.11 |

---

[a] Determinations average of two different trees.

Fairly large differences were obtained in strength and toughness between inner and outer bark. This suggests that it may be possible to remove most of the outer bark by hammermilling and screening procedures. The relatively high bark strength and toughness values are probably related to the presence of fiber in the inner bark.

Toughness tests on a number of species, as reported by Wood Handbook (19) were done on both tangential and radial sections and represented the energy required to rapidly cause complete failure in a centrally loaded bending specimen. Our toughness test is done on a tangential section at 20% moisture content and is a measure of the energy required to rupture a thin specimen by a bending force perpendicular to the grain. At 13% moisture content Wood Handbook results show sweetgum with a tangential value of 260. Again, our results indicate that the wood of sweetgum is weaker than that of sugar maple and southern white oak which agrees with the results obtained in Wood Handbook (sugar maple — 360 *vs*. 1.20, southern white oak — 310 *vs*. 0.98, and sweetgum — 260 *vs*. 0.28). When enough data are obtained, correlations will be run between these values and hammermilling results.

Hammermilling, followed by screening, resulted in a moderate reduction in levels of bark. When the half-sized chips for the two trees investigated were hammermilled, and the material on the 14-mesh screen retained, the result was a 7% loss in wood and a 32% reduction in bark. The bark removed appeared to be mostly outer bark. Since the inner bark has a specific gravity near that of the wood, hammermilling, followed by water flotation, would not, in all probability, result in effective segregation. However, the fiber contained in the inner bark could be of some value. Summarized in Table XXIII are the results of the hammermilling tests run on sweetgum wood and bark. Figure 19 illustrates the effect of hammermilling on the wood and bark of sweetgum. It is possible that improvements could be made in screening by taking advantage of the differences in configuration of wood and bark chips evident in Fig. 19 (12,13). Summary Table XXVI compares bark strength, toughness and reaction to hammermilling of sweetgum to other species tested thus far.

## TABLE XXIII

## SUMMARY OF HAMMERMILLING TEST ON SWEETGUM

| | | Fraction Retained on Standard Screen[a], % | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Tree No. | Type Material | 5 Mesh | 10 Mesh | 14 Mesh | 20 Mesh | 28 Mesh | <28 Mesh | Remarks |
| 3212-25 | Bark | 22 | 30 | 17 | 7 | 8 | 17 | Mostly inner bark on larger mesh screens. Increasing amounts of outer bark on smaller mesh screens |
| | Sapwood | 77 | 14 | 3 | 1 | 1 | 3 | |
| | Heartwood | 74 | 14 | 4 | 3 | 2 | 4 | |
| 3212-26 | Bark | 23 | 27 | 18 | 8 | 8 | 17 | Same as above |
| | Sapwood | 71 | 16 | 5 | 3 | 2 | 4 | |
| | Heartwood | 74 | 14 | 4 | 3 | 2 | 4 | |

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[a] Standard soil screen sizes; 5 mesh has 5 wires per inch and opening of 4.00 mm, 10 mesh has 10 wires per inch and an opening of 2.0 mm, 14 mesh has 14 wires per inch and an opening of 1.168 mm, 20 mesh has 20 wires per inch and an opening of 1.00 mm, and the 28 mesh screen has 28 wires per inch and an opening of 0.589 mm.

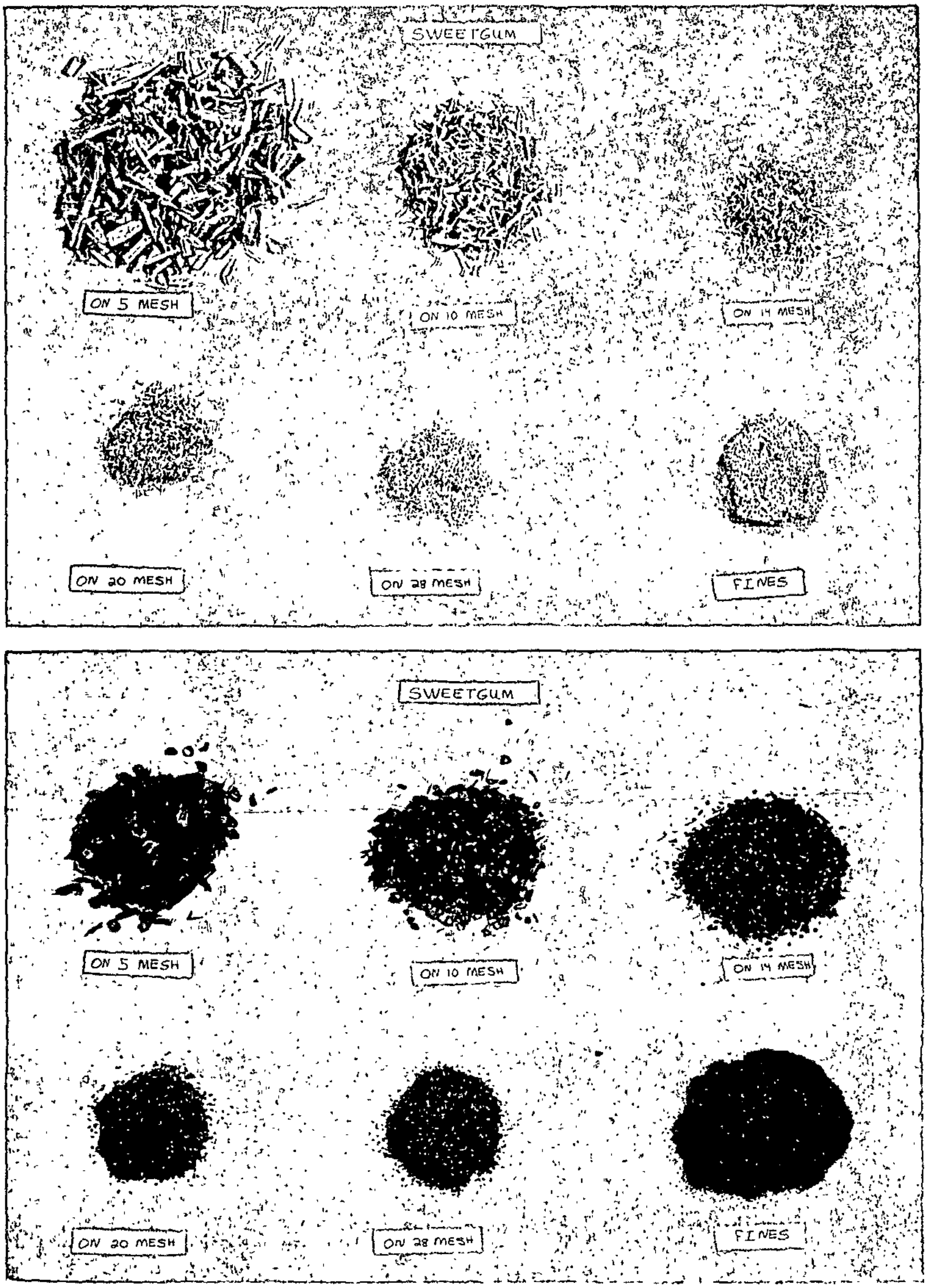

Figure 19. Illustrated Is the Effect of Hammermilling on Sweetgum Wood (Top) and Bark (Bottom)

## WATER FLOTATION BEHAVIOR

One possible method of segregating wood/bark chip mixtures is by water flotation procedures. Knowledge of the flotation characteristics of wood and bark is also expected to be important when certain types of chip washing procedures are employed. Earlier investigations into water flotation segregation (Project 2977) revealed that chip size, specific gravity, moisture content and rate of moisture uptake were factors in the flotation behavior of bark and wood chips. Budget limitations do not permit examination of all factors involved and, as a result, the influence of chip size has been eliminated from the variables considered.

Two procedures were used to examine the water flotation behavior of wood and bark. One procedure involved measuring the density* (green weight divided by green volume) of simulated chips at a number of different moisture contents. The second technique involved measuring the rate of moisture uptake and sinking of wood and bark chips in what have been designated as "dwell time" studies.

### Density Determinations

Simulated chips were used in determining the relationship between moisture content and the density of bark and wood. Wood and bark from two sweetgum trees (IPC 3212-25 and IPC 3212-26) were used in making the determinations. The moisture content of the chip samples was adjusted by equilibrating

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*The term density is used in this report to indicate the weight of wood and bark samples and is expressed in terms of green weight divided by green volume. This is in contrast to the term specific gravity, which also is an expression of the weight of a sample, but in this case it is in terms of dry weight divided by green volume.

in small jars to which had been added appropriate amounts of water. The extremely accurate pycnometer method described in the Experimental Procedures in Report One was used in determining density. Bark samples used were "whole bark" samples, a combination of both inner and outer bark. Small chips of inner and outer bark were also tested. Our limited data showed the inner bark to be higher in density than whole bark and the outer bark slightly lower.

Figure 20 illustrates the relationship that was found between moisture content and density. The linear relationship shown was obtained by fitting the least squares regression line through the data. The dashed lines are two standard deviations above and below the average values. The standard deviation of the regression line is considerably less than would have been obtained if conventional mill-run chips had been used for the water flotation studies, because the simulated chips were uniform in size and shape, had a uniform level of moisture and were relatively free of knots, reaction wood, etc. Water segregation is believed to be possible when one fraction has a density of less than one and the other greater than one at a specific moisture content.

The data indicate that segregation through water flotation would be difficult to achieve. Both wood and bark chips could be expected to float (density less than 1) even at very high moisture contents.

### Dwell Time Investigations

An investigation of dwell time involves nothing more than taking wood and bark chips at some standard moisture content, placing them on a water surface and observing the time it takes the material to pick up enough water to sink. Information on dwell time is useful because moisture uptake rates could have a considerable influence on the success of a segregation procedure (or chip-washing

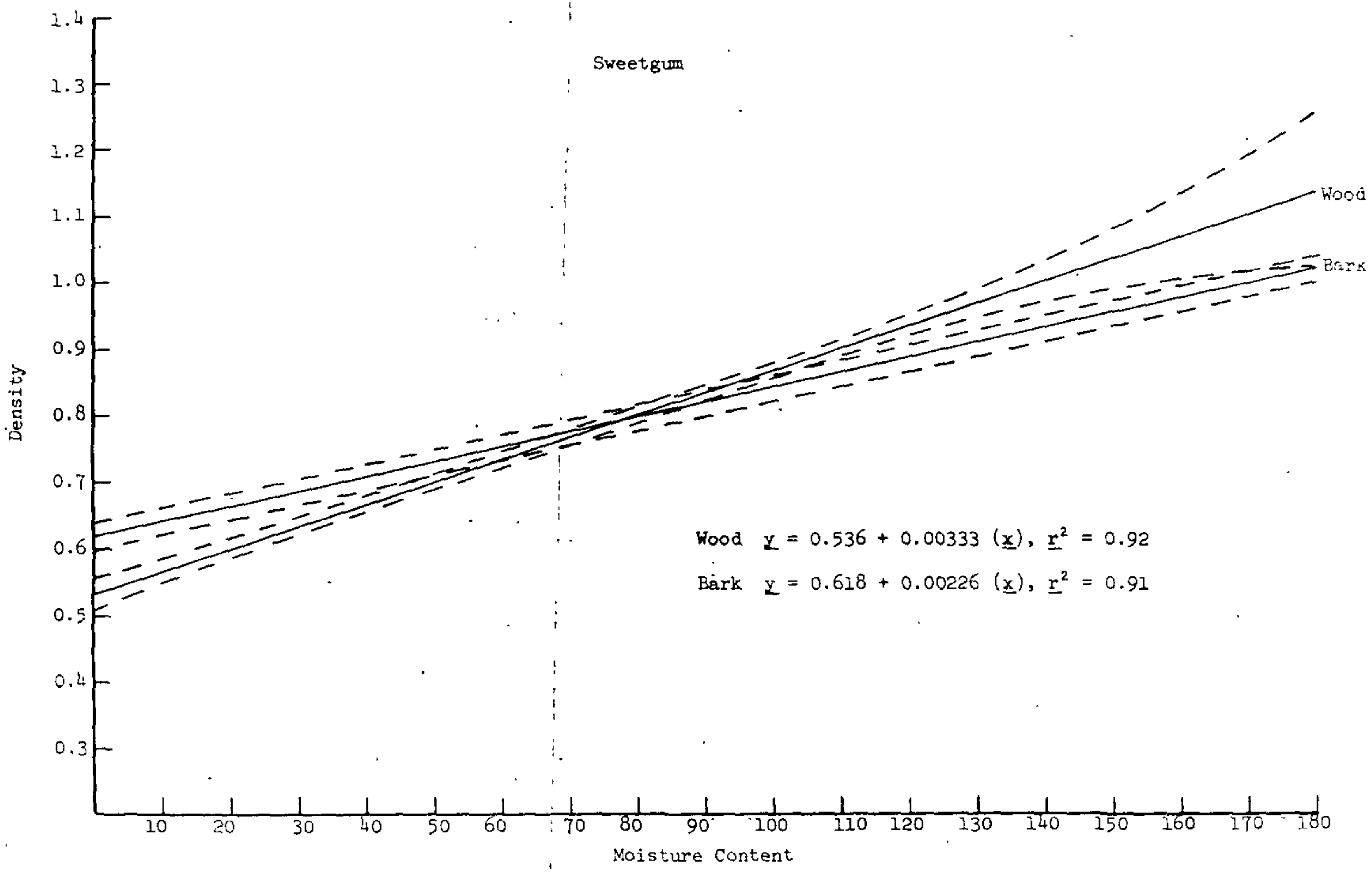

Figure 20. Illustrated Is the Relationship Between Basic Density and Moisture Content for Sweetgum. The Dashed Lines are Two Standard Deviations Above and Below the Mean

procedure) and would provide information on the rate at which segregation could be expected. A species in which either the bark or the wood takes up moisture rapidly could be expected to have a relatively short segregation time. For other species, where specific gravity and density of the wood and bark are similar, and moisture uptake is similar, considerable difficulty in segregation can be anticipated. Time required for chip samples to sink decreases as the sample moisture content at the start of the trial increases.

Half-sized simulated chips (1 x 0.3 x 0.2 inch) were used in the dwell time tests. Prior to testing, the samples were equilibrated in 50% RH and had a moisture content of approximately 20% (ovendry basis). Table XXIV summarizes the results for sweetgum. These results agree with the results obtained in the density determinations which showed both wood and bark floating at 20% moisture content.

DATA INTERPRETATION

Sweetgum had the highest wood/bark growing season adhesion values of any of the species tested so far and also relatively high dormant season values. This makes it appear that many of the methods used to separate wood from bark such as chipper action may not work as well for sweetgum. The high adhesion values appear to be related to low numbers of sclereids and relatively large amounts of fiber.

Density _vs_. moisture content and dwell time investigations showed that segregation of wood/bark chip mixtures through water flotation would be almost impossible to achieve. Both wood and bark chips floated, even at very high moisture contents.

## TABLE XXIV

## SUMMARY OF DWELL TIME RESULTS FOR SWEETGUM

| Sample No. | Time Interval, min | Sinkers, % | Floaters, % |
|---|---|---|---|
| IPC 3212-25 Bark | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-25 Sapwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-25 Heartwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-26 Bark | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-26 Sapwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |
| IPC 3212-26 Heartwood | after 5 | 0 | 100 |
| | 15 | 0 | 100 |
| | 60 | 0 | 100 |
| | 240 | 0 | 100 |

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[a] Starting moisture content 20%.

Hammermilling wood and bark chips and then screening showed moderate results. When the material on the 14-mesh and larger screens was retained, the results were a 7% wood loss and a 32% bark removal. When the size of material retained was increased to 10 mesh and larger, there was a 50% reduction in bark levels but a 12% wood loss. However, as with the previous species, it appears the bark could be concentrated into one or two small-sized chip fractions by screening and those fractions hammermilled and rescreened. It also appears that most of the bark removed was outer bark, leaving behind the inner bark which is high in fiber.

Sweetgum bark is a more suitable candidate for pulping than many others. It contains very few sclereids and relatively high amounts of fiber. For every 100 grams of sweetgum bark that is pulped, about 5 grams of fiber and 3 grams of sieve tubes would be produced. The sieve tubes could be used as filler material in paper but probably would not contribute in any useful way to paper properties.

## RELATED LITERATURE

A paper by Choong, et al. (23) discusses the mineral content of wood and bark of sweetgum. The previously cited papers by Auchter and Horn (14), Hooper (15), and Biltonen, et al. (16) deal with the economics and mechanics of segregating wood/bark chip mixtures.

## BETWEEN-SPECIES COMPARISONS

Between-species comparisons are helpful because they allow establishment of useful relationships and assist in determining those species that should be handled in a similar manner. Tables XXV and XXVI provide a method of quickly comparing basic information available for the first sixteen species investigated. The data for conifers are summarized in Table XXV and Table XXVI contains the hardwood data. As the information on additional tree species becomes available, increasing numbers of useful relationships between morphology, density, bark strength, wood/bark adhesion and other characteristics are developing.

For the species investigated, conifer barks were lower in specific gravity than most hardwood barks (cottonwood was an exception). Conifer bark also tended to be similar or lower in specific gravity than associated sapwood. Conifer barks, with the exception of Douglas-fir, had no fiberlike elements and, as a result, had a low solids yield and no useful fibrous yield when pulped. A number of tree species have bark sclereids (thick-walled, lignified, nonfiber-like cells) and two of the conifers (western hemlock and balsam fir) had high levels of sclereids that survived the kraft pulping procedure and could be expected to be a problem in certain grades of paper. The fibrous yield of hardwood barks varied from 10% for cottonwood and aspen to no usable fiber in white birch. The several oak species studied had three to five percent usable bark fiber. It was also noted that the specific gravity of the bark of the red oaks were higher than of the wood while the reverse was true for white oak and bur oak (studied earlier).

There has been no consistent pattern with regard to level of extractives with the exception that the levels in the bark are from three to eight times as

## TABLE XXV

## WOOD AND BARK CHARACTERISTICS OF CONIFER PULPWOOD SPECIES

| Characteristic | White Spruce | Balsam Fir | Jack Pine | Loblolly Pine | Slash Pine | Douglas-Fir | Westerm Hemlock |
|---|---|---|---|---|---|---|---|
| Specific gravity (o.d. wt./green vol.) | | | | | | | |
| Wood | 0.34 | 0.34 | 0.39 | 0.45 | 0.54 | 0.43 | 0.40 |
| Total bark | 0.39 | 0.40 | 0.41 | 0.33 | 0.35 | 0.41 | 0.45 |
| Inner bark | -- | 0.32 | -- | 0.29 | 0.34 | 0.42 | 0.46 |
| Outer bark | 0.43 | 0.46 | 0.43 | 0.34 | 0.36 | 0.40 | 0.45 |
| Extractives, % | | | | | | | |
| Wood | 2.2 | 2.0 | 3.9 | 3.0 | 3.3 | 4.0 | 1.6 |
| Bark | 16.0 | 19.5 | 15.3 | 8.5 | 8.4 | 16.4 | 11.7 |
| Density at 100% moisture (green wt./green vol.) | | | | | | | |
| Wood | 0.70 | 0.75 | 0.79 | 0.88 | 1.10 | 0.815 | 0.80 |
| Bark | 0.83 | 1.07 | 0.83 | 0.57 | 0.72 | 0.825 | 0.85 |
| Pulp yield, % (bark) | 20.6 | 26.0 | 18.6 | 23.6 | 23.6 | 17.6 | 35.8 |
| Usable bark fiber, %[a] | 0 | 0 | 0 | 0 | 0 | 5 | 0 |
| Sclereids remaining, %[a] | 1.5 | 12.0 | 0 | 0 | 0 | 2 | 11 |
| Fiber location[b] | -- | -- | -- | -- | -- | IB-OB | -- |
| Sclereid location[b] | IB-OB | IB | -- | -- | -- | IB-OB | IB-OB |
| Wood/bark adhesion, kg/cm² | | | | | | | |
| Growing season | 4.4 | 2.4 | 4.0 | 5.8 | 3.5 | 3.4 | 3.6 |
| Dormant season | 10.3 | 9.0 | 10.7 | 5.5 | 9.1 | 8.0 | 8.2 |
| Bark strength, kg/cm² | | | | | | | |
| Inner bark | -- | 1.7 | 2.3 | 3.7 | 6.4 | 5.8 | 6.0 |
| Outer bark | 7.4 | 1.4 | 2.3 | 3.2 | 5.2 | 3.0 | -- |
| Toughness | | | | | | | |
| Inner bark | -- | 0.06 | -- | 0.10 | 0.06 | 0.34 | 0.12 |
| Outer bark | 0.16 | -- | 0.07 | 0.06 | 0.09 | 0.03 | 0.10 |
| Sapwood | 0.34 | 0.42 | 0.34 | 0.54 | 0.54 | 0.58 | 0.28 |
| Hammermilling[c] | | | | | | | |
| Bark removed, % | 23 | 44 | 26 | 34 | 36 | 28 | 24 |
| Wood loss, % | 4 | 6 | 5 | 6 | 5 | 4 | 3 |

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[a] Usable bark fiber and sclereids remaining are the fibers and sclereids retained on the 60 and 100-mesh screens. The percentage given is the yield based on whole bark samples.

[b] Major proportion located in either the inner bark (IB) or outer bark (OB).

[c] Based upon simulated hammermilling followed by screening, using the on 14-mesh screen to remove bark and recover usable fiber from fines.

TABLE XXVI

WOOD AND BARK CHARACTERISTICS OF HARDWOOD PULPWOOD SPECIES

| Characteristic | Quaking Aspen | Eastern Cottonwood | Sweetgum | Sugar Maple | White Birch | Northern Red Oak | Southern Red Oak | Northern White Oak | Southern White Oak |
|---|---|---|---|---|---|---|---|---|---|
| Specific gravity (o.d. wt./green vol.) | | | | | | | | | |
| Wood | 0.38 | 0.38 | 0.44 | 0.59 | 0.49 | 0.56 | 0.60 | 0.64 | 0.67 |
| Total bark | 0.50 | 0.31 | 0.42 | 0.54 | 0.56 | 0.65 | 0.70 | 0.58 | 0.56 |
| Inner bark | 0.40 | 0.29 | 0.51 | 0.69 | 0.57 | 0.53 | 0.68 | 0.65 | 0.70 |
| Outer bark | 0.55 | 0.32 | 0.36 | 0.49 | 0.54 | 0.71 | 0.70 | 0.52 | 0.44 |
| Extractives, % | | | | | | | | | |
| Wood | 3.0 | 1.4 | 2.6 | 1.0 | 4.0 | 4.5 | 4.8 | 2.4 | 4.6 |
| Bark | 15 | 7.9 | 10.2 | 6 | 17 | 11 | 11.6 | 7.2 | 8.6 |
| Density at 100% moisture (green wt./green vol.) | | | | | | | | | |
| Wood | 0.79 | 0.84 | 0.84 | 1.24 | 1.01 | 1.06 | 1.25 | 1.30 | 1.38 |
| Bark | 1.15 | 0.81 | 0.87 | 1.08 | 1.16 | 1.18 | 1.39 | 1.05 | 1.13 |
| Pulp yield, % (bark) | 33.8 | 35.4 | 34.9 | 33.9 | 36.3 | 28.4 | 30.7 | 35.4 | 36.6 |
| Usable bark fiber, %[a] | 10 | 9 | 5 | 3 | 0 | 5 | 4 | 3 | 3 |
| Sclereids remaining, %[a] | 1 | <0.1 | -- | 0.2 | 0.7 | 0.2 | -- | -- | -- |
| Fiber location[b] | IB | IB | IB | IB | -- | IB | IB | IB | IB |
| Sclereid location[b] | IB | -- | IB | IB | IB | IB | IB-OB | IB-OB | IB-OB |
| Wood/bark adhesion, $kg/cm^2$ | | | | | | | | | |
| Growing season | 6.4 | 4.4 | 10.2 | 5.8 | 5.1 | 2.5 | 5.4 | 4.8 | -- |
| Dormant season | 11.4 | 13.5 | 15.3 | 10.1 | 12.0 | 8.4 | 8.2 | 7.8 | 7.2 |
| Bark strength, $kg/cm^2$ | | | | | | | | | |
| Inner bark | 9.0 | 17.7 | 8.1 | 1.4 | 1.6 | 2.1 | 3.6 | 4.6 | 4.7[d] |
| Outer bark | 4.9 | 4.2 | 5.2 | 4.7 | 9.8 | 4.6 | 3.4 | 3.2 | -- |
| Toughness | | | | | | | | | |
| Inner bark | 0.22 | 0.14 | 0.20 | 0.25 | 0.10 | 0.13 | 0.11 | 0.16 | 0.12 |
| Outer bark | 0.10 | 0.11 | 0.11 | 0.10 | 0.10 | 0.18 | 0.14 | 0.10 | 0.09 |
| Sapwood | 0.45 | 0.38 | 0.28 | 1.20 | 0.68 | 0.93 | 0.55 | 0.62 | 0.98 |
| Hammermilling[c] | | | | | | | | | |
| Bark removed, % | 34 | 18 | 32 | 29 | 38 | 34 | 46 | 37 | 38 |
| Wood loss, % | 5 | 5 | 7 | 5 | 6 | 10 | 6 | 5 | 3 |

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[a] Usable bark fiber and sclereids remaining are the fibers and sclereids retained on the 60 and 100-mesh screens. The percentage given is the yield based on whole bark samples.

[b] Major proportion located in either the inner bark (IB) or outer bark (OB).

[c] Based upon simulated hammermilling followed by screening, using the on 14-mesh screen to remove bark and recover usable fiber from fines.

[d] Test mistakenly performed on total bark.

high as that of the wood. With regard to variation in extractive levels, there is evidence in the literature for oaks (18) that indicates the inner bark has higher extractive levels than the outer bark and that inner bark extractive levels are lower during the summer than in the fall, winter and early spring. Seasonal variations and the proportion of inner bark to total bark accounts for some of the variation encountered in extractives.

Wood/bark adhesion during the growing season was low, similar in magnitude for all species investigated (except sweetgum), and the zone of failure quite consistently occurred in the cambium zone or the newly-formed nonlignified wood fibers immediately adjacent to the cambium zone. Dormant season wood/bark adhesion was higher than during the growing season. Dormant season wood/bark adhesion for the hardwoods tends to be slightly higher than for conifiers. Also, for most species, dormant season failure usually occurred in the partially mature sieve and parenchyma cells of the inner bark located just outside the cambium zone. High wood/bark adhesion in hardwoods was often associated with large numbers of phloem fibers in the inner bark.

Mechanical treatment of bark continues to appear promising as a method of upgrading low-quality chips high in levels of bark. Toughness, for example, varies between species but is consistently lower for bark than for wood. Toughness differences suggest hammermilling or a similar treatment (crushing or shredding) could be used to reduce the size of the bark particles and allow the bark to be removed by screening. Correlations between hammermilling results and bark strength and toughness so far are quite low. Also, there appears to be little correlation between wood loss and the density or toughness of the wood. Bark removal, on the other hand, appears to be moderately correlated to toughness, bark strength and density. Low bark density combined with high bark strength and/or toughness (spruce, hemlock and cottonwood) resulted in low bark removal by hammermilling. Extremely low bark strength and toughness (balsam fir) resulted in a high percentage of bark removed. Intermediate bark density and bark strength values were poorly correlated with bark removal. The best correlations were obtained between bark removal and bark specific gravity and between bark removal and the percentage of nonfibrous sclerenchyma (sclereids and phellem cells). High levels of nonfibrous sclerenchyma cells and/or high specific gravity seemed to cause the bark to break up more readily and increased the chances the bark particles could be removed by a screening process.

As reported earlier, breaking the bond between wood and bark (separation) is an important first step in any segregation procedure. A very practical way of separating bark and wood during the growing season, and in some instances during the dormant season, is through the action of the chipper. Arola (24), working with northern hardwoods, found that chipper action during the growing season gave better results than during the dormant season with less than 2% bark remaining on the chips from 4-6 and 8-inch diameter bolts. Erickson (25) obtained similar results with spruce, balsam fir, and jack pine. Results during the growing season were good; however, separation during the dormant season was poor (36-72%) for bolewood and even less for the thin-barked branchwood, with the poorest month for separation being the month of November (36-48%). Erickson (25), working with maple, reported 96% separation during the chipping throughout the year. He also found better separation with winter-cut frozen wood over unfrozen bolts although more fines resulted.

Despite the consistent location of the wood/bark failure zone, there are, particularly in the dormant season, major differences between species in the ability of the chipper to cause separation. Preliminary investigation, Project

2929, suggests inner-bark strength and the chipper knife impact on the cambium zone are important factors. For hardwoods and possibly some conifers, the presence of fibers and sclereids in the inner bark influence inner bark strength. Bark thickness and wood density (or frozen wood) influence chipper knife impact at the cambium zone. Chipper separation during the dormant season is expected to be least effective on thin-barked, low-density woods with fiber in the inner bark. White spruce, although it has no fiber in the inner bark, is an excellent example of a thin-barked low-density wood in which dormant season separation is poor.

Earlier investigation (Project 2929) indicated that there were a number of other procedures (chemical, thermal, and biological) worthy of further consideration in breaking the bond between bark and wood. Use of green kraft cooking liquor at a temperature of 200°F and a treatment time of 60 min gave reduced adhesion. Chemical treatments were also investigated by Haas and Kremers (26) and in their work, dilute acids were effective in reducing adhesion. Pressure chamber treatments also looked promising with reduced treatment time needed when temperatures were in excess of 250°F. Moist storage of chips at temperatures that encourage fungus attack of the cambium zone resulted in greatly reduced wood/bark adhesion at storage times as short as 15-20 days. Another promising approach was microwave heating to create high temperatures in the moist interior of the chips.

One approach that was discussed in earlier reports and which appears to hold considerable promise as a method of reducing the level of bark in whole tree chips for most hardwood species and certain conifers is the use of chip screening. When screening, which concentrates the bark problems in the small size chips (20 to 25% of the total input), is followed by hammermilling or a similar mechanical action on the small-size chips, rescreening can be used to further upgrade the quality. Preliminary results with southern pine suggest the large-size chip fractions have only 4 to 6% bark and this can be handled by a reasonable amount of in-mill equipment modification. A minimum amount of mechanical action on the low-quality chips is expected to cause a 5 to 6% wood loss and a 30 to 36% reduction in the levels of bark. Mechanical action should be continued to the point where the wood loss vs. bark removal was near optimum in view of the relative value of the two types of material for fuel vs. fiber.

Red and white oaks often occur in the same stands and, because the two types of oaks behave in an opposite fashion with regard to water flotation, this approach cannot be used as a segregation procedure. The best overall method for use with the oaks appears to be the use of a "quick and dirty" screening and hammermilling procedure (described above) to reduce the total bark content by 50-60%, combined with a plan to pulp the remaining bark. The bark retained, mostly inner, would produce some usable fiber and the bark removed, along with the wood lost, could be used for fuel.

The screening of the mechanically treated product, in addition to removing bark, could be expected to reduce the amount of embedded sand and dirt. The low-quality chips not going to fuel could either be blended back into the good-quality chips or pulped separately for lower-quality paper or board products.

## PLANS

To date, the bark of 16 pulpwood species has been characterized, including: quaking aspen, sugar maple, white birch, northern red oak (Report One); loblolly pine, slash pine, Douglas-fir, western hemlock (Report Two); white spruce, balsam fir, jack pine, eastern cottonwood (Report Three); and northern white oak, southern white oak, southern red oak, and sweetgum (Report Four).

The bark characterization work has been switched to a formally funded project, meaning that support comes from the Institute general research fund. Plans are to characterize an additional 16 species under the formally-funded arrangement. The next four species to be characterized include Englemánn spruce, lodgepole pine, ponderosa pine, and western larch. Ash and fuel value information will be added to the determinations being made and the report will also contain information on ash and fuel value for the earlier characterized species. The report is scheduled for completion around the end of November.

# ACKNOWLEDGMENTS

The authors of this report are indebted to Floyd Manwiller of the Southern Forest Experiment Station for supplying specific gravity and alcohol-benzene extractives information and Mr. Dale Volkman and Mr. Ted Clark of International Paper Company for supplying samples used in characterizing southern red oak and sweetgum. Thanks also go to Gary Wyckoff, Brent Stewart, and Dave Wautier for their assistance in collecting samples of local species. The authors also wish to acknowledge the help of Roger Van Eperen and John Peckham and his staff within the Division of Materials Engineering & Processes for adhesion, strength, and toughness measurements and the pulping work that was carried out. Special thanks go to Shirley Verhagen for her assistance with density determinations and wood and bark morphology descriptions and to Sandy Berghuis for carrying out some of the experiments described in this report.

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# GLOSSARY

Basic density. Green weight divided by green volume.

Cambium. A cylinder, strip, or layer of meristematic cells, which divide to give cells which ultimately form a permanent tissue. The primary cambium in the stem and root gives rise to xylem and phloem, and the secondary one produces bark.

DBH. Diameter breast height (4.5 feet).

Gelatinous fiber. Fiber, the inner wall of which is more or less gelatinous, or jellylike.

Inner bark. Tissues in the cylindrical axis of a tree immediately outside the cambium; includes the region of the secondary phloem from the cambium to the last-formed periderm.

Outer bark. Tissues in the cylindrical axis of a tree immediately outside the inner bark; includes the tissues from the last-formed periderm to the outer surface of the bark.

Paratracheal. Said of xylem parenchyma which occurs at the edge of the annual ring, around the vessels, but nowhere else.

Parenchyma. Tissue consisting of short, relatively thin-walled cells, generally with simple pits; concerned primarily with storage and distribution of carbohydrates.

Periderm. Term applied to the cork cambium (phellogen) and the tissues (phellem and phelloderm) derived from the cork cambium.

Ray. Ribbon-shaped strand of tissue extending in a radial direction across the grain.

Resin canal. An intercellular space, often bordered by secreting cells, containing resin or turpentine.

Rhytidome. A tissue cut off outside a periderm. The cells die leaving a crust made up of alternate layers of cork and dead phloem or cortex.

Scalariform. Like a ladder.

Sclereid. See Sclerenchyma.

Schlerenchyma. Mechanical tissue consisting of cells with thick, lignified walls and small lumens. If the cells are elongated, they are called fibers and usually occur in bundles. When the cells are oval or rounded, they are called sclereids. They occur singly or in groups.

Secondary phloem. Inner bark.

Segregation. Removal of either the wood or bark fraction from wood/bark chip mixtures.

Separation. Detachment of bark from wood.

Sieve tube. A characteristic element of phloem. It translocates food materials synthesized in the plant. The cells are living, thin-walled and in longitudinal rows. They are connected by perforations in their transverse walls, through which pass strands of cytoplasm.

Specific gravity. Ovendry weight divided by green volume unless otherwise specified.

Storied. Arranged in tiers or in echelon, as viewed on a tangential surface or in a tangential section.

Suberized. Transformed into cork.

Tracheid. Fibrous lignified cell with bordered pits and imperforate ends; in coniferous wood, the tracheids are very long (up to 7+ mm) and are equipped with large, prominent bordered pits on their radial walls; tracheids in hardwoods are shorter fibrous cells (seldom over 1.5 mm), are as long as the vessel segments with which they are associated, and possess small bordered pits.

Tylose. A balloonlike enlargement of the membrane of a pit in the wall of a vessel or tracheid, and a xylem parenchyma cell lying next to it. It protrudes and blocks the cavity of the wood element.

Uniseriate. Arranged in a single row, series, or layer. Also said of a vascular ray which is one cell wide in cross section.

Vasicentric. Paratracheal.

Vessel. Composite, and hence articulated, tubelike structure found in porous wood, arising through the fusion of the cells in a longitudinal row through the partial or complete disappearance of the cross walls.

Xylary initials. The newly formed vascular tissue which conducts water and mineral salts throughout the plant and provides mechanical support.

Xylem. Wood. The vascular tissue which conducts water and mineral salts throughout the plant and provides mechanical support. It consists of vessels, and/or tracheids, fibers and some parenchyma.

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## APPENDIX

### TABLE XXVII

### SAMPLE TREE INFORMATION[a]

| Species | Tree No. | Age, yr | Height, ft | DBH, inch | Location |
|---|---|---|---|---|---|
| Northern white oak | 3212-17 | 35 | 35.5 | 9.1 | N. central Wisconsin |
| | 3212-18 | 34 | 52.5 | 8.1 | N. central Wisconsin |
| Southern white oak | 3212-62 | 42 | 45.0 | 8.8 | Alabama |
| | 3212-63 | 40 | 38.0 | 7.4 | Alabama |
| Southern red oak | 3212-29 | 30 | 30.0 | 8.0 | Alabama |
| | 3212-30 | 30 | 40.0 | 8.3 | Alabama |
| Sweetgum | 3212-25 | 30 | 45.0 | 7.3 | Alabama |
| | 3212-26 | 33 | 35.0 | 7.6 | Alabama |

## TABLE XXVIII

## BETWEEN-SPECIES COMPARISONS OF WOOD/BARK ADHESION

| Species | Wood/Bark Adhesion, kg/cm$^2$ Peeling Season | Dormant Season |
|---|---|---|
| Loblolly pine | 5.8 | 5.5 |
| Slash pine | 3.5 | 9.1 |
| Douglas-fir | 3.4 | 8.0 |
| Western hemlock | 3.6 | 8.2 |
| White spruce | 4.4 | 10.3 |
| Jack pine | 4.0 | 10.7 |
| Balsam fir | 2.4 | 9.0 |
| Shagbark hickory | 5.3 | 26.9 |
| Eastern cottonwood | 4.4 | 13.5 |
| Quaking aspen | 6.4 | 11.4 |
| Bur oak | 5.8 | 9.6 |
| White birch | 5.1 | 12.0 |
| Sugar maple | 5.8 | 10.1 |
| Northern red oak | 2.5 | 8.4 |
| Southern red oak | 5.4 | 8.2 |
| Northern white oak | 4.8 | 7.8 |
| Southern white oak | --[a] | 7.2 |
| Sweetgum | 10.2 | 15.3 |

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[a] Growing season adhesion not measured.

## TABLE XXIX

## BETWEEN-SPECIES COMPARISONS OF BARK STRENGTH

| Species | Bark Strength, kg/cm$^2$ | |
|---|---|---|
| | Inner Bark | Outer Bark |
| Loblolly pine | 3.7 | 3.2 |
| Slash pine | 6.4 | 5.2 |
| Douglas-fir | 5.8 | 3.0 |
| Western hemlock | 6.0 | -- |
| White spruce | 7.4 | -- |
| Jack pine | -- | 0.07 |
| Balsam fir | 0.06 | -- |
| Shagbark hickory | 25.0 | 72.7 |
| Eastern cottonwood | 17.7 | 4.2[a] |
| Quaking aspen | 9.0 | 4.9 |
| Bur oak | 4.5 | 7.0 |
| White birch | 1.6 | 9.8 |
| Sugar maple | 1.4 | 4.7 |
| Northern red oak | 2.1 | 4.6 |
| Southern red oak | 3.6 | 3.4 |
| Northern white oak | 4.6 | 3.2 |
| Southern white oak | 4.7[b] | -- |
| Sweetgum | 8.1 | 5.2 |

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[a] Strength low, test samples failed during preparation, data based upon a single test.

[b] Bark strength measured on total bark rather than inner and outer bark.