THE MICROSTRUCTURE OF A WOOD PULP FIBER

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THE MICROSTRUCTURE OF A WOOD PULP FIBER

By

GEORGE J. RITTER, Chemist
and
G. H. CHIDESTER, Assistant Engineer

The results presented in this paper were obtained in fundamental study that the Forest Products Laboratory is making in its investigation of the behavior of wood, largely for the purpose of bettering the utilization of wood. These results will here be considered in connection with the problems that confront the pulp and paper manufacturer.

I. Constituents of Wood

Constituents Commonly Wasted

The manufacturer of pulp and paper naturally is interested in the nature and the location of the constituents of wood that are removed during the preparation of wood pulp.

Lignin is the major component (28.0 percent) of the part of the wood that is removed during the manufacture of sulphite pulp. It exists in two forms, which differ somewhat in chemical composition.

One form is the binding material (middle-lamella lignin) between the wood fibers; the other is a finely divided amorphous material (cell-wall lignin) in the cell wall. Whether the cell-wall form is chemically combined with the cellulose in the wall or is physically distributed throughout is not known definitely. If all the constituents except lignin are removed from a transverse section of wood, the two forms may be seen with the aid of a microscope. One form, the middle lamella, is revealed as a network; the other, the cell-wall form, appears as a finely divided agglomerated residue in the space formerly occupied by the lignified cellulose in the cell wall. (Plates 1 and 2.)

Further, if all the constituents except lignin are removed from transverse sections, which have been cut thicker than those which were used in Plates 1 and 2 so that the network might remain intact during washing, the two forms of lignin may be separated. By impregnating the network residue with paraffin, thin sections of the middle-lamella lignin may be cut and photographed. (Plates 3 and 4.)

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2Published in Paper Trade Journal, October 25, 1928.
In addition, two other groups of components, the pentosans not in cellulose and the extractives, approximately 6 percent each, are a total loss to the manufacturer of chemical pulp and paper and, still further, the difference between commercially bleached sulfite cellulose and chlorinated cellulose (20 percent of the wood) is another loss of lignin-free material that in all probability is an excellent paper-making material. From work done in the Pulp and Paper Section of the Forest Products Laboratory, it is known that the unbleached sulfite pulp yields can be increased from the usual 40 percent to approximately 50 percent with simultaneous improvement of the quality of the pulp.

Lastly, the viscose manufacturer in using the sulfite cellulose wastes another 7 percent of the spruce wood, leaving but 34 percent to be utilized.

**Constituents Commonly Utilized**

Cellulose, which is practically the sole constituent of chemical wood pulp, consists of various carbohydrates when it is prepared from wood. When isolated by chlorination, it constitutes approximately 60 percent of the total wood forming the major portion of the cell wall. In general, it consists of pointed capsular fibers. A knowledge of the minute structure and the properties of these fibers is of importance to the paper maker, to enable him to best adapt his processes to work in harmony with them rather than against them.

**II. Microstructure of the Wood Fiber**

**Separation of Layers in the Cell Wall**

The cellulosic material used in examining the microstructure of the wood fiber was prepared from thin longitudinal wood shavings. These shavings were delignified by the Cross and Bevan method, were dehydrated with alcohol for several days, and were kept in alcohol until required for use.

Examination of delignified fibers after they had received alternate swelling and shrinking treatments² with alkali and with acids indicated that the cell wall is in a manner similar to that of the cell-wall layers of cotton³ composed of several layers packed together closely. These layers are so close together or else are so embedded in a cementing material of such an index of refraction that the layers are invisible in the original wood. But, by chemical and physical treatments, which the fibers receive through delignification, swelling, and shrinking, the binding material can be removed or the layers can be pushed apart, so that the spacings become visible. The presence of several layers in the cell wall can also be shown plainly by treating delignified fibers with phosphoric acid.

²On neutralization of the alkali with dilute acid the wood sections shrink,

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If fibers that have been treated properly with alkali and acid are examined with the aid of a microscope, stratifications that define the various layers can be seen in the cell wall. (Plate 5.) Since the layers form pointed capsules that are nested, complete layers cannot be separated by sliding certain ones endwise over the others, even though they may have been properly loosened. If short sections of such fibers are used, however, it is possible to remove the loosened, concentric, tube-like sections of the layers by sliding them endwise. (Plate 6.)

At present, the chemical composition of the binding material between the various layers in the cell wall is unknown. It may be composed of an easily hydrolyzable portion of the cellulose. The quantity of this substance may be so minute that it will be necessary to determine it by difference in the composition of the residue before and after its removal, rather than to isolate, recover, and identify the binding material itself. This phase of the study will be undertaken later. The accomplishment reported here is the actual separation of the layers in the secondary thickening of the cell wall in wood fibers.

Orientation and Separation of the Fibrils in the Cell-wall Layers

The swelling properties of bordered pits reported in Part III of this paper, the optical properties observed between Nicol prisms, and the results that will be described in Part II indicate that not only do tiny fibrils form the various layers of the cell wall of wood fibers, but that these fibrils can be separated by chemical means.

The fibrils that compose the outer layers are oriented at approximately 90° to the long axis of the wood fiber. Immediately under the outside layer of some fibers there are occasional stray bands of fibrils wound about the inner layers at approximately 45° to the fiber's axis. Such fibrils do not form a continuous layer. In the remaining layers the fibrils are oriented from 0° to 30° to the axis of the fiber.

A study of the orientation of the fibrils in the various cell-wall layers and of the separation of the fibrils in each layer was made by two methods, alkali-acid and phosphoric acid treatments.

(1) Alkali-acid method.—After alternate alkali and acid treatment of fibers, faint stratifications became prominent and more and more striations became visible. In places at which the outer layer had been dissolved, there was extreme outward swelling of the inner layers. Such swelling made apparent the pronounced constrictions at the places where the outer layer was still intact, and also the less prominent constrictions on the fibers that had stray fibril bands wound about their inner layers at an angle of 45°. (Plate 7.) Continued treatment, with the aid of slight pressure on the cover glass, broke the constricting bands and the inner layers separated into fibril bundles. (Plate 8.) It was possible to separate these bundles into individual fibrils, which naturally have a diameter smaller than that of the bundles. Unfortunately, no satisfactory photomicrographs were obtained.
(2) Phosphoric acid method.—It is possible to control the reaction of the phosphoric acid method upon cellulose better than that of the alkali-acid method. Further, it is possible to reveal the minute structural arrangement of the layers in a manner that can not be accomplished by the alkali-acid method. Phosphoric acid seems to have a specific property for developing striations in wood fibers by loosening the layers and the fibrils before the skeleton structure dissolves.

Fibers treated by the phosphoric acid method show that solution of the outer layer at intervals is accompanied by extreme outward swelling of the inner layers; such fibers are constricted at the places where the outer layer is still intact. (Plate 9.) Extremely high magnification shows that the cell-wall layers separate in the transverse direction. (Plate 10.) Such photographs suggest that the orientation of the fibrils in the outer and in the inner layers is radically different.

Through slowly dissolving the outer layer, it became apparent that striation and separation of the fibrils precede the ultimate solution of the layer as an entirety. Since the fibrils in the outer layer are oriented at approximately 90° to the axis of the wood fiber (pl. 11), it is obvious that the fibers can not swell outwardly beyond the maximum limits that they assume in a water medium, unless the fibrils in the outer layer expand lengthwise or break. Such a structure also accounts for delignified fibers swelling inwardly when the outer layer is still intact. (Plate 18.)

On account of the convexity of the surface of the bead-like swellings in Plate 10, the minute structure of the inner layers is not visible. A swollen surface both flatter and longer must be examined if the tiny fibrils are to be seen in their proper orientation. By proper focusing of the microscope the orientation of the fibrils in the various layers can be studied. (Plate 12.) If the acid treatment is continued, the fibrils are loosened to a greater extent. (Plate 13.) By allowing the reaction to proceed still further, the individual fibrils are isolated. (Plate 14.)

If the minute structural arrangement of the outer layer is contrasted with that of the inner layers, it will be seen that wood fibers are designed to withstand both transverse and longitudinal stresses.

This separation of the cell wall of the wood fiber into fibrils confirms some findings of Waentig. ¹

Separation of the "Fusiform Bodies" in the Fibrils

A careful examination of the isolated fibrils from the inner layers of the cell wall under the high power of the microscope revealed that they were made up of units, the ends of which taper to sharp points; the units are held together by a slight overlapping of the pointed ends ² so as to form the

¹Waentig. Papierfabrikant 25, 115 (1927).
²These units differ from the dermatosomes described by Wiesner in his Elementar Structur, Alfred Holder, Wien, 1892, p. 162.
tiny, slender fibrils of a diameter practically uniform throughout their entire length. The long axis of each unit is parallel to the long axis of the fibril. The reaction of the phosphoric acid under proper conditions slowly opens up the natural planes of cleavage between the tiny units, which are of fusiform shape. (Plate 15.) Since, as far as the senior author knows, these are newly discovered units, which have been separated and photographed for the first time in the investigation now reported, they have been given the descriptive name "fusiform bodies."

III. Properties of Wood

Physical Properties

Some of the swelling properties of wood have been known for a long time through everyday experiences with the increased external dimensions that are produced in wood products when they are changed from dry and wet conditions by soaking in liquids, such as water, and solutions of acids and alkalis. Water swells dry wood approximately to its green volume. Strong acids and alkalis swell dry wood beyond its green volume.

With the aid of a microscope, it may be seen that the cell walls of wood also swell internally, and that the internal swelling, when an alkali or a strong acid is used as the reagent, may be sufficiently severe to fill the cell cavities. If a section of wood in its original state is swollen, the fibers retain their external shape, which in cross section shows definite angles. (Plates 16 and 17.)

From Plate 17 it is evident that a swollen condition retards the movement of impregnating liquors, prior to cooking, through the pits and the cell cavities. On the other hand, a condition such as that shown in this plate indicates that the fibers have been impregnated by diffusion of the alkali through the actual cell walls. The appearance of the section shown suggests that impregnation of wood chips with alkali liquors takes place principally by diffusion through the cell wall itself rather than through the various natural openings in the wall.

Acid solutions of ordinary concentration produce very little swelling of the cell wall beyond its green volume. The original sizes of the openings, therefore, remain practically unaltered. Such a condition suggests that impregnation of wood chips with an acid cooking liquor takes place principally through the pits and cell cavities.

If delignified fibers are treated with alkali or with strong acids, they, too, swell by crowding the cell wall into the lumen, but their cross-section area is changed from a polygonal to a circular shape. (Plate 18.) On reacting with the drastic reagents, the fibers become slightly plastic and they tend to assume shapes that have the minimum external surface in both the transverse and the longitudinal dimensions.
By alternately swelling wood fibers beyond their green volume and then shrinking them quickly, markings are developed that suggest the minute microstructure described in Part II of this paper.

Sodium hydroxide solution (15.6 percent concentration) was used for swelling the fibers shown in Plate 18. The appearance of those fibers gives an idea of the appearance of wood fibers in cross section after the alkaline treatment in the viscose process, and also in the alpha-cellulose determination. The numerous pit openings of the swollen cell walls, which do not appear in the photomicrograph, are changed to oblong slits that are practically closed.

Optical properties that suggest the structure of the cell wall about the bordered pits are manifested when the pits are examined in polarized light. It has been known for a long time that the secondary layer of the cell wall rotates the plane of polarized light and that the face of a bordered pit shows the commonly observed dark cross when it is placed between Nicol prisms that are crossed at 90°. The optical properties of the secondary layer are commonly considered to be due to an orderly arrangement of cellulose molecules in chains (Fagel's hypothesis); X-ray diagrams of Sponsler and Dore suggest that these chains are, in general, parallel to the longitudinal axis of the fiber. The fibrils in the secondary layer about the pit are bent around the opening, making their arrangement somewhat circular. The fibrils in the outside layer are present and are oriented at 90° to the fiber's axis. Bending around the opening they superpose a layer of concentric rings over the slight distortions in the circular structure of the inner layers. It is because of this involved total structure that the bordered pits exhibit a symmetrical dark cross through a complete rotation of the microscope stage. (Plates 21 and 22.)

IV. Significance of Fiber Microstructure to Chemical Pulping

Treatments that tend to separate wood fibers into fibrils and, in turn, tend to separate the fibrils into the fusiform bodies, are of interest to the paper maker. If a definite percentage of the fibers in a pulp are in a physical condition similar to the conditions in Plates 5, 6, 7, 11, and 12, it may aid immensely in the felting qualities of the pulp. On the other hand, if the reaction should be carried on sufficiently to put a large percentage of the fibers in the condition shown in Plate 13, the pulp might be useless for making paper.

Dippel, "Das Mikroskop," 2, p. 264.
Further, from the results already presented in this paper, it appears that pulps of different qualities and varying yields, produced by different cooking conditions, should show some difference in the microstructure of the fibers. Also, pulps cooked under the more drastic of the usual commercial conditions might respond more readily to the treatments previously described than pulps cooked under milder conditions. In addition, pulps beaten for varying periods might show a tendency to respond to the acid treatments more readily as the beating time increased.

The physical properties of the wood fibers determine to a large degree the ease with which phosphoric acid reacts with the cell wall. A slight rupture of the woody tissues, such as frayed ends, aids in starting the reaction. This fact may be demonstrated by treating short sections of fibers with the acid. With such a section, the solution of the outer layer begins at the frayed ends, and progresses toward the middle. By arresting the reaction before all the outer layer is removed, it is possible to obtain a residue that consists of loosened bundles tied with the spiral bands that form the remainder of the outer layer. (Plate 19.)

Optical Properties

Isolated "fusiform bodies," fibrils, and fibril bundles between Nicol prisms exhibit the same property as wood fibers, in that they transmit polarized light when they are oriented at an angle to the axes of the crossed prisms, but do not do so when oriented parallel to either of the axes. (Plate 20.)

The bead-like swelling shown in Plate 10, if placed between Nicol prisms, exhibit a "dark cross" when the axis of the fiber is parallel to the axis of one of the prisms. When the microscope stage is rotated 45°, the dark cross becomes slightly distorted. The bead-like swellings disappear, in general, as spherical bodies with a slight distortion in the direction of the fiber's axis. A cross section of such a body is composed of an approximate circle made up of concentric rings of visible fibrils, which are distorted in a manner similar to that of the swellings. With such a structure, the optical phenomenon of the swellings can be explained.

Some tests were made to determine whether such relationships could be shown. Preliminary experimental work was done on two series of sulfite cooks of white spruce and Eastern hemlock, respectively. Each series consisted of a pulp showing high strength and high yield in contrast to one showing low strength and low yield. These pulps were subjected to the Laboratory standard strength-development procedure by use of the ball mill.

The bleachabilities of the pulps were also determined. The essential data are recorded in Table 1.

The differences in the two spruce pulps are greater than those in the hemlock pulps. In maximum bursting strength, the second spruce pulp is 0.55 point higher than the first; the yield is 8.6 percent higher. The maximum bursting strength of the second hemlock pulp is 0.21 point higher, while the yield is 2.9 percent higher than the first.
The spruce pulps were stained with Bismarck brown, air dried, treated with a solution of phosphoric acid, heated for 4 minutes at 60°C, and cooled. Slides were then made and photomicrographs taken. The hemlock pulps were treated in the same way except that they were heated for 3 minutes instead of 4. In addition, 4 of the initial samples were treated with a slightly stronger acid, at room temperature, to show more clearly the differences in the fibers before milling. The photomicrographs of these fibers appear in Plates 26, 27, 28, and 29.

Untreated fibers, both milled and not milled, of Pulp 3336-I are shown in Plates 23, 24, and 25. Fibers of the 4 pulps, milled and not milled and treated with phosphoric acid, are shown in Plates 30 to 49, inclusive.

The results show that pulps prepared under mild cooking conditions are less susceptible to the attack of phosphoric acid than those prepared by drastic cooking conditions. Differences in the susceptibility to the attack appear when plates prepared from the two unmilled spruce pulps and the two unmilled hemlock pulps are compared. For example, contrast Plates 26 and 27; 28 and 29; 30 and 31; and 40 and 41.

The results further show that the binding material and the helical winding of fibrils forming the outer layer of the fiber have been partially or wholly dissolved, allowing the inner portion of the fiber to expand. In some cases, the inner fibrils may be seen slightly separated, forming an extended helix.

The effect of the acid is also noticeable as the milling progresses. When the outer portion of the fiber has been ruptured mechanically, the inner part is attacked by the acid at the rupture and swelling takes place. In the refined pulps, also, the stronger pulp shows, in general, less effect of the acid.

By treating fibers from various sulfite pulps with phosphoric acid and examining them under the microscope, it is possible to observe differences in the quality of the pulp. Just how fine a distinction can be made remains to be worked out. It may be possible to evaluate the pulp numerically by using phosphoric acid solutions of different concentrations, noting the strength at which the pulp is attacked.

Although a rapid qualitative test may be developed from the method, more important is the information it gives on the fundamental relation of the microstructure of the fiber to different cooking conditions, yields, and strength properties.

**Summary**

The location in the wood of the two forms of lignin is described. The two forms are shown in photomicrographs.
The possibility of obtaining a yield of 60 percent of lignin-free fibers for paper material is suggested.

The cell wall of wood fibers is composed of several layers, which can be separated by chemical means.

The layers in the cell wall of a wood fiber can be separated into fibrils by chemical means. The fibrils in the outer layer are oriented at approximately right angles to the fiber's axis, while those in the remaining layers are from 0° to 30° thereto.

The fibrils can be separated into regularly shaped "fusiform bodies" with optical properties similar to those of the fibrils.

When either lignified or delignified wood fibers are treated with swelling reagents, the fiber walls thicken outwardly and also inwardly. The polygonal shape of the cross section of delignified fibers is unaltered, but the cross section of delignified fibers is limited by the outer layer of fibrils, which are oriented at 90° to the fiber's axis.

The optical phenomenon, when bordered pits are observed between Nicol prisms, is explained on the basis of the ring-like structural arrangement of the cellulosic material of the cell wall.

The effect of phosphoric acid on pulps obtained from two series of cooks of spruce and hemlock is described. Its effect is more severe on the pulps from the more drastic cooks, both in the raw and refined condition. The effect increases as the period of milling increases.

The swelling and dissecting action of the phosphoric acid on the fibers is explained on the assumption that part of the outer layer and more of the building material between the fibrils in the various layers of the cell wall are removed by the more drastic cooking conditions. Milling has the mechanical effect of progressively rupturing the outer layer of fibrils and of loosening the inner fibrils. Such an effect permits a more rapid attack by the phosphoric acid.

It is suggested that the phosphoric acid treatment developed in the study discussed in this paper may be further standardized to provide a new method for the evaluation of pulp quality.
Legends for Plates on Following Pages

Plate 1.--The middle-lamella lignin and the cell-wall lignin of red alder.

Plate 2.--Another transverse section of red alder which also shows the two forms of lignin. Some of the middle-lamella lignin is slightly out of focus because of making visible larger quantities of the cell-wall lignin.

Plate 3.--A cross section cut from a block of yellow pine middle-lamella lignin which was first impregnated with paraffin. The rough appearance of the paraffin is due to a slight melting and resolidifying of the paraffin on the surface.

Plate 4.--A cross section of yellow pine middle-lamella lignin similar to that of Plate 3. The section shows that the cellulose and the cell-wall lignin can be removed with very little injury to the middle lamella.

Plate 5.--Shows a separation of the delignified cell wall of elm into four distinct layers by means of a 58 percent solution of phosphoric acid.

Plate 6.--Short sections of delignified elm fibers in which the cell wall layers have been separated and slipped endwise.

Plate 7.--Delignified elm fiber which has received alternated treatments with alkali and acid. The outer layer has been removed from a large portion of the fiber. A helical band at approximately 45° keeps the cell wall from rupturing.

Plate 8.--Delignified elm fiber treated alternately with alkali and acid. Shows a separation of the cell wall into fibril bundles.

Plate 9.--Shows the transverse swelling of the inner layers of elm in places at which the outer layer has been dissolved.

Plate 10.--Shows three separate layers of a delignified elm fiber at the constricted places and the transverse swelling of two inner layers.

Plate 11.--Section of the outside layer, showing helical striations extending around the fiber at right angles to the fiber's axis.

Plate 12.--Shows minute fibrils of the inner cell wall layers. The fibrils have been loosened by phosphoric acid treatment.

Plate 13.--Shows appearance of a fiber after the fibrils have been well loosened.

Plate 14.--Shows a more nearly complete separation of the cell wall layers into fibrils.
Plate 15.--Shows how the "fusiform" bodies in the fibrils can be separated.

Plate 16.--Cross section of Western yellow pine soaked in water. Note the general rectangular shape of the cells.

Plate 17.--Cross section of Western yellow pine which has been swollen with 15 percent alkali. Note the puffy appearance of the surface, the thickening of the cell wall, and the general rectangular shape of the cells.

Plate 18.--Cross section of Western yellow pine which has been delignified so as to obtain isolated cells. On treatment with 15 percent alkali the isolated cells assume a cylindrical shape with the lumen closed.

Plate 19.--Short sections of delignified elm fibers, showing the bundle-like residue obtained when the dissolving action of phosphoric acid is arrested before the outer layer is removed completely.

Plate 20.--Shows that the fibrils between Nicol prisms are luminous when oriented at an angle to the axis of either Nicol prism, but dark when parallel thereto.

Plate 21.--Radial face of Western yellow pine. The fibers are oriented parallel to the axis of one Nicol prism. The fibers are dark; lines of the "dark cross" are parallel to the corresponding axes of the Nicol prisms.

Plate 22.--Radial face of Western yellow pine between Nicol prisms. The fibers are oriented at approximately 45° to the axes of the Nicol prisms. The fibers are luminous; lines of the "dark cross" are parallel to the corresponding axes of the Nicol prism.

Plate 23.--Spruce sulphite pulp. Pulp 3236; high yield; not milled.

Plate 24.--Spruce sulphite pulp. Pulp 3236; high yield; milled 40 minutes.

Plate 25.--Spruce sulphite pulp. Pulp 3236; high yield; milled 80 minutes.

Plate 26.--Spruce sulphite pulp. Pulp 3236; high yield; not milled; treated with phosphoric acid.

Plate 27.--Spruce sulphite pulp. Pulp 3226; low yield; not milled; treated with phosphoric acid.

Plate 28.--Hemlock sulphite pulp. Pulp 3314; high yield; not milled; treated with phosphoric acid.

Plate 29.--Hemlock sulphite pulp. Pulp 3314; low yield; not milled, treated with phosphoric acid.

Plate 30.--Spruce sulphite pulp. Pulp 3336; high yield, not milled; treated with phosphoric acid.
Plate 31.—Spruce sulphite pulp. Pulp 3226; low yield; not milled; treated with phosphoric acid.

Plate 32.—Spruce sulphite pulp. Pulp 3236; high yield; milled 20 minutes; treated with phosphoric acid.

Plate 33.—Spruce sulphite pulp. Pulp 3226; low yield; milled 20 minutes; treated with phosphoric acid.

Plate 34.—Spruce sulphite pulp. Pulp 3236; high yield; milled 40 minutes; treated with phosphoric acid.

Plate 35.—Spruce sulphite pulp. Pulp 3226; low yield; milled 40 minutes; treated with phosphoric acid.

Plate 36.—Spruce sulphite pulp. Pulp 3236; high yield; milled 60 minutes; treated with phosphoric acid.

Plate 37.—Spruce sulphite pulp. Pulp 3226; low yield; milled 60 minutes; treated with phosphoric acid.

Plate 38.—Spruce sulphite pulp. Pulp 3236; high yield; milled 80 minutes; treated with phosphoric acid.

Plate 39.—Spruce sulphite pulp. Pulp 3226; low yield; milled 80 minutes; treated with phosphoric acid.

Plate 40.—Hemlock sulphite pulp. Pulp 3317; high yield; not milled; treated with phosphoric acid.

Plate 41.—Hemlock sulphite pulp. Pulp 3314; low yield; not milled; treated with phosphoric acid.

Plate 42.—Hemlock sulphite pulp. Pulp 3317; high yield; milled 20 minutes; treated with phosphoric acid.

Plate 43.—Hemlock sulphite pulp. Pulp 3314; low yield; milled 20 minutes; treated with phosphoric acid.

Plate 44.—Hemlock sulphite pulp. Pulp 3317; high yield; milled 40 minutes; treated with phosphoric acid.

Plate 45.—Hemlock sulphite pulp. Pulp 3314; low yield; milled 40 minutes; treated with phosphoric acid.

Plate 46.—Hemlock sulphite pulp. Pulp 3317; high yield; milled 60 minutes; treated with phosphoric acid.

Plate 47.—Hemlock sulphite pulp. Pulp 3314; low yield, milled 60 minutes; treated with phosphoric acid.

Plate 48.—Hemlock sulphite pulp. Pulp 3317; high yield; milled 80 minutes; treated with phosphoric acid.

Plate 49.—Hemlock sulphite pulp. Pulp 3314; low yield; milled 80 minutes; treated with phosphoric acid.
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