

UTILIZATION OF WHITE-POCKET DOUGLAS-FIR: PULPING AND CHEMICAL CONVERSION

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UTILIZATION OF WHITE-POCKET DOUGLAS-FIR;

PULPING AND CHEMICAL CONVERSION

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Summary

White-pocket Douglas-fir was tested for its suitability for the production of kraft, semichemical, and rayon grades of pulp and for the production of wood sugar. The experiments showed that a considerable amount of white pocket can be present without loss in the yield of kraft pulp and without a great loss in the strength of the pulp. Strength losses can be minimized by mixing the white-pocket Douglas-fir with the sound wood of normally associated softwood species.

White pocket showed no effect on the yield of semichemical pulps made under the mildest conditions and produced in the highest yields, but when stronger conditions were used to give lower yields, the wood with white pocket gave less pulp than did the sound wood.

Corrugating and liner boards of satisfactory quality were made from both the neutral sulfite and the sulfate types of semichemical pulp, but the latter proved to be more economical from the standpoint of chemical requirements. Douglas-fir in the intermediate stage of white pocket was made into textile grade rayon pulp by the prehydrolysis-sulfate process.

No significant difference was noticed between the behavior of Douglas-fir with or without white pocket in the production of wood sugar, except that some modification of procedure would be indicated if advanced white pocket were present in large amounts. The yield of sugar from the white-pocket material was comparable to that from the normal wood.

¹Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

Introduction

In the Douglas-fir region of the Northwest, a large amount of material is left on the ground after logging. These defective logs, broken chunks, and unmerchantable tops are left in the woods because there is no market at present for this type of wood. Much of the defective material is caused by an organism, Fomes pini, that attacks the heartwood of Douglas-fir and leaves it in a pitted or white-pocket condition from which it derives its common name of "white pocket."

Losses from Fomes pini in old-growth vary by stands and range from less than 5 percent to more than 60 percent of the gross volume in board feet. The higher losses are found in the Douglas-fir stands in the southern half of Oregon and northwestern California.² Profitable utilization of these waste materials is necessary to attain maximum returns from extensive forest stands of this type.

The Forest Products Laboratory has conducted experiments to test the possibility of using white-pocket wood for lumber, veneer, pulp, and for other mechanical uses. This report summarizes experiments to determine the suitability of white-pocket old-growth Douglas-fir for the production of kraft, semichemical, and rayon grades of pulp, and for conversion into wood sugar by hydrolysis.

Production of Paper-Grade Sulfate Pulp

Comparison of Sound and Decayed Woods

A description of the six samples of Douglas-fir used in the sulfate pulping tests for the production of strong (kraft) paper-grade pulp is given in table 1. They consisted of sound material from whole logs, sound material adjacent to white-pocket material, and white-pocket material in various stages of development ranging from an incipient stage to an extremely advanced stage.

The density values are important factors in determining the yield of pulp obtainable from a unit volume of wood and the weight of chips that can be charged to a digester. The density values of the samples containing the incipient, firm, and advanced stages were similar to those of the sound wood (lot 1). Chemical analysis indicated that the sound wood surrounding white-pocket areas (lot 1A) and the woods containing incipient, firm, and advanced white pocket all had essentially the same chemical composition, but they all had definitely lower holocellulose and pentosan content and a little lower alpha-cellulose content than did the sound wood of lot 1. Judged by the chemical composition and the density values of these samples, the extent

²Boyce, J. S., and Wagg, J. W. Conk Rot of Old-Growth Douglas-fir in Western Oregon. Oregon Forest Products Laboratory Bull. 4, June 1953.

of the deterioration was less than that indicated by their visual appearance and, fortunately, less than that required to interfere with normal reactions with sulfate cooking liquors.

The sample with extreme white pocket (lot 5) was honeycombed and contained a fairly uniform distribution of the white pocket. Its low density of 14.7 pounds per cubic foot was about one-half that of the other samples tested. Chemical analysis showed it to have a higher alkali solubility and lower alpha cellulose and lignin content than did the sound wood. In general, the chemical analysis indicated that the organism destroyed all constituents with about equal intensity.

Sulfate Pulping for Paper-Grade Pulp

The pulping conditions were established by a series of experiments on the sound material from lot 1. The optimum conditions arrived at were:

Chemicals charged per 100 pounds of moisture-free wood:	
Sodium hydroxide and sodium sulfide.....	20.0 pounds
Calculated as sodium oxide.....	15.6 pounds
Initial chemical concentration.....	39.1 grams per liter
Sulfidity (based on active alkali).....	30.0 percent
Liquor-wood ratio.....	4.0
Maximum temperature.....	170° C.
From room temperature to maximum temperature.....	1.5 hours
At maximum temperature.....	1.5 hours

Under these conditions, the amount of chemical consumed for all samples was about 85 percent of the total charged. The amounts charged and consumed are comparable with general mill practice for the production of kraft pulp.

The results of pulping the white-pocket fir indicated that all lots were as satisfactorily reduced as the sound wood of lot 1. Except for the wood with extreme white pocket (lot 5), the yields of screened pulp (including the sound wood samples) were within a narrow range of 43.5 to 45.1 percent and not in order with the observed degree of white pocket.

The yield of screened pulp from the wood with extreme white pocket was 41.8 percent on a weight basis, which was only a little lower than that from the sound wood. Because of the low density of this sample, however, the yield was 50 percent less than that from the sound wood on a volume basis. This type of material would be difficult to handle in a mill wood room because of its friability.

The moisture content of the freshly chipped wood ready for pulping was much lower for the several lots of wood containing white pocket (22-23 percent) than for the two lots of sound material (33-38 percent). Presuming that this is a natural situation, and since dry chips pack more loosely than moist chips, it appears that the only changes necessary for satisfactory pulping of the white-pocket material would be in the adjustment of the chemical

charge to compensate for the lower weight of wood substance in the digester. This adjustment is necessary so as not to overcook the white-pocket material to the detriment of pulp yield and quality.

The pulps from the white-pocket samples appeared to bleach easier than those from the sound woods.

The results indicate that the following strength properties might be expected in kraft pulps made from the various materials:

(a) Pulp made from sound old-growth Douglas-fir wood will probably be slightly lower in bursting strength and significantly higher in tearing strength than southern pine sulfate pulp and probably could be expected to be used for purposes now served by southern pine pulp except where maximum bursting strength or a fine-fibered pulp are needed.

(b) The pulp obtained from sound wood surrounding white-pocket areas has about the same bursting strength as that from wood containing incipient white pocket. Its tearing strength is about equal to that normally expected from sound Douglas-fir.

(c) Sulfate pulps made from Douglas-fir wood containing white-pocket will be lower in bursting strength than pulps made from sound wood in proportion to the observed amount of white pocket. The bursting strength of the pulp made from wood containing advanced white pocket would probably be about 75 percent of that of southern pine pulp.

(d) The tearing strength of the pulps from the white-pocket woods (except for lot 5) may be higher than that of pulp from the sound Douglas-fir, while the tearing strength of lot 5 may be a little lower than that of southern pine kraft.

Values for the relative bursting and tearing strength of the various Douglas-fir sulfate pulps and southern pine kraft are given in table 2.

It is evident that a considerable amount of white pocket can be present in Douglas-fir without loss in yield of pulp and without a great loss in strength. The lowered strength can be compensated for by mixing pulp from the white-pocket material with pulp from sound wood from other softwoods normally associated with Douglas-fir. For example, the following mixture, which simulates that naturally occurring in some stands, was found to give a yield of about 46 percent of screened pulp and to have good bursting strength and tearing strength (see table 2):

	<u>By weight</u>
Douglas-fir from lots 1 and 1A.....	25 percent of each
Douglas-fir from lot 2.....	10 percent
Douglas-fir from lots 3 and 4.....	5 percent of each
Mountain hemlock, noble fir and logpole pine.....	10 percent of each

Production of Semicheical Pulps
and Liner and Corrugating Boards

Semicheical pulps made by partial chemical pulping followed by mechanical fiberizing are produced commercially in yields of 65 to 80 percent in contrast with the range of 45 to 50 percent for chemical pulps. The purpose of this process is to utilize wood as efficiently as possible and to take advantage of certain properties inherent in this type of pulp. Semicheical pulps are now made mostly from hardwoods for use in container board, particularly corrugating medium, and, after bleaching, in fine papers. There is a small amount of softwood semicheical pulp made, and experimental investigations have shown promising indications that the use of softwoods for this purpose can be expanded. The semicheical process, therefore, was applied to the Douglas-fir of lots 1, 3, and 4, respectively (see table 1). Both the neutral sulfite and sulfate semicheical processes were used, and selected pulps were made into standard-weight corrugating and liner boards, which are the components of the widely used corrugated fiberboard.

Neutral Sulfite Semicheical Pulps and Boards

Douglas-fir was readily pulped with sodium sulfite solution buffered with sodium bicarbonate as the pulping reagent. Pulps were made in yields of 53 to 77 percent. The amount of sodium sulfite, based on the wood, varied from about 28 to 17 percent, and the time at the cooking temperature of 175° C. varied from about 7 hours to 1 hour, respectively, for this yield range. The amount of sodium bicarbonate used to maintain a slightly alkaline condition was about 5 percent of the wood. The pulps made under the mildest conditions and produced in the highest yields showed no effect of white pocket on yield, but with stronger conditions used to give lower yields the wood with white pocket gave about 5 percent less pulp than did the sound wood. The pulping of Douglas-fir with the neutral sulfite reagent required large amounts of cooking chemical and fiberizing energy. For example, 1 ton of pulp (air-dry basis) made in a yield of 76 percent had a chemical requirement of 460 pounds of sodium carbonate and 105 pounds of sulfur and an energy requirement of about 40 horsepower-days per ton of air-dry pulp. (This is in contrast with average requirements of about 250 pounds of soda ash, 60 pounds of sulfur, and 15 horsepower-days per ton of hardwood semicheical pulp.) The chemical requirements increased and the energy requirement decreased with decrease in yield. It would probably be uneconomical to make pulp in yields below 70 percent, if the chemicals were not recovered.

Pulp strength was not affected by white pocket regardless of yield; in fact, the pulps from the white-pocket materials actually were stronger in these few experiments than those from the sound wood, although this would not be expected in the average from a large number of tests or in mill production. The pulps made in yields near 75 percent had bursting- and tearing-strength values about one-half those of the kraft pulps from the sound Douglas-fir. These pulps were also much lower in folding endurance. Pulp strength increased with decrease in yield to the extent that pulps made in yields

of 53 to 57 percent had about 80 percent of the strength of the kraft pulp from sound wood as shown in table 2.

Corrugating boards were made from pulps produced in yields near 75 percent from the wood of lots 1 and 3. These boards were equal in properties, such as bursting, tearing, and folding strength, to the strongest commercial corrugating boards, but were somewhat lower in stiffness as measured by flat-crush resistance after corrugating. (The latter is probably the most important criterion of the quality of corrugating board.) As far as the usual strength properties are concerned, Douglas-fir neutral sulfite pulps could be made in yields as high as 80 percent, but they apparently would need to be blended with other pulps that would impart stiffness to the corrugating board, or a stiffening agent would need to be added.

Liner boards were made from all of the semichemical pulps from the sound woods and from those containing white pocket. Although the boards from the lot 3 wood were somewhat lower in strength than the others, it was not believed that the condition of the wood had a serious effect on the quality of the liner boards within the range of conditions studied. The boards from the pulps made in yields near 75 percent approached the bursting-strength requirement for a test liner, which is a minimum of 100 pounds per square inch, Mullen. They had a high resistance to tearing, but were somewhat low in folding endurance, a possible indication of substandard bending properties. The boards made from the lower yield pulps generally met the test liner specification for bursting strength and showed promise for use for this purpose. The highest yield pulps would probably have considerable use in boards that do not require the highest strength properties, and if blended with strong kraft pulps from Douglas-fir or other softwoods, they might be used in the stronger boards.

Sulfate Semichemical Pulps and Boards

Douglas-fir was also readily made into semichemical pulps in yields of 55 to 75 percent using a sulfate pulping liquor. This range of yields was obtained by reducing the total chemical used from 15 to 8.75 percent and the cooking time at 170° C. from 1.5 to 0 hour in comparison with 20 percent chemical and 1.5 hours at 170° C. for the kraft pulps. Thus, the sulfate semichemical pulps, in contrast with the neutral sulfite type, had low, economical chemical requirements. At the highest yields, the presence of white pocket had no effect on pulp yield, but at the lowest yields about 5 percent less pulp was obtained under the same pulping conditions from the wood with advanced white pocket than from the sound wood. The pulps made in the lowest yields from the wood with advanced white pocket were also weaker in bursting strength by 25 percent than the corresponding pulp from the sound wood. Pulp strength decreased in proceeding from the kraft pulp through to the pulps made in the highest yield of 75 percent. At this high yield, the pulps had roughly 60 percent of the bursting and tearing strength of the kraft pulps and only about 10 percent of the folding endurance. (See table 2 for relative strength of the kraft pulp.)

Corrugating boards were made from selected pulps covering the range of yield and wood conditions. The boards made from the highest yield pulps, although unaffected by the white pocket, tended to be low in strength, except in tearing resistance and in crush resistance of the corrugated board. The results of these few tests were not promising for the commercial use of high percentages of the highest yield sulfate semichemical pulps in corrugating board. The corrugating boards made from the lower yield sulfate semichemical pulps had good strength properties except for stiffness, which was lower than desirable in comparison with the highest quality commercial corrugating board. As with the neutral sulfite boards, some means of stiffening would appear to be needed before these boards could compete with boards made from conventional pulps.

The liner boards made from the strongest sulfate pulps, that is those made in the high yield of 55 percent, had bursting-strength values of 75 and 90 pounds per square inch in boards made from wood with advanced white pocket and from sound woods, respectively. Other strength properties were also lower in boards made from wood containing white pocket. None met the bursting strength requirement of 100 pounds per square inch for test liner board even though made in the relatively heavy weight of 47 pounds per 1,000 square feet, but they would be acceptable in other strength properties. The liner boards made from pulps in the intermediate yield class of 63 percent were even lower in bursting strength. These pulps probably would have limited possibilities if used independently of other stronger pulps. The lower-yield sulfate semichemical pulps would be expected to be useful in container liner boards not having the highest strength requirements and they undoubtedly could be blended with strong kraft pulps for producing higher quality container boards and other board products, as mentioned for the neutral sulfite counterparts. Depending on quality requirements, probably up to 50 percent of the wood with advanced white pocket could be used in many high quality board products.

Production of Rayon-Grade Pulp

Wood Used

White-pocket Douglas-fir was tested as a source for viscose rayon pulp. The wood had an intermediate stage of white pocket, as described by lot 3 in table 1.

In chemical composition, the wood was lower in alpha cellulose, the desirable component for rayon-grade pulp, than sound Douglas-fir. It compared favorably to southern pine, which is used in the commercial production of this kind of pulp. A notable characteristic of white-pocket Douglas-fir is the low content of pentosan, an unwanted material in rayon-grade pulp. In the sample of wood used in this work, the pentosan content was somewhat less than half the amount found in southern pine. Test values for the wood are given in table 3.

Pulping and Purification Treatments

Two purified pulps were prepared from the white-pocket Douglas-fir wood using different pulping and purification treatments that were thought might produce different physical characteristics in the cellulose as well as differences in its chemical nature.

The wood was pulped by the prehydrolysis-sulfate process in which the chips are cooked first with water or steam and then pulped by the usual sulfate process. In the prehydrolysis step, acidic conditions are developed that remove some of the unwanted hemicellulose (including some of the pentosans) and apparently renders most of the remaining portion susceptible to removal in the sulfate pulping stage. Sulfate pulping without prehydrolysis does not remove much of the pentosans, and it does not consistently produce pulps that are satisfactorily reactive (that is, will form easily filtrable viscose solutions) in the conversion to viscose rayon.

By using different amounts of chemical in the pulping stage, two unbleached pulps were obtained that differed appreciably in disperse viscosity but were nearly alike in other characteristics as shown in table 3. The first of these, digestion 4038, was purified by sequential treatments with chlorine, sodium hypochlorite, and sodium hydroxide solutions. Conditions in these treatments were selected so that the disperse viscosity was reduced from 17.2 centipoises to the desired low level of 6.4 centipoises. More than 90 percent of the decrease was achieved in the first two stages so as to permit removal of undesired degraded products by the alkaline-extraction treatment.

The second unbleached pulp, digestion 4042, was purified by sequential treatments with chlorine, sodium hydroxide solution, and sodium hypochlorite. These treatments decreased the disperse viscosity from 10.1 centipoises to about the same level as that of the first pulp, and again the conditions were such that no measurable decrease in viscosity occurred after the alkaline-extraction treatment.

Evaluation of the Purified Pulps

A comparison of the composition of the 2 experimental pulps and 2 commercial viscose pulps of tire-cord grade is given in table 3. As shown by these data, the Douglas-fir pulps were equal in composition and in some respects superior to domestic commercial viscose pulps used in tire-cord manufacture.

Viscose rayon yarns and tire cords were made from the experimental pulps in an industrial laboratory. The better tire cord, from the standpoint of fatigue resistance, was obtained from the pulp (digestion 4038) in which the viscosity was lowered more during its purification than the other. This difference in fatigue resistance is evidence of differences in the physical characteristics of the cellulose obtained by the two methods of preparation. The fatigue resistance was about 20 percent below the resistance of tire-cord rayon now made from commercial prehydrolysis-sulfate pulp.

However, the rayon yarns made from both pulps were adequate in strength for textile grade. On the basis of chemical composition, they appeared to be suitable for the explosives grade of cellulose nitrate.

Wood Sugar from White-Pocket Douglas-Fir

Studies on the chemical conversion of white-pocket Douglas-fir were limited to the evaluation of its sugar-producing properties. To determine the potential sugar content of the material, a quantitative saccharification procedure was used, and the yield of sugar was determined by standard analytical methods. The fermentability of the sugars produced was also determined. The potential sugar values were determined for Douglas-fir containing no white pocket, light white pocket, and extensive white pocket (similar to that of lot 5). The data based on bark-free wood are listed in table 4. Even though nearly half of the wood substance has been destroyed in material containing extensive white pocket, the potential sugar content based on the dry weight has not been seriously changed. The total reducing sugar value has been affected somewhat more than the fermentable sugar value, indicating a preferential removal of the more easily hydrolyzable hemicellulose.

No significant difference was noticed between the behavior of Douglas-fir with or without white pocket in the wood-hydrolysis pilot plant. If the extensive white pocket material had been segregated and hydrolyzed separately, however, it would be expected that this material would have behaved more like sawdust than chips, and lower packing pressures would have had to be used during loading to prevent overdensification of the bed. On passing through the hog, the extensive white-pocket material broke up considerably more than did the sound Douglas-fir. It was found that over 70 percent of hogged light white-pocket Douglas-fir went through a 1/4-inch mesh, while only 30 percent of sound material that had been similarly hogged went through the 1/4-inch mesh screen.

In all of the hydrolysis runs on white-pocket Douglas-fir, the material used represented a cross section of the white-pocket material received at this Laboratory. Because of its similarity to Douglas-fir without white pocket, Douglas-fir with white pocket was used as charge stock for much of our wood hydrolysis development work on this species. An operating procedure that resulted from this research was used on 11 consecutive white-pocket hydrolysis runs. The average yield for these runs was 39 percent, and the average sugar concentration of the composite hydrolyzates was 5.4 percent. These figures are comparable to the values that can be obtained by the same procedure from Douglas-fir of low bark content. To obtain these yields the charge was first steamed to 293° F., and dilute sulfuric acid at 293° F. was added at such a rate as to give a water-to-wood ratio of 2.0 at the end of 30 minutes. The water temperature was then raised to 343° F., and the water-to-wood ratio was increased to 2.5 in 15 minutes. At this ratio, the charge was substantially covered with a 0.7 percent sulfuric acid solution. Sugar solution was then started off at such a rate as to maintain a high liquor level in the vessel, and 0.6 percent acid liquor was pumped in at 10 percent of the weight of dry charge per minute. The temperature was increased at a constant rate to 365° F. in 15 minutes. These operating conditions consume a quantity of acid equal to 6 percent of the charge and require a total pumping time of less than 2-1/2 hours to produce the sugar yield of 39 percent and the concentration of 5.4 percent.

Table 1.--Density and chemical analyses of Douglas-fir logging waste with and without white pocket

Lot No. 1:	Density	Cellulose	Lignin	Pentosans	Solubility in:	Ash
	(moisture-free)	Alpha in		In holo-:	Ethyl:	l per-:
	weight and	Holo-:	Total:	cellulose:	hol:	ether:
	green volume):	cellulose:	hol:	cellulose:	hol:	ether:
				benzene:	NaOH:	
1:	27.4	50.4	27.2	6.8	4.4	1.2
1A:	28.6	48.0	27.6	4.8	4.4	2.0
2:	27.1	47.6	27.8	5.0	4.2	2.4
3:	28.6	48.4	28.5	4.8	3.6	1.4
4:	26.4	49.0	27.7	4.9	3.9	1.4
5:	14.7	43.1	24.8	6.1	7.1	.8

1 Lot 1 consisted of logs considered to be typical of sound, clear wood from small depressed trees and from tops of old-growth trees. Lot 1A was wood taken from the rim surrounding the white-pocket areas of the logs from which lots 2, 3, and 4 were obtained. The material in the rim areas was considered to be sound wood but because it was closely associated with white pocket, the rim-area wood was pulped separately from the sound wood of lot 1. Lot 2 consisted of wood taken from central portions stained with incipient white pocket. Lot 3 consisted of firm wood taken from the central portions containing intermediate white pocket. This type of material has been used for the production of No. 4 Common Lumber. Lot 4 was wood taken from the central parts of logs with advanced white pocket. Lot 5 was a sample containing extremely advanced white pocket, of honeycomb appearance, and much lower in density than the other lots.

2 Results based on holocellulose; all other values are based on moisture-free wood.

Table 2.--Comparison of strength of the experimental sulfate pulps
with commercial southern pine sulfate pulps

Kind of sulfate pulp	: Bursting : : strength :	: Tearing : : strength :
	: <u>Percent</u> :	: <u>Percent</u> :
Southern pine.....	: 100 :	: 100 :
Douglas-fir:	: :	: :
Sound wood from lot 1.....	: 95 :	: 120 :
Wood surrounding white-pocket areas, lot 1A....	: 85 :	: 165 :
Stained wood with incipient white pocket, lot 2:	: 85 :	: 180 :
Wood with firm or intermediate white pocket, lot 3.....	: 80 :	: 155 :
Wood with advanced white pocket, lot 4.....	: 75 :	: 175 :
Wood with extremely advanced white pocket, lot 5.....	: 75 :	: 90 :
Mixture of species.....	: 105 :	: 125 :

Table 3.--Certain test results on white-pocket Douglas-fir viscose pulps of rayon grade

Characteristics measured	White-pocket Douglas-fir		Commercial viscose pulps of tire-cord grade
	Wood ¹	Prehydrolysis sulfate pulps	
Yield:			
Basis wood.....percent	34.3	32.4	30.9
Basis unbleached pulp.....percent		94.5	93.0
Permanganate number.....	14.8		9.6
Dispersed viscosity ²percent	17.2	6.4	10.2
Lignin.....percent	28.3		3.0
Alpha cellulose:			
Basis wood.....percent	31.5	31.0	30.6
Basis pulp.....percent	91.6	95.6	92.8
Beta cellulose.....percent	1.5	2.0	1.4
Gamma cellulose.....percent	2.9	2.0	2.1
Total pentosans.....percent	4.8	.7	1.0
Solubility in:			
Ether.....percent	1.8	.3	.2
Sodium hydroxide:			
7.14 percent solution.....percent		5.6	6.4
1.0 percent solution.....percent	18.1		
Ash.....percent	.05	.30	.06
Iron.....parts per million	15	33	13
			14
			.11
			.13
			.13
			.05

¹Wood containing intermediate white pocket.

²Viscosity of a 0.5 percent dispersion of the pulp in cupriethylenediamine.

Table 4.--Potential sugar in samples of Douglas-fir wood

	: Potential ¹ : reducing : sugar	: Potential ¹ : fermentable : sugar
	: <u>Percent</u>	: <u>Percent</u>
Normal.....	: 67	: 58
White pocket, light.....	: 66	: 57
White pocket, extensive..	: 62	: 56

¹Based on oven-dry method.