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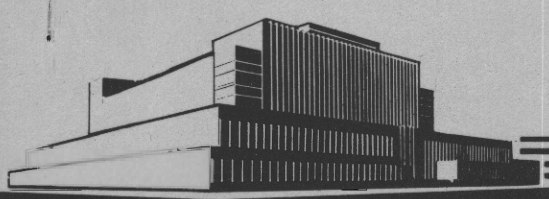

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FOREST SERVICE
In Cooperation with the University of Wisconsin

SULFITE PULPING OF ENGELMANN SPRUCE[1]

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Summary

Sulfite pulps with permanganate numbers ranging from 6 to 15 were made from three green-cut Engelmann spruce trees (Picea engelmannii) under conventional pulping conditions. The wood cooked like eastern spruce, and the pulps showed normal variations with the degree of cooking. Increasing the maximum temperature, acid concentration, and pressure reduced the cooking time for a permanganate number of 8.7 from 9.25 hours to 6.75 with a slight loss in yield. The Engelmann spruce wood gave higher yields than a sample of white spruce (Picea glauca) similarly cooked to equal bleachability. The strength of the Engelmann spruce pulps was greater than that of pulps from this particular sample of white spruce, but was not appreciably greater than that generally obtained with white spruce.

Pulps from beetle-killed wood had approximately the same quality as those from green-cut wood except for a moderately lower resistance to tearing. Compared with a sample of spruce from Wisconsin, the insect-killed material gave a slightly higher yield of pulp that was of comparable quality except that its tear strength was 12 to 15 percent lower.

Introduction

In trials made by the Forest Service more than a quarter century ago, sulfite pulps from Engelmann spruce were shown to equal those from white spruce in nearly every respect.[2] Subsequent work, which is reported here, provides information on pulps that have been evaluated by more modern methods.

This species grows in the Rocky Mountains from Canada to Mexico. Because of the long distance from markets and established pulping centers, comparatively little

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[1] Original report published July 1942.

[2] Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

[3] Wells, S. D. and Rue, J. D. U. S. Dept. Agric. Bull. 1485, 1927. Out of print.

Engelmann spruce has been used by the paper industry. A small amount has been used for sulfite pulping in the Northwest and substantial amounts have been shipped to Wisconsin mills.

A sharp increase in beetle infestation in recent years, which resulted in some millions of cords of dead wood, has underlined the need for more intensive utilization of this species. Since insect-killed trees may stand for as long as 20 years without serious deterioration, much of this material could be salvaged.

Included in this report are (1) the results of cooking green-cut wood over a range of from 6 to 15 in permanganate number, (2) the results of using a shortened cooking schedule, (3) a comparison of the Engelmann spruce pulp with that made from white spruce under similar cooking conditions, and (4) a comparison of pulps made from insect-killed and green-cut woods.

Experimental

The sample of green-cut Engelmann spruce consisted of three trees, sawed to 4-foot lengths, furnished by the Northern Rocky Mountain Forest and Range Experiment Station. Eleven bolts that were representative of the shipment were reduced to standard 5/8-inch chips and screened for pulping. White spruce from Michigan was similarly evaluated and pulped for comparison. The beetle-killed material consisted of a 150-pound composite sample of chips taken from 150 cords of wood intended for mill-scale pulping.

The three lots of chips, all of which were air dry, were cooked in a stainless steel autoclave of about 1-cubic-foot capacity, which was equipped for indirect heating. The cooking liquor was prepared by absorbing sulfur dioxide gas in a suspension of hydrated lime. After the digestion, the fibers were dispersed with a motor-driven agitator and the pulp passed through a 12-cut screen.

The pulps were evaluated by test methods that approximated the contemporary TAPPI standards; the green-cut wood was tested in 1941 and the beetle-killed wood in 1949. Except for the use of the Schopper folding tester, the procedures were very similar to those in use today.

Discussion of Results

The Wood

Engelmann spruce is described as generally light in color, with the heartwood only slightly darker than the sapwood and with a tinge of red.[4] In weight it is the lightest of the spruces, averaging 23 pounds per cubic foot air dry. The fiber length is generally given as 3.0 millimeters.

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[4] Betts, H. S. Engelmann Spruce, American Woods Series. U. S. Department of Agriculture. 1941.

The average density of the green-cut wood used for the pulping experiments was 22.3 pounds per cubic foot on a green volume, oven-dry-basis (table 1). The trees had grown rapidly and were fairly young. Because of the large diameter of the trees compared with typical pulpwood, a racked cord had an unusually high wood volume of 96.7 cubic feet. This offset the low density and gave a weight of dry peeled wood equal to a typical cord of white spruce. Because of the lower density, however, a digester charge of Engelmann spruce will contain less wood than a charge of a denser species. The chemical analysis of the green-cut Engelmann spruce showed lignin, Cross and Bevan cellulose, and pentosan contents typical of spruce. The materials soluble in alcohol-benzene and ether, on the other hand, were relatively small.

The physical and chemical characteristics of the green-cut wood were almost identical with those of the white spruce used for comparison (table 1). This particular lot of white spruce, however, had grown rapidly, was below average in density, and contained a comparatively small amount of extractives.

Effect of Degree of Cooking

The green-cut wood was cooked in a series of digestions in which the time at maximum temperature was varied from 3.5 to 6.5 hours and the total time from 7.5 to 10.5 hours. Details of the cooking conditions are given in table 2. The effects of this variable on the properties of the pulp are given in tables 3 and 4.

Increasing the digestion time from 7.5 to 10.5 hours reduced the permanganate number from 15 to 6.3. The relationship between cooking time and permanganate number was almost linear. Pulp from wood cooked for the longest time contained 2.7 percentage points more total cellulose and 1 percentage point less lignin than did the pulp cooked for the shortest time. The cellulose content increased more or less linearly with time, whereas the lignin content showed little change after 8.5 hours.

The overall decrease in total yield was 2.8 percent, but the rate of decrease was less as the cooking was extended. Except for the shortest digestions, the amount of screenings was very low.

Variations in the strength of these pulps were moderate. The general trend was toward lower strength with increasing digestion time, but several properties showed a maximum. These were the tearing resistance at a freeness of 250 milliliters, the tensile strength (breaking length) at 600 milliliters, and the Schopper folding endurance at both freeness values.

Effect of a Shortened Cooking Schedule

After this cooking-degree series, digestions were made in triplicate in which the cooking cycle was shortened by increasing the maximum temperature, pressure, and the concentration of free sulfur dioxide in the liquor charged. These

modified cooking conditions are included in table 2 along with the basic conditions used in the preceding series. The medium-bleaching pulp obtained with the shortened cooking cycle is compared in tables 5 and 6 with pulp prepared by the basic conditions. Comparison is made at equal bleachability by interpolating the earlier data. The increase in temperature and amount of sulfur dioxide caused a decrease in yield from 48.8 to 47.4 percent. Neither schedule gave a pulp that was consistently stronger, although the pulp cooked under more drastic conditions contained slightly less pentosans and more cellulose. The shorter schedule would increase the daily digester output considerably, inasmuch as the cooking time is reduced from 9.25 to 6.75 hours.

Comparison of Engelmann with a White Spruce Sulfite Pulp

A white spruce pulp was prepared, using the basic pulping conditions given in table 2, to provide a comparison with the Engelmann spruce pulp. The results are given in tables 7 and 8. Comparison was made possible at equal permanganate number by interpolating the results of the first series of digestions in which Engelmann spruce was used. The white spruce pulp was produced in 0.3 hour shorter time, but it was inferior to the other in practically every respect. The yield from white spruce was less, 48.2 compared with 48.9 percent, but the difference was hardly significant. The two pulps did not differ greatly in their chemical analyses although the white spruce showed a slightly greater content of alpha cellulose. The Engelmann spruce pulp was considered to have characteristics normal for spruce pulps, whereas the white spruce pulp was below average in strength.

Beetle-Killed Engelmann Spruce

The cooking conditions used for the beetle-killed Engelmann spruce differed slightly from those used in cooking the green-cut wood by the basic conditions shown in table 2, but insufficiently to greatly affect the pulp. Table 9 shows that the yields were practically identical at equal bleachabilities (permanganate numbers), although the dead-felled trees gave a larger amount of screenings.

While the pulps from insect-killed wood had lower bursting and tearing strength at a freeness of 600 milliliters, the difference in quality was much reduced with further processing (table 10). The data are insufficient to be conclusive, but there is no indication that the value of Engelmann spruce wood is seriously lowered when it comes from a sound but dead and dried-out tree.

When another laboratory compared pulp produced from this lot of insect-killed material with an equivalent pulp made from a shipment of Wisconsin grown spruce, the yield was 1 to 2 percent higher from the western wood. The initial bursting strength was about 15 percent higher for Engelmann spruce, but the tearing resistance was lower by an equal percentage. After these pulps were processed to a freeness of 400 milliliters, the differences in bursting strength were negligible but the differences in tearing resistance had narrowed only slightly.

Table 1.--Physical and chemical characteristics of Englemann and white spruce wood

Characteristics	Engelmann spruce		White spruce
	Shipment 1509[1]	Average[2]	Shipment 1571[1]
Physical properties:			
Density (oven-dry weight per green volume).....pounds per cubic foot	22.3	20.4	21.1
Growth rate.....rings per inch	15.3	25.6	14.2
Age.....years	68	150	55
Diameter.....inches	8.9	11.6	7.6
Heartwood.....percent	54.3	--	--
Chemical analysis:			
Lignin.....percent	28.3	27.3	28.3
Cross and Bevan cellulose.....percent	59.5	--	60.2
Alpha cellulose on Cross and Bevan.....percent	42.7	--	43.2
Holocellulose.....percent	--	68.6	--
Alpha cellulose on holocellulose.....percent	--	45.0	--
Total pentosans.....percent	13.5	--	12.3
Solubility in:			
Alcohol-benzene.....percent	1.5	2.7	2.0
Ether.....percent	.6	1.3	1.0
1 percent NaOH.....percent	8.8	11.4	8.8
Hot water.....percent	1.7	2.3	2.0
Ash.....percent	.15	--	.3

[1]Green-cut wood.

[2]Average of two shipments of green-cut and two shipments of beetle-killed wood from areas ranging from Oregon to Utah (Shipments 2466, 2659, 3030, 3049).

Table 2.--Conditions used in the sulfite pulping of green-cut Engelmann spruce

Cooking conditions	Basic conditions	Shorter cycle
Cooking liquor:		
Total sulfur dioxide.....percent:	6.5	7.0
Combined sulfur dioxide.....percent:	1.25	1.25
Liquor charged..... :		
...gallons per 100 pounds of moisture-free wood:	56.6	55.3
Temperature schedule:		
Time to 110° C.....hours:	2	2
Time at 110° C.....hours:	0	0
Time from 110° C. to maximum.....hours:	2	2
Time at maximum temperature.....hours:	3.5 to 6.5	2.75
Total time.....hours:	7.5 to 10.5	6.75
Maximum temperature.....° C.:	135	145
Maximum pressure.....pounds per square inch:	85	90

Table 3.--Yields and chemical analysis of green-cut Engelmann spruce pulps obtained with different cooking times

Digestion numbers	Cooking time	Screened yield	Screenings	Permanganate number	Chemical analysis of pulps					
					Lignin	Cross and Bevan cellulose		Total pentosans	Solubility in	
						Total	Alpha		Alcohol-benzene	Ether
	Hours	Percent	Percent		Percent	Percent	Percent	Percent	Percent	Percent
335	10.50	48.0	0.3	6.3	0.6	95.4	79.5	5.9	0.6	0.30
338-42	10.00	48.4	.2	7.3	.6	95.6	79.4	6.3	.9	.35
340-43	8.75	48.9	.2	10.6	.6	94.3	77.9	5.9	.9	.35
344-58	7.50	50.2	.9	15.0	1.6	92.7	77.0	6.4	1.1	.45

Table 4.--Strength of green-cut Engelmann spruce pulps from beater tests

Digestion numbers	Bursting strength at freeness[1] of		Tearing strength at freeness[1] of		Folding endurance[2] at freeness[1] of		Breaking length at freeness[1] of		Density at freeness[1] of		Beating time at freeness[1] of	
	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.
	Points per pound per ream[3]		Gr. per lb. per ream[3]		Double folds		Meters		Gr. per cc.		Minutes	
335	0.81	0.92	1.35	0.78	220	520	5,450	6,960	0.66	0.82	11	34
338-42	.75	.78	1.05	.64	280	415	5,600	7,060	.70	.84	11	33
340-43	.98	1.14	1.20	.84	520	865	7,660	8,470	.72	.87	13	36
344-58	1.00	1.16	1.26	.79	435	577	6,450	9,080	.67	.86	10	35

[1] Canadian Standard freeness.

[2] Schopper folding tester.

[3] Ream of 500 sheets, 25 by 40 inches.

Table 5.--Effect of shortened cooking time on yield and properties of sulfite pulps of green-cut Engelmann spruce

Conditions	Total SO_2 in acid	Maximum temperature	Cooking time	Pressure	Screened yield	Screenings	Permanganate number	Chemical analysis of pulps					
								Lignin	Cross and Bevan cellulose		Total pentosans	Solubility in	
									Total	Alpha		Alcohol-benzene	Ether
	Percent	° C.	Hours	Pounds per square inch	Percent	Percent		Percent	Percent	Percent	Percent	Percent	Percent
Basic conditions	6.50	135	9.25	85	48.5	0.3	8.7	0.6	94.8	78.5	6.1	0.9	0.4
Shorter cycle	6.95	145	6.75	90	46.9	.5	8.7	.8	95.7	81.2	5.2	.8	.4

Table 6.--Effect of shortened cooking time on strength values of sulfite pulps of green-cut Engelmann spruce from beater tests

Conditions	Bursting strength at freeness[1] of		Tearing strength at freeness[1] of		Breaking length at freeness[1] of		Density at freeness[1] of		Beating time at freeness[1] of	
	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.
	Points per pound per ream[2]		Gr. per lb. per ream[2]		Meters		Gr. per cc.		Minutes	
Basic conditions	0.93	1.09	1.20	0.81	6,750	8,000	0.70	0.85	12	34
Shorter cycle	1.00	1.00	1.20	.78	6,280	8,620	.67	.87	17	43

[1] Canadian Standard freeness.

[2] Ream of 500 sheets, 25 by 40 inches.

Table 7.--Comparison of sulfite pulps made from green-cut Engelmann and white spruce

Pulp from	Cooking time	Screened yield	Screenings	Permanganate number	Chemical analysis of pulps					
					Lignin	Cross and Bevan cellulose		Total pentosans	Solubility in	
						Total	Alpha		Alcohol-benzene	Ether
	Hours	Percent	Percent		Percent	Percent	Percent	Percent	Percent	Percent
Engelmann spruce	8.7	48.9	0.3	10.9	0.7	94.3	78.0	6.2	0.9	0.4
White spruce	8.4	48.2	.4	10.9	.7	94.8	79.5	6.0	1.0	.4

Table 8.--Strength values of sulfite pulps from beater tests

Pulp from	Bursting strength at freeness[1] of		Tearing strength at freeness[1] of		Breaking length at freeness[1] of		Density at freeness[1] of		Beating time at freeness[1] of	
	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.
	Points per pound per ream[2]		Gr. per lb. per ream[2]		Meters		Gr. per cc.		Minutes	
Engelmann spruce	0.97	1.13	1.21	0.83	7,200	8,550	0.72	0.87	13	36
White spruce	.70	.76	1.01	.65	5,780	7,210	.63	.79	8	29

[1] Canadian Standard freeness.

[2] Ream of 500 sheets, 25 by 40 inches.

Table 9.--Comparison of the sulfite pulps from beetle-killed and green-cut Engelmann spruce

Cooking conditions	Pulp from beetle-killed wood at permanganate No. of		Pulp from green-cut wood at permanganate No. of	
	18.4	10.0	18.4	10.0
Cooking liquor:				
Total sulfur dioxide.....percent	5.85	6.08	6.50	6.50
Combined sulfur dioxide.....percent	1.20	1.20	1.25	1.25
Liquor charged.....gallons per 100 pounds of moisture-free wood	55.2	55.2	56.6	56.6
Temperature schedule:				
Time to 110° C.....hours	2.0	2.0	2.0	2.0
Time at 110° C.....hours	2.0	2.0	0	0
Time from 110° C. to maximum.....hours	2.0	2.0	2.0[1]	2.0[1]
Time at maximum temperature.....hours	3.4	5.1	3.2[1]	5.1[1]
Total time.....hours	9.4	11.1	7.2[1]	9.1[1]
Maximum temperature.....° C.	130	130	135	135
Maximum pressure.....pounds per square inch	84	85	85	85
Total pulp yield.....percent	51.5	48.3	52.0[1]	49.0[1]

[1] Interpolated values based on permanganate number.

Table 10.--Comparison of sulfite pulps from green-cut and beetle-killed Engelmann spruce

Pulp description and strength	Pulp from beetle-killed wood at freeness[2] of				Pulp from green-cut wood[1] at freeness[2] of			
	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.	600 ml.	250 ml.
Permanganate number	18.4	18.4	10.0	10.0	18.4	18.4	10.0	10.0
Yield of screened pulp.....percent	47.1	47.1	47.1	47.1	[3]52	[3]52	48.8	48.8
Screenings.....percent	4.4	4.4	1.2	1.2	--	--	.2	.2
Bursting strength ...pts. per lb. per ream[4]	.90	1.22	.80	1.05	1.00	1.15	.93	1.09
Tearing resistance... gm. per lb. per ream[4]	1.07	.73	1.05	.77	1.25	.78	1.20	.81
Breaking length.....meters	7,300	9,400	6,700	8,000	7,500	9,200	7,100	8,350
Density.....gm. per cc.	0.70	0.93	0.74	0.86	0.68	0.86	0.70	0.84

[1] Interpolated data.

[2] Canadian Standard freeness.

[3] Total yield. Screenings could not be estimated accurately by interpolation.

[4] Ream of 500 sheets, 25 by 40 inches.