SULFITE PULPING OF WESTERN REDCEDAR

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Abstract

Western redcedar (Thuja plicata) was found to be difficult to pulp under normal sulfite pulping conditions. The pulping difficulties could be overcome to some extent by employing a cooking liquor high in calcium bisulfite, together with a long cooking time, although only nonbleaching pulps could be obtained. Sodium and magnesium base sulfite liquors were more effective pulping agents than calcium in that order. The use of a liquor high in magnesium bisulfite (28 percent in comparison with a normal content of 15 percent based on the wood) permitted the production of a bleachable pulp, but with a low yield of only 41 percent. The bleaching and nonbleaching pulps had strength properties in general equal to commercial unbleached sulfite pulp, but their dark color would limit them to uses where color is not a consideration. The bleachable Western redcedar pulp was readily bleached to a satisfactory brightness in a two-stage process using a total chlorine consumption of 5 percent, and the bleached pulp was equal in strength properties to commercial bleached Western hemlock pulp and could perhaps find similar outlets. The low density of the wood and the low percentage yields of pulp from it indicated a yield of pulp per unbarked cord of only 660 pounds (oven dry basis) in comparison with average commercial yields for spruce of about 920 pounds.

Introduction

Although available to a lesser extent than several other western species, Western redcedar can be obtained in fairly large quantities as woods and sawmill waste. This material is not suitable for shingles, poles, or lumber, but it is possible that it may have value for some chemical utilization industry like pulping.

As far as is known, little or no Western redcedar is at present converted into pulp and none is intentionally reduced to sulfite pulp. Wells and Rue[1] in exploratory digestions on this species produced dark, shivey sulfite pulp which was impossible to bleach and whose uses were very limited. Application of the sulfate pulping process, on the other hand, led to strong pulp having properties suitable for general kraft pulp outlets. These pulping data,

[1] Wells, S. D., and Rue, J. D. USDA Dept. Bull. 1485 (1927), out of print.

obtained nearly 25 years ago, appear to constitute the only published information on the pulping of Western redcedar.

The Western redcedar tree is a slow-growing wood found along the North Pacific Coast, generally in mixture with other species. The total stand in the United States has been estimated[2] to be approximately 33-1/2 billion board feet, with a recent annual cut of something like 760 million board feet going principally into shingles, lumber, poles, posts, and piling. The wood is predominantly heartwood of a reddish-brown color, and is classed as one of the most durable woods. Western redcedar is a relatively light wood having a density of 19 pounds per cubic foot, green volume. Its fiber length is generally given as 3.8 mm.

These digestions were made because of the need for up-to-date pulping data for possible application in the utilization of the considerable quantity of Western redcedar waste that is available. This report, therefore, presents the results of a limited number of sulfite digestions made under more or less standard conditions and under certain modifications thereof and of two bleaching trials on the sulfite pulp.

Experimental Part

The wood was available in the form of chips of the size generally used for pulping, with chip lengths of 1/2 to 3/4 inch. The chips on arrival were in a fairly moist condition. At the time of digestion the moisture content was 44 percent. The chips were chemically analyzed and disks of wood which accompanied the chips were measured for various physical characteristics. The results of these tests are given in table 1. For purposes of comparison, chemical data for Western hemlock chips received at the same time are also included in table 1.

Most of the digestions were made in a 1.5-cubic-foot alloy-clad autoclave fitted with a steam jacket for indirect heating. Approximately 11 pounds of chips (moisture-free basis) were used. The cooking liquor was made up as usual by bubbling sulfur dioxide from a cylinder into a suspension of calcium hydroxide or magnesium oxide or a solution of sodium hydroxide in a tank. The acid charge was 6.75 gallons. The cooked chips from the autoclave, after being fiberized with a stirrer, were run through a small flat screen with a 12-cut plate.

One digestion, number 5099, was made in a 13-cubic-foot, alloy-lined, jacketed digester. For this digestion 85 pounds of wood (moisture-free basis) and 61 gallons of acid were used. The cooked chips from the digester were discharged under pressure into a blowpit, the pulp run over a flat screen equipped with 12-cut plates, and the pulp finally lapped by a wet machine.

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[2] Betts, H. S. USDA Forest Service. American Woods, Western Redcedar (1944).

The pressure was 80 pounds per square inch for all digestions. The acid concentrations, temperatures, and times employed in all digestions, and yields of pulp and screenings are given in table 2.

Both the standard TAPPI permanganate number method and a single-stage hypochlorite bleaching method were used to determine the bleachability of the pulps or to characterize them as not bleachable. The strengths of the pulps were judged at freeness levels of 800 and 550 cc. (Schopper-Riegler) after processing in a Valley test beater. The pulps were also chemically analyzed. All these results are found in table 3.

The pulp produced in the larger scale digestion was subjected to two bleaching procedures. The first bleaching procedure was as follows: the pulp was (a) chlorinated with 2 percent chlorine based on the wood, (b) neutralized with lime, (c) extracted with 2 percent caustic soda, (d) washed, (e) given an alkaline hypochlorite bleach using an equivalent of 2 percent chlorine, (f) washed, (g) given a second hypochlorite bleach using an equivalent of 0.3 percent chlorine, (h) washed, and (i) given a sulfur dioxide soak. The second bleaching procedure was as follows: the pulp was (a) chlorinated with 2.7 percent of its weight as chlorine, (b) washed in acid, (c) given an alkaline hypochlorite bleach using the equivalent of 2.2 percent chlorine, (d) washed. The bleached pulps were tested for strength and chemical composition similarly to the unbleached pulps. The results are given in table 4. The properties of a commercial Western hemlock bleached sulfite pulp are also included.

Discussion of Results

The Wood

The Western redcedar used had a characteristically low density of 19.1 pounds per cubic foot (table 1). This fact, coupled with the low percentage pulp yields, as shown later, indicated that a very low yield of pulp per cord and per unit of digester space would be obtained. The heartwood content, likewise characteristically, was high -- 85 percent by volume.

The wood was particularly distinguished by its low total and alpha cellulose contents and high lignin and extractives contents. (See table 1.) This composition is indicative of low pulp yield and possibly difficult pulping by the sulfite process.

Sulfite Pulping Under Standard Conditions

Application of sulfite pulping conditions previously found satisfactory for Western hemlock and other pulping species resulted in incomplete pulping of the Western redcedar (number 431, table 2). The screenings amounted to 4 percent, and the pulp was very dark-colored and shivey, had a very high permanganate number of 35, and a very high lignin content of 6 percent. The strength of the pulp was, however, excellent. Although the very dark color of the spent liquor made its analysis difficult and uncertain, there were indications that unused bisulfite was present at the end of the cook.

The digestion just described was repeated (number 432) with the exception that it was carried to exhaustion of the bisulfite, which required 2.25 hours longer. This procedure aided the pulping somewhat, resulting in lower screenings, a slightly brighter pulp with a permanganate number of 25, and a lignin content of 2.2 percent. However, the pulping was still far from satisfactory and it was concluded that Western redcedar was fairly refractory toward sulfite pulping as ordinarily practiced. The refractoriness differed from that encountered with Douglas-fir and the pines in that the typical decomposition of the cooking liquor with these species before cooking was completed did not occur. The exhaustion of the base was normal.

Digestions with Moderately High Calcium Bisulfite Content

Although the bisulfite content of the liquor used in the digestions under standard conditions, 18 percent of the wood, would be expected to be adequate for most woods, it seemed possible that an appreciable portion of the bisulfite was consumed early in the cook in reactions with certain constituents classed as extractives which are abnormally high in Western redcedar. If this were the case, the remaining bisulfite might be insufficient for completing the desired pulping reactions. A digestion was made, therefore, with a liquor somewhat higher than before in calcium bisulfite content (21.3 percent in number 433 as compared with 18.4 percent in number 432). This digestion was carried practically to exhaustion of the base as shown by liquor control tests. A certain reduction in screenings resulted from this step but the pulp itself was very similar in properties to the first one made (number 431), that is, dark-colored, high in lignin, but excellent strength.

Although the control tests in the digestion just discussed apparently showed exhaustion of the bisulfite, another digestion was made under the same conditions with the exception that the digestion time was arbitrarily lengthened 2.25 hours (number 434). Judged by the low screenings the pulping in this case was complete. The pulp had, however, a high lignin content and permanganate number and its strength properties were very good. The bisulfite content was seemingly high enough in this digestion to permit uniform pulping to the extent that the defibering point was reached by essentially all of the chips.

Digestions with High Calcium Bisulfite Content

The improvement in the pulping of the redcedar resulting from increasing the bisulfite in the cooking liquor from a normal to a relatively high content was followed by making digestions with liquors having a bisulfite content as high as reasonably possible, without encountering precipitation of calcium sulfite during cooking. The first digestion under these conditions (number

436) was made with a liquor having a bisulfite content close to 25 percent or over a third more than normal. The reduction of the wood to pulp was essentially complete and the lignin content of the pulp was fairly low. The permanganate number of the pulp was still very high, however, and the chemical characteristics other than lignin showed no improvement from a standpoint of purification over the previous digestions with lower bisulfite content.

Another digestion was made under the same conditions of high bisulfite content with the exception that the cooking time was lengthened 2 hours (number 437). This procedure resulted in the most satisfactory pulping obtained with the calcium base liquor. The low screenings indicated essentially complete pulping. Although the yield of pulp was low, its lignin content was lower and cellulose content higher than any of the previous pulps. The strength of the pulp was lower, however, than the relatively high-strength pulps with higher lignin contents. The permanganate number of 18 and single-stage bleach value of over 30 percent for this pulp indicated it to be difficult to bleach, except for its low unbleached brightness, the pulp had other properties approaching those of commercial paper-making pulps of similar bleachability. These pulping conditions probably represent the optimum as far as calcium base sulfite liquors are concerned. The low brightness of the unbleached pulp unfortunately limits its use to papers where color is no consideration. Certain board stocks would perhaps be promising outlets.

Digestion at Low Temperature

The use of low pulping temperatures is a known expedient in pulping refractory species like Douglas-fir and the pines. This was tried with the redcedar using a liquor of relatively high calcium bisulfite content. The maximum temperature was decreased from 130° C. (number 434) to 120° C. (number 435). Digestion was carried to exhaustion of the base as far as could be judged by the unsatisfactory control tests. This required a total digestion time of 20 hours as compared with 14 hours at 130° C. No noticeable improvement in pulping was obtained by using the low temperature. There were some indications of overcooking at the low temperature, as shown by the relatively low strength property and low alpha cellulose content of the pulp in relation to its lignin content.

Digestion with Sodium Bisulfite Liquor

Another well-recognized expedient in pulping refractory woods is the use of the soluble sulfites, sodium and ammonium. A digestion with liquor relatively high in sodium bisulfite content (number 438) was more successful in pulping the redcedar than a comparable calcium base digestion (number 434). The permanganate number and lignin content of the sodium base pulp were considerably lower and the cellulose contents higher than these respective values for the base pulp. In spite of the greater purification shown by the sodium base pulp, its yield and strength were only equal to those of the calcium base pulp. The brightness of the sodium base pulp was relatively low and permanganate number high, although very favorable pulping conditions had been employed.

Digestions with Magnesium Bisulfite Liquors

Magnesium bisulfite liquors occupy a position between calcium and sodium bisulfite liquors in their ability to pulp refractory woods. In addition to whatever specific pulping advantage magnesium may have over calcium bisulfite, the former is considerably more soluble than the latter in sulfurous acid, which gives the magnesium liquor a wider range of applicability. The use of magnesium bisulfite liquors has the advantage over sodium of being more completely developed commercially. A digestion with a magnesium bisulfite liquor (number 439) under conditions previously used with calcium and sodium base liquors (numbers 438 and 434) gave pulping results intermediate to the latter two but nearer to the sodium base. The pulping was satisfactorily complete but the pulp was dark-colored and nonbleaching, but had excellent strength properties. It would probably find only limited utilization if produced commercially.

The high solubility of magnesium bisulfite in the cooking liquor permitted further examination of the effect of bisulfite content, which had been previously indicated as an important factor in the pulping of Western redcedar. A digestion was made, therefore, with an abnormally high percentage of magnesium bisulfite (31.2 percent based on the wood in number 440). This modification resulted in the most successful pulping of all the 11-pound scale experiments. The pulp was bleachable and had appreciably higher cellulose contents than any of the previous pulps, although the yield was very low (only 41 percent). When it is considered that Western redcedar will run only about 1,610 pounds of moisture-free wood per cord, the pulp yield per cord is observed to be only about 660 pounds as compared with about 920 for spruce. The strength of this relatively low-bleaching pulp was, however, considerably lower than the pulps with higher permanganate numbers.

On the basis of the relatively successful pulping of the redcedar with the high magnesium bisulfite liquor on the 11-pound scale, the 85-pound-scale digestion (number 5099) was made to provide pulp for more elaborate bleaching experiments. The results on the larger scale verified those on the 11-pound scale. Because the bisulfite content was somewhat less on the 85-pound basis, the permanganate number was slightly higher, as were the strength properties of the pulp.

Bleaching Experiments

The first bleaching procedure (five-stage) used on the 85-pound-scale pulp (number 5099) was adapted from kraft bleaching procedures. The bleaching was entirely successful, the total chlorine consumption being 4.3 percent of the pulp and the brightness 82 percent (see table 3). The simpler two-stage bleach next attempted was equally successful, although the chlorine consumption was somewhat higher than in the first trial (4.9 percent). The two procedures led to pulps having very similar properties (see table 4) except for the higher alpha cellulose in the pulp made by the first procedure with a caustic extraction stage. In comparison with a commercial Western hemlock bleached sulfite pulp recently tested, the Western redcedar was considerably superior in bursting and tensile strengths but somewhat deficient in tearing strength. The bleached Western redcedar sulfite pulp, if it could be produced economically, should be useful for many of the purposes now satisfied by bleached Western hemlock pulp.

Table 1.--Physical test data and chemical analysis on Western redcedar (*Thuja plicata*) (Shipment 2132) and Western hemlock (*Tsuga heterophylla*) (Shipment 2127)

	Shipment numbers	
	2132	2127
Average of physical test data on disks:		
Diameter (inches)	24.5	
Age (years)	205.	
Growth rate rings per inch	8.1	
Density[1] (pounds)	19.1	
Heartwood diameter (inches)	22.7	
Heartwood by volume (percent)	84.8	
	Percent	
Chemical analysis of chips:		
Total cellulose	48.7	57.5
Alpha cellulose	38.0	40.7
Lignin	31.8	31.2
Total pentosans	9.0	8.1
Pentosans in cellulose	6.9	9.6
Solubility in:		
Alcohol-benzene	14.1	3.9
Ethyl ether	2.5	1.3
1 percent NaOH	21.0	11.7
Hot water	11.0	4.2
Ash	.3	.3

[1]Moisture free weight per cubic foot, green volume.

Table 2.--Results of sulfite pulping experiments on Western redcedar

Digestion number	Cooking liquor: Total SO_2	Cooking liquor: Combined SO_2	Cooking liquor: Base	Bisulfite content	Maximum temperature	Total time	Time at maximum temperature	Permanganate number	Yield: Total	Yield: Screenings	Brightness[1]: Unbleached	Brightness[1]: Standard bleaching powder	Brightness[1]: Bleached
	Percent	Percent		Percent of wood	° C.	Hours	Hours	Cc.	Percent	Percent	Percent	Percent	Percent
431	5.21	1.09	Calcium	18.0	130	10.7	4.7	34.8	48.1	4.0	21.7	80	77.8
432	5.35	1.10	Calcium	18.4	130	13.0	7.0	24.7	44.1	2.2	26.5	50	81.1
433	5.32	1.30	Calcium	21.3	130	11.7	5.7	35.5	48.8	1.3	22.3	80	73.9
434	5.27	1.30	Calcium	21.6	130	14.0	8.0	27.1	45.0	.6	27.9	50	77.2
435	5.96	1.32	Calcium	22.0	120	20.0	15.0	27.7	45.5	.8	26.7	50	77.4
436	5.28	1.49	Calcium	24.9	130	16.0	10.0	25.5	46.7	.3	28.8	60	82.9
437	5.35	1.47	Calcium	24.5	130	18.0	12.0	18.0	44.2	.6	33.1	35	80.0
438	5.32	1.26	Soda	21.6	130	15.0	9.0	15.7	44.6	.4	34.2	30	78.7
439	5.99	1.33	Magnesium	20.5	130	16.0	9.0	18.4	44.1	.4	37.5	—	—
440	6.30	2.03	Magnesium	31.2	140	15.0	8.0	11.1	41.3	.1	35.3	20	79.7
5099	5.88	1.58	Magnesium	28.4	130-140	15.2	9.2	13.0	40.7	.4	34.3	21	78.7

[1] Measured with a Hunter Multipurpose Reflectometer calibrated in accordance with TAPPI method T217 sm 42.

Table 3.--Chemical composition and strength properties of unbleached Western redcedar sulfite pulps

Digestion number	Chemical analysis: Lignin	Cellulose: Total	Cellulose: Alpha	Total pentosans	Solubility: Alcohol-benzene	Solubility: Ether	Solubility: 1 percent NaOH	Solubility: Hot water	Ash	Freeness[1]	Interpolated values from beater tests: Bursting[2] strength	Tearing[2] strength	Tensile[3] strength	Folding endurance	Density	Beating time
	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Cc.	Pts. per lb. per rm.	Gm. per lb. per rm.	Lb. per inch width	Double folds	Gm. per cc.	Minutes
431	6.4	86.2	71.8	3.7	0.7	0.2	11.6	2.0	1.1	800 550	0.90 1.09	1.17 .84	28 35	450 430	0.72 .84	9 34
432	2.1	91.4	73.7	3.9	.7	.3	11.4	1.8	.7	800 550	.79 1.02	1.23 .93	28 32	510 600	.72 .84	8 36
433	6.5	84.8	71.1	4.2	.8	.2	12.4	2.0	1.1	800 550	.86 1.08	1.10 .84	26 35	440 520	.67 .80	9 37
434	2.5	90.5	74.2	4.0	.9	.2	11.4	1.7	.9	800 550	.75 1.14	1.15 1.06	27 39	530 830	.71 .84	9 38
435	2.8	90.0	71.6	3.9	.7	.3	10.3	2.1	1.2	800 550	.71 .88	1.02 .77	24 30	360 670	.66 .85	5 28
436	1.5	90.7	74.2	4.0	.8	.3	11.1	1.7	1.0	800 550	.81 1.07	1.17 .90	25 35	530 340	.70 .86	7 30
437	1.3	93.3	77.2	3.9	.8	.2	10.7	1.7	.9	800 550	.78 .94	1.28 .90	26 32	230 420	.71 .89	4 28
438	.6	93.9	78.0	3.9	.6	.3	9.0	1.3	1.0	800 550	.81 1.08	1.30 .99	30 34	620 830	.76 .89	5 31
439	1.5	93.8	77.9	3.9	.9	.6	10.0	1.4	.5	800 550	.82 1.11	1.18 .83	29 37	360 680	.73 .91	5 31
440	1.1	95.7	81.3	3.1	2.1	1.6	10.3	1.0	.5	800 550	.57 .71	1.00 .85	19 25	70 115	.65 .87	2 31
5099	1.0	95.7	77.3	3.2	.6	.5	7.0	1.0	.6	800 550	.80 1.00	1.13 .68	25 33	300 450	.71 .88	5 30

[1] Schopper-Riegler.

[2] Ream weight basis 25 by 40 - 500.

[3] Adjusted to 55 pound ream weights.

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Table 4.--Strength properties and chemical composition of Western redcedar and Western hemlock bleached sulfite pulps

Bleach number	Total chlorine consumption	Brightness[1]	Chemical analysis: Lignin	Chemical analysis: Cellulose, Total	Chemical analysis: Cellulose, Alpha	Chemical analysis: Total pentosans	Chemical analysis: Ash	Freeness[2]	Interpolated values from beater tests: Bursting[3] strength	Tearing[3] strength	Tensile[4] strength	Folding endurance	Density	Beating time
	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Cc.	Pts. per lb. per rm.	Gm. per lb. per rm.	Lb. per inch width	Double folds	Gm. per cc.	Minutes
								Western redcedar						
813	4.2	82.2	0.0	100.0	86.0	3.8	0.8	800	0.76	1.35	22	260	0.73	9
								550	1.08	.87	36	530	.86	63
819	4.9	82.2	.0	100.0	84.3	3.5	1.2	800	.72	1.30	24	220	.67	4
								550	1.13	.96	37	470	.88	47
								Western hemlock						
......	....	81.0	....	....	....	....	....	800	.66	1.63	18	255	.63	31
								550	.76	1.19	25	538	.72	63

[1] Measured with a Hunter Multipurpose Reflectometer calibrated in accordance with TAPPI method T217 SM 42.

[2] Schopper-Riegler.

[3] Ream weight basis 25 by 40 - 500.

[4] Adjusted to 55 pound ream weights.

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