SULFITE PULPS FROM SEVERAL SOUTHERN HARDWOODS

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SULFITE PULPS FROM SEVERAL SOUTHERN HARDWOODS

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Introduction

The sulfite pulping experiments on southern cottonwood (Populus deltoides virginiana), black willow (Salix nigra), and sugarberry (Celtis laevigata) described in this report were made as part of a general investigation of the preparation and properties of a wide variety of pulps from hardwoods grown in the Arkansas Delta region. The objective of the general investigation was to develop possible uses as pulpwood for the large supply of hardwoods from this region. A study allied to the present experiments was reported previously concerning neutral and acid sulfite semichemical pulps from a number of the Delta hardwoods, including the above species (1). One of the woods, sugarberry, was also pulped in connection with the preparation from hardwoods of sulfite pulps suitable for purification to rayon pulps (2).

Of the three species studied, only the cottonwood has received any considerable attention. Carpenter and McCall (3) found southern cottonwood to be readily pulped by the sulfite process to produce a low-bleaching pulp high in alpha cellulose: the purified product prepared from this pulp, however, showed some limitations during conversion to viscose derivatives. This species was considered by Rue and Wells (4) to be similar to aspen in its pulping characteristics. Rue and Wells (4) found black willow to be easily reduced to a low-bleaching sulfite pulp in yields of 50 to 55 percent; the pulp, however, was specky. The pulping of sugarberry by the sulfite process has apparently not been reported, although Rue and Wells (4) found the closely related hackberry (Celtis occidentalis) to produce a light-colored, easily bleached sulfite pulp.

The objectives in the present study were (1) to prepare sulfite pulps by conventional means from southern cottonwood, black willow, and sugarberry, and to determine their properties; (2) to compare the southern hardwood pulps with previously prepared birch (Betula papyrifera) and aspen (Populus tremuloides) sulfite pulps; and (3) to compare the yields and properties of sugarberry sulfite pulps ranging from a high yield semichemical to a low-bleaching pulp.

Experimental Part

The three hardwoods selected for the sulfite pulping experiments were included in a shipment of six Arkansas delta species supplied by the Phillips County Chamber of Commerce, Helena, Arkansas, and cut in that county. The physical and chemical properties of the whole shipment have been described in detail elsewhere (1, 5). Average values for the cottonwood, willow, and sugarberry are given in table 1. Representative logs from each of the three species were converted into standard 5/8-inch chips for the digestions.

The sulfite cooking acid used for digesting the chips was prepared as usual by passing sulfur dioxide from a cylinder into a lead-lined tank containing milk of lime. The digestions were made in a stainless-steel clad, steam-jacketed autoclave having a capacity of 1.5 cubic feet. The pulped chips were defibered with a stirrer and screened through a diaphragm screen with 0.012 inch slots.

The conditions for the digestions of the three southern hardwoods are given in table 2. Conditions for birch and aspen digestions taken from a previous report (2) are also included for comparison. All the digestions were made with indirect steam.

The results from the southern hardwood digestions, as well as those from the northern hardwoods, are found in table 3. These results include yields, pulp bleach requirements, strength values, and chemical analyses of the pulps. In the case of the southern hardwoods the results are averages from two digestions for each species, whereas only one digestion each is represented by the northern hardwoods. The bleach requirement values are the average of data from permanganate number and single stage hypochlorite determinations, expressed in terms of standard bleach powder. The strength values are given for two freeness values, 800 and 550 cc. (Schopper-Riegler), as interpolated from strength-freeness relations obtained by beater processing. Standard Forest Products Laboratory methods were used throughout.

Discussion of Results

The Wood

The chemical and physical properties of the three southern hardwoods given in table 1 indicate certain differences between the cottonwood and willow on one hand and the sugarberry on the other. These differences, as will be shown later, reflect on the qualities of these species as pulpwood.

The cottonwood and willow were very rapid-growing and had relatively low densities but high total and alpha-cellulose contents. The sugarberry, in contrast, had a medium growth rate and density, but rather low contents of alpha and total cellulose. The sugarberry, nevertheless, had a slightly lower lignin content than the others, although it was somewhat high in pentosans and material soluble in 1-percent sodium hydroxide. None of the three woods was high in material soluble in ether.

Sulfite Pulps from the Southern Hardwoods

Application of the pulping conditions outlined in table 2 to the three southern hardwoods resulted in the production of sulfite pulps having bleach requirements of approximately 12 percent, as shown in table 3. The pulping conditions to obtain the same degree of pulping were the same for the three species except for a slightly shorter time, 9.5 compared to 10 hours, for the sugarberry. The shorter time may possibly have been a result of the somewhat lower lignin content of the sugarberry in comparison with the other two species.

The pulp yields from the cottonwood and willow were approximately the same high value of 52 percent, whereas that from the sugarberry was lower by nearly 10 percent. The large difference in pulp yield can be traced to the same relative difference between the woods in their cellulose contents. The chemical analyses of the three pulps, nevertheless, showed them to be very similar in their composition. The sugarberry pulp had a slightly higher lignin content than the others, whereas the willow pulp was slightly lower in alpha cellulose and pentosans. The chemical composition of all the pulps appeared to be typical for hardwoods pulped under the specified conditions.

The strength values of the equal bleaching pulps from cottonwood, and sugarberry, as shown in table 3, were the same for all the strength characteristics, while the same bleaching pulp from the willow had strength values lower throughout than the others. The low values for the willow pulp can not be explained by any differences in the chemical composition of the three pulps or the original chips. The strength values of the pulps on the whole were perhaps somewhat low for hardwood sulfite pulp in general and certainly considerably below the strength of softwood sulfite pulps.

The relatively high density of the sugarberry permitted a larger charge of chips than possible with the cottonwood or willow. The volume of cooking liquor charged for a unit weight of chips was therefore appreciably lower for the sugarberry than the others, as indicated in table 2. The low percentage yield of pulp from the sugarberry, on the other hand, offset to a large extent the advantage of high density. Thus, the yield of pulp for a given digester volume would be about the same for all three species.

Comparison with Birch and Aspen Sulfite Pulps

Birch and aspen pulps prepared for purifying to rayon pulps (2) showed certain similarities and dissimilarities to the southern hardwood sulfite pulps, as indicated in table 3. The pulping conditions for the northern and southern hardwoods were the same except for the low combined sulfur dioxide in the cooking liquor used to digest the birch and aspen. Apparently, because of the low combined sulfur dioxide, the pulping time for the birch and aspen was considerably less than for the southern hardwoods.

The bleach requirement of the birch pulp was the same as that of the southern hardwood pulps, but its yield was 4 percent lower than that of either cottonwood or willow, although 6 percent higher than that of the sugarberry. The aspen pulp had a very low bleach requirement but was produced in about the same yield as were the cottonwood and willow pulps.

Although the birch and aspen pulps were made with an acid low in combined sulfur dioxide, a condition favoring production of a pulp with a high alpha-cellulose and a low pentosan content, they contained about the same general percentage of alpha cellulose and a higher percentage of pentosans than the southern hardwood pulps made with a conventional acid. Further, the southern hardwood pulps had less than half the material soluble in ether contained in the birch and aspen. On the basis of the chemical characteristics shown in table 3, the cottonwood, willow and sugarberry pulp would appear to be more promising pulps for purifying to rayon-type pulp than the birch or aspen pulps.

The strength values of the willow pulp were approximately the same as the low bleaching aspen pulp, whereas those of the cottonwood and sugarberry pulps were intermediate to the aspen and birch pulps.

As might be expected the botanically related species, southern cottonwood and aspen, gave pulps in similar yields and having fairly similar properties. A harder bleaching aspen pulp would perhaps have strength properties nearly the same as the cottonwood pulp. An important difference did exist, however, in the amount of material soluble in ether; this value was relatively high in the aspen pulp.

Sugarberry Sulfite Pulping

Cooking conditions for producing sugarberry pulps ranging from a well-cooked pulp to a partially delignified semichemical pulp and the properties of these pulps are given in table 4. The results for the most delignified pulp, number 1, were taken from table 3; the results for the other pulps were reported previously (1, 2). Although the combined sulfur dioxide in the cooking acid used for number 1 was higher than for the others and the maximum temperature for numbers 3 and 4 was lower than for the others, the main independent cooking variable connecting these data was the cooking time.

The effect of cooking time on the yields and properties of the pulps is plainly evident from the figures in table 4. The yields and some of the chemical and physical properties values were also plotted against cooking time to furnish the curves in figure 1.

There were definite relations between cooking time and yield and many of the pulp properties, as shown by the curves in figure 1. The total yield decreased and the cellulose contents increased rather sharply with an increase in cooking time from 4 to about 8 hours. The changes were more gradual with a cooking time longer than 8 hours. The lignin and pentosans contents decreased gradually with cooking time, the over-all change being less than for yield or cellulose contents. The bursting and tensile strength and solid fraction values increased sharply as the cooking time increased from that producing an undefibered chip to that producing a material approaching the defibering point. The changes in these strength characteristics with cooking times corresponding to yields of 55 percent or less were relatively small. The tearing strength values and the times required to attain certain freeness levels showed no definite relation to cooking time.

It is interesting to note that digestion number 2, table 4, was carried considerably beyond the point of exhaustion of the base in the cooking liquor, but the pulp from it had a considerably higher bleach requirement and was less purified than the pulp from number 1, which was carried just to the point of exhaustion. It appeared that an insufficient amount of chemical was present in number 2 to complete the delignification and purification before the base was exhausted.

Summary

(1) Sulfite pulps having bleach requirements of approximately 12 percent standard bleach powder and an average alpha cellulose content of 82.5 percent were prepared by conventional pulping procedures from southern cottonwood, black willow, and sugarberry. The pulp yield from the cottonwood and willow was 52 percent, whereas that from the sugarberry was lower by 10 percent. The strength properties of the cottonwood and sugarberry pulps was somewhat superior to those of the willow pulp.

(2) Comparison of the southern hardwood pulps with birch and aspen sulfite pulps prepared for purifying to rayon pulps showed the former to have the same alpha cellulose content, lower pentosans content, and lower amounts of material soluble in ether than the latter. The cottonwood and willow pulps were produced in the same yield as the somewhat lower bleaching aspen pulp, but in higher yield than the birch pulp. The birch pulp showed higher values for strength properties than the others.

(3) Sugarberry sulfite pulps covering the range from a semichemical pulp produced in 4.2 hours to a low bleaching pulp produced in 9.5 hours showed consistent relations between pulping time and yields and properties of the pulps. An increase in pulping time caused decreases in pulp yield and lignin and pentosans contents and increases in the total and alpha cellulose content of the pulps and their bursting and tearing strength and solid fractions. The largest part of the changes occurred when the time was increased from 4.2 to 6.5 hours.

References

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(3) Carpenter, Charles and McCall, Frank. Progress in hardwood pulping for viscose rayon. Rayon Textile Monthly 19 (9, 10): 538-9, 618-20 (Sept., Oct. 1938)
(4) Wells, S. D. and Rue, J. D. The suitability of American woods for paper pulp. U.S.D.A. Bull. 1485 (1927)
(5) Pew, J. C. and Schafer, E. R. Physical and chemical properties of Arkansas Delta hardwood pulpwoods: sugarberry, ash, bitter pecan, cottonwood, willow, and elm. Problem D-189; project 1168-5. Mimeographed report.

Table 1.—Average values for physical and chemical characteristics of three Arkansas Delta hardwoods

Wood species	Physical properties					Chemical analyses							
	Density o.d. wt. per green volume	Growth rate	Age	Diameter	Heartwood	Lignin	Cellulose	Alpha cellulose	Total pentosans	Solubility in: Alcohol benzene	Solubility in: Ether	Solubility in: 1% NaOH	Solubility in: Hot water
	Pounds per cubic foot	Rings per inch	Years	Inches	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent
Black willow (*Salix nigra*, Marshall)	23.5	3.7	28	15.5	36.9	21.9	61.6	46.6	18.8	2.2	0.3	17.4	3.6
Cottonwood (*Populus deltoides virginiana* (Foug) Sudworth)	23.7	4.1	26	13.0	43.4	23.6	63.2	46.5	19.0	1.8	.4	15.4	2.0
Sugarberry (Celtis laevigata, Willdenow)	30.5	9.5	47	9.9	None	20.8	54.4	40.2	21.6	3.1	.3	22.7	6.4

Table 2.--Digestion conditions for sulfite pulping of several southern and northern hardwoods

	Cottonwood	Willow	Sugarberry	Birch	Aspen
Wood dryness (percent)	61.2	85.6	87.1	86.0	86.0
Acid-wood ratio (gallons per 100 pounds moisture-free wood)	63.0	61.5	46.8	46.0	63.0
Acid concentration					
Total sulfur dioxide (percent)	6.31	6.46	6.23	5.90	5.90
Combined sulfur dioxide (percent)	1.26	1.26	1.26	1.04	.90
Temperature schedule					
Time to 105°C. (hours)	2.0	2.0	2.0	2.0	2.0
Time at 105°C. (hours)	1.0	1.0	1.0	1.0	1.0
Time from 105° to 135°C. (hours)	2.0	2.0	2.0	2.0	2.0
Time at 135°C. (hours	5.0	5.0	4.5	3.6	3.3
Pressure (pounds per square inch)	80.0	80.0	80.0	80.0	80.0

Table 3.--Yields and properties of sulfite pulps from southern and northern hardwoods

Species	Yield		Bleach requirement	Chemical analysis					Strength values from beater tests - 25 x 40 - 500 ream									
	Total	Screenings		Lignin	Cellulose		Total pentosans	Ether solubility	Bursting strength		Tearing strength		Tensile strength		Solid fraction		Beating time	
					Total	Alpha			Freeness S.R.		Freeness S.R.		Freeness S.R.		Freeness S.R.		Freeness S.R.	
									800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.
	Percent	Percent	Percent standard bleach powder	Percent	Percent	Percent	Percent	Percent	Pt. per lb. per gm.		Gm. per lb. per gm.		Lb. per sq. in.				Minutes	
Southern hardwoods																		
Cottonwood	52.0	0	11.9	0.6	96.2	82.6	6.2	0.6	0.38	0.53	0.56	0.48	3550	6250	0.41	0.52	22	69
Willow	51.9	0	11.8	1.2	95.7	81.4	7.8	.4	.24	.38	.34	.30	2525	4875	.38	.48	6	32
Sugarberry	42.3	0.4	12.3	1.5	95.3	83.6	7.1	.5	.38	.50	.62	.48	3350	6050	.42	.54	17	58
Northern hardwoods																		
Birch	47.8	0	12.0	1.7	96.3	81.8	10.9	1.3	.54	.74	1.05	.83	4300	8100	.43	.59	16	55
Aspen	52.5	1.0	8.9	1.1	94.9	83.3	8.4	1.2	.28	.38	.42	.45	3000	4600	.39	.50	32	111

Table 4.--Digestion conditions and results for sulfite pulping of sugarberry

Number	Cooking Liquor		Combined SO_2 at end	Maximum temperature	Time to maximum temperature	Total time	Yield		Bleach requirement	Chemical analysis				
	Total SO_2	Combined SO_2					Total	Screenings		Lignin	Cellulose		Total pentosans	Ether solubility
											Total	Alpha		
	Percent	Percent	Percent	°C	Hours	Hours	Percent	Percent	Percent standard bleach powder	Percent	Percent	Percent	Percent	Percent
1	6.2	1.26	-0.01	135	5	9.5	42.3	0.4	12.3	1.5	95.3	83.6	7.1	0.5
2	6.0	.92	-.64	135	5	7.5	45.8	.6	22.0	3.4	92.9	81.8	8.4	.4
3	5.6	.90	.48	130	4	5.2	59.9	...	...	11.2	76.1	63.5	12.4	.3
4	6.0	.90	.68	130	4	4.2	76.1	...	...	16.1	63.6	51.7	16.8	.4

Strength values from beater tests -- 25 x 40 - 500 ream

Number	Bursting strength		Tearing strength		Tensile strength		Solid fraction		Beating time	
	Freeness S. R.		Freeness S. R.		Freeness S. R.		Freeness S. R.		Freeness S. R.	
	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.	800 cc.	550 cc.
	Pt. per lb. per gm.		Gm. per lb. per gm.		Lb. per sq. in.				Minutes	
1	0.38	0.50	0.62	0.48	3350	6050	0.42	0.54	17	58
2	.34	.50	.86	.73	2100	5700	.41	.53	9	70
3	.30	.46	.67	.60	2000	4200	.32	.43	24	77
4	.18	.25	.50	.49	600	1250	.20	.25	24	65

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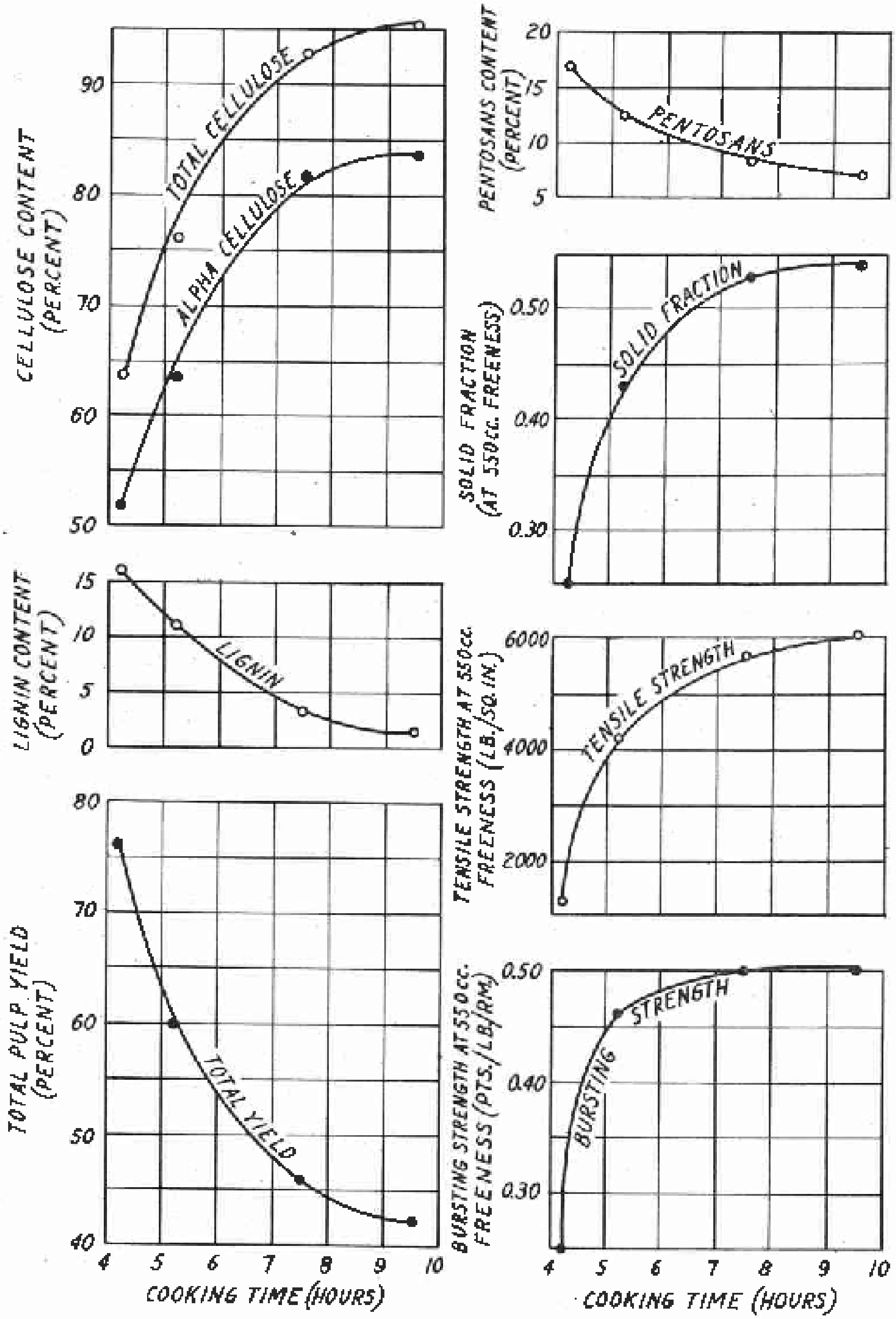

FIG. I
RELATIONS BETWEEN COOKING TIME AND YIELD AND PROPERTIES OF SUGARBERRY SULPHITE PULPS.

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