REFINED NEUTRAL SULFITE SEMICHEMICAL PULPS FROM DOUGLAS-FIR

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REFINED NEUTRAL SULFITE SEMICHEMICAL PULPS FROM DOUGLAS-FIR

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Synopsis

Exceptionally strong, bleached pulps of high yield were made from Douglas-fir by purification of neutral sulfite semichemical pulps by the holocellulose method. A yield of 57 percent of bleached pulp, the alpha-cellulose content of which was 87 percent was realized. The alpha cellulose recovered in this instance amounted to approximately 50 percent of the moisture-free weight of the wood.

Test beater strength data indicated that the bursting strength of this highly purified pulp was much higher than sulfite and considerably higher (approximately 18 percent) than unbleached kraft pulp made from an identical sample of the wood. The high yield of alpha cellulose and its viscosity (43.0 centipoises in cuprammonium solution) indicated that little degradation of the cellulose had resulted from its preparation. Owing to the hemicelluloses present, the bleached pulp hydrated very rapidly in the test beater, requiring only approximately 20 minutes to attain the maximum bursting strength. Further processing to a condition approximating that required for greaseproof or glassine type of paper was accomplished without loss in bursting strength.

Removal of various amounts of the hemicelluloses by extraction with caustic soda solutions of varied concentration changed the physical properties of the pulp and increased its whiteness. By such a treatment the pulps developed strength more slowly to lower bursting and tensile strengths, but showed an increase in resistance to tear. The data indicate that pulps equal in strength to those prepared from spruce on the same method, can be made from Douglas-fir.

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

Introduction

The vast quantities of Douglas-fir available in the western United States have thus far found little use in the pulping field because of the inferior quality of the products obtained by the several commercial pulping processes. Although Douglas-fir pulps usually have high tearing strength, this feature alone does not compensate for their poor felting qualities, their comparatively low bursting and tensile strengths, and for low pulp yields, especially when this species is cooked for bleaching and for alpha cellulose.

Since it was not definitely known whether the low strength properties of Douglas-fir pulps were due to properties inherent in the wood or its fiber or to degradation of the cellulose chains and the removal of certain fractions of the cellulose by the common commercial pulping methods, it was decided to try the mildest pulping methods known. The holocellulose method is known to remove lignin without perceptibly attacking either the cellulose or the hemicelluloses of wood.[2] Recent experiments[3] conducted at the Forest Products Laboratory indicated that stronger pulps than those prepared by the sulfite process can be made from white spruce by the application of the holocellulose method to wood that had been previously fiberized by an Asplund machine.

Owing to difficulties of chlorine penetration and the removal of reaction products from pulpwood chips of the size commonly used in commercial pulping operations, the holocellulose method has been found to be applicable only to finely divided or previously fiberized[3] wood. The mild action of the neutral sulfite semichemical process[4] produces a softened chip of high yield, which is easily fiberized by mechanical means without seriously attacking the cellulose.

Hence, this method offered possibilities of rendering pulpwood chips suitable for further purification by the holocellulose method.[2] Douglas-fir chips were, therefore, subjected to the mild action of these two procedures for the production of a bleachable pulp of high strength and yield.

For comparison with pulps made by this combination of the semichemical and holocellulose methods, identical samples of Douglas-fir chips from the same bolt were pulped by the standard sulfate process under conditions suitable for the production of high strength kraft and bleachable pulps. In addition, a comparison was made with sulfite pulps prepared from wood taken from the same position in a similar tree of like growth characteristics.

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[2] Van Beckum and Ritter. Paper Trade J. 105 (18) 127 (Oct. 28, 1937).

[3] Hajny and Ritter. Paper Trade J. 111 (22) 131 (Nov. 28, 1940).

[4] Rue, Rawling, Wells, and Staidl. Paper Trade J. 83 (13) 50 (Sept. 23, 1926).

[5] Hajny and Ritter. Paper Trade J. 113 (13) 83 (Sept. 25, 1941).

Raw Material

The Douglas-fir (Pseudotsuga taxifolia) pulpwood used in these experiments was part of a shipment (1528) cut in Snohomish County, Washington, and supplied through the cooperation of the Pacific Northwest Forest and Range Experiment Station. The wood used for these and the sulfate pulping experiments was selected from the middle 4-foot section (tree 2, log 11) of a young, rapid-growth tree. Portions of disks cut from top, middle, and butt logs of tree 2 are shown in figure 1. The wood used for the sulfite pulping experiments was also taken from a log midway up a tree (tree 1, log 11) of similar growth characteristics from the same vicinity. The physical properties and chemical analysis of the wood from these two bolts are reported in table 1.

From these data the wood appeared to be in sound condition. The low pentosan content is characteristic of Douglas-fir and certain other western species.

The wood was hand-peeled and converted into 5/8-inch chips by a two-knife, 48-inch chipper running at 500 r.p.m. The over- and under-sized material and knots were removed from the good chips by a shaker screen and hand sorting. The entire batch of chips was thoroughly mixed and stored until used in covered metal containers in a cold room (40° F. and 93 percent relative humidity).

Pulping Experiments

For the initial stage in the delignification of the wood, two neutral sulfite semichemical digestions were made in a 1-1/2-cubic-foot, stainless steel-lined digester provided with a jacket for heating indirectly with steam. Previous to cooking, the equivalent of 10 pounds of moisture-free chips were given an impregnation treatment with 6.25 gallons of liquor containing approximately 127 grams per liter of sodium sulfite and 22.5 grams per liter of sodium carbonate in the form of the bicarbonate. For this treatment, the temperature was increased from that of the room to 120° C. and the pressure to 125 pounds per square inch in 30 minutes, where they were held for a 60-minute period. At the end of this time the excess liquor not absorbed by the chips was withdrawn and analyzed. The chips were then digested at relatively high temperatures with the absorbed liquor. Digestion 1389 was brought to the maximum temperature, 173° C., in 2 hours, where it was allowed to remain for 1 hour. Digestion 1390 was brought to the maximum temperature, 180° C., in 5 minutes, where it was again held for a period of 1 hour before cooling the digester. Digestion 1390 gave approximately the same yield as that of digestion 1389. The results are recorded in table 2.

The softened chips from each of the digestions were disintegrated in a small beater and washed. The pulps were allowed to remain in the beater

for only a short time without weight or pressure applied to the beater roll in order to avoid hydration of the pulp. The Schopper-Riegler freeness values of the disintegrated pulps were approximately 900 cc.

Previous to further purification by the holocellulose method, the pulps were screened through a small 8-cut screen plate, and tested for their strength properties, chlorine requirement, and chemical composition. The results of these tests are also reported in table 2. The data on cooking chemicals and yields are based on the calculated weight of moisture-free chips, while chlorine consumption and the results of chemical analysis are based on the weight of moisture-free pulp tested.

Testing the Pulps

The strength properties of the unbleached neutral sulfite semichemical pulps were determined by testing sheets prepared from stuff processed in 360-gram (moisture-free) portions in 23 liters of water (25° C.) for definite intervals in a 1-1/2-pound standard testing beater provided with a 6500-gram weight on the bedplate. For the unbleached holocellulose[6] and the bleached holocellulose and extracted pulps 5500-gram and 4500-gram weights, respectively, were used. The colors of the bleached and unbleached pulps were determined by an Ives tint photometer. All the pulps were chemically analyzed for their cellulose, alpha-cellulose, lignin, and pentosan contents, and for their chlorine requirements according to standard methods. The analytical data are recorded in table 3.

Purification of the Pulps by the Holocellulose Method

The two neutral sulfite semichemical pulps were thoroughly mixed and converted into unbleached holocellulose by chlorination with chlorine water and extraction of the lignin chloride with alkaline solutions. In order to minimize the formation of hydrochloric acid, which would cause undesired hydrolysis of the cellulose during the chlorination treatment, the chlorine water was freshly prepared before each chlorination by saturation of water at room temperature with chlorine gas. The pulp was divided into four 2.5-pound (moisture-free equivalent) batches for these treatments. The chlorine water added to a water suspension of the pulp was allowed to react for 15 minutes at room temperature, when the treated pulp was thoroughly washed on a 70-mesh wire screen and subsequently pressed to a moisture content of 75 percent. The pressed pulp was then extracted for 15 minutes in a steam-jacketed, glass-lined kettle with a hot (80° C.) 1 percent solution of monoethanolamine in 95 percent ethyl alcohol and washed with hot water until neutral to litmus.

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[6] Since the neutral sulfite semichemical pulps were refined by the holocellulose method, pulps so prepared will be referred to hereafter as holocellulose.

Table 1.--Physical and chemical data on Douglas-fir pulpwood. (Shipment 1528)

	Tree 2, log 11	Tree 1, log 11
	Location in tree	
Physical Data	Middle	Middle
Age (annual rings)	41	37
Rate of growth (rings per inch)	5.3	4.8
Density (pound per cubic foot)	24.9	27.0
Dryness[1] of wood (percent)	54.0	57.9
Heartwood by volume (percent)	59	56.6
Springwood by volume (percent)	78	71
Chemical Data	Percent	Percent
Total cellulose	60.6	61.3
Alpha cellulose	44.3	45.7
Lignin	27.3	25.6
Total pentosans	5.8	8.5
Pentosans in cellulose	6.4	7.1
Solubility in:		
Alcohol-benzene	2.6	4.0
Ethyl ether	1.5	1.1
1 percent NaOH	14.5	11.7
Hot water	4.5	4.7

[1] Moisture content = 100 - Dryness value.

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Table 2.--Neutral sulfite semichemical pulping data on young-rapid growth Douglas-fir (tree 2, log 11), shipment 1526

Digestion Number	Cooking and yield data: Weight of wood: Moist (Lb.)	Weight of wood: Moisture free (Lb.)	Impregnating liquor: Before penetration: Volume (Gal.)	Before penetration: Concentration: Na_2SO_3 (Grams per liter)	Before penetration: Concentration: $NaHCO_3$ (Grams per liter)	After penetration: Volume (Gal.)	After penetration: Concentration: Na_2SO_3 (Grams per liter)	After penetration: Concentration: $NaHCO_3$ (Grams per liter)	Penetration Duration: To maximum temperature (Min.)	Penetration Duration: At maximum temperature 120° C. (Min.)	Cooking chemical absorbed per 100 pounds of moisture-free chips: Na_2SO_3 (Lb.)	Cooking chemical absorbed: $NaHCO_3$ (Lb.)	Cooking chemical absorbed: Total (Lb.)	Duration of digestion: To maximum temperature (Min.)	Duration of digestion: At maximum temperature (Min.)	Maximum digestion temperature (° C.)	Spent liquor: Concentration: Na_2SO_3 (Grams per liter)	Spent liquor: Concentration: $NaHCO_3$ (Grams per liter)	Total yield: Moisture-free pulp (after breaking up in beater) (Per cent)	Physical properties, bleach requirement and chemical analysis of pulps: Test beater strength data on unbleached pulp. Tests made at 75° F. and 50 percent relative humidity: Time of treatment (Min.)	Freeness (Schopper) (cc.)	Ream weight (25 x 40 - 500) (Lb.)	Burst factor (Pt. per lb. per ream)	Tear factor (Pt. per lb. per ream)	Tensile strength (Lb. per square inch)	Solid fraction (Ratio)	Folding endurance (Double fold)	Stretch (Per cent)	Test of bleach requirement: Standard bleach powder added (Per cent)	Ives reading: Red (Parts)	Ives reading: Green (Parts)	Ives reading: Blue (Parts)	Chemical analysis: Cellulose: Total (Per cent)	Cellulose: Alpha- (Per cent)	Lignin (Per cent)	Total pentosans (Per cent)	Chlorine Number (Roe) (Per cent)
																				0	895	54.0	0.00	0.00	0	.11	0	0	0	42	29	23	72.6	59.6	18.6	7.5	21.5
																				20	890	54.5	.26	1.45	1085	.21	3	2.5	25	59	42	31					
1389	13.73	10.00	6.25	126.8	22.9	5.57	109.9	19.6	30	60	15.0	2.7	17.7	120	60	173	.....	......	77.7	40	840	51.3	.51	1.24	2620	.27	51	3.0	50	67	53	41					
																				60	645	52.1	.69	.97	4095	.36	76	3.0									
																				80	405	51.0	.71	.72	6450	.42	86	3.5									
																				0	900	57.0	0.05	0.60	0	.12	0	0	0	49	34	27	73.8	60.8	18.8	7.1	22.0
																				20	890	52.0	.28	1.57	1115	.22	5	2.8	25	62	45	33					
1390	14.42	10.00	6.25	127.2	21.9	5.21	107.9	16.9	30	60	19.3	3.6	22.9	5	60	180	21.7	5.6	76.7	40	845	53.2	.49	1.43	2550	.28	59	3.0	50	72	60	44					
																				60	670	52.8	.63	1.05	4610	.35	100	3.5									
																				80	445	51.7	.72	.82	6405	.44	93	3.5									
																				100	295	52.4	.77	.59	7860	.49	86	3.5									

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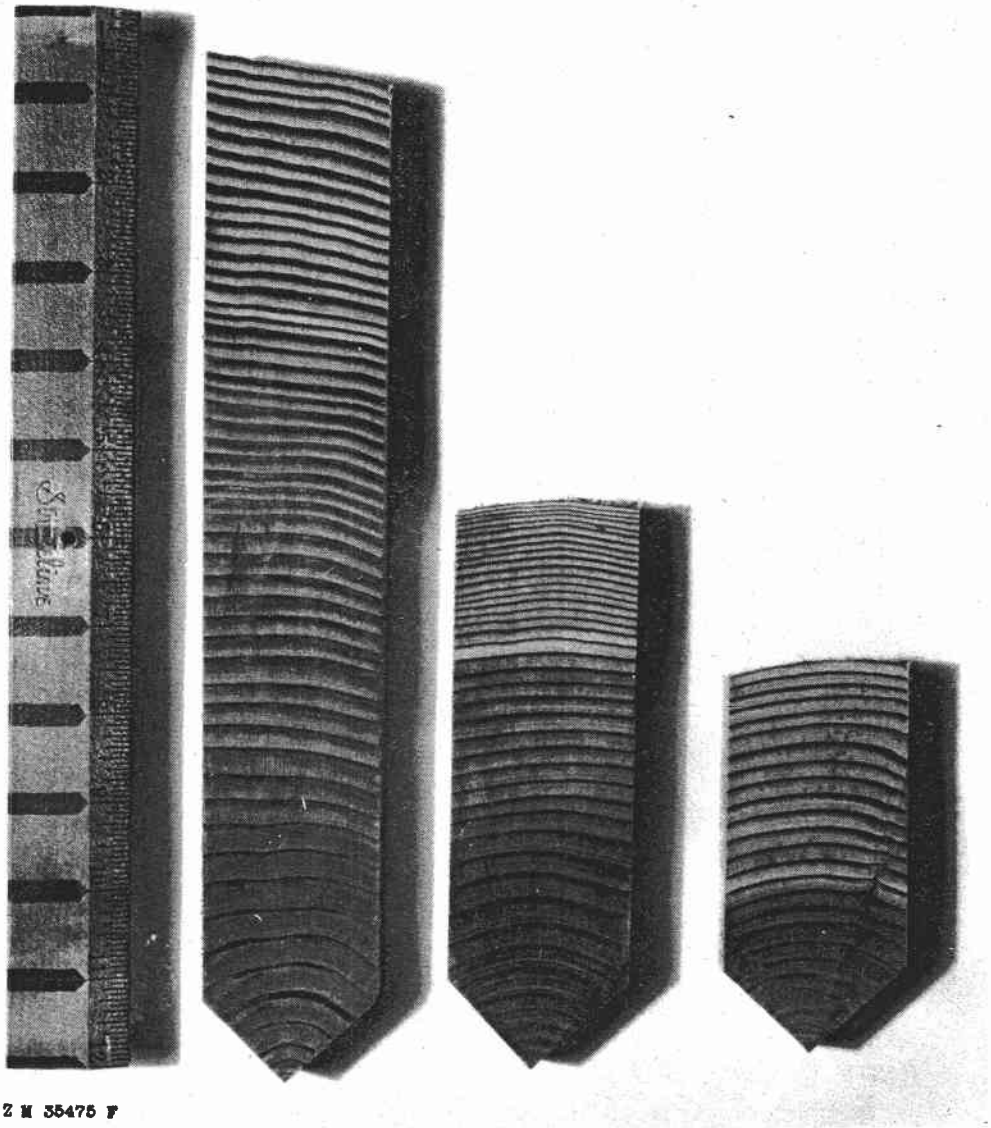

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Figure 1.--Portions of disks cut from the butt, middle (log 11), and top logs, respectively, of tree 2, Douglas-fir pulpwood. Shipment 1528.

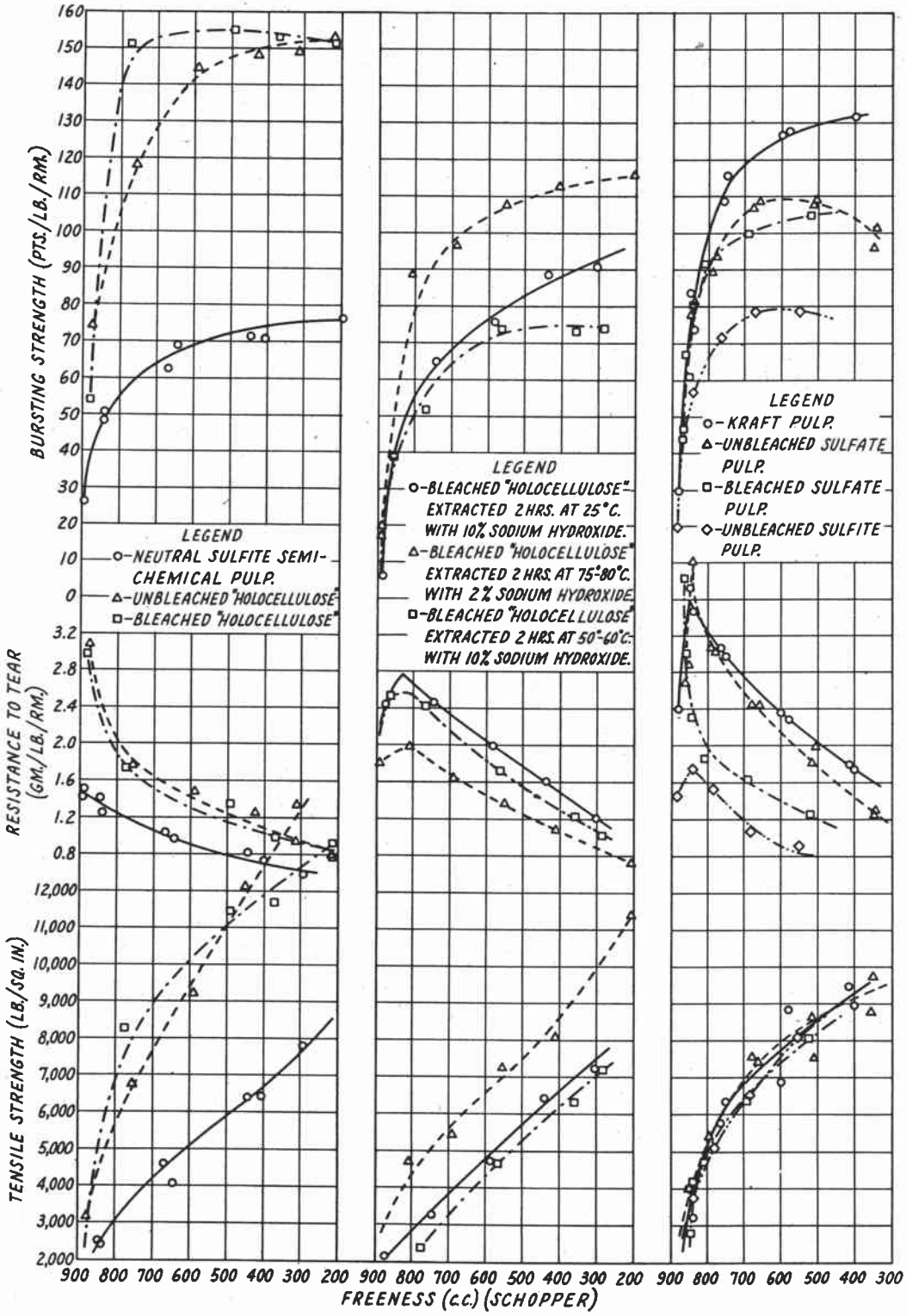

FIG. 2
COMPARISON OF CERTAIN STRENGTH PROPERTIES
OF DOUGLAS-FIR "HOLOCELLULOSE" PULPS
WITH THOSE OF DOUGLAS-FIR SULFATE AND SULFITE PULPS

The pulp was again chlorinated and washed as before but this time it was extracted with a hot 1 percent solution of sodium sulfite in 60 percent ethyl alcohol and again thoroughly washed with hot water. This treatment was repeated to remove the last traces of lignin.

All four batches of holocellulose pulp were combined and thoroughly mixed for the determination of yield, physical properties, and chemical composition according to standard methods. (Table 3.)

The holocellulose, having a permanganate number of 1.5 was bleached in two stages to prevent its degradation. The bleaching was done at approximately 2 percent consistency in a glass-lined tank provided with a motor-driven stainless steel agitator. In the first stage of bleaching, 0.6 percent of calcium hypochlorite solution, equivalent to 0.21 percent of chlorine on the moisture-free weight of pulp charged, was added. Although the bleaching agent was not entirely exhausted, the pulp was removed from the bleacher and washed after the reaction had continued for approximately 30 minutes. In the second stage of bleaching, 0.5 percent of bleaching powder or 0.18 percent of chlorine was added and the pulp treated as before. The washed, bleached holocellulose was then chemically analyzed and subjected to the same physical tests as the unbleached product. (Table 3.)

Treatment of the Holocellulose

Owing to the rapid hydrating properties imparted by the hemicelluloses retained in the holocellulose, certain portions of the hemicelluloses must be removed in order to obtain a pulp suitable for the higher grades of paper. Various portions of the hemicelluloses were, therefore, extracted, first from 10-gram batches of the holocellulose for chemical analysis and later from 1.5-pound batches for strength properties as well by digesting at various temperatures with caustic soda solutions of several concentrations.

For the extraction treatments, samples of bleached holocellulose were digested for 2 hours with 2, 3, 4, and 10 percent solutions of caustic soda. The moisture content of the holocellulose was included in calculating the concentration of the caustic soda solutions. For example, the ratio of caustic soda to moisture-free holocellulose was 0.3 for the 2 percent extraction and 1.5 for the extractions with 10 percent caustic. The temperatures employed were varied from 25° C. to 95° C. For the 1-1/2-pound extractions at elevated temperatures, the pulps were treated in a steam-jacketed, glass-lined kettle equipped with a mechanical stirrer. The extraction at room temperature was done in a 10-gallon earthenware jar without stirring so as to minimize oxidation of the pulp. Strength tests and chemical analyses were made on the pulps by standard methods. The data obtained are reported in table 3.

Results and Discussion

Yield and Chemical Composition

According to the data in tables 2 and 3, the yield of moisture-free softened chips resulting from the first stage of delignification by the neutral sulfite semichemical process averaged 77.2 percent for the two digestions; about one-half of the lignin present in the original wood was removed and 4.1 percent of the cellulose (Cross and Bevan) was solubilized by this treatment. The loss in pentosans (0.16 percent) was much less in proportion to the cellulose removal. Indicative of the mild action of neutral sulfite liquors was the apparent gain (2.15 percent) in the alpha-cellulose content of the Cross and Bevan cellulose obtained from the neutral sulfite semichemical pulps in comparison with that from the original wood.

Purification of the semichemical pulp by the holocellulose method resulted in a yield of 57.2 percent of a lignin-free product containing 100 percent cellulose (Cross and Bevan) and 49.8 percent alpha cellulose. As a result of the holocellulose treatment, the alpha-cellulose content showed a further increase of 3.2 percent over that obtained from the Cross and Bevan cellulose prepared from the semichemical pulp. Thus, nearly 50 percent of the moisture-free weight of the wood was recovered as alpha cellulose. The cuprammonium viscosity of the unbleached holocellulose fraction was 43.5 centipoises which, together with the high yield of alpha-cellulose, indicated but little degradation. The pentosan content of the unbleached "holocellulose" showed a 1.85 percent decrease in comparison with that of the original wood. Most of this loss (1.7 percent) resulted from purification by the holocellulose method rather than as a result of cooking with neutral sodium liquors.

Bleaching the holocellulose with 1.1 percent (0.39 percent chlorine) of standard bleaching powder resulted in no measurable loss in yield or viscosity of the holocellulose, in 0.3 percent loss in alpha cellulose, and in less than 0.2 percent loss in pentosans.

Extraction of portions of the hemicelluloses from the bleached holocellulose with caustic soda solutions for various periods at several temperatures reduced the yield from 57.2 percent to 47.3 percent, depending upon the concentration and temperature of extraction. By this treatment the alpha cellulose in the pulps was increased from 86.7 percent in the bleached holocellulose up to 95.4 percent in one of the extracted pulps. Based on the original wood, losses in alpha cellulose were from 3.1 percent to 7.1 percent, depending upon the degree of treatment.

The degree of degradation is also indicated by the cuprammonium viscosity of the alkali extracted pulps. Viscosity values for the 10-gram experimental extractions were quite uniform for the different degrees of treatment and approximately the same as that of the bleached holocellulose, indicating no degradation of the extracted pulps. On the other hand, the viscosity values of the pulps resulting from the larger-scale extractions (1.5 pounds of pulp) were considerably decreased, especially in the cases where the pulps were stirred during extraction, at the higher temperatures.

Table 3.--Chemical analysis and physical properties of selected pulps from Douglas-fir (Shipment 1528)

Test number	Digestion number	Description and treatment of material	Total yield on moisture-free wood	Total cellulose (Cross and Bevan) In sample	Total cellulose (Cross and Bevan) On moisture-free wood	Alpha-cellulose In sample	Alpha-cellulose On moisture-free wood	Lignin In pulp	Lignin On moisture-free wood	Total pentosans in sample	Chlorine Number (Roe)	Viscosity	Bleaching data: Standard bleach added	Ives Reading: Red	Ives Reading: Green	Ives Reading: Blue	Interpolated beater strength data on pulp. Tests made at 75° F. and 50 percent relative humidity: Freeness (Schopper)	Time of treatment	Ream weight (25 x 40-500)	Burst factor	Tear factor	Tensile strength	Solidification ratio	Folding endurance	Stretch	Permanganate number
Number	Number		Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent		c.p.		Parts	Parts	Parts	c.c.	Minutes	Pounds	Points per pound per ream	Points per pound per ream	Pounds per square inch	Ratio	Double folds	Percent	Number
7015		Douglas-fir wood tree 2 log 11	100	60.6	60.6	73.1	44.3		27.3	5.8																
7783	1389	Neutral sulfite semi-chemical pulp	77.7	72.6	56.4	59.6	46.3	18.6	14.5	7.5	21.5		0	42	29	23	895	0	54	0.00	0	0	0.11	0	0	
													25	59	42	31	700	58	51	.63	1.02	4200	.34	76	3.0	
													50	67	53	41	550	70	51	.70	.88	5550	.39	81	3.3	
7784	1390	Neutral sulfite semi-chemical pulp	76.7	73.8	56.6	60.8	46.6	18.8	14.4	7.1	22.0		0	49	34	27	900	0	57	.05	.60	0	.12	0	0	
													25	62	45	33	700	58	53	.63	1.02	4200	.34	100	3.5	
													50	72	60	44	550	70	51	.70	.88	5550	.39	90	3.5	
7824		Hologellulose (unbleached) prepared from cooks 1389-90	57.2	100	57.2	87.1	49.8	0	0	6.9		43.5	0	77	68	60	875	0	58	.74	3.08	3200	.34	225	7.3	1.5
													1.0	86	82	80	700	24	54	1.27	1.64	7600	.46	488	6.0	
																	550	32	53	1.44	1.32	10100	.50	450	6.6	
7823		Bleached hologellulose	57.2			86.7	49.5			6.6		43.0	1.1	84	80	79	880	0	60	.54	2.98	2530	.34	69	5.5	1.0
																	700	25	54	1.51	1.64	9250	.44	500	5.7	
																	550	36	53	1.55	1.36	10800	.49	400	6.7	
7823	A	Bleached hologellulose extracted with 2 percent NaOH 2 hours at 95° C.	52.8			86.4	45.6			5.93		46.8														
7823	B	Bleached hologellulose extracted with 2 percent NaOH 3 hours at 95° C.	53.0			86.2	45.6			5.76		44.0														
7823	C	Bleached hologellulose extracted with 3 percent NaOH 3 hours at 95° C.	50.9			87.1	44.3			6.21		42.1														
7823	D	Bleached hologellulose extracted with 4 percent NaOH 3 hours at 95° C.	49.7			88.4	43.9			6.18		44.3														

(Continued)

Table 3.--Chemical analysis and physical properties of selected pulps from Douglas-fir (continued)
(Shipment 1528)

Test Number	Digestion Number	Description and treatment of material	Total yield on moisture-free wood	Total cellulose (Cross and Bevan): In sample	Total cellulose (Cross and Bevan): On moisture-free wood	Alpha-cellulose: In sample	Alpha-cellulose: On moisture-free wood	Lignin: In pulp	Lignin: On moisture-free wood	Total pentosans in sample	Chlorine Number (Roe)	Viscosity	Bleaching data: Standard bleach added	Bleaching data: Ives Reading: Red	Bleaching data: Ives Reading: Green	Bleaching data: Ives Reading: Blue	Interpolated beater strength data on pulp Tests made at 75° F. and 50 percent relative humidity: Freeness (Schopper)	Time of treatment	Ream weight (25 x 40-500)	Burst factor	Tear factor	Tensile strength	Solidfraction	Folding endurance	Stretch	Permanganate
Number	Number		Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent		c.p.		Parts	Parts	Parts	c.c.	Minutes	Pounds	Points per pound per ream	Points per pound per ream	Pounds per square inch	Ratio	Double folds	Percent	Number
7888	E	Bleached holocellulose extracted with 2 percent NaOH 2 hours at 75-80° C.	51.3			88.0	45.1			5.08		26.5	0	80	76	71	890	0	60	.17	1.80	900	.28	3	5.5	
																	700	32	54	.99	1.76	5700	.46	400	6.5	
																	550	40	53	1.08	1.40	7100	.49	530	7.0	
7921	F	Bleached holocellulose extracted with 10 percent NaOH 2 hours at 50-60° C.	47.3			89.6	42.4			1.73		20.2	0	90	88	85	895	0	66	.00	.35	0	.18	0	0	
																	700	96	54	.65	2.20	1400	.40	113	4.5	
																	550	123	54	.74	1.72	4700	.44	400	6.0	
7920	G	Bleached holocellulose extracted with 10 percent NaOH 2 hours at 25° C.	48.7			95.4	46.4			1.60		36.7	0	88	86	83	885	0	62	.06	.58	101	.20	0	1.5	
																	700	114	54	.69	2.53	3800	.38	192	5.0	
																	550	148	62	.81	1.88	5100	.42	449	5.5	
7174	1199 1200 1201	Kraft pulp (unbleached)	48.5	92.3	44.8	76.7	37.2	5.2	2.5	7.2	5.3		0 25	38 76	27 62	22 48	880	0	61	.30	2.40	1344	.26	6	5.0	
																	700	48	57	1.21	2.70	7000	.44	2000	7.5	
																	550	64	54	1.28	2.10	8400	.49	3300	6.0	
7323	2514 2515	Bleachable sulfate pulp (unbleached)	44.2	96.6	42.7	82.5	36.5	2.4	1.1	6.4	2.8	38.8	0 25	46 90	36 85	32 80	860	0	64	.34	2.70	1590	.28	9	4.8	
																	700	57	56	1.06	2.60	6700	.46	1500	6.3	
																	550	75	55	1.09	2.00	8100	.51	1400	5.5	
7339	2514 2515	Bleached sulfate pulp. Bleach No. 520				89.7						9.8	[3] 22.5	90	88	86	870	0	57	.47	3.8	1975	.30	94	5.8	
																	700	49	56	1.00	1.6	6400	.45	903	6.3	
																	550	68	58	1.05	1.3	7700	.50	931	5.2	
7240	3900	Sulfite pulp. (unbleached) (tree 1 log 11)	51.1	93.6	47.8	78.3	40.0	2.3	1.2	4.1	4.8	59.8 15.1	0 [2] 25	49 90	37 86	31 81	880	0	59	.23	1.5	1786	.34	4	3.0	
																	700	28	57	.77	1.2	6300	.49	481	4.5	
																	550	40	60	.79	.9	8100	.54	413	4.8	
7450	A	Bleached spruce holocellulose. From asplund fiberized wood, extracted with 5 percent NaOH at 95° C.	58.1			89.6	52.0					37.4	.5	92	90	86	870	0		.46	2.7	2200	.35	50		
																	700	60		1.34	1.52	8000	.57	1400		
																	550	83		1.43	1.30	9000	.54	1450		

[1] Prepared from neutral sulfite semichemical pulps (1389 and 1390) by the holocellulose method.

[2] Bleached in one stage with calcium hypochlorite solution.

[3] Bleached in five stages by chlorination, caustic extraction, calcium hypochlorite, and sulfur dioxide.

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Oxidation of the cellulose may have resulted from such a drastic treatment, as the 1.5-pound batch treated at room temperature with 10 percent sodium hydroxide solution without stirring showed much less evidence of degradation.

The concentration of caustic soda in the extraction liquors had considerable effect on the pentosan content of the extracted pulps. Low concentrations of caustic soda decreased the pentosan content only slightly, whereas the higher concentrations (10 percent) reduced the pentosan content of the pulps from 6.6 percent in the bleached holocellulose to 1.6 percent in the alkali extracted holocellulose.

The concentration of sodium hydroxide used for extraction of the hemicelluloses had more effect on the yield of pulp than did the temperature of extraction. Where the temperature of extraction was the same, an increase in concentration of caustic soda decreased the yield appreciably, whereas the decrease in yield with increase in temperature of treatment was markedly less when the concentration was the same.

Color of Pulps

Purification of the semichemical pulps by the "holocellulose" method increased the color of the pulps from 25 Ives parts blue to 60. The chlorine requirement of the holocellulose was approximately 0.35 percent, whereas that for the Douglas-fir bleachable sulfate pulp was 8.75 percent.

Upon bleaching the holocellulose with 1.1 percent of calcium hypochlorite, an Ives blue reading of 79 was obtained. Extraction of the bleached holocellulose with a 2 percent caustic soda solution for 2 hours at approximately 75° C. decreased this value to 71 parts (Ives blue reading). Of special interest, however, are the results obtained from the removal of the hemicelluloses by extraction with 10 percent caustic soda solution. The color of one of the extracted pulps was increased from a whiteness of 79 parts (Ives blue reading) to 85 and that of the other to a whiteness of 83.

Physical Properties

Except for folding endurance, the strength properties of the Douglas-fir holocellulose prepared from neutral sulfite semichemical pulps were equal or superior to spruce holocellulose[3] made from Asplund fiberized wood. (Table 3, and fig. 2.) They were much stronger than the semichemical pulps from which they were made, and in certain respects they were superior to sulfate and sulfite pulps made from the same batch of wood. Mild bleaching treatments did not materially decrease the strength properties of the holocellulose.

Removal of the hemicelluloses by extraction of the bleached holocellulose with caustic soda solutions greatly retarded both the rate of hydration as measured by Schopper freeness and strength development. This treatment resulted in decreasing the bursting and tensile strength and in

materially increasing the tearing strength of the bleached holocellulose pulp. Upon removal of the hemicellulose and consequent increase in alpha content of the pulps by alkali extraction, the bulkiness of the pulps also increased.

Thus it is possible to produce white pulps of high yield and strength from Douglas-fir, and to control their physical and chemical properties.