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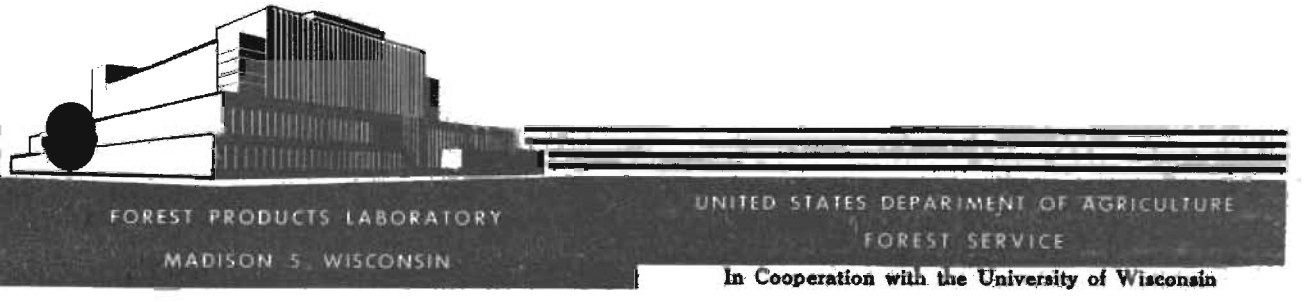

KRAFT PULPING OF WEST FLORIDA SAND PINE AND LONGLEAF PINE

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Summary

Kraft pulps made from the open-cone form of sand pine (*Pinus clausa*, Chapm. Vasey) that grows in western Florida were higher in overall strength and brightness and essentially equal in cooking conditions, bleaching requirements, and yields by weight to kraft pulps made from longleaf pine (*Pinus palustris* Mill.) of equal-growth rate from the same area. Satisfactory bag paper and pulp strength data indicated that sand pine kraft pulps would be suitable for unbleached and bleached kraft papers of high strength.

Introduction

Sand pine (*Pinus clausa*, Chapm. Vasey) of western Florida is commonly known as both Choctawhatchee sand pine and west Florida sand pine. The characteristic open cones of the west Florida trees differentiate this type of sand pine from another type known as Ocala pine, which has closed cones and grows in other parts of Florida. No morphological differences have been found between the two types of sand pine trees from geographic areas in Florida, but common names are used to distinguish the races.[2,3]

Previous work[4,5] at the U.S. Forest Products Laboratory showed that the Ocala type of sand pine produced good kraft pulp and demonstrated the possibility of

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

[2] Little, Elbert L., Jr., and Dorman, Keith W. Geographic Differences in Cone-opening in Sand Pine. Journal of Forestry 50: 204, 1952.

[3] Cooper, Robert W. Silvical Characteristics of Sand Pine. U.S. Forest Service. Southeastern Forest Experiment Station, Station Paper No. 86, September 1957.

[4] Wells, Sidney D., and Rue, John D. The Suitability of American Woods for Paper Pulp. U.S. Department of Agriculture, Department Bulletin No. 1485: 35, May 1927.

[5] Bray, Mark W., and Martin, J. S. Pulping Florida Sand Pine (*Pinus clausa*) for Kraft, High-grade Papers, and Newsprint. Southern Pulp and Paper Journal 5 (1): 7, June 1942.

using the pulp in papers of high grade. Ocala sand pine trees have been cut commercially for pulpwood since 1940.[6] However, the west Florida sand pine is not considered acceptable by all pulpwood users. The production of kraft pulps of acceptable overall quality from only west Florida sand pine or from mixtures of the sand pine with longleaf pine (Pinus palustris Mill.) could be a factor in the greater use of this sand pine as a pulpwood.

Sand pine grown in western Florida was evaluated for kraft pulping by determining: (1) Certain physical and chemical properties of the wood, (2) kraft pulping characteristics over a permanganate number range of 33 to 17, (3) properties of kraft pulp and bag papers, (4) similar factors for longleaf pine of equal rate of growth from the same general area, and (5) pulping characteristics of a mixture of equal parts of the two species.

Woods

Sand pine and longleaf pine pulpwoods were received at the Forest Products Laboratory in the form of peeled logs approximately 5 feet long. The west Florida sand pine shipment was cut from a site that had dry, sandy soil and was at an elevation of 150 feet above sea level in Walton County, Fla. The area had been burned over before 1950. The stand contained 100 percent sand pine, and the trees were in clumps. Shipment selection was made from clumps that had a stand density of 250 trees per acre. The average diameter of the trees at breast height was 7 inches. The longleaf pine shipment was cut from a site that had a mixture of moist sand and clay soil and was at an elevation of 100 feet above sea level in Okaloosa County, Fla. The stand was 100 percent longleaf pine and had 200 trees per acre, averaging 6 inches in diameter at breast height.

Sections 1 inch thick were cut from representative logs and used for the determination of the physical properties of the wood. Each type of wood was converted to chips, which were screened to remove undersize and oversize material. The normal chip size averaged five-eighths inch long. The chemical constituents of each wood were determined on a representative sample of chips.

The physical properties and chemical compositions of the sand pine and longleaf pine are given in table 1.

The pulpwoods of west Florida sand pine and longleaf pine were essentially equal in age (24 years), diameter (7 inches), and growth rate (7 rings per inch). Sand pine had 72 percent springwood, 52 percent heartwood, and a specific gravity of 0.46. Corresponding values for longleaf pine were 62 percent springwood, 15 percent heartwood, and a specific gravity of 0.52. Chip density per cubic foot was 12.6 pounds for sand pine and 14.5 pounds for longleaf pine. The values for chip densities were in direct proportion to those for the specific gravity

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[6] Cooper, Robert W. Sand Pine Regeneration in Florida. Proceedings of the Society of American Foresters, Syracuse, N.Y.: 71, 1957.

of the woods. Chemical composition of the two species differed only in alpha-cellulose and pentosan contents. Longleaf pine had more alpha-cellulose, or 55.9 percent, than sand pine's 51.5 percent; sand pine, however, had more pentosans, or 14.4 percent, than the 12.7 percent for longleaf pine.

Procedure

Kraft Pulping

Kraft pulping experiments on sand pine and longleaf pine were made to produce types of fully cooked pulps with ranges in yield and permanganate numbers that would include high-strength kraft and bleachable-grade kraft. One pulp was made from a mixture of equal parts by weight (moisture-free) of these species.

For the determination of pulp yields, portions of 5/8-inch chips equivalent to 6.0 pounds of moisture-free wood were cooked in cylindrical, tumbling digesters of 0.8-cubic-foot capacity. The steam-jacketed digesters were heated indirectly with steam. Calculations of volumes and chemical concentrations included the moisture in the chips. A series of digestions was made in which active alkali that was calculated as sodium oxide was varied from 14.0 to 20.0 percent of wood on a moisture-free basis. The cooking conditions are given in table 2.

After cooking for the predetermined time, the pulped chips were dumped from the digester, broken up, washed with hot water, screened through a 12-cut (0.012-inch slot width) flat screen, pressed to approximately 30 percent dryness, and sampled for moisture determination. Yields of screened pulps and screenings were based on the original wood and calculated on a moisture-free basis.

After the chemical requirement for the high-strength kraft pulp had been determined in small-scale digestions, both sand pine and longleaf pine pulps of this grade were made in the pilot plant. Chips that were equivalent to 120 pounds of moisture-free wood were cooked in a 14-cubic foot, cylindrical, tumbling digester heated indirectly with steam. At the end of the cooking period, the contents of the digester were blown into a blow tank, washed with hot water, and screened through 12-cut plates of a flat screen. The screened pulp was run over a vacuum washer to reduce the water content to about 75 percent.

Digestions (4401 and 4402) made in the pilot plant for papermaking experiments had the same cooking conditions as small-scale digestions 4186X, 4190X, 4192X, and 4196X listed in table 2, and represented a sand pine kraft pulp with a permanganate of 29.8. A similarly cooked longleaf pine pulp (digestions 4399 and 4400) had a permanganate number of 27.4 and the same cooking conditions as digestions 4205X, 4207X, 4212X, and 4214X listed in table 2. Yield was not determined on the pilot-plant pulps.

Bleaching

A beater-scale bleach by the standard five-stage bleaching procedure for kraft pulp was made on both sand pine and longleaf pine pulps with permanganate

numbers close to 19. The bleaching sequence and chemical requirements for each stage are given in table 4.

Papermaking

Fifty-pound bag papers were made on the 13-inch experimental paper machine from: (1) Sand pine kraft pulp, (2) longleaf pine kraft pulp, and (3) an equal mixture of these pulps. Each pulp was processed by a single pass through a high-angle, conical refiner to reduce freeness. Sulfuric acid was used to reduce the pH of the stock to 6.5. Six-tenths of one percent of rosin size was added to the stock and the pH adjusted to 5.0 with alum.

Results and Conclusions

Evaluation of Unbleached Pulps

The small-scale experiments by the kraft process were designed to produce pulps of high strength and bleachable grades by varying the amount of active alkali over the range from 14.0 to 20.0 percent of the weight of the moisture-free wood. This increase in active alkali for the sand pine digestions (table 2) caused an increase in pulp brightness, and decreases in permanganate number from 33 to 17, in screening rejects from 3.3 to 0.1 percent, in yield by weight of screened pulp from 48 to 44 percent, and in bursting strength from 87 to 78. An excessive amount of screening rejects were obtained with the lowest percentage of active alkali.

The pulp made with 15 percent of active alkali had a satisfactory percentage of screenings, a good screened yield by weight of 47 percent, and a permanganate number of about 29. The bleachable-grade pulp had an active alkali requirement of 18.0 percent, a yield of 45 percent, and a permanganate number of 19. The tearing resistance and folding endurance values were the highest obtained in the sand pine series. A further increase in the percentage of active alkali that was used for pulping caused an additional increase in pulp brightness, but resulted in a decrease in yield, permanganate number, and strength.

The series of longleaf pine digestions showed that longleaf pine and sand pine had the same chemical requirement for pulping, and that the pulps were essentially equal in permanganate number and screenings. Pulps from both species had maximum tearing resistance for the individual series when they were cooked with 18.0 percent of active alkali. The sand pine pulps were about two percentage points higher in brightness than the longleaf pine pulps. For practical purposes, the weight yields of the sand pine and longleaf pine pulps would be considered equal, because the weight yields of the longleaf pine pulps were less than one percentage point higher than those of the sand pine pulps. Volume yields, given in table 2 and calculated in pounds per cubic foot of solid wood, were higher for the longleaf pine pulps than for the sand pine pulps, because of the 12 percent higher density of the longleaf pine wood.

Although there were some variations at different freeness levels, the strength properties of the sand pine pulps (tables 2 and 3) were generally higher in bursting strength, folding endurance, and breaking length, but lower in tearing resistance, than longleaf pine pulps. At the intermediate Canadian Standard freeness of 450 milliliters, the sand pine pulps were about 20 percent higher in bursting strength, 10 percent higher in breaking length, and 10 percent lower in tearing resistance than the longleaf pine pulps; the pulps from sand pine were also much higher in folding endurance. Even though the pulps of both species required nearly equal beating times to reach a given pulp freeness, somewhat higher densities were obtained for test sheets made from the sand pine pulps.

A kraft pulp made with 15 percent of active alkali from the mixture of equal parts of sand pine and longleaf pine was equal in screened pulp yield by weight, screenings, and permanganate number to similarly cooked pulps from the individual species. The pulp made from the mixture was a little higher in brightness, bursting strength, and breaking length, but lower in tearing resistance than the corresponding longleaf pine pulp.

The pulping results indicated that sand pine is essentially equal to longleaf pine in kraft cooking requirements and pulp yields by weight. Sand pine pulps have higher overall strength and brightness than those from longleaf pine. Yields on a volume basis reflected the 12 percent lower density of the sand pine wood.

Evaluation of the Bleached Pulps

Both sand pine and longleaf pine pulps could be bleached to about the same brightness by applying equal dosages of 9.5 percent chlorine equivalent bleaching chemical. Yield of pulp was determined in duplicate on separate small-scale bleaches and gave 93.8 percent based on the weight of sand pine unbleached pulp, and 42.3 percent based on the weight of sand pine wood. Corresponding yields for the longleaf pine pulp were 92.3 and 42.5 percent. Strength properties of both bleached pulps showed a tendency toward higher burst and breaking length, and lower tearing resistance and folding endurance than their unbleached pulps, but the overall strength of each was equal to that of its unbleached pulp.

Evaluation of Bag Papers

The bag paper made from sand pine pulp had adequate tear, tensile, and air resistance to meet requirements of a commercial specification for multiwall sack paper (table 5); the bag paper made from longleaf pine pulp, however, was much higher in tear, better in porosity, and a little lower in tensile strength than the paper made from sand pine pulp. The bag paper made from a mixed pulp of both pine species had adequate tensile and nearly sufficient tear strength to meet the specification requirements. Minor changes in pulp processing would be expected to improve the paper properties of these two pulps to fully comply

with the specifications. Compared with a commercial bag paper, these experimental papers were lower in bursting strength. While this is not a requirement in the specification for multiwall bag paper, it is necessary for wrapping paper. The three experimental papers had adequate strength for grade B kraft wrapping paper.

Papermaking experiments and pulp strength data indicated that sand pine pulps would be suitable for unbleached and bleached kraft papers of high strength.

Table 1.--Physical properties and chemical composition of west Florida sand pine and longleaf pine

Item	Wood	
	Sand pine[1]	Longleaf pine[2]
Physical properties (average)[3]:		
Diameter of logs without bark.....inches	6.7	7.2
Age of logs.....years	24	24
Rate of growth.....rings per inch	7.3	7.0
Specific gravity[4].....	0.46	0.52
Density[4].....lb. per cu. ft.	28.7	32.4
Heartwood content (by volume).....percent	52	15
Summerwood content (by volume).....percent	28	38
Chemical composition[5]:		
Lignin.....percent	28.4	29.2
Holocellulose.....percent	72.2	72.1
Alpha-cellulose.....percent	51.5	55.9
Pentosans.....percent	14.4	12.7
Solubility in:		
Alcohol-benzene.....percent	3.1	2.3
1 percent sodium hydroxide.....percent	11.4	11.0
Hot water.....percent	2.2	1.2
Ash.....percent	0.4	0.3

[1] Shipment 4765. Choctawhatchee race of sand pine (*Pinus clausa*, Chapm. Vasey).

[2] Shipment 4766. Longleaf pine.

[3] Physical tests made on disks cut from logs 5 feet long.

[4] Moisture-free weight and green volume.

[5] Analysis made on samples of chips used for pulping. Percentages based on moisture-free wood.

Table 2.--Kraft pulping of west Florida sand pine and longleaf pine

Digestion No.	Active alkali added[1]	Yield[2] By volume of screened pulp	Yield[2] By weight: Screened pulp	Yield[2] By weight: Screenings	Unbleached pulp: Permanganate number	Unbleached pulp: Brightness	Strength of unbleached pulp at 450 milliliters, C.S.F.[3]: Burst factor	Tear factor	Folding endurance (M.I.T.)	Breaking length
	Percent	Lb. per cu. ft.	Percent	Percent		Percent			Double folds	M.
SAND PINE										
4185X, 4199X	14.0	13.8	47.6	3.3	32.8	22.7	87	144	980	10,400
4186X, 4190X, 4192X, 4196X	15.0	13.7	47.3	1.3	29.1	24.1	85	153	1,250	10,700
4191X	17.4	13.3	46.0	.4	22.3					
4193X, 4195X	18.0	13.1	45.1	.3	19.4	27.5	81	164	1,300	10,000
						[4]89.6	[4]86	[4]148	[4]1,100	[4]10,400
4188X	19.0	13.0	44.7	.2	17.9					
4189X, 4218X	20.0	12.7	43.9	.1	16.6	31.3	78	152	1,020	10,400
LONGLEAF PINE										
4211X, 4215X	14.0	15.8	48.4	2.7	31.5	20.6	75	168	690	9,600
4205X, 4207X, 4212X, 4214X	15.0	15.6	47.9	1.3	28.8	21.6	71	168	780	9,700
4202X	15.5	15.6	47.9	.8	27.4					
4201X, 4206X	18.0	15.0	46.0	.2	19.2	25.4	65	188	790	9,700
						[4]89.5	[4]76	[4]174	[4]760	[4]9,900
4216X, 4217X	20.0	14.6	44.7	.1	16.8	29.2	67	161	820	9,100
SAND PINE AND LONGLEAF PINE MIXTURE (EQUAL PARTS)[5]										
4219X, 4220X	15.0	14.7	47.7	1.4	28.7	23.1	78	144	730	10,500

[1] Digestions were made in steam-jacketed, cylindrical, tumbling digesters of 0.8-cubic-foot capacity, heated indirectly with steam. Constant cooking conditions were: Liquor-to-wood ratio, 4 to 1; sulfidity based on active alkali, 25 percent; time from 30° to 170° C., 1.5 hours; and time at 170° C., 1.5 hours.

[2] Yield by weight on a moisture-free basis. Yield by volume based on moisture-free weight of pulp, moisture-free weight of wood, and green volume of wood.

[3] Strength values for 450 milliliters Canadian Standard freeness are interpolated from beater test curves.

[4] Values for bleached pulp.

[5] Mixture of equal parts by weight (moisture-free).

Table 3.--Strength and other physical characteristics[1] of unbleached and bleached kraft pulps from sand pine and longleaf pine

Digestion No.	Properties of pulp							
	Permanganate number	Beating time	Freeness	Burst factor	Tear factor	Folding endurance (M.I.T.)	Breaking length	Sheet density
		Min.	Ml.			Double folds	M.	G. per cc.
SAND PINE UNBLEACHED PULP								
4185X, 4199X	32.8	43	600	82	170	840	9,400	0.65
		60	450	87	144	980	10,400	.68
		78	250	87	128	1,800	11,200	.72
4186X, 4190X	29.1	38	600	78	173	940	10,000	.66
4192X, 4196X		55	450	85	153	1,250	10,700	.70
		73	250	89	134	1,740	11,100	.73
4193X, 4195X	19.4	32	600	75	178	1,040	9,500	.66
		47	450	81	164	1,300	10,000	.68
		64	250	82	138	1,300	11,100	.72
4189X, 4218X	16.6	36	600	74	174	990	9,600	.67
		57	450	78	152	1,020	10,400	.72
		78	250	80	133	1,120	11,000	.74
SAND PINE BLEACHED PULP (BLEACH NO. 5349)								
4193X, 4195X	.....	23	600	76	172	1,020	9,400	.70
		41	450	86	148	1,100	10,400	.71
		63	250	86	140	1,160	11,100	.76
LONGLEAF PINE UNBLEACHED PULP								
4211X, 4215X	31.5	44	600	68	187	615	9,200	.58
		61	450	75	168	690	9,600	.63
		77	250	75	149	680	10,200	.65
4205X, 4207X	28.8	41	600	60	192	610	8,600	.59
4212X, 4214X		55	450	71	168	780	9,700	.63
		73	250	75	151	800	10,200	.65
4201X, 4206X	19.2	32	600	56	212	470	8,700	.60
		48	450	65	188	790	9,700	.63
		65	250	69	150	930	9,900	.65
4216X, 4217X	16.8	37	600	61	186	600	8,900	.63
		52	450	67	161	820	9,100	.67
		67	250	69	153	770	9,800	.70

(Sheet 1 of 2)

Table 3.--Strength and other physical characteristics[1] of unbleached and bleached kraft pulps from sand pine and longleaf pine--Continued

Digestion No.	Properties of pulp							
	Permanganate number	Beating time	Freeness	Burst factor	Tear factor	Folding endurance (M.I.T.)	Breaking length	Sheet density
		Min.	Ml.			Double folds	M.	G. per cc.
LONGLEAF PINE BLEACHED PULP (BLEACH NO. 5351)								
4201X, 4206X	.....	29	600	71	204	640	9,200	0.65
		42	450	76	174	760	9,900	.67
		61	250	78	167	960	10,200	.71
SAND PINE AND LONGLEAF PINE MIXTURE[2] UNBLEACHED PULP								
4219X, 4220X	28.7	33	600	74	164	680	9,900	.63
		56	450	78	144	730	10,500	.67
		72	250	81	144	800	10,500	.69

[1] Tested according to TAPPI Standard Methods. Values are interpolated from beater test curves.

[2] Equal parts by weight (moisture-free).

(Sheet 2 of 2)

Table 4.--Bleaching of sand and longleaf pine kraft pulps

Bleach No.	Stage No.	Bleaching treatment: Chemical[1]	Amount applied	Amount consumed	Temperature	Duration	pH Initial	pH Final	Brightness
			Percent	Percent	°C.	Min.			Percent
				SAND PINE PULP[2]					
[3]5349	1	Cl_2	6.5	6.30	25	90	2.2	2.0	
	2	NaOH	2.0	.....	70	60	11.4	11.2	
	3	ClO_2	.76	.76	75	120	6.1	3.8	
	4	NaOH	1.0	.....	60	60	11.3	11.1	
	5	ClO_2	.38	.38	75	150	6.1	6.0	89.6
				LONGLEAF PINE PULP[4]					
[5]5351	1	Cl_2	6.5	6.13	25	90	2.2	1.9	
	2	NaOH	2.0	.....	70	60	11.7	11.3	
	3	ClO_2	.76	.76	75	90	6.1	3.7	
	4	NaOH	1.0	.....	60	60	11.5	11.4	
	5	ClO_2	.38	.38	75	180	6.1	5.1	89.5

[1] The amounts of chemicals applied are based on moisture-free weights of pulp. The amount of ClO_2 is in terms of chlorine dioxide. The consistency was 2 percent in the first stage and 10 percent in all other stages.

[2] Digestions 4193X and 4195X yield 45.1 percent, permanganate number 19.4.

[3] The yield of bleached pulp was 93.8 percent based on weight on unbleached pulp and 42.3 percent based on weight of wood.

[4] Digestions 4201X and 4206X yield 46.0 percent, permanganate number 19.2.

[5] The yield of bleached pulp was 92.3 percent based on weight of unbleached pulp and 42.5 percent based on weight of wood.

Table 5.--Properties[1] of 50-pound bag papers made from sand and longleaf pine kraft pulps

Machine run No.	Pulp furnish	Stuff-free-ness	Weight: Ream, 500 sheets 24 by 36 inches	Weight: Square meter	Thick-ness	Density	Bursting strength	Internal tearing resistance: Machine direction	Internal tearing resistance: Cross direction	Internal tearing resistance: Total	Tensile strength: Machine direction	Tensile strength: Cross direction	Tensile strength: Total	Folding endurance M.I.T.-1-kg. tension: Machine direction	Folding endurance M.I.T.-1-kg. tension: Cross direction	Folding endurance M.I.T.-1-kg. tension: Average	Air resistance
		Ml.	Lb.	G.	Mils	G. per cc.	P.s.i.	G.	G.	G.	Lb. per in. width	Lb. per in. width	Lb. per in. width	Double folds	Double folds	Double folds	Sec. per 100 cc.
Commercial specification			50.0	.....	.....	.....	.....	110.0	.....	235.0	.....	19.0	53.0	.....	.....	.....	22
Commercial paper (shipment 4698)			50.2	81.6	5.0	.64	42.3	125.6	136.8	262.4	35.1	21.3	56.4	477	382	429	8
5804	Longleaf pine, Dig. 4399 and 4400	560	53.4	86.8	5.1	.67	30.1	118.8	153.6	272.4	34.3	13.1	47.4	320	200	260	2
5805	Sand pine, Dig. 4401 and 4402	580	49.9	81.2	4.7	.68	37.2	104.0	130.4	234.4	40.7	14.2	54.9	558	134	346	7
5808	Sand pine and longleaf pine[2]	600	48.1	79.4	4.0	.78	36.3	96.0	127.2	223.2	37.0	15.5	52.5	593	215	404	12

[1] Tested according to TAPPI Standard Methods.

[2] Mixture consisted of 50 percent sand pine and 50 percent longleaf pine.