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Recommended Citation

Nihlen, H. and McGovern, J.N. 1943. Sustainability of birch, aspen and sugarberry for rayon pulp: results of certain sulfite pulping and bleaching experiments. U.S. Forest Production Laboratory, Madison. R 1441.

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SUITABILITY OF BIRCH, ASPEN, AND SUGARBERRY FOR RAYON PULP:

Results of Certain Sulfite Pulping and Bleaching Experiments

December 1943



No. R1441

**UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE
FOREST PRODUCTS LABORATORY**

Madison, Wisconsin

In Cooperation with the University of Wisconsin

SUITABILITY OF BIRCH, ASPEN, AND SUGARBERRY FOR RAYON PULP:
RESULTS OF CERTAIN SULFITE PULPING AND BLEACHING EXPERIMENTS

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Summary

Sulfite pulping and bleaching experiments were conducted on three hardwoods, birch, aspen, and sugarberry, to determine the suitability of their pulps primarily for purification to rayon-type pulps and incidentally for paper making purposes. The following results were obtained: (1) Birch sulfite pulps had progressively decreasing bleach requirement and increasing purity with increasing cooking time, other cooking conditions being the same. The pulps had the average strength of hardwood sulfite and, except for a high ether solubility, were fairly well purified. Three-stage bleaching treatments were applied to the easiest-bleaching pulps which included studies of the variables of consistency, extraction temperature, calcium versus sodium hypochlorite in the third stage, and several variations of procedure to reduce the pulp resin content. Pulps of fair rayon quality, but with high ash and resin contents, were produced. An oxidizing pretreatment slightly reduced the ether solubility. (2) Aspen pulps suitable for paper making and purification purposes were produced in high yields. Those suitable for paper making were less delignified and had higher strength properties than the others. The more delignified pulps were given three-stage bleaching treatments, with a comparison made of calcium and sodium hypochlorite in the third stage. Except for their high resin contents, the bleached pulps had promising characteristics for rayon manufacture. (3) Sugarberry yielded a pulp with a low resin content accompanied by low screenings but shivey and hard to bleach. Nevertheless, the pulp was bleached satisfactorily for paper making purposes, and showed possibilities as a rayon-type pulp.

Introduction

The pulping of hardwoods by the sulfite process has long been of interest to investigators and manufacturers in the wood pulping industry

¹Hugo Fihlén was in the United States from January through August, 1941 as a Fellow of the American-Scandinavian Foundation. About two-thirds of his time was spent at the Forest Products Laboratory working on the experiments reported herein.

(1, 6, 7, 10, 11)², because of a desire to utilize more completely large supplies of available pulpwoods and to take advantage of the good formation, finish, bulk and opacity obtainable from hardwood pulps.

For many years relatively small quantities of hardwood sulfite pulps have been produced in the United States, most of the pulp being used as a filler for book-type papers. Some special commercial applications have been fulfilled by hardwood sulfites, such as blending with bleached softwood sulfite for papers suitable for chemical treatment, blending with bleached softwood sulfite for tissue (13), and for conversion to dissolving pulps. It is with the last application that this report is particularly concerned.

Although hardwood sulfites have apparently been already prepared and purified to produce pulp satisfactory for rayon (2, 7, 8) the primary purpose of the present investigation was to determine whether two typical northern hardwoods, birch and aspen, could be pulped by the sulfite process and subsequently purified to a degree equivalent to that of rayon-grade pulp made from softwood. The study was designed to continue work done by one of the authors in which Swedish birch was used. A secondary object in this investigation was the preparation of sulfite pulps, from birch, aspen, and sugarberry, suitable for paper making.

Because of the necessity of limiting the scope of the project, the pulping conditions were varied in cooking time to produce pulps having different bleach requirements. For aspen the variation was also in the maximum temperature in order to produce both rayon-type and paper-making-type pulps. The bleaching experiments involved only the variables of consistency, extraction temperature, the hypochlorite used, and involved several variations of the bleaching procedure for reducing the resin content of the pulp. The project, therefore, should be considered merely as an orientation and the results should be considered as being of indicative character only.

EXPERIMENTAL PART

Wood

The pulping experiments were made with 5/8-inch chips prepared from birch (*Betula papyrifera*), and sugarberry (*Celtis laevigata*) at the Laboratory and with commercially prepared 3/4-inch aspen (*Populus tremuloides*) chips. The birch and aspen chips had moisture contents of 14 and 12 percent respectively; the sugarberry chips had a moisture content of 43 percent. The aspen chips as received were nonuniform and contained numerous knots, chunks, slivers, and chips of extraneous species having the appearance of birch. These chips were rescreened by hand, but a reasonable effort did not produce an entirely satisfactory grade of chip.

²Numerals in parentheses refer to the list of references at the end of this report.

Pulping

The acid sulfite pulping liquors were prepared in the usual laboratory manner of passing sulfur dioxide from a cylinder into milk of lime. The liquors contained approximately 6 percent total sulfur dioxide with a combined sulfur dioxide of about 1 percent in all but two digestions with aspen in which this factor was 1.25 percent. The exact concentration for each liquor is given in table 1.

The chips were packed by hand in the autoclave to give liquor-wood ratios in the range of commercial operation. The ratio varied from 44 to 63 gallons of liquor per 100 pounds of wood (table 1). Sufficient liquor was charged to cover the chips.

The digestions were made in an alloy-lined autoclave, equipped with a steam jacket for indirect heating, having a capacity of 1.5 cubic feet. The autoclave was rotated occasionally by hand during pulping.

The digestions were made under the following temperature and pressure conditions:

Temperature schedule I - The following schedule was used with birch, sugarberry, and two aspen cooks, all employing liquor with 1 percent combined sulfur dioxide:

2 hours, from room temperature to 105° C.,
1 hour at 105° C.,
2 hours, from 105° to 135° C.,
Held at 135° C. to end of digestion.

Temperature schedule II - This schedule was used with two aspen cooks employing acid with 1.25 and 1.30 percent combined sulfur dioxide:

2 hours from room temperature to 105° C.,
1 hour at 105° C.,
3 hours, from 105° to 125° C.,
Held at 125° C. to end of digestion.

Maximum pressure - The maximum pressure was 80 pounds per square inch in all digestions. Samples of cooking liquor were withdrawn during the cooking period and analyzed for combined sulfur dioxide content by both the Sander (12) and the Palmrose (4) methods. The results are shown in table 2. The yields, pulp strength and chemical analyses of the pulps were determined by Forest Products Laboratory standard methods. The pulp bleach requirements were determined by the TAPPI standard permanganate-number test and the Forest Products Laboratory single-stage hypochlorite test. The results are given in table 1.

Bleaching

The bleaching experiments were made with pulp samples equivalent to 50 grams of moisture-free pulp. A three-stage bleaching method was used comprising chlorination, caustic extraction, and final treatment with either calcium or sodium hypochlorite. The bleaching was performed in glass bottles of approximately 3 liters capacity. Except for several experiments made for the purpose of reducing the resin content, described later, the general procedure of the three-stage method used was as follows:

In the first bleaching stage about 70 percent of the chlorine demand, as estimated from the permanganate numbers of the pulps, was satisfied by the use of chlorine water. The reaction time in this stage was 15 minutes, the temperature was 20° C., and the consistency was 2.5 percent. This value for consistency, and subsequent ones given in table 3 for the other stages are in terms of grams of moisture-free pulp per 100 cc. of liquor including the moisture contained in the pulp rather than expressed in customary terms of grams of pulp in 100 grams of suspension. The difference is not great at the low consistencies employed. The suspension was kept uniform during the chlorination by shaking the bottle. On completion of the chlorination stage the liquor was drained from the pulp and the pulp washed thoroughly with cold water. The chlorine consumption was determined by iodometric titration of the initial and residual liquors.

In the caustic extraction stage the temperature, consistency, caustic concentration, and extraction time were varied for the different bleach tests. Since the pulp yields were not determined in each stage, the stock consistencies in the second and third stages were calculated from the weight of pulp used in the first stage. Therefore the values are somewhat higher than actual, but the difference is without practical significance. The temperature was held constant in a thermostatically controlled water bath.

In the final stage, consisting of either calcium or sodium hypochlorite treatments, the amount of active chlorine was 0.5 gram for 100 grams of pulp. The consistency was 5 percent. The bleaches with calcium hypochlorite were made at 35° C. and the suspensions held at a pH value of 8 to 9 by adding, when necessary, a small amount of lime water. After a reaction time of about 4 hours the pulps were drained and washed, first with soft water, then with 2 liters of 0.04 percent hydrochloric acid and finally with soft water again. The bleaches with sodium hypochlorite were made at 70° to 80° C. and the suspensions held at a pH value of 4 to 5 by adding dilute hydrochloric acid. The reaction time was 2 hours and the bleaching was followed with a soft-water wash.

The bleached pulps were analyzed for various chemical constituents by standard methods and their color determined by use of the Ives photometer (table 4).

DISCUSSION OF RESULTS

Pulping Experiments

Birch Pulping.--The conditions for the birch digestions were designed to effect a sufficient removal of hemicellulosic material to produce a pulp which, when purified, would be suitable for rayon manufacture. Therefore, a relatively high maximum temperature and an acid low in combined sulfur dioxide, conditions known to favor solution of hemicelluloses, were employed. Using these selected conditions the time at the maximum temperature was varied to produce pulp of different degrees of delignification.

When the time of pulping at the maximum temperature of 135° C. was varied from 1.2 to 3.6 hours (total digestion times from 6.2 to 8.6 hours) a series of pulps having average hypochlorite bleach requirements between 23 and 11 percent was obtained (table 1). The shortest total cooking time, 6.2 hours used for number 314, was not quite sufficient to reduce all the chips to pulp, the result being a large amount of screenings and a low yield of screened pulp having a relatively high bleach requirement. An increase in the cooking time of 0.7 hour in number 312 resulted in complete reduction of the chips into pulp having a medium-hard bleachability. Increases in the cooking time resulted in further reduction in yield. The relatively low yield obtained with cook No. 323 is possibly an indication of over-cooking. The average bleachability of about 11 percent for this pulp is apparently the minimum value that can be obtained under these conditions.

The chemical purity of the pulps showed a progressive increase with decrease in their bleach requirements. This improvement in purity was indicated by definite increases in the total and alpha cellulose contents of the pulps and decreases in the lignin and pentosan contents, as shown in table 1 by the chemical data for the birch pulps. The amounts of material in the pulps soluble in alcohol-benzene and ether, measures of the so-called resin content, appeared to be unaffected by the degree of pulping. Although the ash contents also appeared to be unaffected by the degree of pulping the rather high values of these, as well as those of the subsequent pulps, were believed to be caused by the hard water used for screening and washing.

The strength characteristics of the pulps, as evaluated by test, showed certain relationships with the degree of pulping. The data for the birch pulps given in table 1 shows that the one with the lowest yield, number 323, was weaker than the others. The bursting strengths of the pulps decreased slightly with decreasing bleach requirement and lignin content.

Pulp from digestion number 323, apparently overcooked, was definitely lower in bursting strength than the others. The tearing strength values did not differ greatly among the various pulps, although number 323 had somewhat lower values than the others. The tensile strengths at the higher freeness showed no definite variation with degree of pulping except that number 323 was lower than the others, but at the lower freeness, the trend with decreasing bleach requirement was similar to that for the bursting strength. The solid fraction values did not indicate any trend. The times of beating required to attain the given freeness values were erratic with respect to degree of pulping. It might be expected that the increase in alpha cellulose and decrease in pentosan contents resulting from the increased pulping action would cause an increase in beating time; but this was not substantiated by the data obtained.

Cooking control was attempted to some extent by an analysis of samples of cooking liquor taken at intervals during the digestion for combined sulfur dioxide content by both the Palmrose and the Sander methods. The results, tabulated in table 2, were erratic from cook to cook but showed consistent trends in any one cook. Negative values indicated the presence in the liquor of an excess of acids whose pH values were lower than four. The Palmrose values were consistently lower than those from the Sander tests, presumably because of the indicators used, methyl red for the Palmrose and bromphenol blue for the Sander. For the same cooking conditions either the Palmrose or the Sander test for combined sulfur dioxide would probably show a practical correlation with the degree of pulping.

Aspen Pulping.--The first two aspen digestions, numbers 327 and 328 in table 1, made with the same general conditions as used for the birch pulping, produced easy bleaching pulps in relatively high yields. Since the screenings contained very few uncooked fibers, the somewhat high value of 1 percent cannot be attributed to inefficient screening operation and, therefore, is believed to be caused by the inferior grade of chips previously described. The percentage yields were appreciably higher for the aspen in comparison with the birch, even though the birch pulps were harder to bleach. An increase in cooking time of 0.8 hour in number 328 over the time used in number 327 effected a small decrease in yield and bleach requirement and a slight increase in pulp purity as shown by an increase in the cellulose contents and a decrease in lignin content. The pentosan content increased slightly, however. Comparing aspen with birch, for the same time of pulping, the aspen pulps were produced in higher yields and with lower bleach requirements, lower total and alpha cellulose, and lignin contents and considerably lower strength characteristics. Thus, the aspen pulps showed more effective lignin removal (hence lower bleach requirement) than the birch, but they were slightly less purified with respect to cellulose. It appeared from these results that the pulping conditions used were not the most suitable for producing an aspen pulp high in alpha cellulose. The solubility in alcohol-benzene and in ether of the aspen pulps, and their ash contents were relatively high.

The strength properties of the aspen pulps produced at 135° C. were uniformly low. The easier bleaching aspen pulp (number 328) had slightly higher bursting, tearing, and tensile strengths than the harder bleaching pulp (number 327).

The other aspen pulps, number 333 and 332 in table 1, were produced under conditions designed to retain the hemicelluloses for paper making purposes by using a low maximum temperature and a medium-high combined sulfur-dioxide content in the cooking liquor. Pulps of two bleach requirements were produced. One digestion, number 333, was made in too short a time to effect complete reduction, as indicated by the screenings value of 2.6 percent, but the yield of screened pulp was high, although the pulp was hard to bleach. A longer digestion time used in number 332 reduced the screenings to 1 percent, apparently the minimum attainable with these chips, and produced a high yield of easy-bleaching pulp. The lignin contents of these pulps increased with the bleach requirements and the pulps were less purified from the standpoint of total and alpha cellulose than the aspen pulps made at the higher temperature of 135° C.

The strength properties for the series of aspen pulps made at 125° C. were generally higher than those of the pulps made at 135° C. when compared at the same bleach requirement. In the pulps produced at the lower temperature, however, the pulp with the higher lignin content was slightly superior to the other. The shorter beating time needed to attain a given freeness value, for the less purified pulps of the second series compared with the first, was perhaps caused by the higher contents of hemicellulosic material in the second series of pulps.

Sugarberry Pulping.--Only one pulping experiment was made with sugarberry chips, number 326 of table 1. The conditions were the same as those used for the birch digestions. Although the wood was reduced satisfactorily from the standpoint of screenings (0.6 percent), the yield of screened pulp was in the medium-low range and the pulp was hard bleaching and very shivey. More suitable conditions need to be developed to produce a satisfactory unbleached pulp from this wood. The lignin content of the sugarberry pulp was relatively high and the cellulose contents somewhat low. The pulp had a lower solubility in alcohol-benzene and in ether than either the birch or aspen pulps. The strength values for this pulp were low, about the same as those of the aspen pulps.

Alpha Cellulose Yield

A considerably higher yield of alpha cellulose resulted from the pulping of aspen than from either the birch or sugarberry. On the basis of the yield of screened pulp and its percentage of alpha cellulose, the yields of alpha cellulose were from aspen (numbers 327 and 328) 42.8 percent, from birch (numbers 322 and 323) 38.9 percent, and from sugarberry (number 326) 37.0 percent. The high value for the aspen pulps can probably be attributed

largely to high alpha cellulose content in the original wood, though that was not tested. On the basis of yield of alpha cellulose per dizester, however, that from the aspen would be expected to be lower than that from the others because of its relatively low density.

Bleaching Experiments

Birch Pulp Bleaching.--The birch pulps, numbers 310 and 323, which represented two fairly well delignified pulps, were selected for bleaching experiments. The bleaching data are given in table 3 and the analyses of the bleached pulps in table 4.

The first variable studied was the temperature of caustic extraction. The results from bleaching experiments numbers 578 and 579, tables 3 and 4, showed that cold extraction at 10 percent consistency resulted in a pulp with a lower alpha-cellulose content and brightness and higher pentosan content than hot extraction at 5 percent consistency. The bleach powder consumption in the third stage was higher following the cold extraction. Hot extraction was employed, therefore, in all subsequent bleaching experiments. It would have been desirable to use a consistency of 10 percent or greater for more effective purification, but uniform mixing at these consistencies was impracticable with the equipment at hand.

A comparison of the use of calcium and sodium hypochlorite bleach liquors in the final stage was made with two pulps, numbers 310 and 323. The respective bleaching experiment numbers are 579 and 580, and 581 and 582, tables 3 and 4. There appeared to be little difference in the action of the two chemicals, even though the sodium compound would be expected to have a milder action than the calcium.

These data also afford a comparison between the bleaching of a pulp less purified by digestion (number 310), and one more purified, (number 323). Pulp number 323 gave a bleached product having a lower pentosan content, a higher alpha cellulose content and a lighter color than pulp number 310. The alpha cellulose contents of the bleached pulp from number 310 were too low and the ether-soluble material too high for the manufacture of rayon of commercial quality. The bleached pulps from number 323 were fairly satisfactory with respect to alpha cellulose content, but the ether-soluble values were very high.

Several experiments were made to reduce the ether-soluble material in the bleached birch pulps below 0.5 percent, the upper limit for purified pulps. Suggestions have been made that resins can be removed during bleaching by treatment with a wetting agent. In an experiment to test this suggestion, (bleach 583, table 3) the first and third stages followed the same procedure as that used with the control bleach (number 581), but the extraction stage was made with 4 percent sodium hydroxide plus 1 percent of the wetting agent tetrasodium pyrophosphate, calculated on the pulp weight. The extraction time was 4 hours, during which the suspension was shaken

repeatedly. After the caustic treatment the pulp was first washed with hot water containing a small amount of tetrasodium pyrophosphate and then with hot water. The chlorine consumption in the third stage appeared to be increased by this operation (table 3) and the resin content showed no improvement (table 4).

It is claimed (3) that treatment with kerosene after chlorination and emulsification of the pitch-solvent mixture following the final washing helps reduce the ether-soluble material. This procedure was followed and the results are given opposite number 584, tables 3 and 4. In this experiment the chlorination stage was followed by a treatment in which kerosene in the amount of 4 percent of the moisture-free pulp was added to the wet pulp with a moisture content of about 70 percent. The caustic extraction and final bleach were carried out as tabulated. After the final bleach and washing, the pulp was diluted with hot water to 2 percent consistency and 2 percent by weight of the pulp of a sulfonated fatty-acid soap was added to emulsify the residual kerosene in the pulp. The ether solubility of this pulp, however, was very little different from that of the control, number 581.

Kichter claims (9) that the hardwood resins become sticky and insoluble in caustic after chlorination and recommends a four-stage bleach with calcium hypochlorite in the first stage, followed either by the usual chlorination, caustic extraction, chlorination, and hypochlorite bleaching or by caustic extraction, chlorination, and hypochlorite bleaching. In trying this process (bleach 585 table 3) the pulp was given a calcium hypochlorite pretreatment at a consistency of 5 percent and a temperature of 20° C. The amount of active chlorine was 2.5 grams for 100 grams of pulp, the same as used in the chlorination stage for the control bleach (number 581). Nearly all of the chlorine was consumed after 2 hours. Following the washing of the pretreated pulp it was given the usual three-stage bleach treatment, following the conditions given for bleach 585 in table 3. Comparison of number 585 with the control number 581 in table 4 shows that the ether-extractive material decreased. Removal of hemicelluloses was also effected as shown by the increased alpha-cellulose content and the decreased pentosan content of the pulp prepared with the preliminary oxidizing treatment. The alcohol-benzene soluble material, in contrast, showed no loss as a result of the treatment while viscosity was reduced to one-half the value of the control pulp.

Aspen Pulp Bleaching.--The standard three-stage bleaching treatment was applied to two aspen pulps, numbers 327 and 328. The only variable was the use of calcium and sodium hypochlorites in the third stage of bleaching. The results are given in numbers 587 to 590 in tables 3 and 4.

Since pulps numbered 327 and 328 were not greatly different in their properties, the bleached pulps showed very little difference. In one instance, use of sodium instead of calcium hypochlorite resulted in an increase in alpha cellulose and color (compare number 589 and 590). Except for their high solubility in ether, the aspen bleached pulps in general were fairly promising for rayon purposes. A lower content of ether-soluble substances

might be expected by use of seasoned wood (5) and a four-stage bleach with an oxidizing treatment in the first stage.

Sugarberry Pulp Bleaching.--The preliminary bleach of the sugarberry pulp by the three-stage method resulted in a shivey pulp with poor brightness. The bleaching conditions used and the results of bleaching a second time are given in number 586, tables 3 and 4. The pulp was similar to the birch pulps in chemical properties, although it had a favorably lower content of ether-soluble materials. The bleached sugarberry pulp appears to be of hardwood paper making quality and might possibly, after further purification for pentosan removal, be used in the manufacture of rayon-type pulp.

CONCLUSIONS

Sulfite pulping and bleaching experiments of limited scope indicate the following:

- (1) Bleached pulps of fair rayon quality, but with high ash and resin contents, were able to be produced from birch.
- (2) Bleached pulps of promising characteristics, except for their high resin contents, were able to be made from aspen.
- (3) Bleached pulps from sugarberry showed possibilities as a rayon-type pulp.

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Table 1.—Digestion conditions and results and the properties of sulfite pulps from birch, aspen, and sugarberry.

2 Average temperature somewhat low.

Z M 51192 R

Table 2.—Concentration of combined sulfur dioxide in cooking liquor during digestion.

Number	Species	Test samples taken at —						Method of analysis
		5 hours	5.5 hours	6 hours	6.5 hours	7 hours	8 hours	
<i>Sander; Palmrose; Sander; Palmrose; Sander; Palmrose; Sander; Palmrose; Sander; Palmrose; Sander; Palmrose; Sander; Palmrose</i>								
								Percent
314	Birch	—	0.22	—	—	-0.06	-0.24	—
312	—	—	.26	—	—	.28	.09	0.16 -0.14
310	—	—	—	—	—	—	—	—
322	—	0.40	.36	0.22	0.07	.40	—	.28 .04
323	—	.40	.01	—	—	.16	—.40	—
327	Aspen	.44	.38	—	—	.16	.12	—
328	—	.48	.42	—	—	.20	.40	—
333	—	—	—	—	—	—	—	—
331	—	.95	.88	—	—	—	—	—
326	Sugberry	.20	.05	.04	—.24	—	—	—

Table 3.—Bleaching conditions and results for birch, aspen, and sugarberry sulfite pulps.

Grams of moisture-free pulp per 100 cc. of liquor.

One percent Na₂HPO₄ (based on pulp) added to NaOH in extraction stage.

Four percent kerosene added after chlorination stage and pulp washed with 2 percent sulfonated fatty-acid soap after final bleach.

"Presentation."

Initially added bleach exhausted and more bleach liquor added.

Table 4.—Chemical analyses and color of bleached birch, aspen, and sugarberry sulfite pulps.