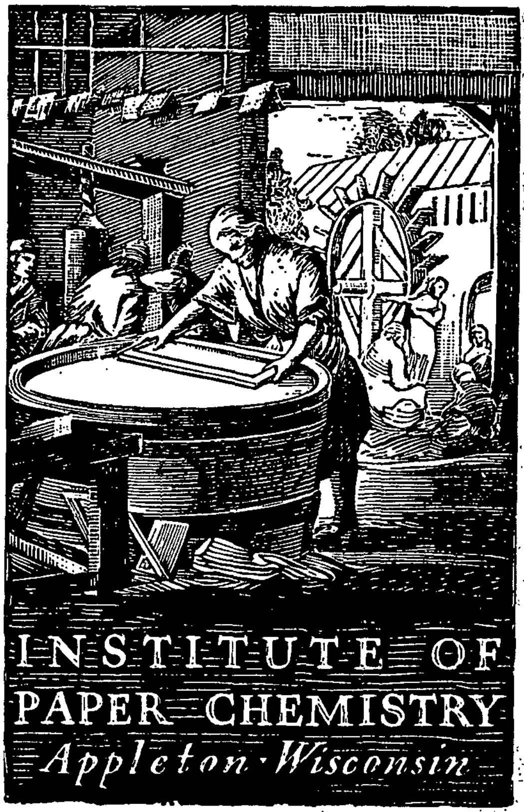

WOOD/BARK ADHESION AND METHODS OF REDUCING ADHESION IN HARDWOOD SPECIES

Project 2929

Report Four

A Progress Report

to

MEMBERS OF GROUP PROJECT 2929

June 28, 1972

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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WOOD/BARK ADHESION AND METHODS OF REDUCING ADHESION IN HARDWOOD SPECIES

SUMMARY

Progress Reports One, Two and Three describe the seasonal variation in wood/bark adhesion and the associated morphological changes that developed in the "cambium zone" for eight selected pulpwood species. Also included in Progress Report Two was a description of preliminary work initiated on ways of reducing wood/bark adhesion in chip samples. The report that follows describes the results of continued efforts to examine alternative methods of reducing dormant season wood/bark adhesion.

Mechanical methods were restricted to observations on the action of the IPC pulpwood chipper on the separation of bark and wood. The "chipper" method appears to have promise for several pulpwood species including bur oak, quaking aspen, sugar maple, and loblolly pine. Evidence in the literature indicates that the procedure is even more effective on frozen wood.

Several chemical methods of reducing adhesion were investigated. The most promising involves the use of green kraft cooking liquor at a temperature of 200°F. and at a treatment time of 60 minutes. The cooking liquor involved has also been shown to be useful in reducing chip deterioration in chip storage piles. The principal disadvantage of the method is the high treatment temperatures and the long treatment time required.

Thermal methods, including pressure chamber procedures, also look quite promising. Time and temperature appear to be interrelated with total treatment times being reduced to 10 to 15 minutes when temperatures in excess of 250°F.

can be employed. Successful use of the thermal procedures to reduce adhesion involves swelling and softening the cell walls of cells in the "cambium zone."

Biological methods of reducing adhesion were also considered. Moist storage of chips under aerobic conditions and at temperatures that encourage fungus attack of the cambium zone and inner bark region resulted in greatly reduced wood/bark adhesion at storage times as short as 15 to 20 days.

One of the most promising approaches tried, although obviously still in the developmental stage, was the use of microwave heating to create high temperatures in the moist interior of the test chips. The result was moderate reduction in wood/bark adhesion at treatment times as short as one minute. Further developmental work with this procedure could conceivably further decrease treatment times and/or increase the effectiveness of the treatment.

Observations on the several general types of approaches (mechanical, chemical, thermal and biological) suggest there may not be any single method that is best for all species. Combinations of methods are worthy of consideration for problem species or when several different species must be processed. Of the trees investigated, dormant season wood/bark adhesion was easy to reduce in the case of white spruce and difficult to decrease for hickory and white birch. Cambium zone specific gravity and maturity (cell size, cell wall thickness, and lignification may be the controlling factors.

INTRODUCTION

Trends in raw material requirements and changes in harvesting methods and utilization standards continue to emphasize the urgency of finding appropriate solutions to the bark utilization problem. One important aspect of the problem is wood/bark adhesion. The objectives of Project 2929 were to: (1) measure accurately seasonal changes in wood/bark adhesion for sugar maple, white birch, quaking aspen, white oak, shagbark hickory, white spruce, southern cottonwood, and loblolly pine; (2) examine between species and seasonal morphological differences in an attempt to correlate morphological differences with measured wood/bark adhesion; (3) develop suitable methods of reducing wood/bark adhesion based upon information regarding the causes of adhesion.

Work on the measurement of wood/bark adhesion was initiated on March 15, 1970 and Progress Reports One through Three described the sampling and testing methods developed and summarize the measurements made on seasonal changes in wood/bark adhesion for the eight tree species listed above. The report that follows describes the studies undertaken to determine ways in which wood/bark adhesion of chip samples might be reduced.

ZONES OF FAILURE

One reason for measuring wood/bark adhesion and for carefully examining the failure zones was to determine those morphological factors which contribute to the differences in dormant season wood/bark adhesion. An equally important objective was to ascertain whether this information could be used to develop methods of reducing wood/bark adhesion and/or improve conventional debarking methods. The wood/bark adhesion test method (see Progress Report One, page eight) was so designed as to confine the failure zone to those cells in the immediate vicinity of the cambium (± approximately 50 cells).

A review of the morphological data for the 8 species studied reveals a surprising between-species uniformity in the location of the zone of failure. This uniformity is in part due to the design of the test method. During the peeling season, despite reported differences between species in difficulty of peeling, the wood/bark adhesion values were uniformly low and appeared to be a valid measure of the zone of weakness. Table I summarizes the wood/bark adhesion data. During the dormant season, although the wood/bark adhesion tests varied considerably between species, the failure zone was quite consistent in location with respect to the cambium. The dormant season adhesion values did not always measure adhesion in the zone of lowest strength. This latter anomaly became evident when the strength of the inner and outer bark was measured. The point of this discussion is that valid measurements have been obtained for wood/bark adhesion in the region near the cambium zone both during the peeling season and during the dormant season. These values, however, did not always explain the differences encountered in debarking. When, however, information on morphology and the strength of the inner bark was considered, the between-species differences in debarking behavior began to make sense. It also appears that appropriate information on outer bark strength and outer bark morphology can be expected to further assist efforts underway aimed at solving wood/bark separation and segregation problems.

TABLE I

SUMMARY OF WOOD/BARK ADHESION DATA

	Zone of Failure		Wood/Bark Adhesion, kg./cm.^{2}		Bark Strength, kg./cm.^{2}	
Species	Peeling Season	Dormant Season	Peeling Season	Dormant Season	Inner Bark	Outer Bark
Shagbark hickory	Cambium zone[a]	Between bands of phloem fibers and immature parenchyma cells	5.3	26.9	25.0	72.7
Eastern cotton-wood	Cambium zone[a]	Immature parenchyma and sieve tubes of inner bark	4.4	13.5	17.7	4.2[b]
Quaking aspen	Cambium zone[a]	Same as above Same as above	6.4	11.4	9.0	4.9
Bur oak	Cambium zone[a]	Same as above	5.8	9.6	4.5	7.0
White birch	Cambium zone[a]	Same as above	5.1	12.0	1.6	9.8
Sugar maple	Cambium zone[a]	Same as above	5.8	10.1	1.4	4.7
White spruce	Cambium zone[a]	Immature inner bark parenchyma and sieve cells	5.0	11.0	7.4	--
Slash pine	Cambium zone[a]	Same as above	3.5	9.1	6.4	5.2

[a]In the cambium zone or the newly-formed nonlignified xylem cells inside the cambium zone.

[b]Strength low, test samples failed during preparation, data based upon a single test.

Looking again to the problem at hand, i.e., the reduction of dormant season wood/bark adhesion, it appears that the inherent weaknesses and the morphology of the cambium zone warrant concentrating attempts at reducing adhesion to the cambium zone and the immature phloem cells just outside the cambium. Table I briefly summarizes the available data on wood/bark adhesion, and inner and outer bark strength data. Also included is a very brief description of the location of the zone of failure. For more detailed information the reader is referred to Table VI, page 49, Progress Report Three.

TESTING PROCEDURES

To facilitate the screening of possible ways to reduce wood/bark adhesion and keep costs at a reasonable level, a procedure using simulated chips was developed. Two sizes of chips were employed. The simulated chips were prepared on a band saw and were tangential sections that contained both wood and bark with the wood/bark interface located approximately equidistant between the wood and bark surfaces. Chips 1-1/2 x 1-1/2 inches and approximately 1/4-inch thick were used in the early trials and the size was later reduced to 1-1/2 x 3/4 x 1/4. Figure 1 illustrates the size and shape of the 1-1/2 x 3/4 x 1/4-inch simulated chips and compares the test chips (tabs) with standard chips. It is fairly evident, because of the differences in the area of the wood/bark interface, that treatments effective on the test chips would be even more effective on actual chips.

Dormant season samples, collected in January, February, and March of 1971 and 1972 were treated and tested within 96 hours after collection. Wood/bark adhesion of most of the samples were rated (from 1 to 10) as to ease with which the bark could be removed and only occasionally were tabs[1] prepared and the samples evaluated on the Instron tester. Table II relates the ranking system to the equivalent Instron test values. Basically, the hope in this series of trials was to reduce wood/bark adhesion to levels equivalent to values obtained during the peeling season (5.1-6.1 kg./cm.^{2}). The treatments applied can be grouped into four categories, namely, mechanical, chemical, thermal and miscellaneous. The discussion that follows describes the use of these four types

[1]The terms test chips, tabs, and simulated chips have been used interchangeably throughout this report to indicate the 1-1/2 x 3/4 x 1/4-inch chips pictured in Fig. 1.

of treatments to reduce wood/bark adhesion. Because of the availability of quaking aspen and the ease of preparing test tabs, most preliminary work was carried out with aspen. Only the most promising approaches were then tried on additional tree species.

Figure 1. Pine Tabs and Pine Chips on the Left and Aspen Tabs and Spruce Chips on the Right Illustrate the Larger Bark/Wood Interface that Existed on the Simulated Chips (Tabs)

TABLE II

COMPARISON OF ESTIMATED ADHESION VALUES
WITH MEASUREMENTS ON INSTRON TESTER

Adhesion Ranking Values	Instron Test Values, kg./cm.^{2}	Description of Adhesion
1	< 2	Falls off to touch
2	2-3	Removed easily by hand
3	3-4	Removed easily by hand
4	4-5	Moderate force required
5	5-6	Considerable force required
6	7-8	Knife required to remove bark — cambium no longer active
7	8-9	Knife required to remove bark — cambium no longer active
8	10	Moderately difficult to remove with knife
9	10+	Bark removed with considerable difficulty using knife
10	10+	Bark removed with considerable difficulty using knife

MECHANICAL METHODS

Presently employed debarking methods use various types of mechanical action to remove bark from logs and pulpwood bolts. Based upon the morphological examinations of the eight species under investigation, similarity in the growing season and dormant season failure zones (cambium zone and inner bark region) suggest that any one of several conventional debarking devices should be equally effective in debarking the species under investigation. Existing devices are not equally effective on the species being evaluated, either during the peeling season or during the dormant season.

Discussions with individuals involved in building and using mechanical debarkers indicate that growing season debarking difficulties are apparently due to the mechanical properties of the bark rather than wood/bark adhesion. Dormant season debarking problems appear to result from a combination of high wood/bark adhesion and some unusual mechanical property of the bark. As discussed in Progress Report Two, such species as basswood, elm, and hickory are described as being difficult to debark and the difficulty appears to be due primarily to the stringy nature of the bark. During debarking, the bark wraps around the cutter heads and tension arms and after a short time renders certain types of debarkers inoperative. White birch is described as being a problem species in debarking because the cutter head type of debarkers tend, at times, to run between the outer papery bark and the reddish-colored inner bark. A quick check into the bark morphology and inner bark strength data for white birch and hickory suggests reasons for existing debarking problems. The inner bark of hickory is very strong (see Table I) and is loaded with fibers which apparently contribute to the stringy nature of the bark. White birch, on the other hand, has very weak inner bark and this is believed to contribute to the tendency for the debarking devices to operate in the inner bark region rather than in the cambium zone.

Chipping prior to debarking is one approach that would eliminate the types of debarking problems described above. Use of specially modified chippers aimed at producing not only suitable chips but designed to provide the necessary additional mechanical action to cause wood/bark separation seem to have considerable promise for certain tree species. With the above considerations in mind, observations were made on the Institute's 41-inch, 4-knife Carthage chipper with regard to the kind of separation being obtained under standard operations.[2] Wood and bark chips were being prepared for use in the work on the characterization of the flotation behavior of wood and bark of several tree species. Progress Report Two (page 41-42) describes the mechanical action of the chipper on dormant quaking aspen, white birch, sugar maple, and bur oak. The results varied between species with the method appearing to have considerable promise for aspen, maple, and bur oak. Additional observations were made during the late fall of 1971 and these were recorded as follows:

Chipper Trial V — Several bolts of shagbark hickory were collected in mid-November, stored outside (under cover) until January, and then the wood was thawed out and chipped. Examination of the bark chips having wood attached revealed that the chipper action was only moderately effective in separating the wood and bark. Approximately 85% of the large bark chips (3/4-inch size), 25% of the medium bark chips (1/2-inch size), and 5% of the small chips (1/4-inch size) had wood attached. After considering the

[2] The chipper runs described were conducted as part of Project 2977.

distribution of the bark by size classes it appears that overall 55% of the bark was "wood-free" bark. Examination of the bark fraction (1/4, 1/2, and 3/4-inch size) revealed that in most instances the chipper action was causing separation at the inner bark/outer bark interface rather than at the cambium zone. Chipping to a smaller-sized chip, it appears, would be one way to improve the separation.

Chipper Trial VI — White spruce bolts collected in early December were thawed out and chipped in mid-December. The chips were screened and the action of the chipper on wood/bark adhesion was evaluated by examining the total weight and the percent of bark chips that had wood attached. Seventy-eight percent of the bark turned up as 3/4-inch size chips, 19% as 1/2-inch and only 3% as 1/4-inch chips. Approximately 80% of the 3/4-inch chips, 90% of the 1/2-inch chips and 0% of the 1/4-inch chips had wood attached. These results indicate very little wood/bark separation occurred due to the action of the chipper. The calculated value, based upon the above percentages, indicated 80% of the total weight of bark still had wood attached. Stated another way, 20% of the total weight of bark was "wood-free" bark. Examination of the wood-free bark revealed the zone of failure to be the cambium zone. Again, it appears, as in the case of hickory, chipping to a smaller chip size might improve the separation.

Chipper Trial VII — Loblolly pine bolts collected in November in central Louisiana were shipped to the Institute, stored until

January and slabs chipped to provide an estimate of wood/bark separation that could be obtained as the result of chipper action. Chips were screened and the amount of bark with wood attached was determined. Twenty-six percent of the bark turned up as 3/4-inch size chips, 41% as 1/2-inch chips and 33% as 1/4-inch chips. Ninty-five percent of the 3/4-inch bark chips, 14% of the 1/2-inch chips, and 17% of the 1/4-inch chips had wood attached. The wood/bark separation for loblolly pine was considered to be better than for either hickory or spruce. Overall it appears that about 65% of the bark was "wood-free" bark. Failure occurred most often at the inner face between the inner and outer bark and to a lesser extent at the cambium zone. Also, the results again suggest that chipping to a smaller chip size may increase the percentage of "wood-free" bark.

Chipper action observations were made on a total of seven tree species. These preliminary observations on a conventional chipper suggest that for certain species the action of the chipper could be used, even during the dormant season, to cause a separation of bark and wood. The procedure seems to have worked best on thick bark species and species which have high enough specific gravity to result in the production of a fairly sizable percentage of small-sized chips. The poorest results occurred with white spruce, which in this trial had thin bark and low specific gravity wood that chipped readily.

Erickson (1) in a chipping study using both frozen and unfrozen wood from winter-cut northern pulpwood species obtained similar results. Spruce and hemlock gave the lowest percentage of "wood-free" bark and frozen wood increased the percentage of small-sized chips and increased the wood/bark separation.

Experimentation with chipper knife design, chipping angle, and ways of forcing the chips at high velocities against baffles can be expected to further improve the effectiveness of this approach to wood/bark separation.

CHEMICAL METHODS

Chemical methods, as a means of reducing wood/bark adhesion, were given some consideration in Progress Report 2. Described briefly was Dr. R. E. Kremers' work with dilute acids. In his work successful tests were made with black ash, quaking aspen, white birch, American elm, jack pine, silver maple, sugar maple, and black spruce. The principal disadvantage of the approach is the relatively long treatment time required, the discoloration of the wood that resulted for certain species, and the lack of effectiveness of the treatment on dried samples. In view of the magnitude of Kremer's work on dilute acids (IPC Research Project 1702), no further preliminary work has been initiated. It appears, however, that by working on chip size, temperature, and ways of increasing acid penetration, a satisfactory method employing dilute acids could be developed.

Also discussed in Progress Report 2 were short-term chemical treatments which employed NSSC, sulfite, kraft, and modified soda waste liquors at temperatures of 100, 150, and 200°F. and at treatment times of 5, 3, and 1 minutes. The best treatments were those conducted at the highest temperature and at the 5-minute treatment time. None of the treatments, however, were better than using water at the same time and temperature. Because it was felt that liquor penetration into the cambium zone region may have been a problem, several additional trials were made using higher temperatures and longer treatment times. The information that follows describes these additional trials that were run employing several new chemicals at higher temperatures and longer treatment times.

RECAUSTICIZED SODA BLACK LIQUOR

This liquor originally came from an IPC experimental cook. The liquor was recharged with calcium oxide (CaO) and made to a saturated solution. The pH after recharging was 12.6.

Tests were made in an oil bath at two different temperatures — 150°F. and 200°F. — at times ranging from 5 to 120 minutes. Five beakers containing the liquor were placed in the oil bath and fifteen standard aspen tabs (test chips) were placed in the first beaker. After five minutes three tabs were removed and tested and the rest of the tabs moved to the second beaker. After an additional ten minutes (total time of 15 minutes), three more tabs were removed and tested and the rest of the tabs were moved to beaker number 3. This procedure was repeated until tabs had been removed and tested at time intervals of 5, 15, 30, 60, and 120 minutes. The tabs were moved from beaker to beaker because it was felt there was a possibility that the tabs, with time, might lessen the effectiveness of the chemical. This was checked by running pH's on the liquor in all five beakers after completion of the treatments. Since the pH remained basically the same for all beakers, it appears the tabs did not lessen the effectiveness of the chemical.

Table III summarizes the results of the treatment with recausticized soda black liquor. There was some question as to whether the liquor treatments were more effective than water in reducing adhesion. The controls indicate that the liquor was somewhat better. The cambium area had an increasingly smoother look, when the wood and bark were separated, as the time of treatment increased. This was particularly true of the tests at 200°F. The area of penetration also appeared to increase with increasing treatment times. The table indicates that 60 minutes at 200°F. would probably be the minimum treatment for good wood/bark separation.

TABLE III

RECAUSTICIZED SODA BLACK LIQUOR RESULTS

Time, minutes	No Heat, water	Estimated Adhesion Values[a,b] 150°F. Water	150°F. Black Liquor	200°F. Water	200°F. Black Liquor
5	--	--	9.5	--	9.5
15	--	--	8.8	--	8.5
30	--	--	8.2	--	7.8
60	--	--	8.2	5.8	4.8
120	9.5	9.2	7.2	4.0	3.2

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressures, above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

KRAFT GREEN LIQUOR

A synthetic kraft green liquor was made up following the procedure of Springer, et al. (2). The liquor consisted of 0.76% sodium sulfide (Na_2S) and 3.0% sodium carbonate (Na_2CO_3). The procedure used for testing the recausticized black soda liquor was followed in this series of tests. Fifteen aspen tabs were placed in the first of five beakers and transferred until three tabs had been tested for each time interval (5, 15, 30, 60, and 120 minutes). The pH of each beaker of liquor was again checked after the tests to be sure the effectiveness of the chemical was not lost. The pH varied from 11.7 to 11.9.

Summarized in Table IV are the results of the treatment with the kraft green liquor. It appears that both liquor treatments were somewhat more effective than water in reducing adhesion with the kraft green liquor performing the best.

The cambium area of tabs treated with the kraft green liquor also had a smoother look when separated as the time of treatment increased. This was again particularly true of the tests at 200°F. Table IV indicates that between 30 and 60 minutes at 200°F. is probably the minimum treatment for good wood/bark separation.

TABLE IV

KRAFT GREEN LIQUOR RESULTS

Time, minutes	No Heat, water	Estimated Adhesion Values[a,b] 150°F.		200°F.	
		Water	Green Liquor	Water	Green Liquor
5	--	--	9.2	--	8.5
15	--	--	9.2	--	8.5
30	--	--	8.5	--	6.5
60	--	--	8.2	--	4.0
120	9.5	9.2	8.2	4.0	3.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

KRAFT GREEN LIQUOR — 6 SPECIES

Since the results of the liquor treatments on aspen showed promise, the chemicals were tried on five additional species. Included in this trial was slash pine, a southern source of eastern cottonwood, white spruce, shagbark hickory, and white birch. Aspen was also included for comparison purposes.

Three beakers containing kraft green liquor were placed in an oil bath heated to 200°F. Three standard tabs from each species were placed in each beaker (tabs had been soaked in water overnight prior to use). Tabs from the first beaker were removed after 30 minutes. Tabs from the second beaker were removed after 60 minutes and tabs from beaker no. 3 were removed after 120 minutes. The results of this trial are shown in Table V.

TABLE V

KRAFT GREEN LIQUOR RESULTS ON SIX SPECIES

	Estimated Adhesion Values[a,b]				
	Oil Bath Treatment, min.				
Species	30	60	120	Control, soaked overnight	Control, kept moist, no soaking
Slash pine	6.2	4.5	3.5	8.0	8.5
Cottonwood	4.5	4.0	3.0	7.0	--[c]
Aspen	6.8	5.2	4.0	9.5	9.5
White spruce	5.5	4.0	3.0	8.2	9.0
Shagbark hickory	10.0	8.5	6.0	10.0	10.0
White birch	8.5	8.0	6.2	10.0	10.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

[c]Supply of tabs exhausted.

Soaking the tabs seemed to have a slight effect on wood/bark adhesion as evidenced by the control. It is possible the slight reduction was due primarily to increased pliability of the bark which made manual separation easier. Again, greater reduction of wood/bark adhesion was evident at increasing treatment times for all species. It appears that 60 minutes at 200°F. would work well for all species except shagbark hickory and white birch. The cambium area was smooth after the 30-minute treatment for all species except hickory and birch. This seems to indicate that even the 30-minute treatment had some effect on the cambium area of these species. This observation is also borne out by the adhesion values in Table V for the 30-minute treatment vs. the controls.

Tests with the two chemicals showed promise as a means of reducing adhesion in all species tested. There did not appear to be any particular discoloration of the tabs except on the immediate surface and even that faded as the tabs dried. The use of the kraft green liquor appears particularly promising. In the work by Springer, et al. (2), investigations were under way using this chemical as a preservative for chip piles stored outdoors. It seems feasible to use the chemical to reduce wood/bark adhesion in chips and at the same time treat the chips for storage.

THERMAL METHODS

Preliminary work on thermal methods of reducing wood/bark adhesion produced some very promising results. These results, which were reported in Progress Report 2, suggested the need to know more about the influence and interaction of treatment time, high temperatures, and pressure on wood/bark adhesion. The following sections describe several experimental trials which were run in an effort to isolate the influence of the above factors and at the same time determine the "species suitability" of one or more of the most promising procedures.

AUTOCLAVE TREATMENTS

As was stated in Project 2929, Progress Report Two, autoclave treatments were used many times in other work to separate bark from wood on disk samples. Based upon this, an autoclave trial was made using standard aspen tabs and promising results obtained for the 15-lb. treatment for a period of three minutes (see Progress Report Two, p. 36). Because the autoclave trials worked well for aspen, it was decided to try the method on an additional five species. Three standard tabs were used for each species and were soaked in water overnight prior to use. The tabs were placed in a Petri dish without water. A small, electrically heated autoclave, which had been heated up in advance of the treatments, was used. The tabs were treated for five minutes at 15 lb. pressure and at a temperature of 250°F. However, 3 minutes and 26 seconds were needed to bring the autoclave to the required pressure and it took 3 minutes for the autoclave to drop to no pressure after the treatment. The total elapsed time was 11 minutes and 26 seconds. Table VI summarizes the results of the treatment.

TABLE VI

AUTOCLAVE TREATMENTS ON SIX SPECIES[a,b]

Species	Estimated Adhesion	Control[c] tabs soaked overnight	fresh tabs
Slash pine	5.8	8.0	8.5
Cottonwood	4.5	7.0	--[d]
Aspen	5.2	9.5	9.5
White spruce	5.0	8.2	9.0
Shagbark hickory	10.0	10.0	10.0
White birch	6.8	10.0	10.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — requires knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

[c]Control samples tested at room temperature.

[d]Supply of tabs exhausted.

As can be seen from the table, shagbark hickory was relatively unaffected by the treatment. The outer bark of the white birch curled up during the test but, although there was some reduction in adhesion, the inner bark was still fairly well attached to the wood. The other species were either separated by hand pressure or easily using a knife.

BOILING WATER TREATMENTS

It appeared from other tests that time was an important factor in any treatments used to reduce wood/bark adhesion. In some of the previous trials the times used had been too short to effect much reduction in adhesion. It appeared desirable to check if longer treatment times at 212°F. might be a simple solution to the adhesion reduction problem.

Standard aspen tabs were boiled in water for up to 30 minutes. At 12 minutes the bark could not be removed by hand but separated easily using a knife. At 15 minutes it was just possible to remove the bark using hand pressure. The adhesion continued to decrease until after 30 minutes the wood and bark separated easily. A control tab was very rough on the failure surfaces. After two minutes of boiling, the separation surfaces were still rough but becoming smoother. At five minutes failure occurred in the cambium area and both separation surfaces were smooth. All surfaces after longer boiling periods were also smooth with failure occurring in the cambium area.

ETHYLENE GLYCOL TREATMENTS

Further information was desired on the effect of temperature on wood/bark adhesion. The autoclave experiments led to the question of whether pressure was important in itself or important because it was then possible to obtain temperatures above the boiling point of water. Several chemicals with boiling points above 212°F. were examined for use in temperature tests. They included tripropylene glycol, ethylene glycol, diisobutyl ketone and perchlorethylene. Ethylene glycol was selected for use because of its availability and high boiling point (379.4-401.0°F.). Standard aspen tabs were soaked overnight in water and immersed in ethylene glycol heated to 370.4°F. on a hot plate. The temperature of the ethylene glycol dropped to 294.8°F. after immersion of the tabs. It began rising after five minutes and at ten minutes read 302.0°F., at 20 minutes read 327.2°F. All tabs were quenched in water at 150°F. after the tests. Other preliminary observations at high temperatures indicated too long a treatment at very high temperatures resulted in the tab "cooking-dry" and a resulting increase in adhesion.

Summarized in Table VII are the results of this experiment. Extensive controls were run to determine whether the chemical itself was having any effect on wood/bark adhesion. It appears the chemical by itself had little or no effect even after long periods of time. The cambium area was smooth on separated surfaces after the five-minute treatment. Cracks appeared between the bark and wood during the ten-minute treatment and the two were easily separated. The wood and bark fell apart during the 20-minute treatment. It appears that both temperature and time are important factors in reducing wood/bark adhesion.

TABLE VII

ETHYLENE GLYCOL TREATMENTS ON ASPEN

Length of Treatment	Estimated Adhesion Values[a,b] Ethylene Glycol at 370.4°F.	Control[c]
5 minutes	4.0	9.2
10 minutes	2.0	9.2
20 minutes	0.0	9.2
8 hours	--	9.2
24 hours	--	9.2
32 hours	--	9.2

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — requires knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

[c]Ethylene glycol at room temperature.

An ethylene glycol experiment was run on five additional species to check the effect of elevated temperatures on these species as compared to aspen. The test was run for ten minutes at 302.0°F. Three tabs from each species were soaked in water overnight prior to use. Tabs from all species were immersed in a beaker of ethylene glycol heated to the 302.0°F. temperature. The temperature of the chemical stayed fairly constant, varying only slightly from the initial 302.0°F. Duplicate trials were run. Table VIII summarizes the results of these treatments.

TABLE VIII

ETHYLENE GLYCOL TREATMENTS ON SIX SPECIES

Species	Estimated Adhesion[a,b]			
	Ethylene Glycol at 302.0°F.		Control[c]	
	Test 1	Test 2	soaked in water overnight	fresh
Slash pine	4.8	4.5	8.0	8.5
Cottonwood	4.0	4.0	7.0	--[d]
Aspen	4.2	4.0	9.5	9.5
White spruce	2.8	2.8	8.2	9.0
Shagbark hickory	5.8	5.5	10.0	10.0
White birch	6.0	5.5	10.0	10.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — requires knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average to three determinations.

[c]Control samples tested at room temperature.

[d]Supply of tabs exhausted.

White birch appeared less affected by the treatment than the other species. As in other tests, the other bark lifted up but the inner bark remained attached to the wood. In all species it was desirable to quench the tabs in warm water.

Because of the extreme heat they dried out quickly when removed from the ethylene glycol and a loss in ease of bark removal was noticed. Some evidence also exists (3) that too rapid cooling will cause the substances solubilized by the heat treatment to again become insoluble and adhesion to increase to near the original level. As was the case with aspen, temperature also appears to be an important factor in reducing adhesion in these additional species.

PRESSURE CHAMBER TREATMENTS

A ten-gallon chamber fitted with a pressure top and a jacket which could be pressurized and heated to create pressure indirectly was being used at The Institute of Paper Chemistry for other phases of wood/bark work. This chamber creates conditions similar to the autoclave and indicated a possibility of scaling up the autoclave method. Use of this pressure chamber could also confirm the usefulness of the autoclave work.

The same six species used in other phases of the thermal treatments were also used for the pressure chamber tests. Three standard tabs from each species were used in each test and all tabs were soaked overnight prior to use. The tabs were placed in a basket and put in the chamber which had been filled with water to approximately one inch below the basket top. The chamber was closed and various pressures employed for the trials. The tabs were tested immediately upon removal from the chamber. Listed in Table IX are the results of the pressure chamber tests.

It appears that 15 p.s.i. and ± 206.6°F. for eight minutes would be adequate for good adhesion reduction in all species tested except white birch and shagbark hickory. As in the other tests, these two species were the most difficult to work with. Based on these tests, 15 p.s.i. at 203.0°F. for 15 minutes would be the minimum treatment for shagbark hickory and white birch for good adhesion reduction.

TABLE IX

PRESSURE CHAMBER TREATMENTS ON SIX SPECIES[a]

Conditions	Trial No. 1	2	3	Control[b]
Pressure, lb./in.^{2}	10	15	15	--
Temperature, °F.	185	207	203	--
Time at pressure, min.	5	8	15	--
Total treatment time, min.	7	10	18	--
Species				
Slash pine	5.8	4.8	3.5	8.0
Cottonwood	4.2	3.5	4.0	7.0
Aspen	7.5	5.5	4.8	9.5
White spruce	--	2.2	2.0	8.2
Shagbark hickory	--	10.0	6.5	10.0
White birch	--	9.5	6.5	10.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure; above 5 — requires knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Control tabs soaked in water overnight and tested at room temperature.

MISCELLANEOUS METHODS

Several additional techniques for reducing wood/bark adhesion were investigated that did not fall comfortably into the mechanical, chemical, or thermal categories. These procedures have been grouped under miscellaneous methods and include ultrasonic treatments, biological treatments, and microwave treatments. The results of the preliminary investigations into these procedures are described below.

ULTRASONIC TREATMENTS

Several researchers have reported the treatment of wood (4) and pulp (5) using ultrasonic techniques and changes in the elastic properties of beechwood and the strength properties of pine pulp were described. With this very minimum background, standard aspen test chips were prepared and treated with low and medium frequency ultrasonic treatments. The water temperature in which the treatments were performed was held constant at 35-40°C. for the medium frequency treatment and 30-32°C. for the low frequency treatment. Such variables as position in the sonic chamber, voltage, and treatment time were evaluated. A Branson automatic cleaner model D-50 was used for the low frequency treatment and a GE ultrasonic generator no. 8665966G3 was used for the medium frequency treatments. The test tabs were from freshly cut trees and were soaked at room temperature in water for one day prior to treatment. Tests for adhesion were made immediately after removal from the sonic chamber. Table X briefly summarizes the conditions of the trial and illustrates the changes in adhesion that resulted. As can be seen from the results, the ultrasonic treatments employed had little or no influence on wood/bark adhesion. Technical advice regarding ultrasonic treatment suggested that the low and medium frequencies would be the most effective. Based upon the limited ultrasonic treatments made at the medium and low frequencies, the use of this procedure appears to have little promise as a method of reducing wood/bark adhesion.

TABLE X

EXPERIMENTAL CONDITIONS AND RESULTS OF ULTRASONIC TREATMENTS

Conditions	Ultrasonic Treatment Medium	Ultrasonic Treatment Low
Frequency, kilocycles	300	20-30
Voltages, milliamperes	150-175	115
Temperature, °C.	35-40	30-35
Treatment Times, min.	Adhesion Ratings[a,b]	
0	9.2	9.2
5	8.8	8.8
10	8.8	8.8
20	8.5	8.8
40	8.5	8.8

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure; above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

BIOLOGICAL TREATMENTS

Biological treatments including reactivation of cambium cells have been suggested as ways of reducing dormant season wood/bark adhesion. Most previous work in this area has involved standing trees or bolts of wood rather than chip samples (6). Springer and Hajny (7) reported that rapid initial increases in chip pile temperatures were believed to be the result of respiration by living ray parenchyma cells. With this information at hand it appeared worthwhile to try several preliminary treatments aimed at reactivating the cambium and ultimately reducing wood/bark adhesion. The chemicals selected were two common growth regulators, indole-3-acetic acid (IAA) and gibberellic acid (GA), and dimethylsulfoxide (DMSO), a chemical noted for its ability to increase the uptake of associated chemicals by plants and animals. The DMSO treatment was used in an attempt to increase the uptake of IAA and GA.

Fresh chip samples were prepared in mid-February and the chips soaked at room temperature for 24 hours in the chemical and at the concentrations shown in Table XI. After soaking for 24 hours the chips were removed and placed in a sealed polyethylene bag with two paper towels treated with a small amount of the original solution. The bags containing the chips were stored at 30 ± 2°C. and wood/bark adhesion checked after 1, 2, 5, 9, 14, and 20 days. The simulated chips were evaluated for wood/bark adhesion at each of the times indicated and the values presented in Table XI are the average of 3 determinations, except as noted for the 20-day treatment. The temperatures in the bags were checked periodically and in no instance were temperatures higher in the bags than the 30 ± 2°C. indicated.

The results of the biochemical treatments, although interesting, did not produce reduced wood/bark adhesion as the result of the reactivation of the cambium. The water (control), IAA and IAA + DMSO treatments had reduced wood/bark adhesion at 20 days but this apparently was a result of fungus infection that caused a deterioration (rotting) of the inner bark regions. The DMSO treatment seemed to inhibit fungus development. In the case of the evaluations made at 20 days, several of the tabs that had inadvertently dried out sometime between the 14th and 20th day exhibited higher wood/bark adhesion than the moist, similarly treated 20-day samples. The result of the water treatment indicates the type of reduced adhesion that could be expected from storing chips in moist chip piles. Considerable discoloration of the wood developed in all treatments with the greatest color changes occurring in the cambium zone. The discoloration decreased following the removal of the bark and the drying of the wood chips. The increased adhesion as a result of drying indicates that to take advantage of the reduced adhesion that developed as a result of moist chip storage, the chips would need to be mechanically treated while still in a moist condition. These results suggest that a simple moist chip storage followed by mechanical action could be used to prepare the chips for segregation treatments using screening, flotation, or the Hosmer compression technique.

TABLE XI

CHANGES IN WOOD/BARK ADHESION AS THE RESULT OF BIOCHEMICAL TREATMENTS[a,b]

Treatment	Treatment Time, days						Remarks
	1	2	5	9	14	20	
Water	9.2	9.2	9.2	6.8	4.5	4.2	Heavy fungus activity
IAA (200 mg./l.)	9.2	9.2	8.8	6.8	6.8	3.0	Very heavy fungus activity
IAA (200 mg./l.) + DMSO (10%)	9.2	9.2	8.8	5.8	6.2	3.0	Moderate fungus activity
GA (100 mg./l.)	9.2	9.2	9.2	6.8	7.2	7.0	Moderate fungus activity
GA (100 mg./l.) + DMSO (10%)	9.2	9.2	9.2	6.8	6.2	6.8	No fungus activity
DMSO (10%)	9.2	9.2	9.2	8.8	8.8	8.2	No fungus activity

[a]Average wood/bark adhesion based upon three determinations, except in several instances on the 20-day treatment where one or more tabs dried out.

[b]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure; above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

MICROWAVE TREATMENTS

Microwaves are electromagnetic waves which have a wavelength in the centimeter range. Microwaves are being investigated industrially for many uses including drying lumber and paper (8, 9). The procedure has the advantage of concentrating the drying action and the highest temperatures in the zone of highest moisture content. This action intends to dry the products more uniformly than normal methods. High temperatures are created by the microwaves with temperature development dependent upon the moisture content, the wavelength frequency and the duration of the treatment. Since fresh wood chips with bark attached tend to have fairly high moisture content in the cambium zone, it seemed reasonable to try using microwaves to create high temperatures in the cambium zone and thus reduce wood/bark adhesion.

All modern microwave generators are electronic devices which produce continuous wave oscillations of a tunable frequency. No microwave generator was available at the Institute and the only feasible way to investigate the idea was to use commercial microwave ovens that were built for home use. Two ovens, one of low frequency (915 megahertz, 1320 watts) and the other of high frequency (2450 megahertz, 700 watts) were available for use in the preliminary work.[3] The high frequency microwaves had little influence on either the temperature or adhesion of the relatively thin chips involved and as a result all subsequent trials involved the 915 megahertz, 1320 watts treatment.[4] Dormant aspen test chips were used in the preliminary treatments. Since moisture content

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[3]Wisconsin Michigan Power Company's assistance in supplying the two ovens used is gratefully acknowledged.
[4]The higher frequency ovens are apparently used for cooking items of greater thickness where greater penetration of the microwaves is required.

was believed to be critical, both freshly prepared chips and chips that had been soaked for a minimum of 24 hours were used as test specimens. Table XII summarizes the results obtained when treatment times ranged from 15 seconds to 5 minutes and where both uncovered and covered glass dishes were used as containers for the chips. Aspen wood/bark adhesion was reduced from 9.5 for the control to as low as 5 on the 3-minute treatments. The temperatures of the test chips appeared to be near 200°F. and wood/bark adhesion was evaluated as soon as the chips cooled enough to be handled. There was little or no difference in wood/bark adhesion for the 1, 2, and 3-minute treatments. The 5-minute treatment caused the test specimens to dry out and the bark to become very tight (10 rating). Treatment of the test chips in a covered container, in order to maintain a high moisture content for as long a period as possible, gave approximately the same results as the treatments made in the uncovered dishes.

The reduction obtained in adhesion for aspen looked promising enough that the 1 and 2-minute treatments were tried on 5 additional tree species. Table XIII summarizes the results of these additional low frequency treatments. The two-minute treatment of wet chips in an uncovered container looks particularly promising for spruce, cottonwood, aspen, and slash pine. Longer, more severe treatments appear to be required for hickory and birch. This will require special manipulation to keep the chips moist and/or increase operating temperature.

At first glance, the results of the microwave treatments do not look spectacular but when the many factors involved are considered, it appears the approach warrants further investigation. The treatments can be of relatively short duration, temperatures tried did not exceed 212°F., treatments can be made o either fresh or wet chips, and treatments can be applied under a variety of conditions (covered, uncovered, or under conditions that create higher than normal atmospheric pressure). In addition, the very restricted microwave frequencies used make it entirely possible that suboptimal frequencies were being employed. Modifications that would permit generation of higher temperatures and use of slightly longer treatment times (without drying) are worthy of consideration.

TABLE XII

CHANGES IN WOOD/BARK ADHESION
MICROWAVE TREATMENTS ON ASPEN TEST CHIPS[a,b]

Treatment Time, min.	915 Megahertz — Covered Dish	
	Fresh Chips	Wet Chips
Control	9.5 — bark tight	9.5 — bark tight
1/4	7.8 — some reduction; cambium rough	7.5 — comparable to fresh chips
1/2	7.0 — cambium becoming smoother	6.5 — cambium smooth
1	5.2 — separated by hand with difficulty	5.2 — comparable to fresh chip results
2	5.2 — same as one-minute treatment	5.2 — same as one-minute treatment
3	5.0 — adhesion slightly less than 1-minute treatment	5.5 — some evidence of drying of test chips
5	10.0 — very tight and dried out	9.8 — very tight and dried out
	915 Megahertz — Uncovered Dish	
1/2	--	6.8
2	5.5 — knife required to separate	5.5 — same as fresh chips
3	--	5.0 — separated by hand with some difficulty

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure; above 5 — require a knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

TABLE XIII

CHANGES IN WOOD/BARK ADHESION
MICROWAVE TREATMENT[a,b]

Species	Control	915 mHz, 1 minute, Covered Dish	915 mHz, 2 minutes, Uncovered Dish
Slash pine	8.0	6.5	5.5
Cottonwood	7.0	5.0	4.5
Quaking aspen	9.5	6.5	6.0
White spruce	8.2	6.5	4.0
Shagbark hickory	10.0	10.0	8.5
White birch	10.0	9.8	8.5

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure; above 5 — require a knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of three determinations.

COMPARISON OF METHODS

After considerable thought regarding both wood/bark adhesion and the segregation of wood/bark mixtures (Project 2977), it became increasingly evident that there was little chance that the studies underway would completely solve the bark problem of a specific mill. Differences in bark characteristics, use of species mixtures, and differences in product bark tolerance, bark markets and existing equipment are a few of the factors that enter into a mill's decision on how they should handle their "specific" bark problem. It appears that the greatest service this project and Project 2977 could provide would be to supply the co-operating agencies with the basic data on wood/bark adhesion and the flotation behavior of wood and bark. It would be up to each company to "put it all together" and in this way develop solutions to specific mill problems.

REVIEW OF PROMISING METHODS

The methods of reducing wood/bark adhesion that were tested included mechanical, thermal, chemical, and biological. The mechanical method was limited to observations on the action of the chipper in separating wood and bark. The "chipper" method appeared to have promise with several tree species including bur oak, quaking aspen, hard maple, and loblolly pine. Tests with shagbark hickory and white birch were only partially successful but, for all species, methods of improving the separation appear to exist.

Several chemical methods also look promising including the use of weak acids and green kraft cooking liquor. In the use of kraft cooking liquor, adhesion values were reduced to acceptable levels after 60 minutes at 200°F. for all species except hickory and birch. The principal disadvantages are the long treatment times and the high temperatures required.

Several types of thermal methods were tried and they looked quite promising with total treatment times being reduced to 10-15 minutes if temperatures above 250°F. can be employed. Time and temperature appear to be interrelated. Use of pressure vessels makes it possible to use temperatures greater than 212°F. and in this way shorten treatment times. The secret of the thermal method appears to be to swell and soften the cell walls of the cambium zone and maintain the cambium zone in this condition until the separation can be effected by mechanical action.

The microwave method is basically a thermal method and because of short treatment times and the flexibility in the ways the treatment can be applied, it appears that this method could be quite easily "engineered" into a chip handling system. Chips could be soaked for a few hours, processed by microwave heating using a pressurized continuous flow procedure that produces temperatures greater than 212°F. and then treated mechanically to complete the required separation. Segregation could then follow via one of several procedures being developed by interested agencies.

Biological methods of reducing wood/bark adhesion were also considered. One of the simplest procedures of all the methods tried appeared to be to store moist chips under aerobic conditions at 80-90°F. and allow the action of fungus to cause a breakdown of the cambium and inner bark region. Fungus action appears to be concentrated in the cambium zone and inner bark because of the higher than average concentrations of sugars and other essential growth nutrients. Times required for the moist storage method appear to be from 15 to 20 days. Possible chip discoloration is one aspect of this approach that would need to be investigated further.

VARIATION BETWEEN SPECIES

Looking back over the procedures described in the previous section, it is apparent that there is no one best method for all species. Shagbark hickory and white birch appear to be the most difficult to handle regardless of the procedure employed. White spruce, because of thin bark and low wood and bark specific gravity did not separate satisfactory as a result of the mechanical action of the chipper but demonstrated considerable reduction in wood/bark adhesion as a result of chemical and thermal treatments. An approximate ranking of the investigated species as to ease of reduction of adhesion results in the following list: (1) white spruce (easiest), (2) cottonwood, (3) slash pine, (4) quaking aspen, (5) shagbark hickory, and (6) white birch (most difficult). The reason for the particularly low response of hickory and white birch to the various treatments is not known. The width of the dormant season cambium zone does not seem to be related to treatment response. One possibility is that narrow, thicker-walled cells are involved in the cambium zone of trees of high specific gravity and this results in the need for longer treatment times, higher temperatures, and/or more drastic measures to swell and soften the cambium zone. Further investigation comparing high and low specific gravity species would be required to confirm this speculative relationship.

A review of species debarking requirements and the methods available for reducing adhesion (chemical, mechanical, thermal, microwave, and biochemical) makes a combination of methods appear as a possible way of handling species mixes and problem species. The mechanical action of the chipper on certain species tends to concentrate the bark in the larger chip fractions. Screening the chips and treating the problem fractions with a combination of microwave and wet thermal methods illustrates one possible use of a combination of procedures. Mechanical chipper action combined with screening and the wet chip storage is an example of another possible treatment combination. Mill requirements will dictate the combination of methods selected and it is obvious there are many possibilities.

PLANS

All field and laboratory work on this project has been completed. Progress Report Four is the final report for the project. A meeting of interested cooperators is planned with the objective of reviewing results and indicating several ways additional wood/bark adhesion research could be carried out and efficiently contribute to solving the many facets of the "bark problem."

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