Laboratory evaluation of the possibility of using sulfuric acid as a wood chip preservative

ABSTRACT

The possibility of preserving wood chips by reducing chip pH with mineral acids was studied on a laboratory scale. Reducing chip pH by treatment with dilute aqueous solutions of sulfuric acid was found to be effective in preserving small quantities of chips. A pH of 1.7 was effective in preserving aspen chips stored in 4-ft³ insulated boxes for 6 months; however, a pH of 1.8 did not preserve slash pine chips stored in a 200-ft³ chip pile simulator. The chip pH of 1.8 allowed the growth of sufficient numbers of micro-organisms to cause substantial heat release which resulted in hydrolytic damage to the wood fibers and a consequent weakening of the kraft pulp produced from the stored chips. To stop microbial growth and the resulting heat release a lower chip pH was evaluated. A pH of 1.2 effectively stopped heat release and preserved slash pine chips stored for 6 months in insulated boxes. Very small to negligible reductions occurred in the strength properties of kraft pulps produced from the treated, stored chips. Further study is needed before mineral acid treatment can be recommended for preserving large quantities of chips.

KEYWORDS

Populus tremuloides
Pinus taeda
Pinus elliottii
Sulfuric acid
Chips
Preservation
Tall oil
Storage
Kraft pulping

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The growth of bacteria, fungi, and other micro-organisms is highly dependent upon the environment in which they are located. One of the most important environmental factors is the substrate acidity or hydrogen ion concentration. Most wood-destroying fungi can grow between pH 2 and pH 8. Optimal growth usually takes place at about pH 5 to 6. Growth of wood-destroying fungi is checked or else is very poor under alkaline conditions (*1*). Treatment of wood chips with alkaline solutions such as kraft green liquor (*2*), white liquor, and sodium hydroxide (*3*) has been shown to inhibit microbial growth. On the acid side, Henningsson has shown that the growth of white rot fungi is stopped at about pH 2 to 3, while brown rot fungi are able to grow even at substrate pH as low as 1.5 (*4*).

A very limited effort has been directed toward preservation of stored chips by lowering the pH. This is probably because of the fear of greatly reducing the strength of pulps produced from the stored chips as a result of acid hydrolysis during storage. Treatment of chips with acidic evaporator condensates from sulfite mills has been reported to inhibit deterioration (*5*). Laboratory experiments on treatment of red alder chips with sulfur dioxide gas showed the treatment to be effective in reducing fungal growth and also resulted in some bleaching of the chips (*6*). Treatment of hardwood chips with sulfur dioxide gas for improving the brightness of bisulfite and chemi-mechanical pulps is now in commercial use at two pulpmills in the United States (*7*). The gaseous sulfur dioxide treatment, no doubt, effectively lowers the pH of the chips. It is not certain whether the preservative effect of sulfur dioxide results from reduced pH or from another mechanism, since aqueous solutions of sodium bisulfite, with a pH of about 4, are about as effective as gaseous sulfur dioxide (*8*). The studies on sulfite evaporator condensates and sulfur dioxide are the only known instances of the use of low pH to inhibit wood chip deterioration.

The purpose of this study was to investigate the possibility of using mineral acids to preserve stored wood chips. It was thought that lowering the pH of the chips using, for example, sulfuric acid might be a cost-effective method for reducing chip deterioration. It was, however, feared that long-term exposure of the wood under conditions of high acidity might result in serious weakening of the resultant pulp.

Initial screening

A simple screening test was devised to study the influence of chip pH on inhibition of the growth of micro-organisms on acid-treated chips. Aspen (*Populus tremuloides*) and loblolly pine (*Pinus taeda*) chips were immersed for 15 s in various concentrations of sulfuric and nitric acids, and the pH of the liquor in the chips was determined by squeezing the liquor from the well-drained chips using a hydraulic press. Samples of each type of treated chips were placed in 1-quart fruit jars having filter paper lids. A small quantity of water was present in the bottom of each jar; it was separated from the chips by a raised

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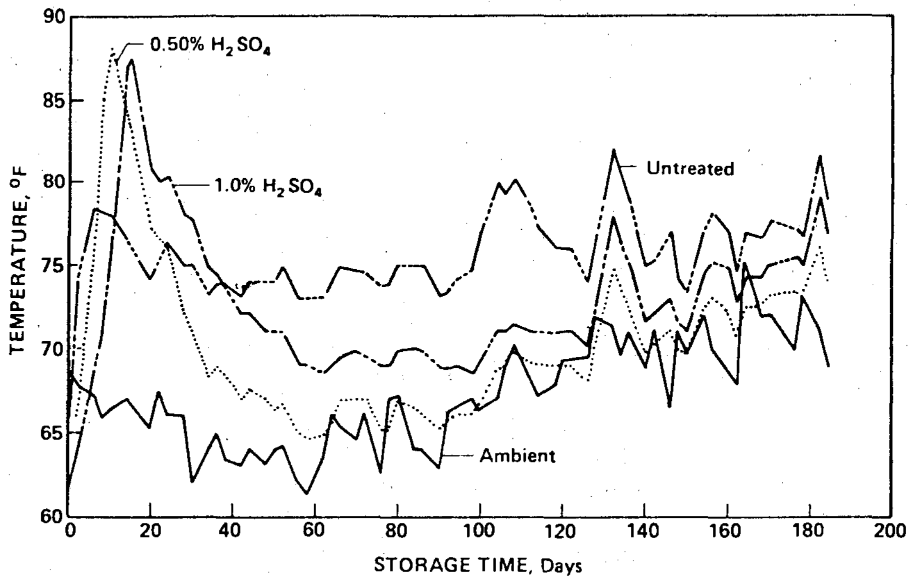
plastic grate. The jars were stored in an 80°F (29°C), 90% relative humidity room, and the chips were visually observed weekly for growth of micro-organisms. It was found that the pH of the chips remained essentially constant during storage times of several months and that a pH below 1.3 was required to completely stop the visible growth of all micro-organisms. Type of wood or mineral acid used did not appear to be of importance.

Study of aspen chips in insulated boxes

To determine the effect of long-term storage on the strength of pulp produced from acid-treated chips, a larger-scale testing procedure was used. Fresh aspen chips (*Populus tremuloides*) were immersed for 15 s in solutions containing 0.50, 0.55, 0.60, and 1.0% sulfuric acid. The chips were drained and then stored in 4-ft^3 (0.113-m^3) insulated boxes for six months. Two boxes were filled with water-soaked chips to act as controls. A small, measured volume of water-saturated air was continuously passed through each box. Three small, weighed samples of chips (300 g of fresh chips) in nylon mesh bags were placed near the center of each box to determine weight loss on storage. Center temperatures were observed with copper–constantan thermocouples. The construction of the boxes and the exact experimental techniques used have been previously described (*9*).

The initial fresh chips and chips from the boxes after storage were subjected to kraft pulping using the following conditions: 14.0% active alkali; 25.0% sulfidity; 4:1 liquor to wood ratio; 60 min from 176°F (80°C) to 338°F (170°C); 75 min at 338°F (170°C).

The pH of the chips before and after storage, the average weight loss, the brightness of the chips, and the number of micro-organisms present in comparison with the control chips are given in Table I. The brightness and the number of micro-organisms present in a box containing stored treated chips were determined by visual comparison with the chips in the untreated control box. The pH of the treated chips showed very little change during storage; the maximum change observed was 0.8 pH unit. Chip pH values above 1.3 were studied since it was thought that completely stopping the growth of all micro-organisms might not be necessary and that pH values as low as 1.3 might seriously weaken the resultant pulp. All treatments kept the chips brighter than the control chips and very significantly reduced weight loss although, as expected, none of the treatments completely stopped the growth of micro-organisms.



1. Relationship between temperature of center of insulated box and storage time for sulfuric acid-treated and untreated aspen chips.

The temperatures observed at the center of the insulated boxes during storage of chips soaked in water, and in 0.50% and 1.0% sulfuric acid, are shown in Fig. 1. The acid-treated chips heated up slower but rose to a higher temperature than did the water-soaked control chips. After about a month of storage, the temperatures in the acid-treated chips fell below those of the control chips and remained lower during the remainder of the storage period. Apparently, the acid treatments stopped the respiration of the living wood cells, but did not inhibit the growth of certain acid-resistant organisms which flourished for a few weeks and then in large part died. Perhaps a specific biocide could be added to the acid solution to control these micro-organisms and thus completely suppress chip heating.

Table II gives the digester and overall yield values from kraft pulping of the original chips and of chips stored six months. It was attempted to achieve a kappa no. of 17 for each cook; however, some deviations occurred. These deviations did not appear to have a significant effect on digester yield. No attempt was made to compensate for the acid content of the treated chips during pulping. For the unstored chips, digester yield was about one percentage point higher for chips treated with 1.0% H_2SO_4 than for the untreated chips. This trend of higher yield with increasing acid concentration in the treating solution also occurred with the stored chips. Here digester yield was more than 3 percentage points higher for the chips treated with 1.0% H_2SO_4 than for the untreated chips. Based on the data for the untreated chips, it is unlikely that this much larger difference is entirely due to reduction of the active alkali during pulping. It is, no doubt, principally due to the lack of deterioration of the treated chips during storage. The difference in digester yields between the stored, water-soaked chips and the unstored, untreated chips was probably due to the much higher extractives content of the unstored chips. Digester yields have often been found to increase during the first several months of chip storage.

I. Results on sulfuric acid-treated and untreated aspen chips stored six months in insulated boxes

H_2SO_4 in treating solution, %	H_2SO_4 pickup, % of o.d. wood	pH		Weight loss, %	Brightness[a]	Micro-organisms[b]
		Before storage	After storage			
None	None	6.7	6.2	9.5	. . .	. . .
None	None	...	5.7	8.7	. . .	. . .
0.50	0.11	2.8	3.0	3.0	+	+
0.55	0.14	2.8	3.5	2.8	+	+
0.60	0.15	2.7	3.1	4.1	+	+
1.0	0.22	1.7	1.6	4.7	+	0

[a]Brightness scale: - = less bright than untreated; 0 = same as untreated; + = brighter than untreated: + + = much brighter than untreated. [b]Micro-organism scale: - = more micro-organisms than untreated; 0 = same number as untreated; + = fewer than untreated: + + = very few micro-organisms.

Overall yield is a much better indicator than digester yield of the deterioration, or lack thereof, that takes place in the stored chips. In this experiment, stored, untreated chips lost about 5 percentage points in overall yield; those treated with 0.50, 0.55, and 0.60% H_2SO_4 lost about 2 percentage points. The chips treated with 1.0% H_2SO_4 and stored six months suffered no loss in overall yield and responded to kraft pulping as if they were fresh chips.

Table III gives the pulp strength properties of the original and six-month stored chips. Pulp produced from the water-soaked control chips stored for six months was nearly identical in strength properties. to those produced from the unstored chips. Only tear factor showed a small decrease. The pulps produced from the sulfuric acid-treated, six-month stored chips were at least as strong as those produced from the original unstored chips. Even the pulps produced from chips treated with 1.0% sulfuric acid and stored for six months were as strong as those produced from the original unstored chips. Apparently, under these rigorous conditions of acidity, temperature, and storage time, fiber degradation caused by acid hydrolysis is negligible.

Based on these findings, it appeared to be possible to preserve stored wood chips using a mineral acid. Sulfuric acid would be the logical choice since it is the cheapest mineral acid. Since some heating occurred in the small mass of 1.0% sulfuric acid-treated chips in the insulated box, it might be expected that heating would take place in larger masses of acid-treated chips and thus higher temperatures be attained. These higher temperatures might give rise to serious fiber degradation by acid hydrolysis. Since it is impossible to calculate the temperatures that would be encountered with a larger mass of chips and, even knowing this, to also calculate the extent of pulp strength loss due to acid hydrolysis at these temperatures, it was considered necessary to adopt an experimental approach. In response to these considerations, slash pine chips were immersed in 1.0% H_2SO_4 and loaded into a chip pile simulator. Slash pine chips were used because they were readily available, and the initial screening indicated that wood species was not of importance.

Study of slash pine chips in chip pile simulator

Fresh slash pine (*Pinus elliotti*) chips were raked through a trough containing a 1.0% aqueous solution of sulfuric acid and then loaded into a chip pile simulator. Care was taken to fully submerge all chips, and contact time was about 15 s. Based on ovendry wood, the sulfuric acid pickup was 0.17% or 3.3 lb (1.5 kg)/ton of ovendry wood. The simulator was a 4.0-ft (1.2-m)-diameter by 16.0-ft (4.8-m)-high insulated cylinder [volume: 200 ft^3 (5.66 m^3)] lined with polyethylene film and fed at a very low rate (0.25 empty simulator volumes per day) with water-saturated air. All experimental equipment and techniques employed were identical to those used in previous work (*2,10*). For purposes of comparison, an identical simulator was filled with untreated slash pine chips.

Center-temperature profiles of the simulators filled with the sulfuric acid-treated and untreated chips are shown in Fig. 2. The acid treatment somewhat inhibited initial heating; however, the center temperature did rise to 114°F (46°C) and remained at about 100°F (38°C) during most of the six-month storage period. As expected, some growth of micro-organisms did occur.

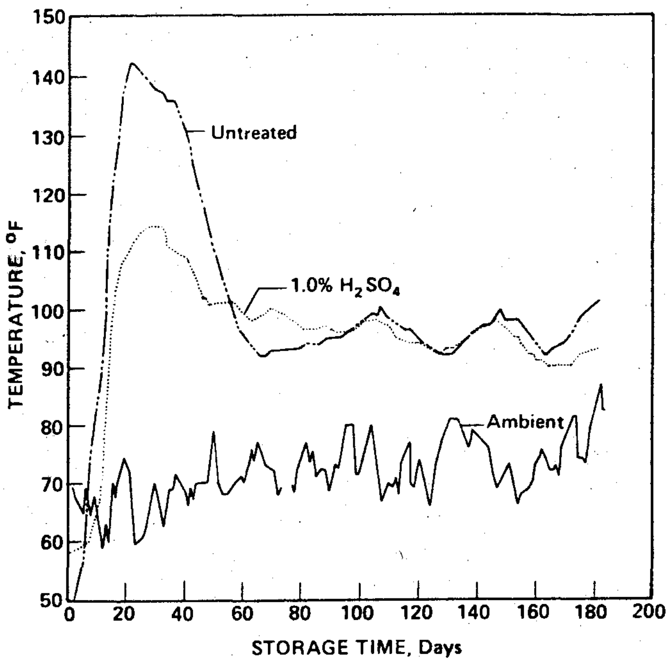
Nylon mesh bags containing weighed samples of chips, for determining wood substance loss, were placed in the simulators as in previous studies (*2,10*). After six months of storage, the chips were removed from the simulators, the sample bags retrieved, and weight losses

II. Digester and overall yields from kraft pulping of stored and unstored aspen chips

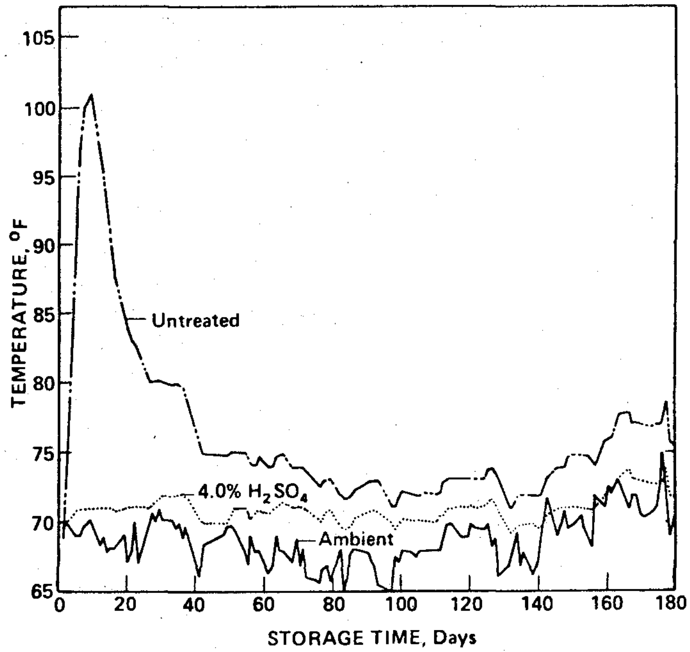
H_2SO_4 in treating solution, %	Active alkali, %	Weight loss, %	Kappa no.	Digester yield, %	Screenings, %	Overall yield, %
			Unstored			
Untreated	14.0	0	17.0	54.8	6.1	54.8
0.50	14.0	0	15.8	55.8	4.3	55.8
1.0	14.0	0	16.9	56.2	5.0	56.2
			Stored six months			
Water soak	14.0	9.5	18.3	56.2	0.6	50.9
Water soak	15.0	8.7	18.1	55.3	1.1	50.5
0.50	14.0	3.0	15.3	55.7	2.5	54.0
0.55	14.0	2.8	16.5	56.0	1.0	54.4
0.60	14.0	4.1	15.8	56.7	3.1	54.4
1.0	14.0	4.7	14.6	59.0	6.4	56.2
1.0	14.0	4.7	18.4	58.3	6.3	55.6
1.0	14.0	4.7	20.5	58.8	11.2	56.0

III. Pulp strength properties of stored and unstored aspen chips

H_2SO_4 in treating solution, %	Kappa no.	Brightness, %	C.S. freeness, ml	Beating time, min	Burst factor	Tear factor	Breaking length, km	Sheet density, g/cm^3
				Unstored				
Untreated	17.0	35	500–300	13–30	59–89	89–84	11.6–12.6	0.76–0.84
0.50	15.8	38	500–300	13–31	64–91	95–78	11.1–14.2	0.74–0.82
1.0	16.9	36	500-300	13–28	72–91	94–90	12.1–13.5	0.76–0.83
				Stored six months				
Water soak	18.3	29	500-300	14–36	69-95	84–73	11.2–13.5	0.77–0.85
Water soak	18.1	31	500–300	12–33	71–89	82–71	11.1–13.2	0.75–0.83
0.50	15.3	38	500–300	16–38	79–99	94-78	12.2–14.6	0.77–0.84
0.55	16.5	35	500–300	14–32	78–96	89–81	11.6–14.0	0.78–0.84
0.60	15.8	38	500–300	17–38	72-94	94-77	11.5–13.2	0.76–0.84
1.0	14.6	37	500–300	20-44	85-98	89–78	12.7–14.0	0.78–0.84
1.0	18.4	37	500-300	18–42	83–100	99-83	12.1–13.9	0.76–0.83
1.0	20.5	37	500–300	21–44	83–101	89–77	12.8–14.0	0.76–0.84



2. Relationship between temperature at center of chip pile simulator and storage time for sulfuric acid-treated and untreated slash pine chips.



3. Relationship between temperature at center of insulated box and storage time for sulfuric acid-treated and untreated slash pine chips.

determined. Table IV lists the ovendry wood substance losses at the various sample locations. The sulfuric acid treatment showed only a very small positive effect in retarding the loss of wood substance. The average pH of the chips going into the simulator was 1.9. At the end of storage, the average pH was 1.8. This level of acidity is apparently not very effective in inhibiting the growth of wood-consuming micro-organisms on slash pine chips.

IV. Loss in ovendry wood substance of untreated and treated slash pine chips after six months in the simulators

Sample location: Vertical, feet from bottom	Sample location: Cross section, feet from center	Sample size, kg	Weight loss, %: Untreated	Weight loss, %: Sulfuric acid
8	0	0.15	4.1	3.8
	1	0.15	4.4	2.5
	2	0.15	3.6	3.1
10	1	8	3.9	4.5
12	0	0.15	4.0	3.0
	1	0.15	4.5	2.9
	2	0.15	4.4	3.2

Samples of the initial chips and of stored chips contained in the large nylon mesh bags were subjected to kraft pulping using the conditions given in Table V. Pulp yields and properties are also shown. The sulfuric acid treatment reduced both digester and overall yields and seriously weakened all pulp properties except for tear factor. In contrast to the insulated-box results with aspen chips, the immersion of slash pine chips in 1.0% sulfuric acid and storage for six months in the 200-ft^3 (5.66 m^3) simulator had a detrimental effect on the resultant kraft pulp. The pulp properties were significantly lower than the properties of the pulp from the untreated, stored chips.

A sample of black liquor from each kraft cook was analyzed for tall oil using the method of Saltsman and Kuiken (*11*). Unstored chips contained 410% tall oil (ovendry wood basis). The untreated, stored chips retained 46% of their original tall oil, and the sulfuric acid-treated, stored chips retained 42%. The sulfuric acid treatment thus had no effect on losses of tall oil. Apparently, the larger mass of chips in the simulator resulted in a higher average chip temperature than in the insulated boxes, which promoted the growth of thermophilic micro-organisms and also the acid hydrolysis of the wood fiber carbohydrate polymers.

From the screening work, it was known that a chip pH below 1.3 was required to completely stop the growth of all visible micro-organisms on the chips. If the growth of all micro-organisms was stopped, all heating would be stopped and the chips might be preserved. At such a low pH, the danger of fiber damage due to acid hydrolysis would be increased. To investigate the possibility of preserving slash pine chips using a chip pH below 1.3 while at the same time avoiding fiber damage from acid hydrolysis, some additional work was done using the insulated boxes.

Study of slash pine chips in insulated boxes

Fresh slash pine chips were immersed for 15 s in a 4.0% solution of sulfuric acid and stored in the 4-ft^3 (0.113-m^3) insulated boxes for periods of time ranging up to six months. For purposes of comparison, one box was filled with untreated chips and the chips stored for six months.

V. Yields and physical properties of kraft pulps from unstored, untreated, and treated slash pine chips after six months storage[a]

Treatment	Weight loss, %	Digester yield, %	Overall yield, %	Kappa no.	Brightness, %	C.S. freeness, ml	Beating time, min	Burst factor	Tear factor	Breaking length, km	Sheet density, g/cm³
Unstored	0	47	47	47	21	500–300	44–59	74–81	189–182	9.8–10.0	0.61–0.62
Untreated	3.0	47	46	46	24	...	45–55	67–71	175–161	9.7–10.2	0.60–0.62
Sulfuric	4.5[b]	46	44	48	23	...	37–47	59–64	208–182	8.7– 9.2	0.59–0.62

[a]Pulping conditions. 17.5% active alkali. 25% sulfidity; 4:1 liquor-to-wood ratio. 90 min from 80° to 170°C. 75 min at 170°C. [b]Not corrected for chemical pickup

As shown in Table VI, the pH of the treated chips was 1.1 before storage and 1.2 after storage. Figure 3 gives the center-temperature profiles for the treated and untreated chips stored in the insulated boxes for six months. Very little heating occurred in the treated chips. Three nylon mesh bags containing weighed samples of chips for determining weight loss were placed at the center of each box. Table VI shows that no wood substance losses occurred during storage of the treated chips, whereas the untreated chips lost 5.1% of their ovendry weight. The treatment also had a beneficial effect on chip-brightness retention; however, some growth of micro-organisms was found on the treated chips after only 60 days of storage.

Randomly selected samples of initial untreated and treated chips, and of the stored untreated and treated chips were subjected to kraft pulping using the conditions listed in Table VII. For the treated chips, a 0.5% increase in active alkali was used to compensate for the acid present in the chips. In three instances cooking times were adjusted so as to attempt to obtain a pulp kappa no. of 50. The pulp yields and kappa numbers obtained are shown in Table VII. At equal kappa numbers, no significant change in digester yield occurred as a result of storage for either the treated or the untreated chips. A 2 percentage point drop in overall yield was found for the untreated chips stored for 180 days. No drop in overall yield occurred for the treated, stored chips. Although some growth of micro-organisms occurred, the treatment was very effective in stopping wood substance and yield losses. Table VIII gives the strength properties of the kraft pulps produced. The untreated chips stored for 180 days suffered small but significant losses in all strength values. The treated, stored chips showed some reduction in strength values but, in every case, the reduction was less than for the untreated chips. Storage of the treated chips for six months at a pH of about 1.1 had very little effect on pulp strength. Apparently, the reaction conditions were not severe enough to result in significant hydrolysis of the wood carbohydrate polymers.

Samples of black liquor from each kraft cook were analyzed for tall oil. Unstored chips contained 1.5% tall ail (ovendry wood basis). The untreated, stored chips retained only 31% of their original tall oil; the treated, stored chips retained 100%. The treatment was thus totally effective in preservation of tall oil. This was probably due to the fact that few micro-organisms were present and, consequently, little heating and no consumption of tall oil occurred.

Use of sulfuric acid to maintain the pH of slash pine chips at about 1.2 is thus a highly effective means for preserving small quantities of chips for at least six months. Since little heating occurred, it may also be effective for large masses of chips. This needs further study. In previous research with biocides, treatments which were highly effective in small scale tests often proved relatively ineffective in large scale or field testing. For example, one of the most promising biocides studied to date for preservation of wood chips is sodium N-methyldithiocarbamate (*10*). Unfortunately, in an actual outside pile test this chemical was effective only for approximately one month (*12*). Microencapsulation of sodium N-methyldithiocarbamate in a suitable material could slow its release and subsequent reaction and thus greatly prolong its preservative effect. This is a very promising area which warrants further study.

VI. Results on slash pine chips treated by immersion for 15 s in 4.0% sulfuric acid and on untreated chips stored in insulated boxes

Storage time, days	H_2SO_4 pickup, % of o.d. wood	pH: Before storage	pH: After storage	Weight loss, %	Brightness[a]	Micro-organisms[a]
180	None	4.9	4.7	5.1	...	...
60	0.76	1.1	1.2	0	++	++
120	0.75	1.1	1.2	0	++	+
180	0.75	1.1	1.2	0	+	+

[a]Refer to footnote in Table I.

VII. Digester and overall yields from kraft pulping[a] of stored and unstored slash pine chips

Storage time, days	H_2SO_4 pickup, % of o.d. wood	Weight loss, %	Active alkali, %	Kappa no.	Digester yield, %	Screenings, %	Overall yield, %
None	None	0	17.5	48.2	49.2	0.85	49.2
None	None	0	17.5	49.8	49.5	1.85	49.5
None	0.76	0	18.0	54.7	51.7	2.23	51.7
None	0.76	0	18.0	39.6[b]	49.5	0.79	49.5
180	None	5.1	17.5	50.1[c]	49.2	0.51	46.7
60	0.76	0	18.0	50.7	50.0	0.74	50.0
120	0.75	0	18.0	47.7	50.1	0.45	50.1
180	0.75	0	18.0	47.5[c]	49.4	0.49	49.4

[a]Pulping conditions: active alkali as above, 25% sulfidity. 4:1 liquor-to-wood ratio. 90 min from 80°C to 170°C. 75 min at 170°C. [b]90 min at 170°C. [c]65 min at 170°C.

Summary

Use of sulfuric acid to reduce chip pH is an effective means for preserving small quantities of chips for at least six months. The pH level required to stop chip heating and deterioration is about 1.2. It was initially assumed that long-term exposure to such low pH conditions would have a very adverse effect on the

VIII. Pulping strength properties of stored and unstored slash pine chips

Storage time, days	H_2SO_4 pickup, % of o.d. wood	Kappa no.	Brightness, %	C.S. freeness, ml	Beating time, min	Burst factor	Tear factor	Breaking length, km	Sheet density, g/cm^3
None	None	48.2	22	500–300	54–70	75–81	173–158	10.0–10.7	0.60–0.63
None	None	49.8	22	500–300	55–74	76–81	173–162	10.3–10.5	0.60–0.61
None	0.76	54.7	20	500–300	51–67	71–79	176–163	10.2–10.4	0.59–0.62
None	0.76	39.6	23	500–300	49–68	74–82	172–153	10.2–10.4	0.60–0.62
180	None	50.1	23	500–300	33–45	69–74	152–143	9.4 – 9.9	0.60–0.62
60	0.76	50.7	22	500–300	53–71	73–77	175–163	9.7 – 9.8	0.59–0.62
120	0.75	47.7	22	500–300	48–65	70–77	178–145	9.6 – 9.9	0.59–0.62
180	0.75	47.5	24	500–300	35–48	70–76	203–155	9.6–10.1	0.60–0.63

strength of pulp made from the acid-treated chips. Very little effect on pulp strength was observed. The treatment might be effective for large quantities of chips in piles or bins, but the possible heating of the chips under such circumstances needs very careful investigation.

If it were found that reducing chip pH to 1.2 was effective even in large masses of chips, such as in large chip piles, acid treatment might still be impractical. In an outside pile, rain would leach the acid from the chips located along natural channels down through the pile. The chips in these channels would be attacked by micro-organisms with resultant heat evolution. The pile, at least in these areas, would heat up and nearby acid-treated chips would undergo serious hydrolytic attack. Sheltering the chips from rain would be technically feasible; however, the cost of both acid treatment and shelter would probably be in excess of the benefits gained. Because of these considerations, no further experimental studies on acid treatment were undertaken.

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