Pilot trials of hemicelluloses extraction prior to thermomechanical pulp production: Part 1

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ABSTRACT: Pilot data indicate that wood chip pretreatment with oxalic acid reduced the specific energy required to make thermomechanical pulp. A combined oxalic acid/bisulfite treatment resulted in 21% refiner energy savings and 13% increase in brightness for aspen. A low level of oxalic acid treatment was effective for spruce. Energy savings of 30% was observed with no significant change in strength properties. Adding bisulfite did not significantly increase the brightness of the spruce pulp. For pine, the optimum treatment was a moderate level of oxalic acid, which resulted in 34% energy savings and an increase in strength properties. For all of these treatments 1–3 w/w % carbohydrates were recovered, which can be fermented to produce ethanol. The extract sugar solution contained significant quantities of arabinose.

Application: The energy required to refine pulps may be significantly reduced with oxalic acid treatment.

Previous publications have shown that low levels of wood chip pretreatment using oxalic acid (OA) solutions were effective for reducing refiner energy, increasing strength properties, and increasing carbohydrate extraction [1-3]. These benefits are associated with yield and brightness loss. Our study provided pilot-scale data that can be used in an economic evaluation of the impact of pre-extraction on mill profitability [4]. This work was funded by a consortium of paper companies, universities, and government agencies as part of Value Prior to Pulping (VPP).

EXPERIMENTAL

All trials were conducted at the Forest Products Laboratory in Madison, WI, USA. The pretreatment was conducted with a pilot-scale 1.2 kg o.d./min wood chips and a PREX pretreatment system supplied by Sunds Defibrator (Metso; Helsinki, Finland). The pretreatment system was modified to provide a second plug-screw feeder on the discharge of the pressure vessel. This additional equipment allows liquid to be expressed from the chips as they leave the system. Typically, the chips are 65% solids as they leave the second plug-screw. All pulps were subjected to one stage of pressurized refining at 30 psig, 12-in. Sprout-Bauer 12-ICP (Andritz; Gratz, Austria), followed by either one or two stages of atmospheric refining, Sprout-Waldon 12 in. (Andritz; Gratz, Austria), to obtain a target freeness of 150 mL CSF. We tested the physical properties of the pulps using TAPPI T220 sp-10 "physical testing of pulp handsheets," and we determined brightness using TAPPI T525 om-06 "diffuse brightness of paper, paperboard & pulp (d/0 diffuse)." We analyzed the extracts using high-performance liquid chromatography (HPLC) with amperometric detection [5].

	Aspen	Spruce	Pine
Control	X	X	X
OA	X	X	X
SO_2		X	
OA/SO_2	X		X

I. Conditions and wood species explored during pilot trials.

Table I shows the chemical conditions used for each wood species. The general operating conditions were:

- 135ºC for 10 min
- Chemical concentration varied between 0% and 1.2% on o.d. wood
- 1.2 kg/min commercial chips
- 2–3 L/min liquid flow
- Chips leave pretreatment at 65% solids

Aspen treatments with hot water for 120 min at 130ºC, 142ºC, and 151ºC were also conducted with this same system.

Statistical methods

For all trials, we analyzed the results using effect screening of a full quadratic model with OA and sulfite concentration as independent variables. The terms "OA const" and "SO_2 const" represent effects of the treatment that did not depend on concentration. These terms were included in the model to allow for the possibility that the chemical loading levels were above the active range and had reached an asymptotic value. The values in the results tables are fitted parameters for each particular term in the model. Our method was an iterative least-squares procedure, implemented using the JMP (SAS Institute Inc., Cary, NC, USA) statistical package. Terms were deemed significant if their calculated *p*-value was less than 0.05. An entry of "ns" indicates a *p*-value above 0.05. Also calculated were the corrected R^2 coefficients for each model,

Species	OA	SO_2	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
	% on o.d. wood	% on o.d. wood	% of o.d. wood	MWh/MT	N-m/g	mN-m^2/g	kPa-m^2/g	cm^3/g	% (TAPPI)
Aspen	0.00	0.00	1.6	2.60	24.4	0.887	2.80	2.44	53.6
Aspen	0.00	0.00	1.7	2.63	25.2	0.867	2.85	2.30	53.9
Aspen		Variance	0.1	0.00	0.3	0.000	0.00	0.01	0.0
Aspen	0.70	0.00	7.1	2.12	19.9	0.675	2.06	2.13	49.3
Aspen	0.75	0.00	4.2	2.05	18.6	0.644	2.05	2.23	48.4
Aspen		Variance	3.9	0.00	0.9	0.001	0.00	0.01	0.4
Aspen	0.73	0.73	5.1	2.18	23.2	0.866	2.90	2.29	60.8
Aspen	0.73	0.73	3.2	2.14	21.2	0.764	2.59	2.56	59.9
Aspen		Variance	1.8	0.00	1.9	0.005	0.05	0.04	0.4
Spruce	0.00	0.00	1.0	2.62	35.7	1.648	5.87	2.61	52.4
Spruce	0.00	0.00	1.0	2.70	37.7	1.676	5.79	2.36	52.5
Spruce		Variance	0.0	0.00	2.1	0.000	0.00	0.03	0.0
Pine	0.00	0.00	0.0	2.36	21.5	1.130	5.43	2.94	55.5
Pine	0.00	0.00	1.0	2.26	20.8	1.125	5.62	2.90	55.4
Pine		Variance	0.5	0.00	0.2	0.000	0.02	0.00	0.0
	Total variance		6.4	0.01	5.4	0.006	0.07	0.08	0.9
	Pooled SD		1.1	0.05	1.0	0.036	0.12	0.13	0.4
	95% Significant difference		**3.1**	**0.14**	**2.9**	**0.099**	**0.33**	**0.36**	**1.2**

II. Data used to determine the minimum significant difference.

including all the significant entries in a particular column of a table. For all dependent variables, models were significant if their *p*-values were less than 0.0001. Reduced models showing only the statistically significant terms were implemented in Microsoft Excel to allow for optimization.

Estimates of variance

Properly interpreting the experimental results required an estimate of the minimum significant difference. While it would have been desirable to conduct replicate trials for all conditions, cost and time constraints did not allow for this level of effort. Instead, we used a pooled estimate of standard deviation (SD) to develop an estimate of the minimum significant difference between two data points. **Table II** shows the data used to obtain these estimates. The data span the ranges of treatment and include values for all three wood species. The values in the bottom row of the table are the minimum differences that can be interpreted as significant at the 95% confidence level, which were calculated in Excel by the following formula:

= SQRT(2)*NORMSDIST(0.975)*STDEV(data)

For example, we interpreted any differences in refiner energy greater than 0.14 MWh/MT as significant. We ignored differences less than this value. Table II shows that differences in brightness of 2%, energy of 6%, and strength properties of 10% are significant. The carbohydrate removal values appeared to be the most prone to variation, as the estimated significant difference was nearly the same as the average value.

RESULTS AND DISCUSSION

Aspen

Table III shows experimental data acquired during the trials with aspen. In general, OA acid treatment resulted in significantly reduced refiner energy, increased carbohydrate removal, and decreased pulp properties. Because aspen thermomechanical pulp (TMP) is added to pulp blends to improve opacity and bulk, their loss of strength is not critical. The brightness loss, however, presents an unacceptable degradation. The addition of sulfite improved brightness compared to the control. For purposes of this paper, sulfite loading was expressed as sulfur dioxide (SO_2) equivalents.

Values in **Table IV** indicate that OA had a strong effect on all of the measured values presented, except bulk. We found that the addition of sulfite had effects that tended to be opposite in sign to the OA effects. Interpretation of the parameters shown in Table IV is complex, because of nonlinear terms. **Table V** shows the effect of a 1% loading of OA, SO_2, or both combined. Entries of "ns" indicate that the calculated effect

OA	SO_2	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
% on o.d. wood	% on o.d. wood	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
0.00	0.00	1.6	2.60	24.4	0.887	2.80	2.44	53.6
0.00	0.00	1.7	2.63	25.2	0.867	2.85	2.3	53.9
0.08	0.00	1.5	2.54	19.3	0.656	2.28	2.38	50.7
0.17	0.00	3.0	2.62	19.9	0.695	2.34	2.32	50.5
0.34	0.00	3.8	2.32	20.2	0.686	2.68	2.15	49.9
0.70	0.00	7.1	2.12	19.9	0.675	2.06	2.13	49.3
0.75	0.00	4.2	2.05	18.6	0.644	2.05	2.23	48.4
1.37	0.00	8.4	1.57	15.7	0.544	1.47	2.41	50.2
0.36	0.71	2.5	2.41	22.3	0.799	2.70	2.31	60.3
0.65	0.33	2.8	2.02	21.6	0.849	2.50	2.38	59.1
0.73	0.73	5.1	2.18	23.2	0.866	2.90	2.29	60.8
0.73	0.73	3.2	2.14	21.2	0.764	2.59	2.56	59.9
0.66	1.00	2.5	2.16	23.2	0.809	2.79	2.34	61.1
1.06	0.70	2.8	1.95	22.5	0.809	2.73	2.39	59.6

III. Experimental data with aspen wood.

Aspen	Mass	Energy	Tensile	Burst	Tear	Bulk	Brightness
Intercept	1.879	2.722	26.064	0.877	2.479	2.331	52.794
OA	4.460	-0.758	-0.873	-0.079	-0.458	ns	-1.765
SO_2	-2.447	0.958	4.005	ns	0.632	ns	3.015
OA const	ns	ns	-5.732	-0.182	ns	ns	-2.574
SO_2 const	ns	-0.518	ns	0.176	ns	ns	8.843
OA	ns	ns	-4.320	ns	ns	ns	3.147
SO_2	ns	-1.020	ns	ns	ns	ns	ns
OA*SO_2	ns	ns	ns	ns	1.331	ns	ns
R^2	0.656	0.973	0.843	0.874	0.779	ns	0.992

IV. Effect screening results for treatment of aspen wood.

	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	1.9	2.63	24.8	0.877	2.69	2.33	53.7
OA effect	4.5	-0.76	-6.2	-0.261	-0.86	ns	-4.6
SO_2 effect	ns	Ns	4.0	0.176	ns	ns	11.9
OA and SO_2	ns	-0.73	ns	ns	0.38	ns	7.3

V. Estimated effects for a loading of 1% of chemical agents of aspen wood.

	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	1.9	2.63	24.8	0.877	2.69	2.33	53.7
Treat	3.2	2.02	23.2	0.804	3.01	2.33	60.9
% change	ns	-23%	ns	ns	ns	ns	13%

VI. Predicted optimum property values for 0.85% oxalic acid and 1.0% sulfur dioxide treatment of aspen wood.

OA	SO_2	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
% on o.d. wood	% on o.d. wood	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
0.00	0.00	1.0	2.62	35.7	1.648	5.87	2.61	52.4
0.00	0.00	1.4	2.41	36.2	1.799	6.04	2.28	50.6
0.00	0.00	0.0	2.70	37.7	1.676	5.79	2.36	52.5
0.12	0.00	1.3	1.84	38.0	1.988	6.16	2.19	52.4
0.23	0.00	1.0	1.63	29.7	1.313	5.05	2.22	46.1
0.47	0.00	2.8	1.46	27.3	1.231	4.62	2.24	45.0
0.93	0.00	9.1	1.25	24.6	0.966	3.64	2.16	45.3
0.00	0.05	0.0	2.45	27.1	1.441	5.59		57.3
0.00	0.11	0.3	2.52	28.3	1.648	7.48		54.1
0.00	0.21	0.6	2.49	27.6	1.706	7.35		55.3
0.00	0.38	1.9	2.40	27.0	1.641	7.25		53.9
0.00	0.71	2.0	2.29	27.9	1.691	7.24		54.1

VII. Experimental data with spruce wood.

was below the minimum difference shown in Table II.

While OA did increase hemicellulose extraction and energy savings, it had a detrimental impact on the strength properties and brightness. Yet, the addition of sulfite can dramatically improve brightness. We performed a statistical optimization to maximize energy savings without a significant loss in properties, as defined by the error analysis. This optimum occurred at 0.85% OA loading and 1.0% SO_2 loading. Entries of "ns" indicate that the calculated effect was below the minimum difference shown in Table II. These loading levels resulted in both strength and brightness improvement. The property values for this optimum are shown in **Table VI**.

Spruce

Table VII shows the experimental data from the trials with spruce. In general, increasing levels of OA treatment resulted in increased hemicellulose removal rates and energy savings. These benefits were accompanied by significant losses in physical properties and brightness. Treatment with sulfite did not significantly improve the brightness, as had been observed for aspen.

Values in **Table VIII** show that OA had an effect on all properties except bulk. Sulfite had an effect only on refiner energy and tear strength. The effect of sulfite on pulp brightness for spruce was much less than that observed for aspen.

Table IX shows the effect of a 1% loading of OA, SO_2, or both combined. Entries of "ns" indicate that the calculated effect was below the minimum difference shown in Table II.

We performed a statistical optimization using these models. We maximized energy savings while requiring that there not be a significant loss in properties, as defined by the error analysis. This optimum occurred at 0.052% OA. Because sulfite is less effective at brightening spruce pulp, we did not use it. Entries of "ns" indicate that the calculated effect was below the minimum difference shown in Table II. The property values for this optimum are shown in **Table X**. The sensitivity of spruce to OA treatment caused this optimum to occur at very low acid loadings.

Pine

Table XI shows the experimental data acquired during trials with red pine. As with the other wood species, increasing

Spruce	Mass	Energy	Tensile	Burst	Tear	Bulk	Brightness
Intercept	0.914	2.553	36.311	1.679	6.068	2.294	51.878
OA	1.211	-0.670	-14.244	-0.797	-2.545	ns	-18.464
SO_2	ns	-0.379	ns	ns	5.575	ns	ns
OA const	ns	-0.714	ns	ns	ns	ns	ns
SO_2 const	ns	ns	-8.740	ns	ns	ns	2.681
OA	11.494	ns	ns	ns	ns	ns	17.051
SO_2	ns	ns	ns	ns	-8.310	ns	ns
OA*SO_2	ns	ns	ns	ns	ns	ns	ns
R^2	0.948	0.964	0.841	0.644	0.848	ns	0.842

VIII. Effect screening results for treatment of spruce wood.

	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	1.2	2.55	36.3	1.679	5.95	2.29	52.2
OA effect	9.3	-1.38	-14.2	-0.797	-2.54	ns	-6.4
SO2 effect	ns	-0.38	-8.7	ns	-0.72	ns	2.7
OA and SO2	9.3	-1.76	-23.0	-0.797	-3.26	ns	-3.7

IX. Estimated effects for a loading of 1% of chemical agents of spruce wood.

	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	1.2	2.55	36.3	1.679	5.95	2.29	52.2
Treat	1.1	1.80	35.6	1.637	5.81	2.29	51.1
% change	ns	-29%	ns	ns	ns	ns	ns

X. Predicted optimum property values for 0.052% oxalic acid treatment of spruce wood.

OA	SO_2	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
% on o.d. wood	% on o.d. wood	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
0.00	0.00	0.0	2.36	21.5	1.130	5.43	2.94	55.5
0.00	0.00	1.0	2.26	20.8	1.125	5.62	2.9	55.4
0.21	0.00	1.0	1.91	18.6	1.131	6.20	2.88	55.4
0.41	0.00	2.6	1.75	24.4	1.403	6.16	2.47	56.8
0.81	0.00	5.6	1.54	28.0	1.526	6.55	2.49	49.7
0.86	0.86	2.0	1.77	18.9	0.937	5.87	3.09	50.6

XI. Experimental data with red pine wood.

Pine	Mass	Energy	Tensile	Burst	Tear	Bulk	Brightness
Intercept	ns	2.213	22.033	1.112	5.639	2.795	55.442
OA	6.678	-0.732	ns	0.526	1.221	ns	-10.689
SO_2	-4.328	ns	ns	-0.730	-0.950	ns	0.706
OA const	ns	ns	ns	ns	ns	ns	3.666
SO_2 const	ns	ns	ns	ns	ns	ns	ns
OA_2	ns	ns	ns	ns	ns	ns	ns
SO_2^2	ns	ns	ns	ns	ns	ns	ns
OA*SO_2	ns	ns	ns	ns	ns	ns	ns
R^2	0.915	0.723	ns	0.893	0.723	ns	0.604

XII. Effect screening results for treatment of red pine wood.

	Mass Removal	Energy to 150 mL CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	0.0	2.21	22.0	1.112	5.64	2.80	55.4
OA effect	6.7	-0.73	ns	0.526	1.22	ns	-7.0
SO_2 effect	-4.3	ns	ns	-0.730	-0.95	ns	ns
OA and SO_2	ns	-0.73	ns	-0.204	ns	ns	-6.3

XIII. Estimated effects for a loading of 1% of chemical agents of pine wood.

	Mass Removal	Energy to 150 ml CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	0.0	2.21	22.0	1.112	5.64	2.80	55.4
Treat	3.0	1.88	22.0	1.349	6.19	2.80	54.3
% change	ns	-15%	ns	21%	10%	ns	ns

XIV. Predicted optimum property values for 0.45% oxalic acid treatment of pine wood.

levels of OA treatment resulted in increased hemicellulose extraction and reduced refiner energy. In contrast to aspen or spruce, the physical properties of pine pulps tended to improve with higher levels of treatment. Because SO_2 did not appear to have a significant effect on brightness, we only conducted one trial.

Inspection of the values in Table XII indicates that, except for brightness loss, OA treatment seemed to improve the pulp compared to the control. While sulfite treatment had a small impact on brightness, it negatively affected burst and tear. Many of the terms in the model were not significant, which is more an indication of the small size of the data set than lack of higher-order effects.

Table XIII shows the effect of a 1% loading of OA, SO_2, or both combined. Entries of "ns" indicate that the calculated effect was below the minimum difference shown in Table II.

A statistical optimization was performed using the models. We maximized energy savings while requiring that there not be a significant loss in properties, as defined by the error analysis. This optimum occurred at 0.45% OA. Sulfite is less effective at brightening pine pulp and it was not used. Entries of "ns" indicate that the calculated effect was below the minimum difference shown in Table II. **Table XIV** shows the property values for this optimum.

We observed a dramatic loss in pulp brightness at higher loadings during the optimization. If a drop in brightness to 49% or increased bleaching costs can be tolerated, the energy savings can be increased to 30%. **Table XV** shows the results of calculations for higher loadings of OA.

Hot water treatment of aspen

Hot water extractions were also conducted with aspen wood where no OA was added. **Table XVI** shows the conditions of these extractions, as well as the standard H-factor value

	Mass Removal	Energy to 150 ml CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
Control	0.0	2.21	22.0	1.112	5.64	2.80	55.4
Treat	6.0	1.55	22.0	1.585	6.74	2.80	49.5
% change	6.0	-30%	ns	43%	19%	ns	-11%

XV. Property values for 0.90% oxalic acid treatment of red pine wood.

Trial Number	Pressure	Temperature	Time	H-factor
	psig	C	min	h
29	30	135	10	7
30	30	135	10	7
38	30	135	120	79
39	40	142	120	154
40	55	151	120	345
41	55	151	120	345

XVI. Conditions for hot water extraction experiments with aspen.

calculated from the displayed time and temperature. H-factor was developed for controlling kraft processes and accounted for the Arrhenius dependence of pulping rate on temperature. Because the majority of the chemical action in both pre-extraction and kraft pulping is hydrolysis, one might expect that the H-factor could correlate with prehydrolysis data as well. **Table XVII** shows the property data for these conditions.

While H-factor accounts for both time and temperature, not all of the changes may be related to hydrolysis. **Table XVIII** shows the results of a least squares fitting procedure, where H-factor and time were used as independent variables. The data suggest that the H-factor did an excellent job of capturing the variation of sugar extraction with time and temperature.

The significant loss in brightness can also be modeled with a linear dependence on H-factor. Clearly, the hot water extraction did not produce a printing-quality pulp. The brightness was 22% and could not be recovered by bleaching.

Carbohydrate extract composition

While the estimated total carbohydrate removal rate has been analyzed for each wood species in the previous sections, the composition of the hydrolyzed extract streams has not. In general, the composition changes with the extent of hydrolysis. In the case of all three wood species, the first carbohydrate to be removed is arabinose. As the hydrolysis proceeded further, oligomers were released. The degree of polymerization of the typical extract carbohydrate was less than seven. Table **XIX** shows the hydrolyzed carbohydrate composition for the optimal treatment identified in each of the previous sections.

The comparison of the composition of extracts from aspen shows the dramatic shift toward xylose as the extent of extraction increased. The OA instance corresponded to 3.2% mass removal and the water instance had extract removal of 36%. While arabinose and glucomannans dominated the initial stages of extraction, the majority of the subsequent materials removed were xylans. Similarly, comparing the spruce and pine data shows that low extents of extraction enhanced the amount of arabinose in the extract.

Trial Number	Mass Removal	Energy to 150 ml CSF	Tensile	Burst	Tear	Bulk	Brightness
	% of o.d. wood	MWh/MT	N-m/g	mN-m²/g	kPa-m²/g	cm³/g	% (TAPPI)
29	1.6	2.60	24.4	0.887	2.80	2.44	53.6
30	1.7	2.63	25.2	0.867	2.85	2.30	53.9
38	8.9	2.04	26.7	1.030	2.63	1.84	38.4
39	18.0	1.99	23.1	0.824	1.87	1.96	38.5
40	35.4	1.92	20.8	0.854	2.53	1.93	22.4
41	36.0	1.82	21.2	0.838	2.50	1.94	22.8

XVII. Properties for hot water extraction experiments with aspen.

	Mass	Energy	Tensile	Burst	Tear	Bulk	Brightness
Intercept	ns	2.668	25.590	0.883	2.530	2.411	51.758
Time	ns	-0.005	ns	ns	ns	-0.004	ns
H-factor	0.105	-0.001	-0.013	ns	ns	ns	-0.087
R^2	0.995	0.987	0.702	ns	ns	0.922	0.927

XVIII. Effect screening results for treatment of aspen with water.

	Aspen OA	Spruce OA	Pine OA	Aspen Water
	%	%	%	%
Mass removal	3.2	1.1	6.0	35.8
Arabinose	12.3	25.7	4.8	3.3
Xylose	22.3	1.9	22.9	84.8
Glucose	43.6	14.8	27.9	3.0
Galactose	6.7	31.9	7.4	3.6
Mannose	15.1	25.7	37.1	5.3

XIX. Hydrolyzed carbohydrate composition for each wood species.

CONCLUSIONS

Oxalic acid treatment can be used to significantly reduce the energy required to refine pulps. For aspen, a combined OA/SO_2 treatment gave 30% energy savings and 13% brightness increase.

Spruce was very sensitive to OA treatment. Pulp properties rapidly degraded with increasing levels of treatment. The high sensitivity to treatment may indicate that spruce, with its thin cell walls, is more easily hydrolyzed than other wood species. The optimum treatment level was near 0.05% on o.d. wood.

Pine showed a much higher tolerance for treatment. In fact, since pine wood is known to have thick-walled cells and coarse fibers, the softening effect of the pretreatment may be making the fibers more compliant, resulting in better bonding. The contrast between spruce and pine may be attributed to the differences in wood cell morphology. Pine, with its thicker cell walls, makes better paper when its fibers are made more flexible, while spruce, with its thin cell walls, is rapidly degraded.

Successful commercialization of this pretreatment method will depend on clearing both economic and engineering hurdles. These factors are thoroughly discussed in Part 2 [4]. **TJ**

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A Forest Service focus area is to develop energy sources from wood resources. As energy costs continue to increase, oxalic acid pretreatment may provide a method to reduce TMP-energy requirements. Oxalic acid has an advantage over other pretreatment methods in that it provided significant energy savings for softwoods.

Our study provided the first continuous pilot-scale data for an oxalic acid process. The equipment has a plug-screw feeder on the outlet of the pretreatment vessel to express extracts before refining. Developing this novel equipment was the most difficult aspect of our work.

A surprising finding was how much difference there was between the response of spruce and pine to the pretreatment. Spruce seemed very sensitive to fiber damage.

The next step is to take these pilot results and the economic analysis of Part 2, which is published in this issue of *TAPPI Journal*, and explore the economics of installing the technology in a specific mill, with its own particular constraints.

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