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Removal of Wax and Stickies From OCC by Flotation

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Recycling Research Area: Separation Technologies

Progress Report #2, April 1, 1998 to June 30, 1998

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Executive Summary

In the first quarterly report, we focused on methods for measuring the concentration of wax. Image analysis methods with transmitted or reflected light are not suitable. Therefore, we are employing a solvent extraction method for the quantification of wax. Secondly, we have developed sedimentation-flotation method (S-F method) for distinguishing between bound wax and free wax. The S-F method will be quite useful in analyzing pulping and flotation data.

In this quarter we completed high consistency pulping trials. Pulping results were analyzed in terms of defibering index or yield and the concentration of free wax. The objective of these trials is to identify pulping conditions that will give higher yield and higher concentration of free wax.

The yield from high consistency pulping trials ranged from 65% to 97%. In general, high consistency (10 to 11%) and high pH (9.7 to 11) conditions resulted in higher yield. The temperature range of 110 to 130 °F did not show a significant effect on yield. The amount of free wax in the pulp depended on pulping conditions as well as the surfactant. In either case, lower hardness (less than 50 ppm as calcium carbonate) seems to be preferable to get more free wax. The pulping time of 20 minutes seems to be satisfactory for both defibering and dewaxing. We need to do more experiments to better understand the effect of temperature by carrying out experiments at somewhat higher temperature.

Preliminary experiments were conducted to study the effect of enzymes, ultrasonic treatment and washing. Results indicated that enzymatic treatment and washing are not very effective while ultrasonic treatment appears to be promising.

Future plans include pulping trials at lower consistency (less than 6%). Flotation trials will begin (probably in Fall) once the ideal pulping conditions have been identified.

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2.0 INTRODUCTION

During the second quarter of the study we examined the conditions necessary for repulping a mixture of wax-coated boards that would be conducive to the flotation of detached wax. Also important for the economic viability of a waxed-board repulping process is adequate defibering of the recovered paper. Several methods for the dewaxing of pulped waxed-boards were investigated.

We have continued to survey the literature to determine what other efforts are being made to ameliorate the impact of waxed boards during the recycling of OCC. Two noteworthy articles are discussed below.

The Thermo Black Clawson Company has published information about a method that employs reverse screening, or washing (1). In the trials discussed, wax saturated boxes containing 30% wax (straight paraffin) by weight were pulped at 9% consistency, 65° C (150° F) and pH 9 for 20 minutes. Specially formulated waxes were not considered in this trial. The report states that the furnish was 90% defibered in a mid consistency Hydrapulper after 20 minutes and that the wax releases from the fibers and remains in suspension as long as the temperature of the slurry remains higher than the melting point of the wax (1). In order to simulate a full scale system, pulp from the pulper was washed across a high-speed belt washer to generate wax-laden white water as dilution. Pulp was screened through 0.25 mm (0.010 in) slotted pressure screens to remove undefibered flakes and then processed across three stages of counter-current reverse screening. The reverse screens are conventional screens fitted with a special high frequency rotor and screen cylinder with 0.15 mm (0.006 in) holes. The screen cylinder allows wax-laden water to pass through the holes while the washed pulp is retained on the plate and "rejected". In this application, the "rejects" are the washed, accepted fiber, hence the name reverse screening. The report states that dissolved air flotation (DAF) clarification is used to remove wax from the wash water to allow reuse of the water in the process. The author claims that a proposed system using furnish with 30% wax by weight would have a yield of 60% and that the final de-waxed pulp would have a 1% extractable content.

A major chemical manufacturer has published research results demonstrating the use of a dispersant chemistry in which, under specified pulping and processing conditions, wax can be washed from pulped wax-containing furnish (2). Several components are present in the dispersant blend used in this method. An additive depresses the melting temperature to ensure that the wax is melting before dispersion occurs. Research had indicated that prior to the onset of melting, paraffinic wax is difficult to disperse, and has a high tendency to redeposit. This manipulation of wax melting temperature, according to the report, appears to be key in the application of this technology. Anionic dispersant and nonionic wetting

agent stabilize the dispersed, colloidal wax, thus aiding the removal of the wax from the pulp by washing. According to the authors, through the use of this dispersant chemistry, wax can be removed from the process by coagulating the colloidal particles at a clarifier or on the fiberline (2).

2.0 CURRENT ACTIVITY

During the past quarter our main research emphasis was on repulping studies to determine the optimum conditions to prepare wax for removal by froth flotation. Concurrent with the repulping work we examined other mechanisms for the detachment of wax from fiber. These methods, proposed in the *Methods Used to Remove Wax from Fiber* section of the first report to DOE, included ultrasonic treatment, enzyme treatment, and the application of a hyperwashing technique described in a paper presented by Thermo Black Clawson at the International Recovered Paper IX Conference. The use of a hydrophobic collector for the quantification of free wax was examined (Doshi microfoam method). An additional task that was deemed important was the determination of the association of wax and fines. Experimental data are presented and discussed in the next section.

3.0 EXPERIMENTAL

3.1 Pulping Experiments

Experiments were designed using the Plackett-Burman matrix design so that the effects of six parameters on defibering and creation of free wax could be examined.

Wax-containing board samples representing three types of wax coated products were obtained from converters. These products included curtain-coated corrugated board, wax-coated medium, and cascade coated corrugated board. Furnish for the pulping experiments consisted of 4% curtain-coated board, 2% cascade-coated board, and 4% waxed medium. The remainder of the furnish consisted of wax-free OCC. The percentage of wax by weight in each product was determined by soxhlet extraction using 1,1,1-trichloroethane as a solvent (Table 1). Subsequent analysis of the wax-free OCC indicated that the extractable level (background) was 0.28%.

Table 1. Wax Percentage in Three Wax Containing Products.

<u>Product</u>	<u>% Extractable</u>
Curtain-coated board	6.00
Cascade coated board	29.39
Waxed medium	3.89
Control (wax-free OCC)	0.28

Table 2. Parameters Examined in Repulping Studies

Parameters	High	Low
Consistency	11%	6%
Temperature	130°F	110°F
pH	11	5
Chemical A or B	1 lb/ton	0 lb/ton
Time	40 minutes	20 minutes
Hardness	Hard water	Soft water
(Calcium carbonate)	> 250 ppm	< 50 ppm

Table 2 shows the parameters that were considered during the repulping trials. Table 3 shows the Plackett-Burman experimental design. The highest and lowest consistencies that were attainable during this phase of the study were 11% and 6%, respectively. A laboratory pulper with a helical rotor was used for these experiments. Based on preliminary repulping experiments, the pulping durations were set at 20 minutes for the low treatment, and 40 minutes for the high treatment. Hardness was adjusted with calcium chloride. In those experiments where chemical was high, 1.0 g surfactant was added. Surfactant was obtained from two major manufacturers of paper processing chemicals. The surfactant used in experiments W1, W4, W5, and W6 was designated as Chemical A, and in experiments W9-W12 the chemical is designated as Chemical B. Four additional experiments were conducted, W13-W16, at neutral pH to evaluate the effect of pH on defibering and wax detachment.

Table 3. Plackett-Burman Experimental Design

Run No.	% C	pH	Dummy	Temp.	Chemical	Hardness	Time
W1	H	H	H	L	H	L	L
W2	H	H	L	H	L	L	H
W3	H	L	H	L	L	H	H
W4	L	H	L	L	H	H	H
W5	H	L	L	H	H	H	L
W6	L	L	H	H	H	L	H
W7	L	H	H	H	L	H	L
W8	L	L	L	L	L	L	L

Run Nos. W9-W12: Repeat Runs W1, W4, W5, W6 with chemical B. (Note: replaces runs with chemical A by runs with chemical B)

Run Nos. W13-W16: Repeat runs W2, W3, W7, W8 at pH 7. (Note: all these runs have no chemicals added)

Table 4. Pulping Experiment Results.

Run (no.)	cons (%)	pH	Temp. (°F)	Chem. (g)	Hardness (ppm)	Time (min)	Debris (%)	Yield (%)	Wax total %	Wax bound %	Wax free %
W1	9.28%	10.7	109	1	82	20	6.36%	93.64%	1.23%	0.92%	32.63%
W2	10.92%	10.3	132	0	82	40	2.88%	97.12%	0.99%	0.58%	57.75%
W3	12.66%	4.0	111	0	306	40	11.71%	88.29%	1.54%	1.34%	15.87%
W4	6.0%	9.7	109	1	306	40	25.90%	74.0%	1.78%	1.65%	8.67%
W5	11.26%	4.3	127	1	306	20	16.92%	83.08%	1.2%	0.60%	68.63%
W6	5.39%	4.8	123	1	84	40	34.86%	65.14%	1.49%	0.62%	71.90%
W7	5.57%	10.4	127	0	306	20	8.68%	91.32%	1.20%	0.70%	54.35%
W8	4.40%	4.4	113	0	76	20	23.49%	76.51%	1.43%	0.33%	95.65%
W9	9.61%	11.2	117	1	70	20	4.5%	95.3%	1.31%	0.30%	98.06%
W10	5.09%	10.5	105	1	290	40	9.59%	90.41%	1.66%	0.27%	100.00%
W11	11.36%	4.3	134	1	290	20	6.14%	93.86%	1.34%	1.32%	1.89%
W12	6.03%	4	124	1	92	40	14.77%	85.23%	1.22%	0.93%	30.85%
W13	11.89%	7.0	122	0	88	40	4.08%	95.92%	1.44%	1.16%	24.14%
W14	10.1%	6.9	116	0	316	40	7.91%	92.09%	1.02%	0.58%	59.46%
W15	6.52%	6.8	127	0	322	20	18.11%	81.89%	1.2%	0.84%	45.10%
W16	6.03%	7.4	111	0	92	20	21.93%	78.07%	1.32%	0.51%	77.88%

Note: Extractable% of wax-free OCC (= control) is 0.28%.

Results from the first set of 16 experiments are given in Table 4.

Pulping Experiments Measurements:

1. Measure defibering index in terms of debris captured on 0.15-mm (0.006-in) slotted laboratory screen.
2. Conduct sedimentation-flotation test to determine "free" wax and bound wax content of samples.
3. Conduct solvent extraction of pulper sample and bound wax (settled fibers) sample following sedimentation-flotation test.

Following pulping, consistency of the stock in the pulper was determined. A sample was collected from the pulper for solvent extraction. This extraction indicated the total wax concentration. The yield, or defibering index, was determined by processing a 100 g subsample of the pulped stock through a 0.15-mm (0.006-in) laboratory flat screen. Debris captured on the screen was dried and weighed, and yield was determined as:

$$\text{Yield} = \frac{(\text{Feed g} - \text{Debris g})}{\text{Feed g}}$$

The two key parameters that were of interest during these pulping experiments were the defibering index and the free wax. Ideally, the pulping conditions should be such that the defibering index and free wax are maximized.

Summarizing the discussion of "free wax" and "bound wax" from Report 1, the term "free" wax describes the condition during which the wax contaminant is detached from the fibers. Hypothetically,

the free wax portion should be removed to the greater extent by flotation, washing, and reverse cleaning processes, while bound wax, still attached or coating fibers, may be removable by screening, but not removed to a significant degree by flotation, washing, and reverse cleaning. The pulping experiments were designed so that the influence of pulping time, pulping consistency, temperature, pH, surfactant, etc., on the distribution of the free and bound fractions of wax could be determined.

Free and bound wax quantities in pulped samples were determined using the sedimentation-flotation method. This method characterizes the distribution of free and bound wax at the existing temperature. It was assumed that all free wax occurring at the pulping temperature would float to the surface of a gently stirred sample at low consistency (0.5%). Free wax was decanted from the sample, and the remaining settled fiber (containing bound wax) was concentrated. Solvent extraction was conducted on a sample of settled fiber to determine the bound wax. The concentration of free wax was determined by difference:

$$\text{Free Wax} = \text{Total Wax} - \text{Bound Wax}$$

The data from Table 4 were analyzed to compare the low and high treatment of each parameter for the first eight pulping experiments (W1-W8). The background extractable level of the wax-free OCC was 0.28%. Background extractables were deducted from extractable results of the total and bound wax in order to calculate the free wax% as follows:

$$\text{Free Wax\%} = \frac{(\text{Total Wax\%} - \text{Background\%}) - (\text{Bound Wax\%} - \text{Background\%})}{(\text{Total Wax\%} - \text{Background\%})}$$

Table 5 compares the effects of high and low treatments on defibering index and free wax. To determine if a parameter, i.e., consistency, was important, the difference between the "high" and "low" treatment was divided by the number of treatments (= 4), and the result, either negative or positive was compared to the value for the dummy treatment. Positive values that were significantly larger than the dummy value indicated that the "high" treatment is important in producing the effect being analyzed. Negative values that are larger in absolute value compared to the dummy value indicate that the low treatment is important in producing the effect.

Table 5. Effects of Pulping Conditions On Defibering Index and Free Wax. When Present, the Surfactant Was Chemical A.

Parameter	Cons.%	pH	<u>Effects on Defibering Index</u>				Dummy
			Temp. °F	Chem	Hardness	Time (min)	
High	362.13%	356.18%	336.66%	315.96%	336.79%	324.65%	338.39%
Low	307.07%	313.02%	332.54%	353.24%	332.41%	344.55%	330.81%
Δ/4	13.76%	10.79%	1.03%	-9.32%	1.09%	-4.97%	1.89%
Parameter	Cons.%	pH	<u>Effects on Free Wax</u>				Dummy
			Temp. °F	Chem	Hardness	Time (min)	
High	174.88%	153.39%	252.62%	181.83%	147.51%	154.19%	174.75%
Low	230.57%	252.05%	152.82%	223.62%	257.93%	251.26%	230.69%
Δ /4	-13.92%	-24.67%	24.95%	-10.45%	-27.60%	-24.27%	-13.98%

Results compiled in Table 5 indicate that high consistency and high pH are important in maximizing the defibering of this furnish when the helical rotor was used in the laboratory pulper. The negative values for the chemical and time indicate that, in this case, the use of Chemical A negatively impacted defibering. Surprisingly, short pulping time had greater effect on defibering index than longer pulping time. Hardness and temperature were not significant to defibering within the range of these parameters.

Important factors in promoting the detachment of wax were low pH, low hardness, short pulping time, and high temperature. Chemical and consistency were not important.

Table 6 shows a comparison of high with low treatments of the various pulping parameters and includes Chemical B as surfactant. Factors that are important in maximizing defibering were high consistency, high pH, and high temperature. Use of chemical and high hardness had a subtle influence on defibering, and time was not important.

The generation of free wax was enhanced by low consistency, high pH, and low hardness. Pulping temperature and time were not important factors, and use of Chemical B only slightly increased the generation of free wax.

Table 7 examines the effect of neutral pH compared to low and high pH on defibering and free wax.

Table 6. Effects of Pulping Conditions On Defibering Index and Free Wax. When Present, the Surfactant Was Chemical B.

Parameter	Cons.%	pH	Effects On Defibering Index				
			Temp. °F	Chem	Hardness	Time (min)	Dummy
High	374.67%	374.25%	367.53%	364.90%	363.88%	361.05%	360.24%
Low	343.47%	343.89%	337.48%	353.24%	354.26%	357.09%	357.8%
Δ /4	7.7%	7.59%	7.51%	2.92%	2.41%	0.99%	0.59%
Parameter	Cons.%	pH	Effects On Free Wax				
			Temp. °F	Chem	Hardness	Time (min)	Dummy
High	173.56%	310.15%	144.83%	230.80%	172.11%	204.47%	199.13%
Low	280.85%	144.262%	220.41%	223.62%	282.31%	249.95%	255.29%
Δ /4	-26.82%	41.47%	-18.89%	1.79%	-27.55%	-11.37%	-14.04%

Table 7. Effect of Neutral pH Compared to High and Low pH.

	Defibering Index (Yield)	Dewaxing Index (% Free Wax)
(W2 + W7)/2 High pH	94	56
(W13 + W15)/2 Neutral pH	89	35
(W14 + W16)/2 Neutral pH	85	69
(W3 + W8)/2 Low pH	83	56

3.1.1 Discussion of Pulping Experiments

Analyses of the interactions between the pulping parameters were not conducted, and we recognize that these occur. For example, surfactants are pH sensitive, perform differently at different temperatures based upon their cloud point, and may be affected by the water hardness.

In this set of pulping trials with the helical rotor, high consistency (9-11%) was more effective in promoting defibering than medium consistency (6%). The converse was true when considering wax dispersal, or the creation of free wax. Surprisingly, pulping time was not a major factor affecting defibering. The two chemicals caused opposite effects. Chemical A at high temperature favored the generation of free wax. In contrast, the application of Chemical B at low temperature generated more free wax, but to a lesser degree than in the former case.

Results from Table 7 indicate that when all parameters except pH are the same, increasing the pH helps in both defibering and wax detachment. Note that results from high and low pH experiments should not be compared since the other parameters are different.

A serious concern during the mid and high consistency pulping experiments was the amount of undefibered flakes remaining. Analysis with solvent extraction showed elevated wax levels in this debris. The loss of fiber and wax in the screen debris would skew the results for bound and free wax in the defibered pulp.

Near the end of the reporting quarter, Voith Sulzer fabricated a low consistency rotor for the laboratory pulper. Preliminary trials on wax-coated board showed excellent defibering at 5% consistency.

3.2 Wax Deposition on Microfoam (Doshi Method)

Two experiments were conducted to determine if dispersed wax from 5 g of pulped cascade-coated board would deposit on a hydrophobic collector. Two tests were run, one at 20° C and one at 50° C. Preweighed strips of Microfoam™ polypropylene foam packing material were suspended in a stirred pulp slurry at approximately 1.8% consistency for 30 min. Following the treatment period, microfoam was removed from the pulp, rinsed in cold water, dried, and reweighed. Weight gain was an indication of the amount of deposited wax.

For the 20° C temperature trial, no weight gain was noted in the microfoam. At this temperature, the wax was not sufficiently tacky or its surface properties were not amenable to attraction to the hydrophobic media.

At 50° C, a weight gain of 0.1976 g was observed on the microfoam indicating that at this temperature the wax was attracted to the hydrophobic collector. However, observation of the container holding the pulp slurry indicated a substantial amount of wax floating on the surface that was not captured by the microfoam. In addition, fibers attached to the microfoam causing some of the weight gain to be attributed to fiber rather than wax.

Based on these results and observations, the deposition test was deemed unsuitable for the quantification of wax at this time.

3.3 Application of Enzymes for the Removal of Wax from Board

Enzymes have been used in deinking applications to detach ink from fiber. Using this premise, it was hypothesized that enzymes could be of value in defibering as well as detaching wax from fiber during the pulping process.

A chemical supplier that produces enzymes for the paper industry was contacted for assistance in providing a suitable enzyme for this application. Three enzymes and two surfactants were supplied, and experiments were designed to analyze the effects of enzymes on wax removal. Table 8 lists the pulping conditions and results for the enzyme study. Pulping trials were conducted using the laboratory pulper with helical rotor.

Table 8. Enzymatic Pulping Dewaxing Study.

Test 1: Surfactant A, 0.1%; Enzyme G, 0.2%; Enzyme H, 0.2%; Enzyme I, 0.2%

Cons.%	pH	Temp °F	Hardness (ppm)	Time (min)	Debris%	Defibering Index	Total Wax%	Bound Wax%	Free Wax%
9.45	6.06	102.8	259	20	9.32%	90.7%	1.18%	0.79%	43%

Test 2: Surfactant B, 0.1%; Enzyme G, 0.2%; Enzyme H, 0.2%; Enzyme I, 0.2%

Cons. (%)	pH	Temp (°F)	Hardness (ppm)	Time (min)	Debris%	Defibering Index	Total Wax%	Bound Wax%	Free Wax%
10.47	6.59	100.4	250	20	12.88%	87.12%	1.93%	0.49%	87%

Note: Total and bound wax are corrected for background extractable% (0.28%) prior to calculation of free wax%.

3.3.1 Discussion of Enzyme Trials

Defibering index results for the enzyme pulping trials were unsatisfactory. Based on observations and the bound wax and free wax results, it does not appear that under these conditions, the use of enzymes enhanced the dewaxing of the fiber. Due to the pH conditions at which enzymatic activity is optimum (pH 6-7), a direct comparison to the earlier pulping tests (Table 4) is not possible.

The use of enzymes during low consistency pulping may be feasible if pulping times can be reduced as a result of their application. This approach may be revisited when low consistency pulping is available in the next quarter.

3.4 Ultrasonic Dewaxing Trials

Research in deinking with ultrasound indicates that the cavitation generated by the ultrasonics frequencies focused on a very small volume is sufficient to detach ink from fiber. Two studies were conducted to investigate the feasibility for dewaxing using ultrasound.

A local supplier of parts washing equipment was invited to the Voith Sulzer Technology Center to demonstrate an ultrasonics apparatus. The portable parts washing apparatus consisted of an approximately 35 L tub, pumping apparatus, heater, and ultrasound generator. Tap water was heated in the apparatus to approximately 130° F, a detergent, provided by the ultrasonic parts washer supplier, was added, and a subsample from pulping experiment W4 (Table 4) and three board samples—waxed medium, cascade-coated, and curtain-coated, were introduced. The board samples were intact prior to treatment. The W4 pulp samples was confined within a wire mesh (150 mesh) enclosure. The samples were subjected to the ultrasound within the parts washer for approximately 45 min. Results from the parts washer trial are shown in Table 9.

Table 9. Ultrasonic (US) Treatment of Pulp from Wax-Coated Board.

<u>Sample</u>	<u>Total Wax%</u>	<u>Bound Wax%</u>	<u>Free Wax%</u>
W4 Control	1.78	1.65	8.67
W4 US Treated	2.12	0.23	100

Note: In the W4 samples background extractable is subtracted from total and bound wax values before free wax is calculated.

3.4.1 Discussion of Ultrasonic Treatment (Parts Washer Trials)

The quantity of free wax in the W4 pulp sample increased substantially as a result of ultrasonic treatment from 8% in the control to 100% in the US treated sample. The total wax content, based on solvent extraction, increased from 1.78% in the control sample to 2.12% in the US treated sample. The increase in total wax may have been due either to experimental error or to extraction of the organic detergent added to the parts washer.

Board samples remained intact throughout US treatment and did not appear to be affected by the US treatment.

3.5 Ultrasonic (US) Treatment of Pulp from Wax-Coated Board Using Laboratory Sonic Dismembrator

A Fischer Scientific Model 550 Sonic Dismembrator was obtained for use in this study. Three waxed board samples were pulped separately in a 4-L Waring blender until no visible flakes were apparent at the following conditions:

4% consistency, 125° F, pH 7.5, ~5 minutes

A subsample of each waxed board pulp was removed, analyzed for total wax, and processed through the sedimentation-flotation process to determine bound and free wax.

Ultrasonic treatment consisted of subjecting 1800 ml of each pulp at 0.5% consistency to 20 kHz frequency, 45-50% of full output power (or 213-238 watts), for 10 min under continuous agitation. After US treatment, samples were tested by the sedimentation-flotation method for bound and free wax. Results for the ultrasonic dewaxing study are shown in Table 10.

Table 10. Ultrasonic (US) Treatment of Wax-Coated Pulp by Laboratory Dismembrator.

<u>Sample</u>	<u>Total Wax%</u>	<u>Bound Wax%</u>	<u>Free Wax%</u>
Curtain-Coated Board Control	6.0	2.52	58
Curtain-Coated Board Treated		1.15	81
Cascade-Coated Board Control	29.39	21.37	27
Cascade-Coated Board Treated		24.87	15
Waxed Medium Control	3.89	3.92	0
Waxed Medium Treated		4.05	0

Note: Free Wax = (Total - Bound) + Total

3.5.1 Discussion of Ultrasonic (US) Treatment of Pulp Using Laboratory Dismembrator

The wax in the curtain-coated board was more vulnerable to ultrasonic treatment than the waxes in the medium and cascade-coated board. The percentage of free wax increased from 58% in the control curtain-coated board pulp to 81% in the US treated pulp. For the other pulps, positive results relative to generation of free wax as a result of US treatment were not realized. More work needs to be done to examine the energy input and frequency of ultrasound required to detach bound wax from these pulps.

3.6 Wash Dewaxing Study

A trial was devised in which a mixed waxed board furnish was pulped under aggressive conditions to maximize the amount of dispersed wax and hyperwashed to remove wax from the pulp. Pulping conditions are as follow:

Consistency: 9% Temperature: 150° F pH: 9 Time: 20 min
 Furnish: 50% wax-free OCC, 20% curtain-coated board, 20% cascade-coated board,
 10% waxed medium.

Following pulping in the high consistency laboratory pulper, samples were analyzed for total wax and debris (6-cut laboratory screen). Two washing experiments were conducted on this pulp. In the first, pulp was processed through a Bauer-McNett Classifier with a 100-mesh screen using tap water (cold washed). In the second, pulp was processed through the Bauer-McNett with 100-mesh screen, but this time using hot water at 150° F. Sedimentation-flotation was conducted to determine bound and free wax in the two washed pulps. Results for these washing trials are shown in Table 11.

Table 11. Wash Dewaxing of Pulp from Wax Coated Board.

Sample	Total Wax%	Bound Wax%	Free Wax%	Yield from Bauer-McNett
Unwashed	4.2%	3.45%	18%	
Cold Washed	2.22%	1.90%	14%	84.2%
Hot Washed	2.56%	—		76.2%

3.6.1 Discussion of Wash Dewaxing Study

Yield from the pulping of mixed board furnish was only 86.1%. This low yield probably indicates that pulping was not aggressive enough to adequately detach wax from the fiber. Cold washing with the Bauer-McNett removed approximately 48% of the total wax, and 55% of the bound wax present in the pulp. Hot washing, expected to be superior to cold washing, removed slightly less of the total wax, or 40%. Another set of washing experiments was designed to determine if wax redeposition onto the fibers was occurring following pulping. Also surprising was that there was free wax remaining in the cold washed sample following processing in the Bauer-McNett. Results for bound and free wax from hot washing are not available. When examining the yield from the washing, a question arose as to the association of wax with fines removed from the pulp by washing.

3.6.2 Wax Redeposition onto Fibers During Storage

A furnish of 100% cascade-coated board was repulped in the 4-L Waring blender. A sample was analyzed for total wax following pulping. One sample was processed through the Bauer-McNett immediately after pulping. A second sample was stored overnight in a cold-room at approximately 4° C and then processed through the Bauer-McNett. Water used for washing these pulps in the Bauer-McNett was approximately 160° F. Test results are shown in Table 12

Table 12. Wax in Washed Stored Pulp and Unstored Pulp.

Sample	Test #1	Test #2
Non-stored A	5.15	5.12
Non-stored B	5.51	5.49
Cold-stored A	2.18	2.20
Cold-stored B	3.16	3.02

3.6.3 Discussion of Washing Trials

It was hypothesized that storing the pulp at the cold temperature would enhance the redeposition of wax onto the fibers thus lowering the effectiveness of subsequent washing. Under this condition, one might have expected that the wax concentration in the cold-stored pulp after washing to be higher than that of the non-stored pulp. Alternatively, during the cooling down period in cold storage, wax resolidified into particles that were not tacky. During subsequent washing, the wax particles became dispersed

in the diluted stock before they reached the melting point. In the dilute stock, there is less probability of wax contacting with fiber and redepositing, so during washing the wax was removed.

3.7 Wax in Fines

Cascade-coated board was repulped in the 4-L Waring blender at 120° F and neutral pH for 5 min. Pulp was processed through the Bauer-McNett configured with 4 screen mesh sizes. Screen sizes were 35, 60, 100, 200. Tap water was used for the washing. For the first 3 min., water passing the 200-mesh screen was drained to the sewer, after which time the water from the 200-mesh screen was collected. Fines in the collected water were allowed to settle for approximately 24 hr. Supernatant was siphoned off and fines were collected on a Buchner funnel with filter paper. After air drying, fines were analyzed for total and bound wax (160° F) following standard procedures. Test results are shown in Table 13.

Table 13. Wax in Fines Compared to Wax in Whole Pulp.

Sample	Total Wax%	Bound Wax%	Free Wax%
Fines (passing 200-mesh)	42.44%	43.28%	0
Whole Pulp (= fibers and fines)	29.39%	21.37%	27%

3.7.1 Discussion of Wax in Fines

Results indicate that the fines contain twice as much bound wax as the pulp. Moreover, a considerable fraction of the fines in this pulp is comprised of wax. Free wax would be expected to be very low in the fines, since during the concentration of the fines following washing, the free wax would have been removed with the supernatant. See Appendix I for a copy of the article, "Measurement of Bound and Free Wax," by Mahendra R. Doshi, Jeffrey M. Dyer, Oliver U. Heise and Bangji Cao, *Progress in Paper Recycling* 7 (4): 83-85 (August 1998).

This association of wax with the fines has some interesting ramifications. The applicability of flotation on this pulp is uncertain since the wax-fine particle may not have sufficient hydrophobicity to attach to air bubbles. Analysis of the fines by the Fiber Quality Analyzer (FQA) indicates that the arithmetic average length is 0.11 ± 0.002 mm and the length weighted average length is 0.142 mm. These fines and associated wax would be washable by the "reverse screen" process which uses a 0.15-mm slotted screen. Based on FQA data, when the pulp is washed through a 100-mesh screen (0.15-mm), nearly all of the fines are removed.

4.0 Comments on Progress Report #1

Dave South of St. Laurent Paper and Board Co. and Mahendra Doshi had the following discussion in reference to the first progress report issued in April 1998.

In the report there was a detailed discussion of measuring the concentration of stickies by image analysis using water soluble black dye and reflective light vs. transmitted light etc. However, these methods worked fine for curtain coated wax board but were not suitable for cascade coated board.

The wax from cascade coated board coats the handsheet so instead of getting distinct specks we get large patches and streaks that are not measurable by image analysis.

We therefore, resorted to solvent extraction method for measuring the concentration of wax. We realize that this method is not attractive for mill application for two reasons: it involves the use of chlorinated solvent; and relatively low concentration of wax in OCC makes it difficult to distinguish between background and wax. For our laboratory studies solvent extraction is fine but we may have to look at image analysis or some other method when we are ready for mill application.

The question of implementation of our approach in the mill is not clear at this time. If we intend to take a side stream from an existing mill system, it is unclear as to how we will implement higher pH, higher temperature, chemical addition, etc. (assuming that will be recommended by our experiments). The mill may not be interested in, or be unable to change pulping conditions for our trials. This issue merits further discussion and thinking but need not hinder our current investigation.

4.0 FUTURE ACTIVITIES

As stated previously, preliminary trials with the low consistency rotor in the Voith Sulzer laboratory pulper appear to be favorable in terms of defibering and wax detachment from fiber. Our immediate focus will be to optimize pulping conditions at low consistency to prepare pulp for flotation studies. Also, pulping conditions will be selected for furnishes containing hot-melt coated board and pressure sensitive adhesive contaminated board.

We will continue, as time permits, to explore innovative methods for the detachment of wax from fiber. Investigations into ultrasonic treatment and enzyme dewaxing will be ongoing.

LITERATURE CITED

1. Vitori, C.M. Counter-current reverse screening technology for recycling wax saturated corrugated containers. Proc. International Recovered Paper IX Conference and Exhibition, Paper II-3, Miller Freeman, Inc., San Francisco, CA (May 6-8, 1998).
2. Severtson, S.J.; Coffey, M.J.; Nowak, M.J. Dispersion of waxes during the recycling of old corrugated containers. Nalco Chemical Co., Naperville, IL (1998).

*Appendix 1 removed for
Separate processing*