DOE/ID/13563

# Removal of Wax and Stickies from OCC by Flotation

Final Report – 01/01/1999 – 01/31/2000

M. R. Doshi
J. Dyer

January 2000

Work Performed Under Contract No. DE-FC07-97ID13563

For
U.S. Department of Energy
Assistant Secretary for
Energy Research
Washington, DC

By
Doshi & Associates, Inc.
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**DOE Project DE-FC07-97ID13563**

**Recycling Research Area: Separation Technologies**

## Final Report, January 1, 1999 to January 31, 2000

**Mahendra R. Doshi and Jeffrey Dyer, Doshi & Associates, Inc., P.O. Box 2771, Appleton, WI 54913-2771**

**Subcontractor: Oliver Heise and Bangji Cao, Voith Sulzer Papertechnology, P.O. Box 2337, Appleton, WI 54913-2337**

## Executive Summary

Our original goal in this project was to study the removal of wax and stickies from OCC by flotation.

Laboratory research indicates that wax is amenable to removal by froth flotation provided it is free or detached from the fiber. The only effective means, at this time, of maximizing detachment of wax is through the use of low consistency pulping at temperatures above the melting point of wax. Wax removal from WCC through washing, flotation, or a combination of both was approximately 90% in these laboratory studies, indicating that not all of the wax is detached from fibers. These results were summarized in Annual Report 1, December 1, 1997 to November 30, 1998.

Pilot trials were conducted in which we simulated a conventional OCC repulping process with and without flotation. Additional aggressive washing and water clarification were also examined during the study. The inclusion of flotation in the OCC stock preparation system significantly improved the removal of wax spots and extractable material from the furnish. Based on this study, we predict that a compact flotation system with 2 lb surfactant/ton of fiber would improve the OCC pulp quality with regard to wax spots by 60% and would not negatively affect strength properties. Flotation losses would be in the 2-5% range.

Two mill trials were conducted during the last quarter of the project. One trial was carried out at Green Bay Packaging, Green Bay, WI, and a second trial was conducted at Menasha Corporation, Otsego, MI. A 250-liter Voith Sulzer Ecocell was used to evaluate the removal of wax and stickies from the OCC processing systems at these two mills.

The inclusion of flotation in the OCC stock preparation system significantly improved the removal of wax spots from the furnish. The data indicate that flotation was more effective in removing wax and stickies than reverse cleaners.

The mill trials have demonstrated that flotation can be substituted for or replace existing reverse cleaning systems and, in some cases, can replace dispersion systems. In this manner, the use of flotation can provide significant energy savings when compared to reverse cleaning or dispersion.

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### 1.0 INTRODUCTION

Wax and stickies continue to be the number one challenge for most mills recycling OCC. Due to their particle size and density, wax and stickies are difficult to remove with existing screens and cleaners. As a result, this project was initiated with the objective of removing wax and stickies from OCC by flotation.

Recognizing the presence of wax contamination in the recycle stream entering board mills, we examined three approaches for confronting the issue. The choice in selection depends upon whether we want to view the wax-coated board, now going to landfill, as a potential fiber source, or whether we want to continue to isolate the wax contaminated board to the highest extent possible.

The selection of the system depends upon the degree of wax contamination in the furnish accepted at the facility. The systems are as follow:

*Existing System:* Currently, cascade-coated wax boxes are kept out of the OCC stream by most grocery stores and are sent to landfills. Curtain-coated wax cartons, OCC with wax coated medium and wax from hot melts will continue to enter the existing system. A strategy developed by mills to handle these materials is to pulp at a temperature below 120 °F. We explored the feasibility of adding a flotation stage to the existing system to enhance the removal of wax, hot melts and other stickies.

*Satellite System:* Cascade-coated wax cartons can be processed in this specially designed system to remove wax by flotation rather than in the washing process proposed by Thermo Black Clawson. Pulping temperature in the satellite system should be 160 °F to maximize defibering and the detachment of wax from fiber.

*Future System:* Cascade-coated wax cartons would not be segregated at the generation source as is the case now. The estimated wax concentration in OCC can be 5 to 10% if WCC is included. The addition of flotation in the existing system may allow mills to accept commingled bales.

We expanded the scope of the project by including the removal of wax from WCC. This investigation was undertaken in light of the recent developments in the industry. As a result the timeframe for the completion of the project was longer than we had anticipated.

Based upon our laboratory and pilot work, it appears that, at this time, a satellite system processing 100% WCC by washing and/or flotation will yield a recycled pulp with 2-3% residual wax. This level of wax is generally not acceptable in most situations. Furthermore, the economics of this type of system do not seem to be justified under current market conditions.

Accordingly, our scope was modified to adhere more closely to our original intent- to investigate the removal of wax and stickies from existing OCC recycling systems by using the froth flotation process. Subsequently, we examined optimum pulping and flotation conditions for OCC in the laboratory to be used in pilot plant trials. In addition, we examined the clarification of wax-laden filtrate and trialed innovative chemical agglomeration/dispersant technologies targeted specifically for wax.

In pilot plant trials at the Voith Technology Center, we used OCC from three different sources: Green Bay Packaging, Green Bay, WI, U.S. Papers, Menasha, WI, and Golper Supply, Appleton, WI.

During three separate trials, each furnish was processed through the Voith pilot plant configured as a conventional OCC stock preparation process with and without flotation. Additional washing and DAF clarification was also evaluated. Stock was pulped at low consistency, processed through HD cleaner, coarse screened, fine screened, flotation, through flow cleaning, and Hydrodisk washing. Stock temperature was 120 °F.

During the final phase of this project we conducted two mill trials – one at Green Bay Packaging and the second at Menasha Corporation. The intent of these mill investigations was to determine the feasibility of treating recycled OCC stock through froth flotation. In each case, the flotation cell was positioned following coarse pressure screening and preceeding either through-flow or reverse cleaning. In this manner, we were able to compare the effectiveness of stickies and wax removal by flotation to that of lightweight centrifugal cleaning. It was hypothesized that flotation could be used to substitute for lightweight cleaning, thus reducing the horsepower requirements for this stage of stock preparation.

## 2.0 EXPERIMENTAL RESULTS AND DISCUSSION

### 2.1 Wax Removal from WCC (Wax Corrugated Container) by Washing-Flotation Sequence

Several laboratory pulping trials were conducted at high temperature. Cascade-coated WCC with approximately 29% wax by weight was pulped at 160 °F, pH 10, and 6% consistency for 60-70 minutes. Following pulping, stock was processed through theVoith laboratory flotation cell at 1% consistency, 155 °C for 30 minutes. Fatty acid soap and nonionic surfactants were applied as flotation aids. The presence of melted, emulsified wax in the pulp following hot pulping posed a challenge with regard to froth flotation since the ability to form stable foam is severely curtailed by the wax. Lacking stable foam, a considerable portion of the wax that is carried to the surface can, under the moderately turbulent conditions in the flotation cell, be remixed with the pulp. Pulp samples were examined for wax using solvent extraction. Residual wax levels in pulp after flotation ranged from 10-15%.

We hypothesized that by using single stage of washing, perhaps a sufficient quantity of wax would be removed from the pulp so that the remaining wax would not negatively affect the foaming characteristics of the flotation surfactant. Cascade coated OCC was repulped under the following conditions:

| | |
|---|---|
| Consistency, % | 6.18 |
| Initial temperature, °F | 126.5 |
| Final temperature, °F | 155 |
| Initial pH | 10.4 |
| Final pH | 9.2 |
| Pulping time, min | 70 |

Pulp was washed with 140 °F water on 150 mesh screen immediately after pulping. The pulp yield following washing was 73.4%. Washed pulp was then processed through lab flotation in two batches, one using a fatty acid soap, and the other using a flotation surfactant. Conditions during flotation are described in Table 1. When the flotation aid was the fatty acid soap, the water hardness was adjusted to 150 ppm with $CaCl_2$.

**Table 1. High Temperature Flotation Using Fatty Acid Soap and Surfactant.**

| | Fatty acid soap | Surfactant |
|---|---|---|
| Temperature, °F | 145 | 147 |
| pH | 8.4 | 8.36 |
| Cons, % | 1.0 | 1.0 |
| Chem dosage | 0.5% | 6 lbs/ton |
| Air intake, L/min | 25 | 25 |
| Reject rate, % | 4.7 | 6.8 |

| | | |
|---|---|---|
| Test results for wax content in pulp are as follow: | After washing | 8.29% |
| | Flotation accepts, fatty acid soap | 5.10% |
| | Flotation accepts, surfactant | 4.44% |

**Discussion**

*Based on the amount of foam and persistence of foam observed during flotation, we suggest that one stage of washing* should precede flotation. The surfactant showed slightly better performance than the fatty acid soap. The relatively high levels of wax in the accepts pulp suggest that a considerable amount of attached wax was still present.

**2.2 Laboratory Pulping Trials Using Nalco Wax Dispersant**

*2.2.1 Background*

Nalco Chemical Company has developed a product which is intended to minimize the impact of wax in recycled OCC systems. The Nalco dispersant chemical alters the behavior of wax by combining the following three components:

1. melting point reduction agent
2. wax dispersant
3. wetting agent

According to Nalco's laboratory research, the chemical depresses the melting point of paraffinic waxes during pulping and disperses the wax into a fairly narrow range of particles sizes (0.5-0.8 mm diameter). One component of the chemical stabilizes the wax emulsion so that the wax does not reagglomerate when the pulp cools. Mills using the Nalco technology have the option of either washing the dispersed wax from the pulp, or adding a retention aid to "fix" the finely dispersed wax onto the fibers, whereby the wax leaves the mill in the final product.

The objectives of our research with Nalco were as follow:

1. Chemically treat wax-containing furnish in the pulper to disperse and stabilize wax.
2. Process treated pulp through combination of flotation and washing.
3. Compare the treated pulp to control (untreated) pulp in terms of mechanical properties, angle of slide, wax in handsheets (image analysis), and wax content (solvent extraction).

*2.2.2 Particle size control in existing OCC mills*

The first series of experiment were designed to examine the dispersant chemical treatment of a "worst case" wax-contaminated furnish. The furnish contained 3% cascade-coated waxed board, 6% curtain-coated board, 6% waxed medium, and 85% clean OCC. Two trials, one without chemical, and one with 3 lb chemical/ton of fiber, were conducted using the Voith laboratory pulper at low consistency and moderate temperature. The dispersant dosage recommended by Nalco is 0.1 - 0.25 lbs of chemical per lb of wax. The furnish combination used in this experiment contained 30 lbs of wax/ton so the 3 lb/ton dosage of chemical represented the lower limit of the recommended dosage. The lowest recommended treatment level was chosen to confirm the lower level of treatment under pilot plant conditions.

Flake content was determined by 0.006-in slotted laboratory screen following pulping. Each of the two pulps were divided into two portions. Runs 1 and 2 were untreated pulp, while runs 3 and 4 were chemically treated. Runs 2 and 4 were washed on the Britt jar (150 mesh). Runs 1 and 3 were not washed. Pulping conditions and results are shown in Table 2.

**Table 2. Mixed Wax Furnish Pulping Conditions and Flake Content.**

| | Run 1 | Run 2 | Run 3 | Run 4 |
|---|---|---|---|---|
| Cons., % | 6.33 | | 6.31 | |
| Pulping Temp., °F | | | | |
| Initial | 116 | | 116 | |
| Final | 144 | | 142 | |
| pH | 7.3 | | 7 | |
| Pulping Time, min | 30 | | 30 | |
| Nalco Dispersant | Zero | | 3 lb/ton | |
| Flakes, % | 5.2 | | 3.7 | |
| Britt jar washing | No | Yes | No | Yes |

Britt jar washing: Five grams (o.d. basis) pulp was washed four times (800 mL each) with tap water at 120 °F. Stirring speed was 750 rpm. A sufficient number of pulp was washed to generate 20 g of washed pulp.
Handsheets: TAPPI sheets at 1.2 g were formed. A retention aid was added to the pulp prior to making the sheets to retain wax . The retention aid application rate was:

8 lbs/ton of Nalco 7583 coagulant + 1 lb/ton of Nalco 7520 flocculent

Wax pad preparation: Pads were prepared on filter paper (30 mm pore). Each pad contained 3-5 g (o.d. basis). Wax content in pulp was determined by solvent extraction using 1,1,1-trichloroethane.
Water clarification: Filtrate from Britt jar washing, runs 2 and 4, was treated with polymers to test effectiveness of wax removal from water. Testing was conducted on filtrates at room temperature. Several concentrations of coagulant and flocculent were dosed. Turbidity was determined on the clarified layer decanted from the sample.
Physical testing: Slide angle, burst index and tensile index were determined from handsheets.
Slide angle testing: One set of handsheets from each run was hot pressed to determine this effect on the angle of slide.
Image analysis: A set of handsheets from each run were dyed prior to examination by image analysis. Due to the excessive wax levels in the sheets, image analysis was not feasible.
Particle size analysis: Pulps from the four runs were filtered through Whatman no. 4 filter paper (25 mm pore) and the filtrate was analyzed through Mastersizer®.

Test results for the first four runs are shown in Table 3. Filtrate clarification results are given in Table 4.

**Table 3. Mixed Wax Furnish Test Results Using Nalco Wax Dispersant.**

| | Run 1 Untreated No Wash | Run 2 Untreated Washed | Run 3 Treated No Wash | Run 4 Treated Washed |
|---|---|---|---|---|
| Wax content, %[a] | 1.32 | 0.72 | 1.46 | 0.94 |
| Slide angle, degrees[b] | | | | |
| Felt/Felt w/o heating | 17.4 | 20.3 | 15.5 | 19.2 |
| Felt/Felt with heating | 14.9 | 13.6 | 15.1 | 16.3 |
| Wire/Wire w/o heating | 18.5 | 22.0 | 20.0 | 21.9 |
| Wire/Wire with heating | 16.9 | 18.4 | 15.4 | 17.0 |
| Burst index, kPa.m²/g | 1.75 | 1.15 | 2.01 | 1.27 |
| Tensile index, N.m/g | 37.7 | 31.2 | 37.6 | 31.8 |

[a] Wax content of original mixed wax furnish was 1.48%
[b] Slide angle of "clean" OCC (repulped in disintegrator at 1.2% cons., 30 min, 130 °F): Wire/Wire 24.5° Felt/Felt 21.3°

**Table 4. Filtrate Turbidity for Pulps Washed in Runs 2 and 4, NTU.**

| Coagulent, ppm | Flocculent, ppm | Run 2 (untreated) | Run 4 (treated) |
|---|---|---|---|
| 0 (Control) | 0 (Control) | 270 | 137 |
| 5 | 0 | 160 | 85 |
| 10 | 0 | 101 | 73 |
| 20 | 0 | 75 | 62 |
| 40 | 0 | 75 | 56 |
| 20 | 2 | 71 | 57 |
| 40 | 4 | 58 | 63 |

**Discussion**

The flake content result from the pulping including the dispersant (Runs 3 and 4, Table 2) indicated that the chemical had a significant effect on defibering. Flake content decreased from 5.2% in the untreated pulp to 3.7% in the pulp treated with dispersant.

The wax content in unwashed pulp was similar to the starting wax content, which is what would be expected. Wax content in the washed, untreated pulp (0.72%) was less than that in the washed dispersant-treated pulp (0.96%). This was not expected, since the dispersant should yield emulsified, stable particles which are amenable to removal from the pulp by washing.

Although the angle of slide (AOS) results were inconclusive, it should be noted that the samples with dispersant contained 10-20% more wax, yet no difference in the AOS was noted between the samples with and without dispersant. The difference in AOS between washed and unwashed pulps is likely due to the presence of fines in the unwashed sheets which affected surface smoothness. Hot pressing also had a significant effect, as AOS decreased slide angle by 3-7°. This decrease in AOS was probably caused by melting of wax increasing the surface smoothness.

Burst index was slightly higher in the dispersant-treated sample than in the untreated sample, while tensile indices for treated and untreated were about the same. The removal of fines through washing had a negative impact on burst and tensile in both the untreated and treated pulps.

The effect of dispersant chemical on wax particle size was inconclusive since the particle size could not be measured in handsheets. Particle size of filtrate samples was similar for all samples, untreated and treated, with the bulk of particles in the 0.5-0.6 mm range. Note that these were filtered samples. Therefore if large particles were present in the untreated samples, these particles would have been removed prior to particle size analysis. Cooling the sample did not appear to affect the particle size.

Filtrate from untreated washed pulp was easier to clarify compared to that from treated washed pulp. Thus, turbidity of untreated pulp filtrate decreased from 270 to 58, or 78.5%, while turbidity of treated pulp filtrate decreased from 137 to 63, or 54%.

*2.2.3 Flotation of 100% cascade-coated OCC*

In this set of trials we examined the combination of Nalco dispersant chemistry and froth flotation. Of secondary importance during these trials was to identify a flotation surfactant that is effective in removing wax at high temperature.

Cascade-coated board was pulped at two temperatures, 90 and 140 °F, with and without wax dispersing chemical, for a total of four runs (Runs 5-8). The wax content of the cascade-coated board was ~29% (solvent extraction). A control consisting of clean OCC pulped at ~120 °F was also evaluated (Run 9). Flake content (0.0006-in laboratory screen rejects) of each pulp run was determined. The pulps from runs 5 and 6 were processed through flotation. Due to the excessive flake content in runs 7 and 8 (lower pulping temperature), flotation was not conducted on the pulps. A subsample of flotation accepts from runs 5 and 6 was washed in the Britt jar, and an unfloated sample from runs 5 and 6 was also washed in the Britt jar. All samples were tested for wax content (solvent extraction), AOS, tensile, and burst index. Pulping conditions are given in Table 5.

**Table 5. Pulping Conditions for 100% Cascade-Coated Board With and Without Nalco Wax Dispersant.**

| | Run 5 | Run 6 | Run 7 | Run 8 | Run 9[a] |
|---|---|---|---|---|---|
| Consistency, % | 5.95 | 5.82 | 6.63 | 6.20 | 6.49 |
| Pulping Temp., °F | | | | | |
| Initial | 143 | 141 | 91 | 87 | 117 |
| Final | 158 | 150 | 138 | 135 | 141 |
| pH | 10.4 | 10.0 | 10.8 | 10.2 | 6.7 |
| Pulping time, min | 70 | 70 | 70 | 70 | 30 |
| Dispersant chemical | 0 | 60 lb/ton | 0 | 60 lb/ton | 0 |
| Flake content, % | 5.9 | 5.8 | 10.4 | 10.3 | 4.0 |

### *2.2.4 Flotation surfactants*

Using run 5 pulp in the Voith laboratory flotation cell, several surfactants from High Point Chemicals were evaluated at two dosages, 10 lbs/ton and 20 lbs/ton, for foam volume, persistence, and amount of fiber loss. Two surfactants, DI-2803 and X-91057, were selected from this group. Flotation conditions used to process pulp from runs 5 and 6 are shown in Table 6.

**Table 6. Flotation Conditions.**

| | Run 5 (no dispersant) | | Run 6 (with dispersant) | |
|---|---|---|---|---|
| | DI-2803 | X-91057 | DI-2803 | X-91057 |
| Surfactant dosage, lb/ton | 20 | 20 | 20 | 20 |
| Temp.[a] °F | 141 | 142 | 144 | 143 |
| pH | 8.2 | 8.5 | 8.1 | 8.6 |
| Cons., % | 1.0 | 1.0 | 1.0 | 1.0 |
| Air intake, L/min | 25 | 25 | 25 | 25 |
| Reject rate, % | 35 | 34 | 37 | 84 |

[a] Temperature loss was approximately 10° F after 20 minutes.

**Table 7. Test Results from Washing and Flotation of Cascade-Coated Board.**

| | Run 5 (no dispersant) | | Run 6 (with dispersant) | |
|---|---|---|---|---|
| Wash accepts (no flotation) | 6.75 | | 8.08 | |
| Surfactant | DI-2803 | X-91057 | DI-2803 | X-91057 |
| Flotation accepts | 6.19 | 9.15 | 4.79 | 9.13 |
| Flotation-Wash accepts | 4.86 | 6.19 | 4.14 | 5.58 |

**Table 8. Test Results from Washing and Flotation of Cascade-Coated Board.**

| | Run 5 (no dispersant) | | Run 6 (with dispersant) | |
|---|---|---|---|---|
| Wash accepts (no flotation) | 6.75 | | 8.08 | |
| Surfactant | DI-2803 | X-91057 | DI-2803 | X-91057 |
| Flotation accepts | 6.19 | 9.15 | 4.79 | 9.13 |
| Flotation-Wash accepts | 4.86 | 6.19 | 4.14 | 5.58 |

**Table 9. Angle of Slide (AOS), Tensile Index and Burst Index.**

| | Run 5 (no dispersant) | | Run 6 (with dispersant) | |
|---|---|---|---|---|
| **AOS** | | | | |
| Wash accepts Felt/Felt | 15.1 | | 14.2 | |
| Wash accepts Wire/Wire | 17.8 | | 16.1 | |
| | DI-2803 | X-91057 | DI-2803 | X-91057 |
| Flotation-Wash accepts Felt/Felt | 16.7 | 17.2 | 18.0 | 15.1 |
| Flotation-Wash accepts Wire/Wire | 19.2 | 17.9 | 19.2 | 17.7 |
| **Tensile Index, N.m/g** | | | | |
| Wash accepts | 37.51 | | 33.91 | |
| | DI-2803 | X-91057 | DI-2803 | X-91057 |
| Flotation-Wash accepts | 32.82 | 33.66 | 32.09 | 27.13 |
| **Burst Index, kPa.m²/g** | | | | |
| Wash accepts | 1.91 | | 1.49 | |
| | DI-2803 | X-91057 | DI-2803 | X-91057 |
| Flotation-Wash accepts | 1.35 | 1.47 | 1.59 | 1.06 |

**Discussion**

As evidenced by the flake content values, the presence of the dispersant chemical in the furnish did not appear to aid in the repulping of the wax-coated board. At both pulping temperatures, the flake content was similar in pulp treated with dispersant and without dispersant. It is possible that under the conditions of the experiment, i.e., long pulping time, the effects of the dispersant in releasing wax, and thus promoting defibering, were masked.

The wax removal by the flotation-washing sequence was more effective than that of washing alone. In run 5 (without dispersant) the wax content after the washing sequence was 6.75%, and 4.86% after the flotation-washing sequence. Surprisingly, the wax content in the dispersant-treated pulp after washing was 8.08% and after flotation-washing sequence it was 4.14%. It appears that the wax particles formed during the pulping of dispersant-treated furnish were more effectively removed by flotation than by washing.

The slide angle of the run 6 wash accepts was slightly less than that of the run 5 wash accepts suggesting that there was more wax remaining in the dispersant treated pulp. Slide angle reflects the amount of wax remaining in the handsheet sample. Those factors that lowered wax content in the pulp also increased slide angle. Slide angle of the flotation-washed accepts was higher than that for the washed-only pulp. The AOS of dispersant-treated flotation-washed accepts was slightly higher than that of the untreated flotation-washed accepts, but the difference was probably not significant.

The particle size distribution suggests that the wax agglomerates during flotation, likely due to the interaction between wax particles and the flotation surfactant. Particle size before flotation was 0.6 mm (pulp filtered on Whatman 4 filter paper), while the largest concentration of wax particles in the flotation rejects (sample not filtered) were ~100 mm. The average size of particles in the run 6 (dispersant-treated pulp) flotation rejects was slightly larger than in run 5 (untreated pulp) rejects.

Burst and tensile indices declined in flotation-washed accepts compared to wash only pulp. This may be related to the loss of some long fiber during flotation or may be attributed to the presence of flotation chemical.

Wax results indicate that the flotation surfactant DI2803 was more effective than X-91057. Foaming was greater with X-91057. However, more fibers were entrained in the foam, especially in run 6, where the reject rate was 84%.

Overall, while some subtle differences were observed when comparing the trials conducted with dispersant-treated wax-coated furnish to untreated furnish, the cost of introducing the chemical to treat 100% wax coated OCC does not justify its use at this time. Consider that in the trials using 100% cascade-coated board, the wax dispersant was applied at 60 lbs/ton. Note that the wax dispersant was designed for lower levels of wax contamination (0.5-3%) and that the goal of using the product in these experiments was to determine if any benefits could be observed with the products as it is currently available. Based on these results, custom formulations will be developed which will use only certain components of the product, most notably, the melting point reduction functionality and the redeposition functionality.

## 2.3 Evaluation of Water Clarification Polymers

The mentoring group recommended that we investigate procedures for increasing the efficiency of wax removal from filtrate. Several different clarification coagulent and flocculent polymers from Buckman Laboratories were tested on wash filtrate. Cascade-coated wax board was pulped at low consistency and 160 °F in the Voith laboratory pulper for 70 minutes. Pulp was immediately washed on 150 mesh screen with water at 150 °F. The effectiveness of the polymers in flocculating the dispersed wax in the wash filtrate was evaluated. Testing was conducted using gang stirrers.
Anionic polymer was not effective in clarifying the wash filtrate, with the wax remaining relatively dispersed after treatment. In contrast, cationic polymers were more effective, producing better results visually. The results shown in Table 10 were from testing at two temperatures at the same polymer dosage of 10 ppm. Flocculated water was filtered through Whatman #4 paper, and this filtrate was measured for turbidity.

**Table 10. Clarification of Wash Filtrate from Pulped Waxed OCC. Results in NTU (Nephelometric Turbidity Units).**

| | Polymer | | | |
|---|---|---|---|---|
| Condition | 5394 | 590 | 591 | control |
| 126 °F, 10 ppm | 126 | 162 | 419 | 462 |
| 150 °F, 10 ppm | 529 | 177 | 227 | 374 |

**Discussion**
It was concluded that Buckman 590 was the most consistent performer at both high and low temperatures, while at low temperature 5394 gave the best results with regard to turbidity.

## 2.4 High Temperature Washing and Kneading Trials

A persistent problem that accompanied the laboratory pulping trials was the incomplete detachment of wax from the cascade-coated wax board. A set of experiments were designed to determine if the wax nits that exist following pulping at high temperature, not removable by screening, could be disintegrated by kneading. Cascade-coated wax board was pulped under the following conditions:

| | |
|---|---|
| Consistency, % | 6.4 |
| Initial Temperature, °F | 159 |
| Final Temperature, °F | 187 |
| Initial pH | 10.05 |
| Final pH | 8.3 |
| Pulping time, min | 70 |

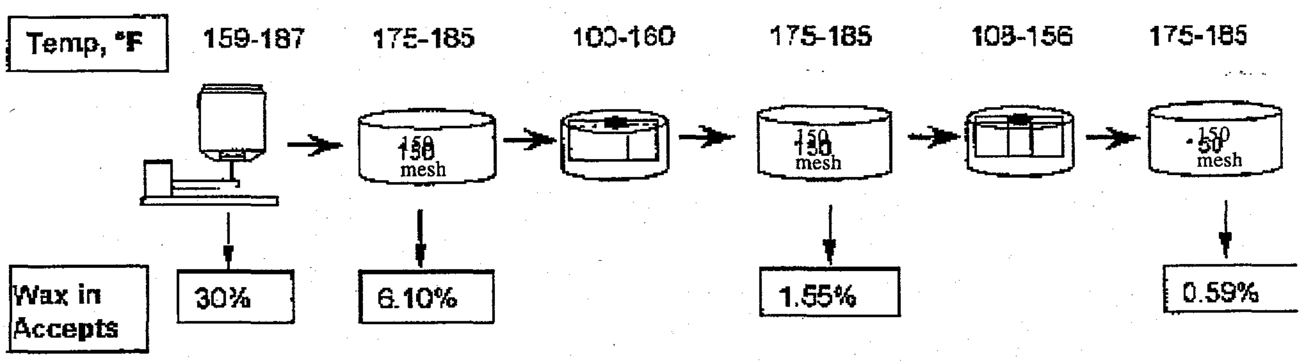
*Washing*
Immediately following pulping, five samples of pulp were washed in a Britt jar under the following conditions:

5 g pulp per washing series
175-185 °F wash water temperature
Stirring speed 750 rpm
Wash with 6 volumes water, 800 ml each

Washed pulp was tested for wax content by solvent extraction.

*Kneading-Washing*
Three samples from the pulper of approximately 100 g each were measured. Each sample was washed on a 150 mesh wire with 175-185 °F water. The three washed pulps were combined and placed into a Quantum Technology, Inc., Mark V Laboratory Mixer/Reactor bowl. Consistency of the pulp was 23%. Pulp was kneaded for 20 min at 15 Hz (approximately 600 rpm). The starting temperature was 100 °F and the final temperature was 160 °F. After kneading, pulp was washed again on the 150 mesh wire for 10 min. A sample was taken for wax determination. Following thickening, the remainder of the pulp was placed in the Quantum Mixer and kneaded for an additional 20 min. Starting temperature was 108 °F and final temperature was 156 °F. Pulp was removed from the Quantum Mixer and washed on 150 mesh wire for 10 min. A schematic of the kneading sequence is shown in Figure 1.

Figure 1: Pulping, washing, kneading sequence used in laboratory study.




**Discussion**
Figure 1 shows the wax content in pulp following each step of the washing/kneading sequence. Nearly all of the wax was removed by washing following the second kneading stage. This exercise suggests that a major deterrent to complete wax removal is the bound wax remaining in the fiber nits. Flotation and washing are not able to remove this bound wax in nits, and screening is not effective since the nits are small enough to pass 0.006-in slots.
The results of this experiment suggest that aggressive mechanical action applied through kneading or dispersion may be sufficient to detach bound wax. Although the kneading action of the Quantum mixer is relatively mild, after two kneadings

and washings, extractable content of the pulp was 0.6%. The cascade-coated wax board that has been used in the laboratory experiments is very difficult to defiber and dewax without the additional energy afforded by kneading or dispersion.

**2.5 Pilot Plant Trials I-III**

OCC supplied by three sources was processed in the Voith Sulzer Paper Technology Center pilot plant at Appleton, WI. The objective was to examine the removal of wax and stickies from OCC. These included two recycling mills, Green Bay Packaging, Green Bay, WI, and U.S. Papers, Menasha, WI, and one scrap paper dealer, Golper Supply, Appleton, WI. The furnish provided by Green Bay Packaging was considered to be of marginal quality, i.e., material that is normally rejected by the mill was included for evaluation purposes. The material sent by U.S. Papers was considered to be their typical furnish. The material supplied by Golper supply was considered a normal OCC; however, it contained approximately 1% carrier containers and 1% WCC.

During three separate trials, each furnish was processed through the Voith Sulzer pilot plant configured as a conventional OCC stock preparation process. Alternatively, froth flotation was coupled with the conventional system. Additional washing and dissolved air flotation (DAF) clarification was also evaluated. Stock was pulped at low consistency, processed through HD cleaner, coarse screen, fine screen, flotation, through-flow cleaner, and Hydrodisk washer. Stock temperature was 120 °F. A schematic diagram of the stock preparation system is shown in Figure 2.

Three different methods were used for the quantification of wax and stickies. CPPC/FPL method of using water soluble black dye and reverse image analysis was employed to measure wax and stickies spots. The method developed by Voith Sulzer, TAPPI T277 pm-99 was used for measuring tacky contaminants. Extraction using trichloroethane solvent was used to determine the concentration of extractable in the pulp samples. The amount of extractable in clean OCC averaged 0.28%.

*2.5.1 Trial I: OCC from Paper Stock Dealer*

The OCC for the first pilot plant trial was obtained from a scrap paper dealer. The furnish was inspected on the conveyor to the pulper where it was determined that approximately 1% carrier boxes and 1% WCC were present.

Batch pulping was conducted at 49 °C (120 °F) and approximately 5% consistency. A commercially available flotation surfactant was added to the pulper at the rate of 1 kg/Mg (2 lb/ton) furnish. The pH was not adjusted. The water as supplied by the local municipality was slightly alkaline, pH 8.0, and the hardness was 80 mg/L as $CaCO_3$. Following pulping, stock was pumped to a 4.53 Mg (5 ton) dump chest where it was blended with preceding pulper dumps. When the pulping of a sufficient quantity of OCC to fill the dump chest was completed, stock was processed through the system on a continuous basis. The stock preparation sequence is shown the Figure 1. When the system was operated without flotation, stock passed from the fine slotted screens directly to the through-flow cleaners, bypassing the flotation cells. Filtrate from the washer and from the belt press was clarified through a 5.2 meter (17') diameter DAF clarifier. Clarified effluent was returned as dilution water to the various unit processes. A medium charge, high molecular weight, cationic polymer was used to flocculate the solids in the influent sent to the clarifier.

Samples collected at the various points in the process were analyzed for the following:

· Consistency, Canadian Standard Freeness, pH,

· Wax spot count was determined by dyed handsheet image analysis; Wax% was determined by solvent extraction; Stickies were determined by TAPPI T277 pm-99 "Tackies" procedure.

Samples collected before and after the thru-flow cleaners and after the belt press (with and without the flotation unit operating) were tested for the following physical properties:

Basis weight, bulk, tensile index, tear index, burst index, ring crush, slide angle (wire side and felt side of sheet), and STFI.

The effectiveness of water clarification was evaluated by turbidity testing and conductivity measurement.

Figure 2: Stock preparation flow diagram of Voith Sulzer Technology Center pilot plant.

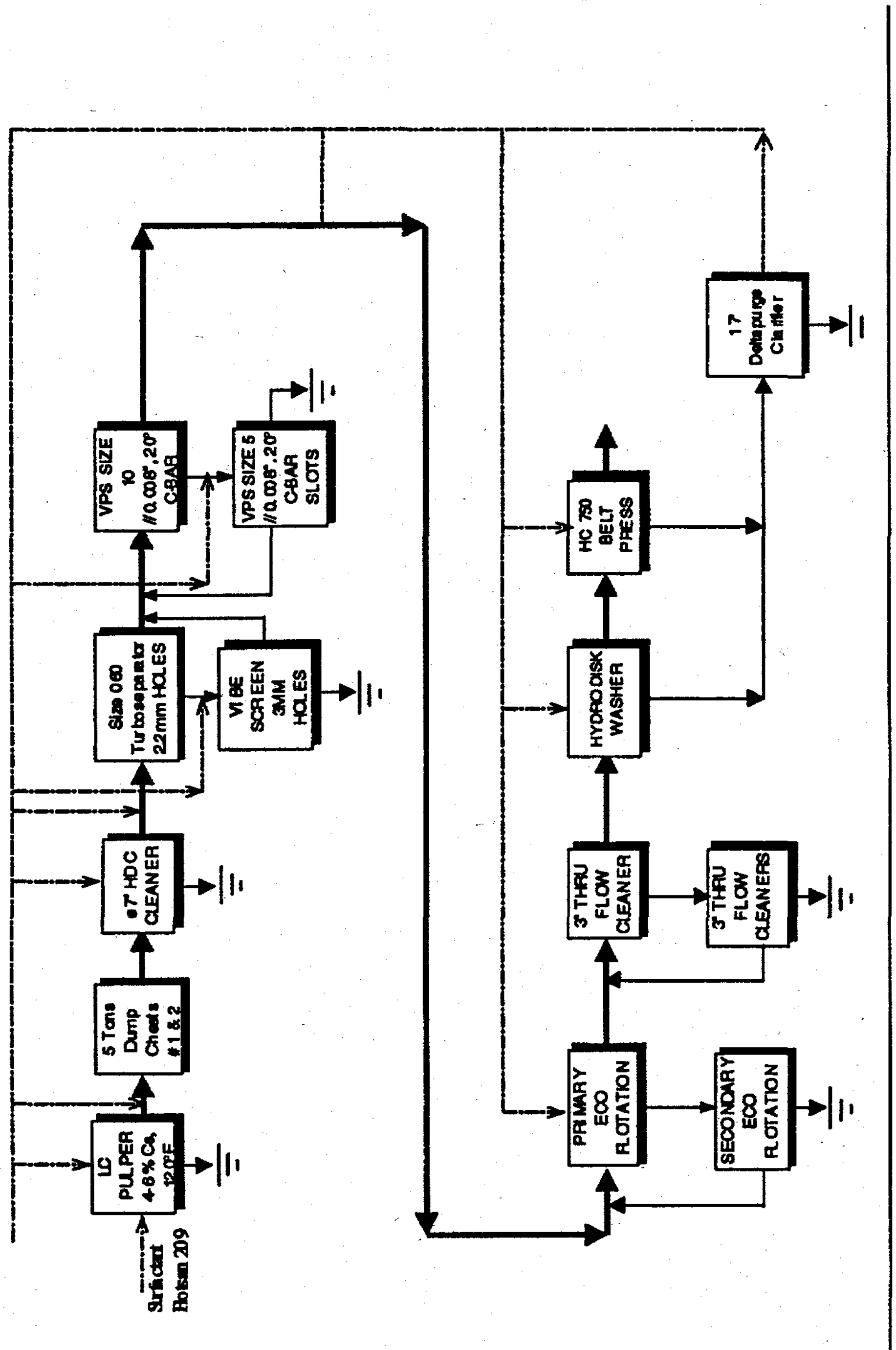


**Table 11. Test Results for Trial I, OCC from Paper Stock Dealer.**

| Sample | Cons. | CSF | Wax Spot | pH | Temp. | Stickies | Wax |
|---|---|---|---|---|---|---|---|
| | % | (ml) | Test (ppm) | | (°F) | (T277) | (%) |
| | | | | | | @0.006 in | |
| | | | | | | ($mm^2$/kg) | |
| Dump chest | 3.35 | | | | | | |
| Inlet Fine Screen | 1.75 | 570 | 34158 | 6.60 | 103 | 29202 | 0.65 |
| Fine Screen Accept | 1.4 | 510 | 18348 | | | 4002 | 0.38 |
| Flotation Feed | 0.92 | 520 | 39449 | 7.00 | 104 | 4801 | 0.31 |
| Flotation Accept | 0.94 | 505 | 12935 | | | 2358 | 0.28 |
| Sec. Flotation Reject | 0.47 | | | | | | 1.54 |
| Inlet Thru-flow cleaner | 0.63 | | 13538 | | | 3922 | 0.22 |
| Thru-flow cleaner accept | 0.69 | 525 | 16722 | 7.50 | 104 | 2350 | 1.58 |
| Washer accept | 6.24 | 660 | 9650 | 7.50 | 104 | 2695 | 1.17 |
| Belt press dewatered pulp | 36.06 | 640 | 7511 | | | 1879 | 1.35 |
| Filtrate to Clarifier | 0.06 | | | | | 0.7 | |
| Clarified effluent | 0.01 | | | | | 0.1 | |
| | | | | | | | |
| Inlet Thru-flow cleaner NF* | 0.75 | | 27771 | | | 4833 | 1.17 |
| Thru-flow accept NF | 0.77 | 540 | 15813 | 7.45 | 106 | 3665 | 1.63 |
| Belt press dewatered pulp NF | 35.94 | 630 | 17682 | | | 2419 | 1.38 |
| Filtrate to Clarifier NF | 0.06 | | | | | 0.3 | |
| Clarified effluent NF | 0.01 | | | | | 0.5 | |

Note: microstickies of samples 1.200 and 1.201 were determined using Robert DeJong method, based on kg of process water.

NF* Operation without using flotation cells

**Table 11a. Summary of Physical Properties from Trial I.**

| Sample | Basis | Bulk | Tensile | Tear | Burst | Ring | Slide | Slide | STFI |
|---|---|---|---|---|---|---|---|---|---|
| | Weight | ($cm^3$/g) | Index | Index | Index | Crush | Angle | Angle | |
| | ($g/m^2$) | | (N m/g) | ($mNm^2$/g) | (kPa $m^2$/g) | (lb f/in) | WW | FF | lb f/in |
| Thru-flow cleaner accept | 69.5 | 2.35 | 30.3 | 12.53 | 1.562 | 11.5 | 29 | 29 | 5.465 |
| Belt press dewatered pulp | 65.5 | 2.51 | 30.8 | 11.26 | 1.395 | 9.4 | 30 | 26 | 5.089 |
| Thru-flow accept NF* | 67.2 | 2.39 | 30.5 | 11.45 | 1.549 | 10.1 | 30 | 29 | 5.699 |
| Belt press dewatered pulp NF | 66.5 | 2.34 | 29.0 | 11.80 | 1.358 | 8.6 | 33 | 26 | 4.714 |

NF* Operation without using flotation cells

**Table 12. Test Results for Trial II, OCC from Green Bay Packaging.**

| Sample | Cons. % | CSF (ml) | Wax Spot Test (ppm) | pH | Temp. (°F) | Stickies (T277) @0.006 in (mm²/kg) | Wax (%) |
|---|---|---|---|---|---|---|---|
| Dump chest | 4.87 | | | 6.6 | 110 | | |
| Inlet Fine Screen | 2.04 | 550 | 186920 | 5.72 | 109 | 52,000 | 1.59 |
| Fine Screen Accept | 2.00 | 540 | 65550 | | | 5650 | 1.33 |
| Flotation Feed | 1.17 | 490 | 119120 | 5.13 | 107 | 3170 | 1.28 |
| Flotation Accept | 0.85 | 520 | 26450 | | | 4087 | 0.53 |
| Sec. Flotation Reject | 0.74 | | | | | | 12.9 |
| Inlet Thru-flow cleaner | 0.74 | | 24830 | | | 3479 | 0.89 |
| Thru-flow cleaner accept | 0.76 | 530 | 25450 | 5.20 | 108 | 1729 | 0.35 |
| Washer accept | 5.92 | 650 | 6880 | 5.10 | 108 | 2235 | 0.48 |
| Belt press dewatered pulp | 36.04 | 630 | 17360 | | | 1523 | 0.21 |
| Filtrate to Clarifier | 0.07 | | | | | 0.3 | |
| Clarified effluent | 0.01 | | | | | 0.1 | |
| | | | | | | | |
| Inlet Thru-flow cleaner NF* | 0.74 | | 227675 | | | 2076 | 1.52 |
| Thru-flow accept NF | 0.84 | 510 | 67340 | 5.11 | 103 | 2269 | 0.91 |
| Belt press dewatered pulp NF | 25.4 | 615 | 61226 | | | 7051 | 0.74 |
| Filtrate to Clarifier NF | 0.07 | | | | | 0.6 | |
| Clarified effluent NF | 0.01 | | | | | 1.1 | |
| | | | | | | | |

Note: microstickies of samples 1.200 and 1.201 were determined using Robert DeJong method, based on kg of process water.

NF* Operation without using flotation cells

**Table 12a. Summary of Physical Properties from Trial II.**

| Sample | Basis Weight (g/m²) | Bulk (cm³/g) | Tensile Index (N m/g) | Tear Index (mNm²/g) | Burst Index (kPa m²/g) | Ring Crush (lb f/in) | Slide Angle WW | Slide Angle FF | STFI (lb f/in) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | |
| Thru-flow cleaner accept | 66.2 | 2.30 | 22.8 | 10.79 | 1.448 | 9.30 | 31 | 28 | 5.675 |
| Belt press dewatered pulp | 64.7 | 2.36 | 22.9 | 11.28 | 1.295 | 8.95 | 30 | 27 | 5.397 |
| Thru-flow accept NF* | 66.2 | 2.39 | 28.7 | 10.08 | 1.479 | 10.55 | 25 | 26 | 5.181 |
| Belt press dewatered pulp NF | 66.5 | 2.37 | 20.1 | 11.33 | 1.182 | 9.38 | 29 | 25 | 4.938 |

NF* Operation without using flotation cells

**Table 13. Test Results for Trial III, OCC from U.S. Papers.**

| Sample | Cons. % | CSF (ml) | Wax Spot Test (ppm) | pH | Temp. (°F) | Stickies (T277) @0.006 in ($mm^2/kg$) | Wax (%) |
|---|---|---|---|---|---|---|---|
| Dump chest | 3.75 | | | 6.00 | 112 | | |
| Inlet Fine Screen | 1.37 | 405 | 7059 | 6.40 | 107 | 25220 | 1.35 |
| Fine Screen Accept | 1.32 | 360 | 6058 | | | 8579 | 1.02 |
| Flotation Feed | 0.93 | 350 | 5123 | 6.60 | 106 | 9622 | 0.98 |
| Flotation Accept | 0.6 | 510 | 3251 | | | 6021 | 0.43 |
| Sec. Flotation Reject | 0.15 | | | | | | 12.03 |
| Inlet Thru-flow cleaner | 0.51 | | 4218 | | | 5912 | 0.48 |
| Thru-flow cleaner accept | 0.73 | 505 | 2546 | 6.60 | 107 | 3541 | 0.22 |
| Washer accept | 6.94 | 605 | 3212 | 7.10 | 107 | 2736 | 0.15 |
| Belt press dewatered pulp | 40.18 | 565 | 2811 | | | 2602 | 0.24 |
| Filtrate to Clarifier | 0.06 | | | | | 0.2 | |
| Clarified effluent | 0.01 | | | | | 0.2 | |
| | | | | | | | |
| Inlet Thru-flow cleaner NF* | 0.67 | | 5594 | | | 6120 | 0.93 |
| Thru-flow accept NF | 0.76 | 375 | 3968 | 6.90 | 103 | 11515 | 0.83 |
| Belt press dewatered pulp NF | 27.5 | 500 | 3502 | | | 6954 | 0.43 |
| Filtrate to Clarifier NF | 0.06 | | | | | 0.7 | |
| Clarified effluent NF | 0.01 | | | | | 0.2 | |
| | | | | | | | |

Note: microstickies of samples 1.200 and 1.201 were determined using Robert DeJong method, based on kg of process water.

*NF* Operation without using flotation cells*

**Table 13a. Summary of Physical Properties of Samples from Trial III.**

| Sample | Basis Weight ($g/m^2$) | Bulk ($cm^3/g$) | Tensile Index (N m/g) | Tear Index ($mNm^2/g$) | Burst Index (kPa $m^2/g$) | Ring Crush (lbf/in) | Slide Angle WW | Slide Angle FF | STFI (lb f/in) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | |
| Thru-flow cleaner accept | 65.4 | 2.33 | 27.2 | 9.60 | 1.386 | 10.35 | 29 | 27 | 4.919 |
| Belt press dewatered pulp | 68.6 | 2.28 | 27.2 | 10.41 | 1.352 | 10.10 | 32 | 27 | 5.344 |
| Thru-flow accept NF* | 63.5 | 2.24 | 27.6 | 7.04 | 1.330 | 8.55 | 29 | 28 | 6.170 |
| Belt press dewatered pulp NF | 63.9 | 2.41 | 26.5 | 8.47 | 1.462 | 7.85 | 29 | 26 | 5.352 |

NF* Operation without using flotation cells

A summary of test results from Trial I is shown in Table 11. Physical properties are shown in Table 11a.

*2.5.2 Trial II: OCC from Green Bay Packaging*

In addition to OCC, the furnish provided by Green Bay Packaging, Green Bay, WI, consisted of approximately 4% carrier stock, 2% wet-strength, 2.5% black construction paper, and 2.5% file stock. Mill personnel indicated that this supply included material that is normally rejected by the mill.

The furnish was processed and samples were collected and tested in the same manner as described in Trial I. The data from Trial II are displayed in Table 12. Physical properties data are shown in Table 12a.

*2.5.3 Trial III: OCC from U.S. Papers*

The typical furnish used at U.S. Papers, Menasha, WI, was supplied. It included approximately 1/3 core stock, 1/3 box cuts, and 1/3 mixed papers. The box cuts were comprised of 50% carrier stock and other kraft clippings. The mixed papers included mixed office papers with ONP and OMG.

The furnish was processed in the manner described in Trial I. Table 13 includes data collected from Trial III. Physical properties are shown in Table 13a.

*2.5.4 Water Clarification*

Washer filtrate from the Hydrodisk washer was routed to the DAF clarifier. A cationic flocculent, Buckman Bufloc™ 590 was used to flocculate suspended solids. During each of the three WCC trials, temperature, pH, turbidity, conductivity, TSS and sludge solids were monitored at the DAF. Flocculent dosages varied for each of the trials in the range 5-10 ppm, but were normally in the 8 ppm concentration. The data are presented in Table 14.

**Table 14. Water Clarification Data from Pilot Plant Trials.**

| Sample | Influent solids, ppm | Effluent solids, ppm | Removal efficiency, % | Sludge solids, % | Flocculent dosage | pH | Temp. °C | Conductivity μS/cm | Turbidity NTU |
|---|---|---|---|---|---|---|---|---|---|
| Trial I control | 776 | 247 | 68.2% | 2.84% | 0 | 7.5 | 40 | 350 | 247 |
| sample 1 | 668 | 14 | 97.9% | 4.0% | 10 ppm | 7.5 | 40 | 350 | 139 |
| sample 2 | 789 | 42 | 95% | 5.2% | 10 ppm | 7.5 | 40 | 350 | 130 |
| sample 3 | 774 | 58 | 92.5% | 4.7% | 7 ppm | 7.5 | 39 | 350 | 136 |
| Trial II control | 834 | 505 | 39.4% | 2.34% | 0 | 6.6 | 39 | 350 | 560 |
| sample 1 | 794 | 74 | 91% | 3.38% | 8 ppm | 7.0 | 38.5 | 350 | 116 |
| sample 2 | 639 | 29 | 95.5% | 4.0% | 7 ppm | 7.2 | 38.8 | 350 | 136 |
| sample 3 | 803 | 61 | 92.4% | 3.92% | 5 ppm | 7.25 | 38.5 | 350 | 154 |
| Trial III control | 1338 | 589 | 56.0% | 3.16% | 0 | 6.9 | 38.3 | 350 | 816 |
| sample 1 | 694 | 38 | 94.5% | 5.16% | 8 ppm | 7.3 | 39.5 | 350 | 171 |
| sample 2 | 496 | 44 | 91.1% | 5.35% | 8 ppm | 7.4 | 39 | 350 | 111 |
| sample 3 | 1163 | 89 | 92.3% | 4.9% | 8 ppm | 7.3 | 39.1 | 350 | 203 |

## DISCUSSION

The furnish used in Trial II (Green Bay Packaging) furnish was the lowest quality of the three commercial OCC furnishes evaluated during these trials. In addition to OCC, a considerable amount of wet-strength container stock was present along with file stock, potential sticky contaminant source. The Trial II furnish also contained a relatively higher percentage of mixed papers than the other furnishes. Surprisingly, the level of hydrophobic material in the handsheets from Trial III furnish (U.S. Papers), as determined by the wax spot test, was much lower than in the other furnishes. Stickies concentration in Trial III furnish was comparable to the OCC supplied by the paper stock dealer (Trial I), and about one-half that of the Trial II furnish.

In Trial I, approximately 50% of the stickies entering the flotation stage were removed by that process. When flotation was not used, through-flow cleaners removed only 24% of the stickies, but when flotation preceded through-flow cleaners, removal efficiency was approximately 40%. Stickies removal efficiency through the entire process was 94% when flotation was in operation, dropping to 92% when flotation was not used. Process yield in Trial I decreased by 5.6% when flotation was used. Wax spots as the Trial I furnish progressed through the system decreases from a value of 34,200 ppm after the perforated coarse screen, to about 13,000 ppm after flotation. The wax spot values for the flotation feed samples from Trials I and II appear to be incorrect since they do not follow the trend shown by the stickies and extractable (wax %) data from these sample locations.

Overall system efficiency in reducing wax spots, stickies, and extractable levels in the Trial II furnish was very good. Again, as seen in Trial I, wax spot removal efficiency was 91% when flotation was applied, and 67% when flotation was not used. The wax spot concentration in Trial II furnish was reduced from 186,900 ppm after the perforated screen to about 26,500 ppm after flotation. The Trial II furnish contained considerably more hydrophobic particles that became evident as spots in the handsheets (wax spot test) than the other two commercial furnishes.

Removal of wax spots during Trial III (U.S. Papers furnish) was 60% when flotation was present and 50% when flotation was not present in the system. Stickies removal was 90% when flotation was used, dropping to 72% when flotation was not present in the system. Considerably less hydrophobic contamination, as evidenced by the wax spot test, was present in this furnish than in the other two furnishes. Additional yield loss was a modest 1.8% when flotation was used with the Trial III furnish.

Since mixed papers were present in the Trial III furnish, the effect of flotation, washing, and clarification on ash levels was of interest. Ash concentration, 7.85% prior to flotation, decreased to 3.9% after through-flow cleaning, and was 2.6% following washing. Freeness increase was correlated with the decrease in ash, increasing from 350 ml prior to flotation, to 605 ml following washing.

The overall process yield when using flotation, washing, and DAF clarification was 85%.

*Water Clarification Discussion*

The control data (Table 4) for each trial depicts the operating condition when the water sent to the clarifier was not treated with polymer. During all trials, solids removal efficiency of greater than 93% was attainable. Influent solids levels in the three furnishes was in the range, 600-1000 ppm. When polymer was not used, the water from Trial II furnish was treated less effectively by DAF clarification alone than the water of the other two trials. All furnishes were clarified to about the same degree in terms of effluent solids and turbidity when polymer was applied. Due to the limited running time in trials of this nature, it was not possible to optimize the polymer dosage and DAF operating conditions.

*Sheet Properties Discussion*

A number of sheet properties were also measured. Burst indices of thickened process pulp (including flotation) were 1.395, 1.295 and 1.352, respectively, for dealer OCC, Green Bay Packaging, and U.S. Papers supplied furnishes. Corresponding values of burst indices without flotation were 1.358, 1.182 and 1.462. Therefore, burst index increased when flotation was included in two of the three trials. Furthermore, tensile index increased in all three trials when flotation was applied.

Due to the relatively low amount of wax in all three furnishes, slide angles (AOS) were in the acceptable range for all.

## 2.6 Mill Trial I: Green Bay Packaging, Green Bay, WI

The Green Bay Packaging mill in Green Bay produces approximately 725 ADMT/day (800 t/day) of linerboard and corrugated medium from OCC, mixed paper, and DLK cuttings. Stock preparation system for top stock and base stock is combined for the processes of pulping, high density cleaning and coarse screening. Following coarse screening, pulp flow is split and directed along two lines: base stock receives fine screening and thickening, and top stock receives forward and reverse cleaning, fine screening, and refining. Approximately one-quarter of the production is top stock and three-quarters is base stock. Wax contamination is not a major issue, however, periodic outbreaks occur and wax spots are observed on the linerboard.

The test Voith Sulzer Eco-Cell (250 liter capacity) was configured to process a stream from the forward cleaner accepts in the top stock cleaning system. A simplified diagram of the stock preparation system with the Eco-Cell configured is shown in Figure 3. This location was chosen for two primary reasons: 1) the consistency of the stock was in the optimum range for flotation, and 2) the effectiveness of flotation in removing stickies and wax could be compared to the existing reverse cleaning system which follows the forward cleaners.

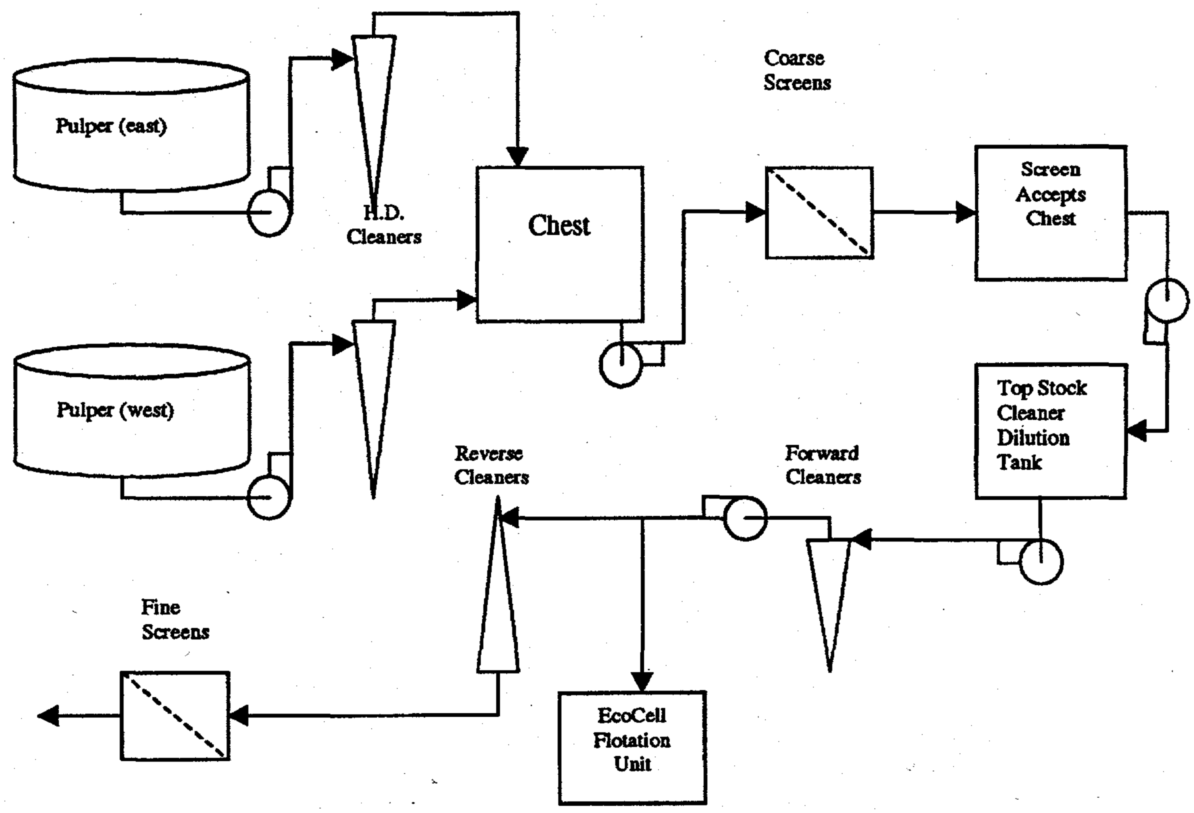



Figure 3: Green Bay Packaging topliner preparation flow chart. Test flotation cell was installed following the forward cleaners (= accepts from forward cleaners).

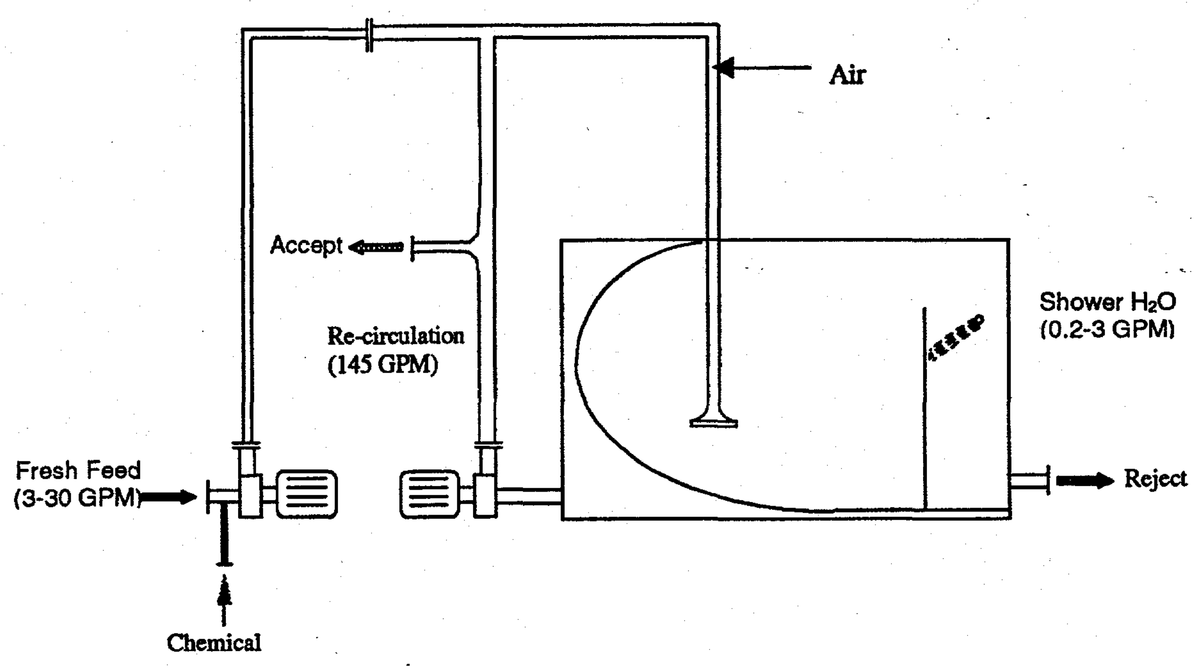



Figure 4: Eco-Cell 250 flow diagram.

*2.6.1 Test Flotation Cell*

A schematic of the Eco-Cell used for the mill trial is shown in Figure 4. The test cell features a recirculation pump which permits the simulation of a flotation system with several stages of flotation. The flow rate of the recirculation pump is approximately 550 L/min (145 gpm). Feed rate to the cell via a feed pump was varied in the range 19 to 114 L/min (5 gpm to 30 gpm) during these mill trials. The volume of air in the stock in the flotation cell was estimated to be about 65% and was determined by measuring the air flow rate through two orifices on the feed pipe to the cell. Air flow rate was measured using a pitot tube placed in the orifices.

Accepts flow rate from the cell is controlled by a valve positioned directly above the discharge of the recirculation pump. Foam containing rejected solids is displaced by surface movement over a weir at one end of the cell. A shower directed into the rejects chamber was used to break the foam and rinse the rejects through a pipe near the bottom of the rejects chamber. This shower was adjusted to about 9.5 L/min (2.4 gpm). The reject rate is controlled based on the difference between the feed flow to the cell and the accepts flow, i.e., reject flow increases as the accepts valve is closed.

The flotation surfactant used during the mill trials, an emulsion-based fatty acid soap, Calsan50T®, was supplied by BASF Corporation. The surfactant was made down to a 2% solution by weight of active ingredient and delivered by a chemical metering pump to the line directly preceding the feed pump. During these trials, surfactant dosage was varied in the range 2 - 4 kg chemical/MT fiber (4 - 8 lb chemical/ton fiber).

*2.6.2 Sampling Plan and Testing*

Samples were collected from the forward cleaner accepts (= feed to the test flotation cell), flotation cell accepts and rejects. Prior to sampling, flow rates of accepts and rejects were determined. Sufficient time passed to allow the volume of the cell to turn over twice (two residence times) before samples were collected. Tests were conducted at three different feed flow rates: 23, 42, and 113 L/min (6, 11, and 30 gpm). On the first day of trials, the flotation tests were run with and without flotation chemical at each flow rate, for a total of six trials. On the second day, two runs were conducted at a feed flow rate of 83 L/min (22 gpm), with and without surfactant. Samples of feed, accepts and rejects were collected at each flow interval. To evaluate the effectiveness of the existing system, samples were also collected from high density cleaner accepts, reverse cleaner feed, reverse cleaner accepts and top stock refiner feed.

Wax spot tests and consistency measurements were conducted on each sample. Five standard handsheets (60 $g/m^2$) for feed and accept samples were pressed at 300 °F and 1 bar (14 psi) for 10 min between two stainless steel plates. Under these conditions, contaminants melt and migrate into the fiber network forming dark spots in the sheet. Following hot pressing, handsheets were treated with hydrophilic black dye. The dyed sheets were dried and the light specks which contrast against the dark fiber background were quantified using scanner-based image analysis in reverse mode. Table 15 shows baseline data on consistency and wax spot area for the existing mill system.

*2.6.3 Trial Results for the Existing Mill System*

Baseline data for the top stock system, which includes coarse and fine screening and forward and reverse cleaning, indicate that overall hydrophobic contaminant removal efficiency was 72%, based on wax spot test. The efficiency of the reverse cleaner system was approximately 9% during the time of the sampling.

**Table 15. Wax Spot Test Results and Consistencies from Existing System at Green Bay Packaging**

| Sample | Test 1 | | Test 2 | | Test 3 | | Average Efficiency |
|---|---|---|---|---|---|---|---|
| | Cons.% | ppm* | Cons.% | ppm | Cons.% | ppm | |
| HD Cleaner Accept | - | - | - | - | 3.26 | 10648 | - |
| Reverse Cleaner Feed | 0.94 | 4031 | 0.96 | 7052 | 0.91 | 4566 | - |
| Reverse Cleaner Accept | 1.16 | 4136 | 1.27 | 6882 | 1.19 | 3193 | 9.2 |
| Top Stock Refiner Feed | - | - | - | - | 2.44 | 2979 | - |

*** Wax spot area in parts per million**

| Run # | Calsan 50T ml/min | Calsan 50T lb/ton | Stock Temp °F | Shwr Water GPM | Accepts GPM | Accepts % Cs | Rejects GPM | Rejects %Cs | Mass Reject Rate % | Wax Spot Feed ppm | Wax Spot Accept ppm | Spot Removal Efficiency % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 90 | 4 | 134 | 2.4 | 25.7 | 1.02 | 6.1 | 0.28 | 6.1% | 6646 | 2832 | 57% |
| 2 | 44 | 4 | 134 | 2.4 | 8.7 | 0.98 | 4.7 | 0.21 | 10.4% | 6646 | 2276 | 66% |
| 3 | 15 | 4 | 134 | 2.4 | 2.7 | 1.09 | 6.1 | 0.23 | 32.3% | 6646 | 1640 | 75% |
| 4 | 0 | 0 | 134 | 2.4 | 26.4 | 1.1 | 5.6 | 0.28 | 5.1% | 6646 | 2289 | 66% |
| 5 | 0 | 0 | 135 | 2.4 | 8.7 | 1.08 | 4.7 | 0.2 | 9.1% | 6646 | 1991 | 70% |
| 6 | 0 | 0 | 135 | 2.4 | 2.6 | 1.28 | 6.0 | 0.17 | 23.5% | 6646 | 1263 | 81% |
| 7[a] | 92 | 4 | 135 | 2.2 | 14.4 | 1.11 | 9.0 | 0.29 | 14.0% | 2527 | 1425 | 44% |
| 8[a] | 0 | 0 | 135 | 2.2 | 14.4 | 1.16 | 10.0 | 0.28 | 14.0% | 2527 | 1016 | 60% |

**Table 16. Operating Parameters and Data from Green Bay Packaging Flotation Trial.**

* Runs 7 and 8 were performed during the second day of testing. Wax spots in feed samples from the second day were considerably lower than in the first day.

### *2.6.4 Flotation Conditions and Results*

Two runs were conducted consecutively at each flow rate: without surfactant and with surfactant. As the feed rate to the cell was reduced, the reject rate increased. This occurred because the balance was established by adjusting feed and accepts flow, without adjusting the reject rate. Table 16 lists the parameters for each run and wax spot test results for feed and accept samples.

The highest wax spot removal efficiency (81%) occurred at the lowest feed flow rate. During the high feed flow rate, the wax spot removal efficiency was about 57% when surfactant was used and 66% when surfactant was not used. During low feed flow rate conditions, which simulated more stages of flotation, the reject rate was considerably higher than at the higher flow rates . This, however, is not indicative of real world conditions where fiber rejected in primary flotation stages would be processed through secondary flotation cells and recovered.

The flotation surfactant dosage was not optimized for these trials and the addition of surfactant to the flotation feed did not appear to improve flotation performance. Foam levels during operation of the test flotation cell were high probably due to the presence of esters and fatty acids in the OCC furnish. As a consequence, any additional benefit derived from using the surfactant was not observed.

During these trials, flotation outperformed reverse cleaning in removing hydrophobic contaminants such as wax and stickies.

## 2.7 Mill Trial II: Menasha Corporation, Otsego, MI

The Menasha Corporation mill produces approximately 450 ADMT/day (500 t/day) of corrugating medium from a blend of neutral sulfite semichemical (NSSC) pulp and recycled fiber. The NSSC production is approximately 181 ADMT/day (200 t/day). The recycled furnish consists of OCC and mixed paper. Stock preparation for recycled fiber processing is comprised of two systems with a total production of about 271 ADMT/day (300 t/day): 14-foot system and 16-foot system. The designation is based on the pulper size in each system. The 16-foot system, which primarily processes OCC, includes low consistency continuous pulping, high density cleaning, coarse screening, fine screening, forward cleaning and thickening. The 14-foot system processes a combination of OCC and mixed paper. Mixed paper may consist of up to 15% of the recycled furnish and is made up of OCC, office papers, carrier stock and some ONP. The

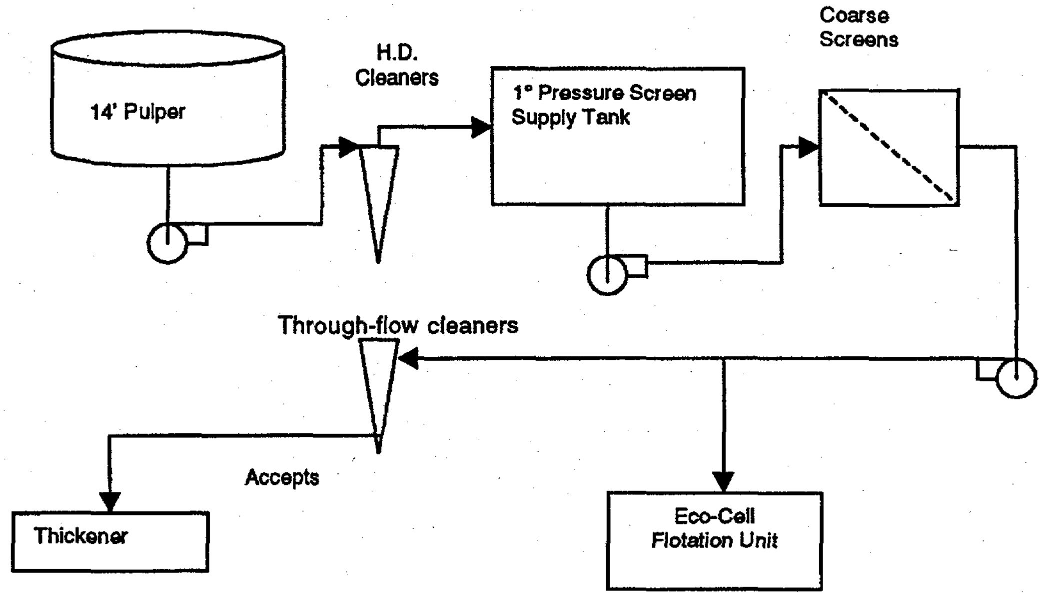



Figure 5: Simplified diagram of Menasha Corporation 14-foot system. Test flotation cell was installed at through-flow cleaner feed line.

processing sequence in the 14-foot system includes continuous pulping, high density cleaning, coarse screening, through-flow cleaning and thickening.

The test Eco-Cell was positioned in the 14-foot system (Figure 5) to process a side stream of the feed to the first stage through-flow cleaners. The intent of this approach was to compare the effectiveness of flotation in removing wax and stickies to that of the through-flow cleaners. As in the previous mill trial, the consistency at this point in stock preparation was nominal for flotation. Calsan® 50T, supplied by BASF Corporation, was the flotation surfactant. During the trials at Menasha Corporation, surfactant was made down to a 1% solution by weight of active ingredient and delivered by a chemical metering pump to the line directly preceding the feed pump. Surfactant dosage was 1 kg chemical/MT fiber (2 lb chemical/ton fiber).

The furnish processed through the 14-foot system changed twice during the flotation trial. The first six flotation tests were conducted when regular OCC was present. The next four flotation tests were run when mixed papers were being processed by the system, and the last two tests were run with OCC and roll wrap in the system.

The mill has found that one component of the mixed paper furnish, roll wrap, is especially troublesome. A problematic contaminant, wax, has been identified in this furnish. The roll wrap looks like linerboard, however, a polymer-fortified wax layer is found between two board laminates. The wax from the roll wrap plugs the paper machine felts, necessitating downtime for cleaning. The inclusion of roll wrap is regulated to minimize the buildup of wax in the system.

*2.7.1 Sampling Plan and Testing*

Three feed flow rates to the test flotation cell were evaluated: 11.3, 53, and 106 L/min (3, 14 and 28 gpm). Accepts and rejects flows were determined at each feed flow rate. Sufficient time was allowed to process at least two volumes of feed through the cell before samples were collected. Consistency was determined at each sample point, and the wax spot test was conducted on the handsheets formed from each sample.

Samples were collected from first stage (P1) through-flow cleaner feed and accepts and secondary stage (P2) through-flow cleaner accepts to determine the efficiency of the existing system. Samples for Tests 1 and 2 were taken when OCC furnish was present, Test 3 samples were collected when mixed papers were present, and Test 4 samples were taken when roll wrap was in the system. Data from samples collected from the through-flow cleaners is given in Table 17. The average wax spot removal efficiency on regular OCC with two stages (P1 and P2) of through-flow cleaning was 61.4%. When the through-flow cleaner stages were evaluated individually, the average efficiency of the first stage (P1) was 33% and the average efficiency of the second stage (P2) was 43%.

**Table 17. Wax Spot Test Results from Through-Flow Cleaners at Menasha Corporation.**

| Sample ID | Test 1 (OCC) | | Test 2 (OCC) | | Test 3 (Mixed Waste) | | Test 4 (Mill-Wrap)[b] | |
|---|---|---|---|---|---|---|---|---|
| | %Cs | ppm | %Cs | ppm | %Cs | ppm | %Cs | ppm |
| P1 X-clone Feed | 1.30 | 23266 | 1.13 | 21492 | 1.52 | 25157 | 1.15 | 1000000 |
| P1 X-clone Acc | NA | 15126 | 1.25 | 14960 | 1.82 | 38736 | 1.25 | 950000 |
| P2 X-clone Acc | 1.26 | 6351 | 1.29 | 10714 | 1.83 | 29924 | 1.27 | 900000 |
| Removal Efficiency% | 72.7 | | 50.1 | | -18.9 | | 10.0 | |

[b] Wax in handsheets was too high to be determined by image analysis. The values were estimates.

When mixed paper was processed, our results indicated that the removal efficiency was negative. This may have been caused by coating particles with high specific gravity present in the carrier stock which concentrated in the accepts stream of the through-flow cleaners. Due to the extremely high wax concentration in the roll wrap furnish, an evaluation of wax spot levels was not practical, since the entire handsheets for the feed samples were impregnated with wax.

*2.7.2 Flotation Conditions and Test Results*

During operation of the test flotation cell the foam level was high even without the addition of flotation surfactant. Two runs were carried out consecutively at each flow rate: one with surfactant and one without surfactant. Similar to the previous trial, as the feed flow rate was reduced the reject rate increased. Data from Trial II is given in Table 18. The flotation feed samples were collected from the primary (P1) through-flow cleaner feed line.

Consistent with results observed during the first mill trial, the highest wax spot removal efficiency occurred at the lowest feed flow rate to the flotation cell. In contrast to the first trial, the addition of flotation surfactant increased the wax spot removal efficiency during these trials. When comparing similar furnishes, the residual wax spot concentration in flotation accepts was significantly less than wax spot levels in second stage through-flow cleaner accepts samples. This indicates that flotation was more effective than through-flow cleaners at removing the hydrophobic contaminants found in each of the furnish types— OCC, mixed papers, and roll wrap.

**Table 18. Operating Parameters and Data from Menasha Corporation, Otsego Mill Flotation Trial.**

| Run # | Furnish | Calsan 50T ml/min | Calsan 50T lb/ton | Stock Temp °F | Shwr Water GPM | Accepts GPM | Accepts %Cs | Rejects GPM | Rejects %Cs | Mass Reject Rate % | Wax Spot Feed ppm | Wax Spot Accept ppm | Spot Removal Efficiency % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | OCC | 0 | 0 | 132 | 0.83 | 27.6 | 1.28 | 3.3 | 0.39 | 3.5% | 22380 | 9574 | 57% |
| 2 | OCC | 0 | 0 | 132 | 0.83 | 14.2 | 1.21 | 2.4 | 0.48 | 6.3% | 22380 | 6350 | 72% |
| 3 | OCC | 0 | 0 | 132 | 0.83 | 2.7 | 1.06 | 1.3 | 0.31 | 12.3% | 22380 | 5006 | 78% |
| 4 | OCC | 134 | 2 | 132 | 0.83 | 27.6 | 0.94 | 3.3 | 0.4 | 4.8% | 22380 | 3813 | 83% |
| 5 | OCC | 69 | 2 | 132 | 0.83 | 14.2 | 1.11 | 2.4 | 0.44 | 6.3% | 22380 | 3784 | 83% |
| 6 | OCC | 22 | 2 | 132 | 0.83 | 2.7 | 0.89 | 1.0 | 0.24 | 9.1% | 22380 | 4172 | 81% |
| 7 | MIXED | 0 | 0 | 130 | 2.18 | 11.53 | 1.49 | 3.96 | 0.51 | 10.5% | 25157 | 24994 | 1% |
| 8 | MIXED | 65 | 2 | 130 | 2.18 | 11.53 | 1.13 | 3.96 | 0.41 | 11.1% | 25157 | 12268 | 51% |
| 9 | MIXED | 137 | 2 | 130 | 2.18 | 25.05 | 1.17 | 5.88 | 0.43 | 7.9% | 25157 | 5261 | 79% |
| 10 | MIXED | 0 | 0 | 130 | 2.18 | 25.05 | 0.97 | 5.88 | 0.39 | 8.6% | 25157 | 3403 | 86% |
| 11 | RWP[a] | 0 | 0 | 126 | 2.18 | 13.59 | 0.99 | 2.58 | 0.38 | 6.8% | 1000000[b] | 105404 | 89% |
| 12 | RWP | 68 | 2 | 126 | 2.18 | 13.59 | 1.01 | 2.58 | 0.22 | 4.0% | 1000000 | 22609 | 98% |

[a]Roll wrap [b]Feed handsheets were entirely coated with wax.

The mass reject rates for each flotation cell feed rate interval during Trial II were considerably less than reject rates observed at similar feed flow rates during Trial I. While the reasons for this difference is not entirely clear, factors such as background foaming compounds (fatty acids, resins, etc.) present in the Trial I furnish and the feed valve and accepts valve settings during each trial may have contributed to the loss of more solids with the reject flow.

## 3.0 SUMMARY

Froth flotation is an effective process for removing wax from recycled fiber provided the wax is detached from fiber. This process offers a distinct advantage over washing in that the size of the rejects stream that must be treated is minimal. Our investigations demonstrated that flotation was more effective in removing hydrophobic contaminants than reverse or through-flow cleaners. Application of flotation to improve the cleanliness of OCC/ mixed paper furnishes may allow the reduction or elimination in use of existing dispersion or reverse cleaning systems, thus reducing the energy requirements of the stock preparation system.

Water clarification with polymers at the temperatures existing in a high temperature, washing operation may be difficult since polymer performance is severely degraded at high temperature. More importantly, in existing mill systems considering the application of aggressive washing to remove wax, considerable upgrades in DAF clarification capacity will be necessary to process the additional washer filtrate. Work on optimizing polymer selection and dosage during our trials has shown promise and needs to continue.

The use of a chemical dispersant/surfactant to depress the melting point of wax and particle size of dispersed wax may provide some relief to mills experiencing low to moderate wax contamination levels. At high levels regardless of whether the wax is dispersed, if wax is retained with the fiber to pass through the paper machine, the same problems that occur without the treatment, i.e., slippery sheet and strength losses, would be expected. If dispersed wax is washed from the fiber, an effective process water clarification program must be implemented to prevent the eventual recontamination of the system as wax accumulates.

Pilot plant studies demonstrated that the application of froth flotation in the stock preparation system significantly enhanced the removal of stickies and promoted a decrease in the area of wax spots in handsheets. Several observations made during the pilot plant trials are worth noting:

· Under the conditions of these trials, froth flotation was more effective in removing wax and stickies than conventional through-flow cleaners. The application of froth flotation can be considered as a quality improvement tool for the reduction of stickies and wax in an OCC recycled system.

· Based on the analysis of the data collected during these trials, we conclude that flotation cells used in an OCC system need not be as sophisticated as those found in a deinking system. Three stages of flotation would be adequate, whereas in the deinking plant, 4 or 5 stages are generally necessary. In addition, based on our observations, significant yield losses are not to be expected from the application of flotation in an OCC system.

· Efficient water clarification, essential to the prevention of stickies and wax accumulation in process water, is achievable when an effective polymer program in combination with DAF is applied

The two mill trials conducted during this study demonstrated that froth flotation effectively removes waxes, small particles of hot melts, coatings and other hydrophobic contaminants from OCC and mixed papers. These trials confirmed the previous findings from laboratory studies. In both mill trials, froth flotation outperformed reverse cleaners.

OCC contains rich resin content (fatty acids and esters). These resins can behave similar to flotation surfactant. The efficiency of flotation can be further improved by adding Calsan 50T or other surfactant (such as Floatsan 209), as indicated in our laboratory and pilot trials. Results from Trial II, conducted at Menasha Corporation, indicated that fiber losses by flotation are minimal.

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