PROCESS WATER RECOVERY: DISSOLVED AIR FLOTATION COMPARED TO HIGH SHEAR RATE SEPARATION

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ABSTRACT

Process water from recovered paper pulp recycling operations is often discarded from papermill operations because even low percentages of hydrophobic contaminants, such as stickies, are a problem to papermakers. This study compared the use of dissolved air flotation (DAF) and a high shear field (HSF) separator for removing a pressure-sensitive/hot-melt adhesive (PSA/HMA) from process water. The adhesive was applied to dried pulp sheets, pulped, and then screened for subsequent preparation of low-consistency process water. An experimental DAF cell (about 200 L/min) was constructed and used to compare with an experimental HSF separator (about 40 L/min). Total stickie removal efficiency of the DAF cell was between 83% and 84%. Total stickie removal efficiency of the HSF separator was improved to about 60% for all stickie sizes measured by adding dissolved air and flocculating polymers. The HSF separator performed best (about 80% total stickie removal efficiency) when calcium hydroxide was added to the feed without dissolved air or with the addition of dual polymer flocculating polymers. Although fiber loss was not optimized for the HSF separator, it was about half that measured for the DAF cell. We expect to improve both the DAF cell operation and the HSF by adding more dissolved air capacity. We also intend to investigate the effect of pacifying stickies by precipitating calcium carbonate with carbon dioxide after the addition of calcium hydroxide.

INTRODUCTION

Process water rejects from wastepaper pulp recycling operations are often discarded from papermill operations because of a low percentage of hydrophobic contaminants such as stickies, which are a problem in papermaking [1]. An effective process for removing contaminants has become increasingly important as papermills begin closing the process water loops [2,3]. One of the most effective methods for removing contaminants from process water is dissolved air flotation (DAF). However, two aspects of the DAF process [4-6] could be improved [7]. First, the DAF process typically is designed to remove all particulate matter from process water. This is an effective method for removing contaminants, but it also removes filler and fiber, which theoretically could be used to increase production. Second, flotation processes use gravity as a driving force for the separation and thus are relatively slow processes compared to screening and cleaning, for example [8].

In this study, we compared and contrasted the separation efficiencies and corresponding fiber loss of a DAF cell to those of a high shear field (HSF) separator using a commercial pressure-sensitive hot-melt adhesive (PSA/HMA). An experimental DAF ceil (about 200 L/min) was constructed and used to compare to an experimental HSF separator (about 40 L/min). Earlier experiments at the Forest Products Laboratory had shown that the addition of mineral spirits and calcium hydroxide could improve the removal of stickies by flotation.

This paper reports progress to date on four aspects of DAF compared to HSF separations:

1. operating procedure and initial effectiveness of DAF cell,
2. effectiveness of HSF separator without additives and with dissolved air and flocculating polymers,
3. addition of mineral spirits to HSF separator feed slurry, and
4. effect of calcium hydroxide shocking of feed slurry on HSF separation.

EXPERIMENTAL

Materials

A commercial pressure-sensitive hot-melt adhesive (PSA/HMA) was applied to commercial dry-lap bleached northern mixed hardwood pulp. The PSA/HMA equivalent to 3% of fiber weight was placed between two 270- by 270-mm sheets of dry-lap pulp. Stacks of 4 to 5 pairs of sheets were then placed in a laboratory hydraulic press, and the press was heated to 120°C to melt the adhesive. The sheets were pressed at 280 kPa for 3 min to allow PSA/HMA distribution and then allowed to cool to room temperature. The adhesive covered 25% to 50% of the sheet area. Sheets with PSA/HMA were matched with uncontaminated sheets as needed for experiments.

Treated and untreated dry lap sheets were pulped in a laboratory medium-consistency pulper at 8% to 9% consistency for 45 min at a starting temperature of 82°C and cooled to 70°C by the end of pulping. The fiberized pulp was screened with a laboratory-scale 0.15-mm-wide slotted screen. Rejects were discarded and accepts were collected in a sump pump and then fed directly to a 3000-L stock tank. Consistency of stock in the tank was usually between 0.2% and 0.4%.

The dual polymer system used for coagulating and flocculating the feed pulp slurries was obtained from a chemical supplier. The polymers consisted of a low molecular weight cationic polymer, which was initially fed to the stock tank, and a high molecular weight flocculant, which was fed to the tank after the dissolved air.

Equipment

The DAF design was obtained from an industry source [9]. We reduced the flow capacity to accommodate our needs by constructing a narrower DAF cell. The cell was constructed of Plexiglas to visually monitor the operation. The capacity of the cell was about 1,100 L. The cell operated by recycling a portion (20%-30%) of the accepts or cleaned process water.

The recycled portion of the accepts consisted of a small line off the accepts pump, which fed the stock first to a flow meter, next to a tee where compressed air was injected, and finally to a stationary in-line static mixer. The pressure of the compressed air was about 620 kPa and that of the recycled stock about 590 kPa. This small differential in pressure allowed air to flow into the recycle stock stream at about 3.6 L/m.

After the air and stock were mixed in the static mixer tube, the mixture was fed to the retention tank. This pressurized tank was added to the recycle line to allow more time for the air to dissolve and for large bubbles to separate and vent from the recycle flow. Large bubbles tend to scavenge smaller bubbles, which are needed for good separation of particles from the stock flow. The pressure in the retention tank was typically held at 425 to 550 kPa, while pressure in the inlet to the DAF cell was about 425 kPa. The recycle flow was then mixed with the main feed at the inlet to the DAF cell. The inlet flow without the recycle portion was usually held at about 110 L/m.

The entire flow to the DAF cell was controlled by inlet flow control valves, which were set to maintain back pressure at about 425 kPa. When the dissolved air section is operating correctly, fresh water appears milky with dissolved air. This milkiness lasts for 1 to 2 min. This process was examined visually. No attempt was made to measure bubble size or amount of dissolved air in the feed.

We were unable to get the flow in the DAF cell to follow the ideal loop. Instead, we observed short circuiting to the outlet, which reduced the residence time needed for particle removal. To remedy this problem, we added additional baffling, which directed flow upwards and reduced short circuiting. This is not thought to be the best solution, but it improved operation compared to operation without additional baffling.

In all runs, floating rejects were not removed until the run was over. The removal mechanism caused some of the floating floc to sink and re-disperse.

The FPL-constructed disk separator used for HSF separation [10] consisted of a top-fed, motor-driven disk The desired fiber furnish was fed directly to the spinning disk. The disk was fitted with a 76-mm-high cone; the diameter of the disk was 152 mm, with a 50-mm-wide lip 22.5° from horizontal. The disk was operated at about 5,000 r/min and the feed was 35 to 40 L/m.

The spinning disk set up rotational flow as the pulp slurry moved out across the disk surface. At the shoulder, most of the film turned the comer, followed the lip of the spinning disk, and was discharged at the lip edge. Adhesive particles typically could not turn the comer at the disk shoulder and were separated in the hydraulic split. Discharged material was prevented from rejoining the material discharged at the lip edge by a surrounding separator shelf.

Tests

Handsheets (1.2 g) were made according to TAPPI T-205 om-88 and dyed with Morplas blue dye so that the adhesive particles would be highly visible. The handsheets were then scanned using an HP flat-bed scanner and an Optomax Speckcheck Dirt Counter system to determine the level of visible adhesive particles.

The Doshi method [11,12] was used for dissolved stickies. A 12-L stainless steel bucket was filled with 7 L of process water and placed on a hot plate. Three monolayers of microfoam (polypropylene packaging material) cut to 25.4- by 25.4-mm squares were weighed, fastened together with a metal wire, and submerged in the process water. An adjustable stirrer was submerged to within 100 mm of the bottom of the bucket to ensure adequate mixing. The temperature was raised to 65° C and stirring was continued for 30 min. The microfoam was then removed, oven-dried, and weighed. The difference in weights showed the amount of dissolved stickies in the sample. Results were expressed with respect to amount of dry fiber in the sample in parts per million.

Efficiency calculations for stickies removal and fiber loss were calculated on mass balances based on flow rates and concentrations in respective flows. That is, the amount of stickies in each flow stream was obtained by multiplying the flow rate of the stream by the concentration of the stickies in parts per million. Stickies above and below 0.02 mm^2 were measured by the same handsheet measuring technique. However, dissolved stickies were measured by a different technique, the Doshi method. We realize that this change introduced an error in our calculations of total removal efficiency. Nevertheless, we think that our method for measuring total stickie removal efficiency was sufficiently accurate for ranking the different processes.

RESULTS AND DISCUSSION

DAF Cell Operating Time and Chemical Dosage

We first investigated the effect of operating time and chemical dosage rate on the efficiency of the DAF cell. The results of Runs 1 and 2 are given in Table I. The feed rate of the cationic polymer for Run 1 (1.66 mL/m) was approximately 10% that for Run 2. Accepts for Run 1 were recycled to the feed tank for 10 min whereas accepts for Run 2 were sewered, which resulted in approximately equal total dosage of cationic polymer for both runs. Accepts for Run 1 were sewered for 9 min without additional cationic polymer before sampling, while accepts for Run No. 2 were sampled at the end of 10 min.

The anionic polymer feed rate was held the same for both runs on the assumption that above a certain dosage the anionic polymer was not as critical in forming flocs of adhesive contaminants for removal by flotation. The results of the two experiments indicate that our assumption was correct. Even though the anionic polymer was 10 times higher for Run 1, stickie removal efficiency for this run after 10 min recycling was about the same as that for Run 2.

For both runs, total stickie removal efficiency ranged from 83% to 84%. The best efficiency values were for the >0.02-mm^2 stickies. For these stickies, removal efficiency was 92.4% and 91.14% for Runs 1 and 2, respectively. Removal efficiency was somewhat lower for the <0.2-mm^2 particles (82.67% and 76.42% for Runs 1 and 2, respectively) and even lower (58.37% and 62.96%, respectively) for dissolved stickies. This result was somewhat surprising since we had expected the DAF cell to be the most effective in removing the smallest stickie particles.

The results can perhaps be explained by the fact that there were more >0.02-mm^2 than <0.2-mm^2 stickie particles for the feed. For dissolved stickies, the type of test method was so different from that used for the other stickies that comparisons between the two methods are probably not very accurate. Removal efficiency values for the dissolved stickie method should be used to compare different runs rather than larger stickies in the same run.

We think that the amount of dissolved air needs to be increased for the DAF cell to improve stickie removal efficiency. The consistency of the feed was 0.085% and 0.104% for Runs 1 and 2, respectively; consistency for the accepts was 0.066% and 0.081%, respectively. Removal of fiber was in the range of only 22% to 23% compared to

>90% for stickie removal. Higher stickie removal rates are needed for reusing process water. Higher removal rates are not always needed for fiber and filler, especially if the process water is to be reused in the pulper.

For future experiments, we plan to modify our equipment to permit greater amounts of dissolved air in the feed slurry. One method we will examine is to add greater retention tank capacity.

HSF Separation

Addition of dissolved air and chemical. We next investigated the effect of adding dissolved air and flocculating polymers using the HSF separator (Table II). Although one pass of the HSF separator was considerably less effective in removing stickies than was the DAF cell after 10 min, we did find ways to improve the efficiency of the separator.

Using a similar feedstock, but with about twice the level of stickies, we measured total stickie removal efficiency of 33.87% (Run 3). Total efficiency was improved to 37.17% (Run 4) by adding dissolved air to the feed, and to 60.84% (Run 5) by adding dissolved air and flocculating polymers.

As in Runs 1 and 2, we observed greater removal efficiency values for the >0.02-mm^2 stickies than for the <0.02-mm^2 stickies in Runs 3 to 5. However, the difference diminished with the addition of dissolved air and diminished even more with the addition of dissolved air and flocculating polymers. This was especially true for the dissolved stickies; stickie removal efficiency increased from -54.25% for Run 3 to 14.62% for Run 4 and to 60.84% for Run 5. We think that increasing the amount of dissolved air in the feed will produce even greater increases in stickie removal efficiency.

Consistency values for accepts in Runs 3 to 5 did not decrease from that for the feed compared to consistency values for DAF cell experiments in Runs 1 and 2. Actually, consistency values tended to increase somewhat for the accepts compared to the feed in Runs 3 to 5. Feed consistency was 0.088% in Runs 3 to 5; consistency for accepts was 0.088%, 0.103%, and 0.096% in Runs 3, 4, and 5, respectively. Loss of solids ranged from 10% to 15% in Runs 3, 4, and 5, lower than that in DAF Runs 1 and 2 (22% to 23%). The lower fiber loss for the HSF experiments indicates that the HSF separator potentially could result in less fiber and filler loss compared to DAF separation. Of course, we will need to improve the separation of stickies for the HSF separator to be comparable to the DAF cell.

Addition of mineral spirits. In Runs 6 to 8, we evaluated the effect of adding mineral spirits to the feed slurry with respect to stickie removal efficiency (Table III). In Run 6, we added 1.0% mineral spirits (dry fiber basis) without any flocculating chemicals or dissolved air. Total stickie removal efficiency was 40.13%. Again we observed the best removal efficiency values for the largest stickies particles. Increasing the mineral spirits to 2.0% (Run 7) and adding flocculating polymers and dissolved air raised total stickie removal efficiency to 58.43%. Once again we observed a greater increase in efficiency for smaller stickie particles.

When the mineral spirits level was raised to 5.0% (Run 8) along with the same level of dissolved air and flocculating polymers as for Run 7, we observed a decrease in total stickie removal efficiency to 52.70%. This decrease was caused by the mineral spirits, which produced more dissolved stickies in the accepts than in the feed.

Adding mineral spirits could possibly be an effective method for increasing stickie removal efficiency for the DAF cell, but again we think that increasing the amount of dissolved air will be more advantageous.

Addition of calcium hydroxide and varying feedstock consistency. Our final experiments involved determining the effect of calcium hydroxide and feedstock consistency on stickie removal efficiency (Runs 9 and 10, Table IV). By adding calcium hydroxide to the feedstock without flocculating polymers or dissolved air, total stickie removal efficiency increased from 24.00% to 79.73%.

The greatest increase in stickie removal efficiency was for dissolved stickies. Stickie removal efficiency was 24.00% for the HSF separator without calcium hydroxide. Adding 10% calcium hydroxide (dry fiber basis) yielded dissolved stickie removal efficiency of 92.61%. Again, the improvement in stickie removal efficiency was greater for stickies < 0.02 mm^2 (7.27% to 42.30%) than for those > 0.02 mm^2 (37.58% to 62.48%).

Feed consistency values for Runs 9 and 10 were 0.738% and 0.217%, respectively. Lower consistency values are expected to improve stickie removal efficiency, but the results obtained from reducing feed consistency in Run 10 (compared to Run 9) were much greater than we expected and were probably due to shocking the stickies and producing larger and more readily removed stickies. Shocking stickies may be a way to improve the removal of stickies during simultaneous recovery of fiber and filler.

Fiber Loss

Fiber losses for the HSF separator were not minimized by experimental methods; fiber loss was about half that measured for the DAF cell. Although operating conditions were not optimized for the DAF cell and HSF separator, we expect that at optimum conditions DAF cell fiber loss will greatly increase and HSF separator cell fiber loss will be reduced. A DAF cell typically removes all the fiber; when operated at optimum conditions, an HSF separator loses only approximately 5% fiber [10].

CONCLUSIONS

- The DAF cell was not greatly affected by varying the length of operation. Total stickie removal efftciency was lower than expected (83% to 84%). We anticipate that this can be improved by adding more dissolved air capacity.
- For the HSF separator, stickie removal efficiency was improved for all size ranges of stickies measured by adding dissolved air and flocculating polymers. Total stickie removal efficiency was increased from 33.87% to 60.84% by adding dissolved air and using a dual polymer flocculating treatment.
- Mineral spirits did not improve total stickie removal efficiency because they formed dissolved stickies.
- The HSF separator performed best without dissolved air or dual polymer agglomerating polymers, but with the addition of calcium hydroxide to the feed slurry. We think that the calcium hydroxide shocked the stickies and the resulting agglomerating action (especially on dissolved stickies) improved stickie removal. Stickie removal efftciency was 79.73% for dissolved stickies with addition of calcium hydroxide, compared to 58% to 63% for the DAF cell. Total stickie removal efficiency was 79.7% for the HSF separator with the addition of calcium hydroxide.
- Fiber loss was not optimized for the HSF separator; fiber loss was about half that measured for the DAF cell.
- In future experiments, we intend to examine increasing the dissolved air capacity for both the DAF cell and HSF separator. For the HSF separator, future work will examine pacification of stickie contaminants by precipitating calcium carbonate after addition of calcium hydroxide.

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Table I. Stickie removal with dissolved air flotation: operating time and chemical dosage

Process variable	Run 1[a]	Run 2[b]
Feed		
Rate (L/m)	136	136
Consistency (%)	0.085	0.104
Stickie concentration (ppm)		
>0.02 mm^2	1,104	1,844
<0.02 mm^2	121	132
Dissolved	403	628
Chemical feed (mL/m)		
Cationic polymer	1.66	14.8
Anionic polymer	5.4	5.4
Accepts		
Rate (L/m)	136	136
Consistency (%)	0.066	0.080
Stickie concentration (ppm)		
>0.02 mm^2	108	210
<0.02 mm^2	27	40
Dissolved	216	299
Stickie removal efficiency (%)		
>0.02 mm^2	92.40	91.14
<0.02 mm^2	82.67	76.42
Dissolved	58.37	62.96
Total	83.25	83.60
Fiber loss (%)	22.3	22.1

[a] Accepts recycled to feed for 10 min, accepts sewered for 9 min, and accepts sampled at end.
[b] Accepts sewered for 10 min and sampled at end.

Table II. High shear field separation: dissolved air and chemical addition

Process variable	Run 3	Run 4	Run 5
Feed			
Rate (L/m)	38.0	38.4	39.2
Consistency (%)	0.088	0.088	0.088
Stickie concentration (ppm)			
>0.02 mm^2	3,998	3,998	3,998
<0.02 mm^2	333	333	333
Dissolved	341	341	341
Chemical feed (mL/m)			
Cationic polymer	0	0	0.8
Anionic polymer	0	0	5.4
Air feed	0	yes	yes
Accepts			
Rate (L/m)	33.0	32.8	34.2
Consistency (%)	0.088	0.103	0.096
Stickie concentration (ppm)			
>0.02 mm^2	2,540	2,349	1,070
<0.02 mm^2	389	285	169
Dissolved	601	290	89
Stickie removal efficiency (%)			
>0.02 mm^2	44.40	41.02	60.36
<0.02 mm^2	-2.64	14.08	51.84
Dissolved	-54.25	14.62	75.23
Total	33.87	37.17	60.84
Fiber loss (%)	12.9	14.6	13.2

Table III. High shear field separation: mineral spirits

process variable	Run 3	Run 6	Run 7	Run 8
Feed				
Rate (L/m)	38.0	34.8	35.8	35.7
Consistency (%)	0.088	0.075	0.075	0.075
Stickie concentration (ppm)				
>0.02 mm^2	3,998	2,916	2,916	2,916
<0.02 mm^2	333	154	154	154
Dissolved	341	418	418	418
Chemical feed (mL/m)				
Mineral spirits	0	1.0	2.0	5.0
Cationic polymer	0	0	0.8	0.8
Anionic polymer	0	0	5.4	5.4
Air feed	0	0	0	0
Accepts				
Rate (L/m)	33.0	30.6	30.0	30.3
Consistency (%)	0.088	0.075	0.075	0.082
Stickie concentration (ppm)				
>0.02 mm^2	2,540	1,479	1,029	1,062
<0.02 mm^2	389	122	81	63
Dissolved	601	769	603	664
Stickie removal efficiency (%)				
>0.02 mm^2	44.40	55.31	70.13	66.41
<0.02 mm^2	-2.64	30.20	55.48	62.27
Dissolved	-54.25	-62.10	-22.09	-46.49
Total	33.87	40.13	58.43	52.70
Fiber loss (%)	12.9	12.0	16.2	15.2

Table IV. High shear field separation: calcium hydroxide and feedstock consistency

Process variable	Run 3	Run 9	Run 10
Feed			
Rate (L/m)	38.0	45.0	47.3
Consistency (%)	0.088	0.783	0.217
Stickie concentration (ppm)			
>0.02 mm^2	3,998	385	286
<0.02 mm^2	333	86	76
Dissolved	341	831	605
Chemical feed (mL/m)			
Calcium hydroxide (%)	0	10.0	10.0
Air feed	No	Yes	Yes
Accepts			
Rate (L/m)	33.0	39.3	39.1
Consistency (%)	0.088	0.852	0.226
Stickie concentration (ppm)			
>0.02 mm^2	2,540	238	125
<0.02 mm^2	389	79	51
Dissolved	601	663	52
Stickie removal efficiency (%)			
>0.02 mm^2	44.40	37.58	62.42
<0.02 mm^2	-2.64	7.25	42.30
Dissolved	-54.25	19.44	92.61
Total	33.87	24.00	79.73
Fiber loss (%)	12.9	12.5	17.4