A novel unit operation to remove hydrophobic contaminants

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ABSTRACT: For mills making paper with recovered fiber, removal of hydrophobic contaminants is essential for trouble-free operation of paper machines. Significant cost savings on paper machine operation can be achieved by reducing deposits, which results in better quality, reduced downtime, increased fiber yield, and reduced energy consumption.

Bubble nucleation separation (BNS) is a relatively new process for removing hydrophobic particles. When vacuum is applied to a slurry, dissolved gas bubbles nucleate on hydrophobic particles and drag them to the surface for easy removal. We constructed a 16-L batch unit to evaluate the effect of operating parameters on removal of hydrophobic particles, using statistical design of experiments. These results were used to guide our design of a 16-L continuous unit. We tested this unit on laboratory and mill samples. The removal of 60%–80% of hydrophobic particles was achieved with a low reject rate of < 2%.

Following on this success, we built a 200-L pilot unit and tested it in our pilot plant. With promising results there, we installed the pilot unit at a commercial paper recycling mill. Over the course of several mill trials, we showed that it was possible to remove a considerable amount of suspended solids from paper machine white water with less than 2% rejects. Unfortunately, due to the unit only treating 50 L/min and the mill flow being 12000 L/min, we were not able treat a sufficient portion of the white water to know whether a large-scale implementation of BNS would improve paper machine runnability.

Application: If implemented in a paper machine white water loop, this technology could provide a way to control stickies and dirt, without adding any chemicals.

According to the American Forest and Paper Association (AF&PA), 77% of paper mills in the United States use some form of recovered fiber [1]. In spite of advances in screens and cleaners, we do not have a satisfactory process for the removal of hydrophobic contaminants, especially small particles that pass through slotted screens. Hydrophobic particles in paper recycling, also called stickies, arise from pressure sensitive adhesives, natural pitch, book bindings, postage stamps, envelopes, inks, etc. Often, stickies are comprised of mixtures of materials, including wax, polystyrene butadiene, polyvinyl acetate, silicones, wood extractives, and any other polymers in the recovered paper.

In paper recycling, the removal of stickies is of paramount importance for trouble-free operation of paper machines. Stickies accumulate in the paper machine white water and eventually agglomerate and deposit on wires and felts. These deposits have drastic effects on the paper machine productivity. Cost savings on paper machines due to reducing stickies deposits can be substantial. Tom Friberg estimated in 1996 that the cost impact of stickies to all paper recycling mills combined in the United States is about $700 million per year [2].

Flotation processes are widely used in the industry to selectively or completely remove suspended solids from slurries. Two widely used flotation processes are froth flotation (FF) and dissolved air flotation (DAF). In this project, the bubble nucleation separation (BNS) process is introduced. Important features of these three flotation processes are shown in **Table I**.

Froth flotation is widely used in ore processing and pulp deinking for selective separation by dispersing air through the slurry. Surfactants are added to enhance particle attachment to bubbles. Relatively large bubbles, 0.1 to 1.0 mm^2, collect hydrophobic particles and float to the surface of the slurry, where the foam is removed. Controlled turbulence is maintained to minimize fiber loss.

Dissolved air flotation completely removes suspended solids from slurries for water clarification. In DAF, air is dissolved at high pressure (about 65 psi). Coagulation and flocculation chemicals are added to flocculate suspended solids. When the pressure is released, dissolved air comes out of solution as fine bubbles that are entrapped in the flocs, which causes everything to float to the surface where it is skimmed off. Relatively quiescent condition is maintained to avoid breaking up flocs.

In most industrial water streams, there are sufficient dissolved gases, including air and carbon dioxide. When vacuum is applied to a tank containing a slurry, dissolved gases form bubbles that nucleate on hydrophobic particles and float to the surface. In this respect, BNS is somewhat similar to DAF except that no agglomeration chemicals are added.

There are other types of flotation processes like electroflotation, centrifugal flotation, and cavitation air flotation as reviewed by Rubio et al. [3]. However, at present these processes are not commercially used.

	Froth Flotation	Dissolved Air Flotation	Bubble Nucleation
Separation	Selective separation	Complete removal	Selective separation
Mechanism	Hydrophobic particles attach to bubbles	Bubble entrapment in flocs	Bubble nucleation on hydrophobic particles
Chemistry	Surfactant	Flocculation agents	None
Bubble size	0.1 to 1.0 mm^2	0.01 to 0.1 mm^2	0.01 to 0.1 mm^2
Fluid mechanics	Turbulent	Somewhat quiescent	Quiescent
Air source	Air sparged into the system	Air dissolved at elevated pressure	Residual dissolved air and other gases

I. Comparison of flotation processes.

In the BNS process, vacuum is applied to a vessel containing a slurry to be cleaned. Our hypothesis is that as the pressure inside the vessel decreases, dissolved gases form bubbles on hydrophobic particles that serve as nucleation sites. Because hydrophobic particles have lower energy surfaces than fiber, they are more likely to nucleate bubble formation. The bubble-particle aggregates rise to the surface, where they meet other aggregates and coalesce. The fiber-containing slurry is continuously removed from the bottom of the vessel while agglomerated particles may be removed from the upper surface. The removal efficiency depends on many parameters, including the magnitude of applied vacuum, residence time, temperature, solids concentration (consistency), pH, conductivity, particle size, bicarbonate ion concentration, and carbon dioxide content.

The principle behind BNS was used in a patented process for the measurement of stickies and pitch in paper mill process streams [4]. The unit was successfully tested in laboratory and mill trials [5-7]. It is now commercially available for monitoring stickies and pitch. Even though our focus is on the paper industry, the new unit operation could potentially be applied to other manufacturing processes for the removal of hydrophobic contaminants.

The BNS process removes dissolved gases from the process water, which is not accomplished in DAF or FF. In fact, the amount of air in the accepts from DAF or FF increases. Excess air or foaming, which can be detrimental to papermaking, could be virtually eliminated by using the BNS process. Unit operations that are used to remove air from the pulp slurry (e.g., deculators) might no longer be needed after the implementation of the BNS process. The removal of carbon dioxide could potentially reduce scaling due to calcium at constant pH.

The first objective of the project is to understand the mechanism and the role of various parameters on the vacuum flotation particle removal efficiency. The second objective is to design a continuous process, take it through several stages of scale-up, and try it in a commercial paper mill.

MATERIALS AND METHODS

Equipment design

For this work, we conducted three types of experiments: (1) batch with 16-L volume, (2) continuous with 16-L volume, and (3) continuous with 200-L volume. **Figure 1** is a photograph of the Plexiglas tube with an aluminum lid that was used to conduct the bench-scale experiments. The addition of pumps and tubes to this vessel allowed us to conduct bench-scale continuous experiments as well. The flowsheet of both continuous processes is also shown in Fig. 1. While the tanks were approximately ten times larger, the components were the same.

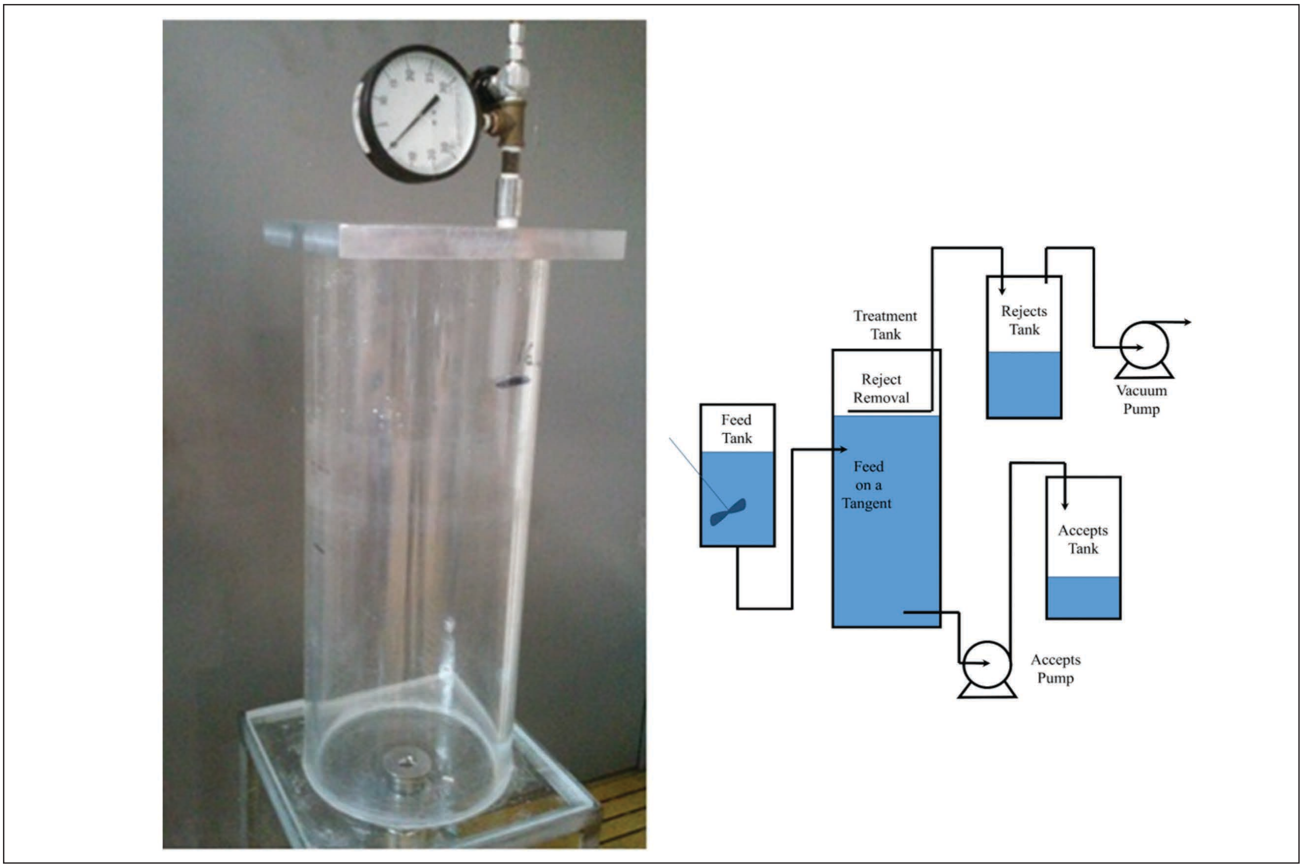
Synthetic process water generation

To evaluate the role and importance of parameters affecting the removal efficiency of hydrophobic contaminants, we needed reproducible synthetic process water (SPW). While using mill water was desirable, it was determined that it was too variable to give reproducible results.

We used pressure sensitive adhesive (PSA) labels, which were a tackified 2-ethylhexylacrylate-based, emulsion-polymerized label coated on a plain white facestock similar to Avery Matte White Paper Labels (Avery Dennison; Glendale, CA, USA). This material is likely representative of many labels currently in North American mixed office paper. The procedure for producing reproducible SPW from PSA label stock is outlined below.

- Attach PSA labels to completely cover one side of the envelope paper and discard the silicone-coated liner. The PSA in this case is a typical acrylic-based PSA.
- Pulp the mixture at high temperature (greater than 45°C) for more than 10 min in a Waring blender. Water and labels attached to paper were added to provide a slurry of 2% consistency.
- Dilute the pulp to 0.1% consistency to represent SPW.

A control sample was generated by a similar procedure, except the label stock was not added and additional envelope paper was added to produce the same consistency.



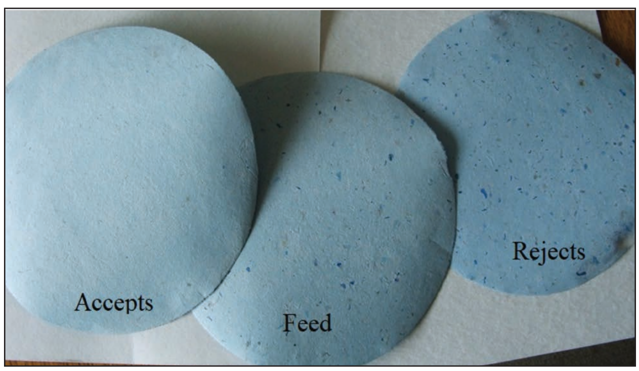
1. Batch bench-scale unit (left); the black mark indicates 16 L volume. Schematic of the two continuous processes (right).

Mill samples

In some experiments, we used SPW and in others we used water/stock from two cooperating recycle mills. Mill samples were transported in 5-gal buckets. Transportation to our laboratory resulted in these samples being used between 3 h and 6 h after collection. We attempted to remove stickies from paper machine white water, tertiary fine screen feed, and tertiary fine screen rejects.

Batch experiments

The sample was added to the 16-L batch vessel. Vacuum was applied for 15 min. At the end of the treatment time, the vacuum was released, and the cover removed. Reject samples (stickies) were collected from the liquid surface by aspiration by applying a vacuum to a side-arm flask fitted with a hose. The rest of the sample was then drained and collected in a bucket. This was the accept sample. A small number of stickies, < 1%, attached to the walls of the vessel. They were washed into the chamber and added to the reject fraction. Temperature, pH, and conductivity were measured in the feed and accepts samples. Handsheets were made directly from the feed and accepts samples. Because the reject sample contained only limited amounts of fiber, we added clean pulp before making handsheets. Stickies were counted and their area measured using the blue dye method [8]. Examples of dyed handsheets are shown in **Fig. 2.**



2. Dyed handsheets showing accepts are relatively free of adhesive particles.

Design of experiments

A statistical half fraction design, with replicates, was used to determine the effect of the variables. **Table II** shows the experiments. With replicates, this design represents 27 experiments.

Carbon dioxide (CO_2) was added by carbonating a portion of the water with a home carbonation unit (SodaStream; Kefar Sava, Israel). The amount of carbonation is expressed as the amount of carbonated water added, where 100% is the addition of 1 L of fully carbonated water to the vessel.

Pattern	BNS Variable						
	Temperature, °C	Time, min	Vacuum, in. Hg	Bicarbonate, g/16 L	Filler, %	CO_2, %	Sparge Air
++----+	40	15	-20	0	0	0	No
+-+--+-	40	5	-10	0	0	100	Yes
+--++--	40	5	-20	20	10	0	Yes
-++-+--	20	15	-10	0	10	0	Yes
-+-+-+-	20	15	-20	20	0	100	Yes
--++--+	20	5	-10	20	0	0	No
----+++	20	5	-20	0	10	100	No
+++++++	40	15	-10	20	10	100	No
000000-	30	10	-15	10	5	50	Yes
000000+	30	10	-15	10	5	50	No

BNS = bubble nucleation separation; CO_2 = carbon dioxide.

II. Experimental design.

Continuous experiments

As described previously, the 16-L batch vessel was adapted by the addition of two pumps and a reject skimmer to allow for continuous operation. The vacuum was applied to the tank via the slotted reject skimmer, which withdrew both air and reject material. The reduced pressure in the vessel allowed the feed to be drawn into the tank. The accepts were removed by a gear pump, which is a positive displacement pump and is sufficient to withdraw liquid against the reduced pressure in the tank. Typical flow rates were 2–6 L/min., which provided 3–8 min of retention time. A typical bench-scale experiment involved generation of 150 L of stock for the feed in a stirred tank. Handsheets and image analysis were used to determine removal efficiency.

A pilot-scale unit was constructed that was based on our experience with the bench-scale unit. The flowsheet was the same as shown in Fig. 1. The primary vessel was a steel tank with 2.5-cm-thick aluminum plate as a top. A 1-hp, piston-type pump was used to generate vacuum. A 1.5-hp vane pump was used to withdraw accepts. As with the bench-scale unit, we used the reduced pressure to draw stock into the primary vessel, which entered at a tangent to the tank wall. Magnetic flow meters allowed measurement of the feed and accept rates. The feed, primary, and reject collection tanks were fitted with level sensors. With the addition of control valves, we were able to provide automated operation using National Instruments (Austin, TX, USA) CompactDAQ modules and LabView software. Typical feed rates were between 30 and 50 L/min. The rejects were collected in a closed tank fitted with automated valves. The control system watched the level in the reject tank, and periodically emptied it.

When the unit was operated in the pilot plant, 1000 L of feed stock was generated by diluting mill samples. This stock was introduced to the feed tank by a third pump. In the mill, the feed to the unit was only successfully operated on paper machine white water. The water recirculation line returning to the machine chest was under sufficient pressure to fill the feed tank directly.

RESULTS AND DISCUSSION

Batch treatment of SPW

Using the design matrix shown in Table II, we conducted 27 experiments. Three sets of handsheets were made: feed (F), accepts (A), and rejects (R). The feed handsheets were made from a portion of the diluted stock. The accepts were made from the stock remaining in the batch column (**Fig. 3**) after BNS treatment. The rejects were collected by aspiration of all the material at the water surface after the vacuum was released (Fig. 3).

The stickies removal efficiency (SRE) was calculated using the following formula, which is based on area of adhesive in the accepts (A) and feed (F) as determined by image analysis:

$$SRE = 100*(1 - A/F)$$

A Yates algorithm [9] was used to determine the effects for a full model, including cross and quadratic terms. At the 95% confidence level, $p < 0.05$, the significant variables affecting stickies removal efficiency (SRE) were CO_2 and vacuum level, as shown in **Table III**. A response surface for a reduced model including only CO_2 and vacuum levels is shown in **Fig. 4**.

3. Surface of the liquid after the vacuum has been released (left). Tube at the end of the experiment showing the settled fiber (right).

Effect	Estimate	Standard Error	t	*P*	
Intercept	1.1966	0.1489	8.0354	0.0000	<-- significant
Temperature, °C	0.0028	0.0030	0.9390	0.3663	
Time, min	0.0027	0.0061	0.4456	0.6638	
Vacuum, in. Hg	0.0309	0.0058	5.3318	0.0002	<-- significant
Bicarbonate, g	0.0040	0.0030	1.3302	0.2082	
Filler, %	0.0090	0.0061	1.4779	0.1652	
CO_2 , %	-0.0046	0.0006	-7.4963	0.0000	<-- significant
$CO_2{}^2$, %	-0.0001	0.0000	-5.6253	0.0001	<-- significant
Sparge air, yes/no	0.0178	0.0254	0.7028	0.4956	

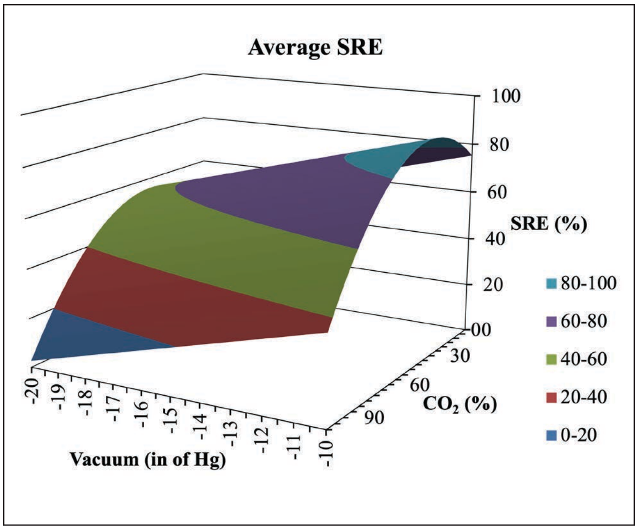
III. Results from the analysis of the statistically designed experiments.

The response surface shows that greater than 80% SRE was obtained when the CO_2 level was near 50% and the vacuum level was -11 in. Hg. High vacuum and high CO_2 content resulted in the lowest SRE. We interpret this result to mean that the turbulence generated by rapid bubble formation and larger bubble size results in the detachment from stickies, and thus the stickies are not brought to the surface.

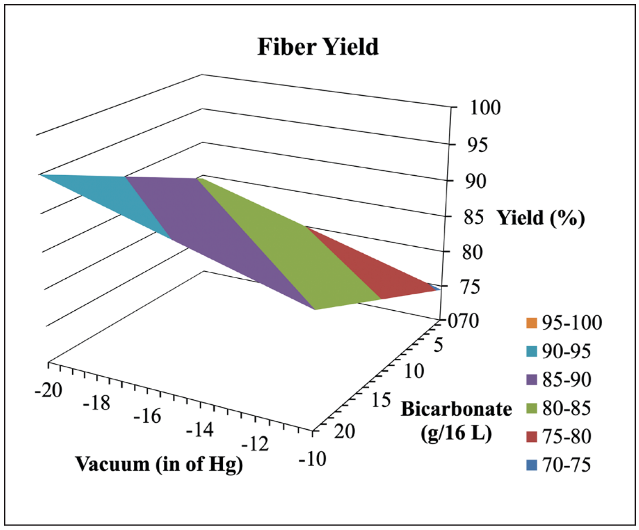
A test of this conclusion was conducted by repeating a subset of the experiments only varying the vacuum. Results are shown in **Table IV**. Inspecting the average total stickies area at -10, -15, and -20 in. Hg vacuum showed that the best SRE (83%) was obtained during the experiments with -15 in. Hg vacuum.

Yield results

Label stock (15 g) containing 10% adhesive was used in each experiment. Rejects removed from the top of the batch column were weighed to determine the percentage



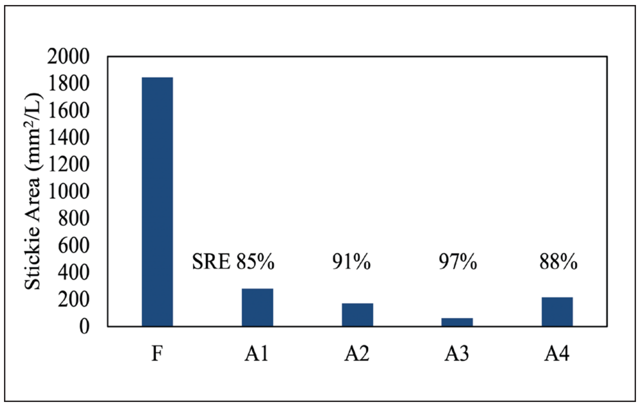
4. *Response surface for carbon dioxide (CO_2) and vacuum levels (SRE = stickies removal efficiency).*



5. *Response surface for fiber yield vs. vacuum and bicarbonate level.*

# Trials	Vacuum, in. Hg	Final pH	Rejects, g	Stickies Area, mm²	SRE, %
9	-10	8	1.36	2341	68
5	-15	7.6	1.7	1209	83
8	-20	8.3	0.92	4181	43

IV. *Average values of stickies removal efficiency (SRE) for three vacuum levels.*



6. *SRE for diluted tertiary fine screen feed sample (F = feed, A1 = 5, A2 = 10, A3 = 15 min under 18 in. Hg vacuum, A4 = 30 min with no vacuum).*

based on 15 g of stock input, i.e., yield = 100*(1-R/15), where R is the reject weight. An analysis of the yield data showed that the only significant effects for yield were vacuum and bicarbonate levels. The resulting response surface is shown in **Fig. 5**. The signs of the coefficients of the fit equation show a positive correlation with both terms, i.e., higher vacuum and higher bicarbonate leads to higher yields. This correlation with vacuum is to be expected in that higher vacuum results in more bubble movement to the surface, where bubbles release the fibers. Interestingly, CO_2 concentration did not show a significant correlation. The addition of bicarbonate will change the ionic strength of the slurry and may lead to lower bubble nucleation on the fiber surface.

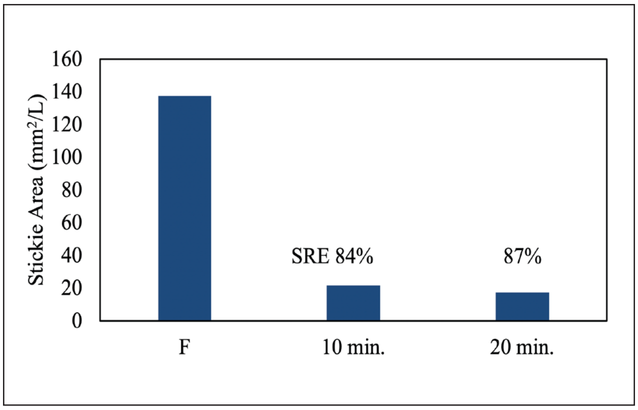
Batch experiments with mill samples

In an attempt to recover fiber from a fine screening system, we tested diluted tertiary fine screen feed (TFF) with BNS. The sample was diluted to a consistency of 0.5%. The initial sample had a large number of stickies (**Fig. 6**). As treatment time increased (Sample A1 to A3), the SRE increased from 85% to 97%. Sample A4 shows the removal achieved by just letting the stock sit with no vacuum for 30 min with no agitation, which is followed by removing the material at the top surface. This indicates that most of the stickies are less dense than water. By applying a vacuum, we are able speed the separation process, but just letting the tank set for a longer time is nearly as efficient.

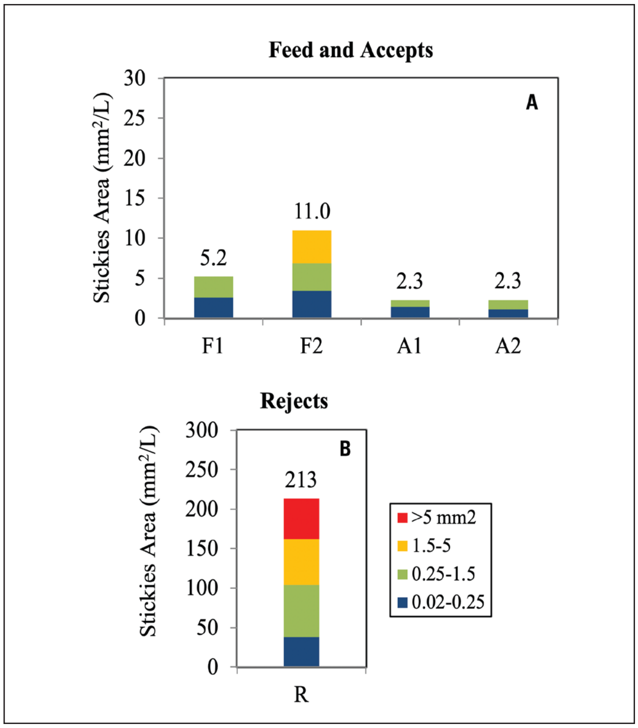
Continuous bench-scale experiments with mill samples

After the successful run of the continuous unit with SPW at 0.5% consistency, a continuous run was made with TFF. The TFF sample was diluted to 0.5% consistency using hot tap water (pH 8.09, temperature 43.1°C, and conductivity 523 mS/cm). Vacuum was maintained between 10 and 20 in. Hg and accepts flow rate was about 1.9 L/min. Accepts samples were taken at 10 min and 20 min, and handsheets were made for the determination of stickies.

The three feed handsheets had an average of 137.5 mm² stickie area. While running diluted TFF stock through the BNS process, accept samples were collected at 10 min and 20 min. The accept handsheets averaged 21.8 mm² and



7. SRE during continuous operation of bubble nucleation separation (BNS) with tertiary fine screen feed (diluted to 0.5% consistency).



8. A) Stickies distribution of feeds (F1, F2) and accepts (A1, A2), and B) rejects during "white water" trial. (Note the difference in y-axis scales between A and B.)

17.3 mm^2, respectively (**Fig. 7**). The SRE was therefore 84% and 87%, respectively, which is well above the minimum target of 60% for the process water samples.

Automated continuous pilot-scale experiments

After successfully testing the pilot unit with water, two SPW, three tertiary fine screen rejects (TFR), and two simulated white water (WW) runs were conducted. The results are summarized in **Table V**.

The previous trials showed that it was possible to remove a large amount of highly-agglomerated stickies with only 1.8%–2.7% rejects by volume. SRE of samples varied considerably from 64% to 81%, depending on the source.

The stickies size distributions for two samples of the run with 0.1% WW are shown in **Fig. 8**. The stickies distribution of rejects (R) is based on the average of five reject samples. Comparison of the size distribution between the feed and rejects clearly shows a significant amount of agglomeration of stickies.

One observation of note was that the TFR sample had an appreciable amount of unpulped paper pieces, which are also known as "tags". At higher consistency, these tags collected in the slot of the reject skimmer and ultimately caused it to plug. If one wanted to implement this technology to recover fiber from a commercial screening system, one would likely need a small reject refiner to deflake this stock before BNS.

Mill pilot plant runs

The pilot unit with a tank, pumps, and controls was transferred to a Wisconsin tissue mill (**Fig. 9**). The pilot unit ran

Consistency and Source	Vacuum, in. Hg	Feed Flow, L/min	Temperature, °C	Time, min	SRE	Comments
0.08% SPW	14	40	53	25	64	Temperature too high
0.08% SPW	14	35–40	32	25	73	
0.1% TFR	20–16	44	36	18	81	
0.1% WW	20	48	40	73	70	
0.1% WW	15	40	40	32	75	
0.5% TFR	20	42	40	14	nd*	Plugged reject
0.25% TFR	20	42	40	9	nd*	

*nd = not determined.
SPW = synthetic process water; TFR = tertiary fine screen rejects; WW = white water; SRE = stickies removal efficiency.

V. Summary of automated pilot plant.

9. Pilot unit being unloaded.

10. Samples from mill trial.

for four to seven hours each day without any downtime. The paper machine was producing heavy basis weight brown towel grades that contained wet strength resin during those trial days. A sample of white water was filtered but showed mostly black solids and no stickies. The mill typically does not have stickies-related issues on heavy basis weight grades.

The trial was conducted at 12–14 in. Hg vacuum and 1 L/min of rejects. Suspended solids results shown in **Table VI** indicated a removal efficiency of 46%–54%. We decided to add dilution water. Results of the 12:30 pm run were similar to earlier morning results, but later results were inaccurate due to inadequate mixing with dilution water added to the feed tank. A mixer is needed, especially when adding dilution water, since solids tend to float to the top of the feed tank where the sample is taken. Higher suspended solids in the feed after dilution were probably caused by floating solids in the feed tank. **Figure 10** shows feed, accepts, and rejects samples from the mill trial.

Microscopic analysis was also performed on the accepts and rejects. The accept sample contained fibers, while the reject sample contained few fibers and a lot of debris and ink (**Fig. 11**). The rest of the material consisted mostly of colloidal particles.

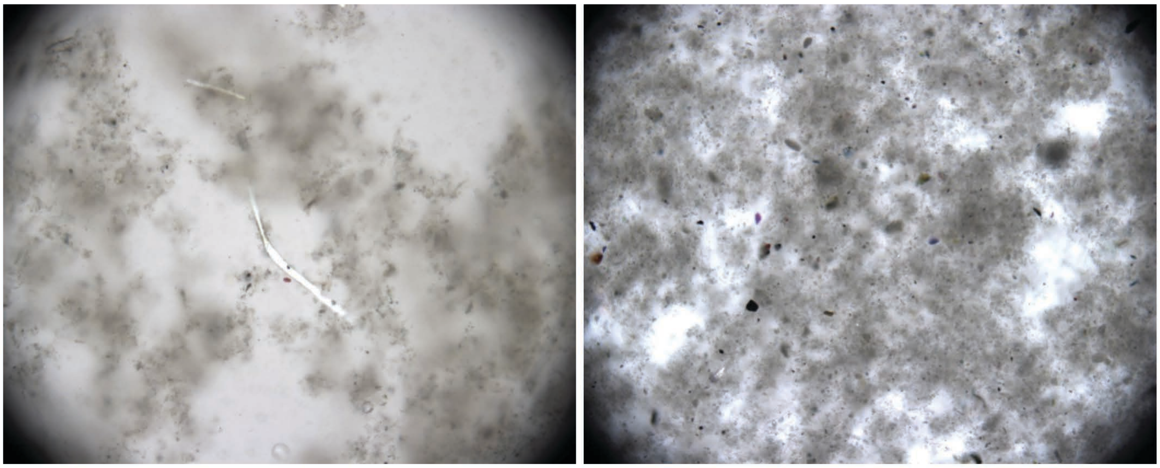
Discussions with the mill personnel indicated strong interest in the stickies removal and fiber recovery capability of the unit. If it works as demonstrated, then there are many opportunities open such as:

- Use of inexpensive lower grade furnish.
- Use of higher reject ratios in screens followed by fiber recovery.
- Less stickies downtime on paper machines.
- Lower chemical costs for defoamers, flocculants, etc.

Time	8:30 am	10:30 am	12:30 pm	2:20 pm	2:50 pm	3:30 pm
Vacuum, in. Hg	12	12	14	12	12	14
Feed flow, L/min	25	25	46	45	45	46
Accepts flow, L/min	24	24	45	44	44	45
Rejects flow, L/min	1	1	1	1	1	1
Dilution water flow, L/min	0	0	20	20	20	20
Sample Test Results	**Suspended Solids, mg/L**					
Feed	433	475	437*	830*	907*	1377*
Accepts	207	265	210	105	131	155
Rejects	2,740	4,067	3,734	2,668	2,274	-
Efficiency	54%	46%				

* Higher suspended solids in feed after dilution with clean water caused by inadequate mixing in feed tank and solids rising to top of tank where sample was taken in spite of stirring the tank by hand.

VI. Suspended solids removal during trial on brown towel white water.



11. Microscopic images of samples from the mill trial (left = accept sample; right = reject sample). Cross polarizers used to highlight the fibers.

CONCLUSIONS

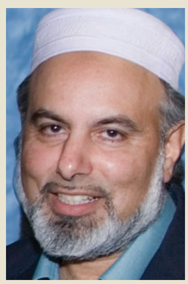
By conducting a large number of experiments, we have demonstrated the bubble nucleation separation (BNS) process. Under the correct conditions, dissolved gas bubbles form on hydrophobic particles that serve as nucleation sites. The bubble-particle aggregates rise to the surface, where they meet other aggregates on the surface and coalesce. They can then be collected and removed from the slurry. We have demonstrated stickies removal efficiencies of greater than 80% using BNS.

Using design of experiment methods, we have determined that vacuum level and gas concentration are the two major effects. In the range of conditions used here, we found an optimum. The data show that too little or too much bubble formation reduces the efficiency. Using moderate vacuum, 12–14 in. Hg, we were able to dramatically remove contaminants from paper machine white water in a mill setting.

We attempted to do fiber recoveries on pulps from a commercial tertiary fine screen. For consistencies greater

ABOUT THE AUTHORS

Houtman



Aziz

De Jong

Doshi

Microstickies continue to plague the paper industry. Robert de Jong made the original observation while working in a mill. He found that pulp samples seemed to collect stickies on the surface after storage. We then found that stickies could be collected more rapidly by applying vacuum. Collectively, we have a long history with stickies, and this is the only way we know to selectively remove microstickies from white water.

Studying colloidal stability was the most difficult aspect of this research. Many factors can influence whether microparticles agglomerate. We used a broad statistical design to narrow our focus to the few variables that were significant.

This project spans the whole cycle of innovation. We started with an observation in the lab, took it through several stages of scale-up, and then did a side-stream trial at a commercial facility.

If implemented in a paper machine white water loop, this technology could provide a way for mills to control stickies and dirt, without adding any chemicals. We are working with various cooperators to commercialize the technology.

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than 0.1%, more fibers were rejected, so high levels of dilution would be required. We also observed that fiber "tags" clogged the reject system. We have concluded that a rejects refiner might be needed to implement BNS in this situation. Given the technical and economic challenges of treating tertiary fine screen rejects, we have chosen to focus on paper machine white water as we go forward, looking for opportunities to demonstrate BNS on a commercial scale. **TJ**

ACKNOWLEGDEMENTS

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