Washing the fibers from foams for higher yields in flotation deinking

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ABSTRACT

The purpose of this work was to evaluate the possibility of washing pulp fibers from a flotation froth. A steady-state froth column was created in which the formation of foam balanced the collapse of foam. Dilute pulp and wash water were sprayed on the top of the froth, and measurements were made of the concentration of fibers that broke through into the liquid area below the froth. The results showed that it is possible to increase pulp yield in deinking flotation by washing the froth. A minimum wash water flux is required to keep fibers out of the froth. A mechanism is proposed to explain how fibers are washed from relatively dry froths.

Application:

This work shows the conditions under which fibers and fines can be washed from foams. This information can be used to design flotation deinking cells which will give higher yields while giving good ink removal.

FLOTATION DEINKING IS USED TO remove dispersed ink from wood pulp suspensions. Collectors such as calcium soaps of fatty acids agglomerate the ink into hydrophobic flocs that adhere to air bubbles and are transported to the foam phase (*1*). Unfortunately, as much as 10% of the added fibers and fines are entrained in the foam as well and are wasted. In previous work, we showed that the primary mechanism by which fibers and fines end up in the foam is hydraulic entrainment (*2*). By this mechanism, some fibers and fines simply go with the water into the froth.

As has been shown in the mineral processing industry, entrainment can be minimized by using column flotation technology (*3*). In this process, air is sparged into the bottom of a column partially filled with surfactant solution to generate a foam that is removed from the top of the column. A wash water stream sprayed on top of the foam performs two functions. First, it replaces liquid that drains naturally from the bubble films. Replacing the liquid makes the froth more stable, which in turn makes possible deep froth layers. Second, the action of wash water flowing down removes hydrophilic gangue from the froth, taking it to the bottom of the column where it can be ejected (*4*).

The net flux of water in the froth zone of the column is called "bias," and it is considered positive when the net flux of water is downwards. To ensure proper separation and cleaning, the bias must be positive but not too large. A high bias results in decreased efficiency as a result of channeling and axial mixing. To give steady-state operation, the flows of the input streams are balanced with the flows of the output streams. The input streams are the feed stream, which enters halfway up the column, and the wash water stream, which is sprayed on top of the foam. The output streams are the accept stream, which is removed from the bottom, and the froth, which is removed from the top.

One can imagine two types of mechanisms by which wash water lowers the entrainment of hydrophilic material in a froth. In one, the entrained material not adhering to the bubbles is simply washed out with the downward flux of water. In the other, the downward flux of wash water prevents that material not adhering to air bubbles from entering the foam phase.

In the case of paper recycling, the entrained material spans a broad range of sizes and shapes. Clay and other valuable filler particles are of the order of 10 µm in size, whereas wood pulp fibers are 1 mm to 5 mm long and are 20 µm wide. It was not obvious that it is possible to wash such large wood pulp fibers out of a froth made of bubbles 1 mm to 10 mm in diameter.

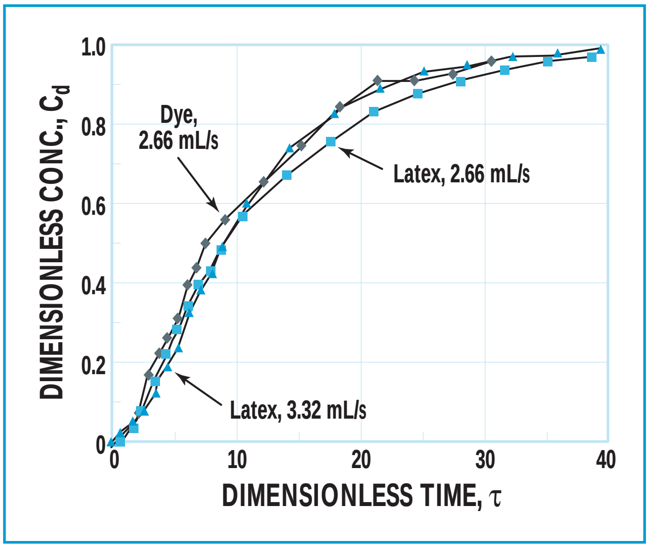
The objective of this work was to identify the conditions under which wood pulp fibers can be washed from a foam. The objective was met by two types of experiments. In the first, steady-state columns of foam were formed, and a small volume of pulp was placed on top of the foam together with a steady flow of wash water. The breakthrough of fibers was measured by monitoring the concentration of fibers in the water layer below the foam phase. It was shown that wood fibers can indeed be washed through a foam.

In the second series of experiments, small scale column flotation experiments were conducted to determine the wash water flow requirements to prevent fibers from becoming entrained in foams.

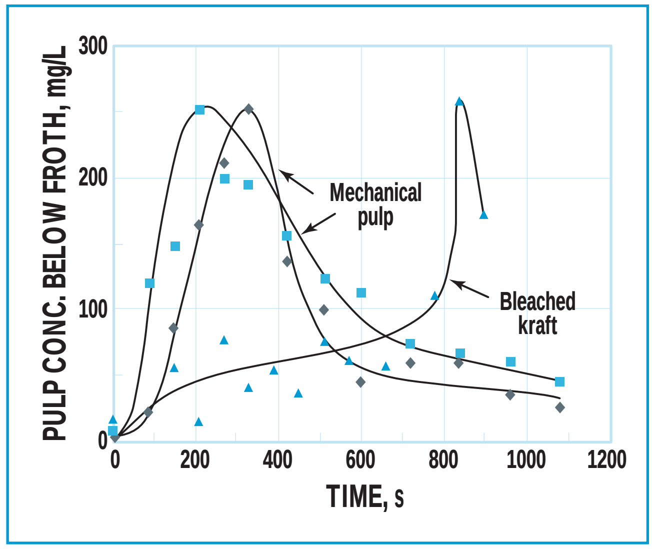
EXPERIMENTAL PROCEDURES

Materials

The red dye tracer was Liquid Food Color, Brilliant Red from the Bowes Co. Another tracer used was an aqueous polystyrene latex dispersion, 7.7% by weight, with a diameter of



1. Breakthrough curves for step injection of pulp onto the foam column. (The number beside the label to each curve is the wash water flow rate.)



2. Breakthrough curves for pulse injection of pulp onto the foam column.

0.775 ±0.06 µm, which was prepared in our laboratory. Sodium dodecyl sulfate (SDS) was BDH "Specially Pure Grade." Solutions were made daily using distilled water.

The mechanical pulp mixture, which was a blend of thermomechanical pulp and groundwood pulp, contained 39% fines and 61% long fibers. The bleached kraft pulp mixture contained 11% fines and 89% long fibers. Arithmetic average fiber lengths were 0.34 mm and 1.21 mm, respectively, for the fines and the fibers. Fiber suspensions were made by adding distilled water to the stock fiber and blending it until a uniform consistency was achieved.

Breakthrough curves

All experiments were conducted with a laboratory Plexiglas column 80.5 cm tall and 7.6 cm in internal diameter. A peristaltic pump was used to pump wash water from a reservoir to a nozzle fixed 1–2 cm above the top of the column. The spray from the nozzle uniformly distributed wash water on the foam column and helped to break foam and keep the foam volume constant.

Nitrogen gas flow, which was controlled by a Matheson gas flowmeter, was introduced through an orifice plate with holes 1/20,000 inch in diameter, spaced 1/8 in. apart. For one experiment, 43 bubble images of the froth phase were analyzed, giving an average bubble diameter of 3.81 mm with a standard deviation (of the mean) of 0.67 mm.

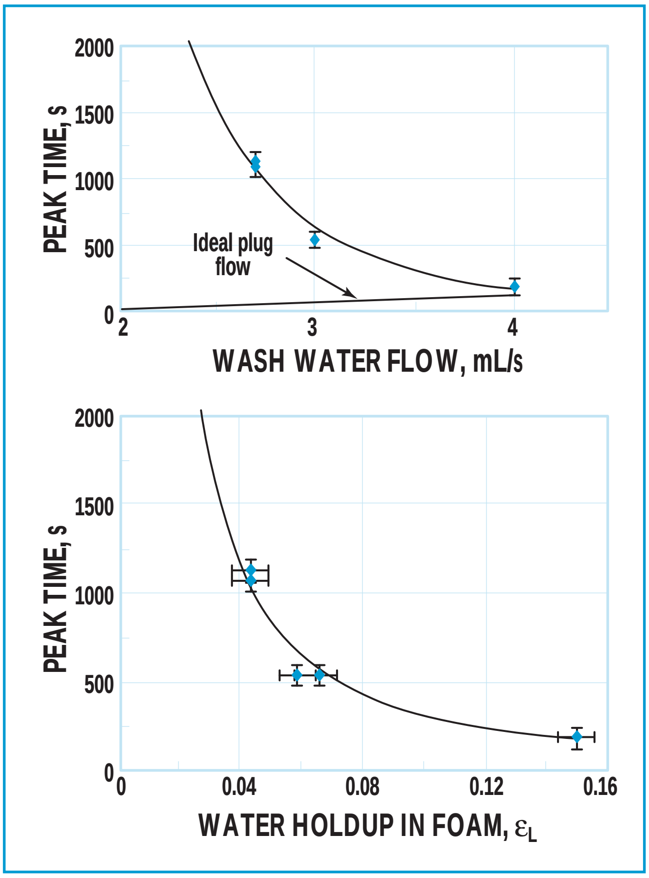
Liquid was removed from the column via an accepts tube that joined the side of the column 2 cm from its base. In steady-state operation, the column had a liquid zone of either 500 mL or 1100 mL, supporting froth layers either 65 cm or 55 cm deep, respectively. The liquid level in the column was controlled by the vertical position of an open tee joint, acting as a weir, on the accepts line. For the breakthrough experiments, samples were taken from the accepts line. For the flotation experiments, everything passing through the accepts line was collected and filtered to give the total amount of recovered pulp.

In a typical foam breakthrough experiment, 3.47 g/L SDS was added to the column, and the leveling T was adjusted to give 1100 mL of liquid in the column. Four liters of 0.2 g/L SDS was added to the wash water reservoir. The nitrogen flow was set to 1800 std. cm³/min. The wash water flow pump was started with the flow rate typically at 2.7 mL/s. The gas flow rate was reduced to 1250 std. cm³/min when the foam reached 5 cm from the top of the column. Small changes in wash water flux or air flow were sometimes required to achieve a steady-state froth volume.

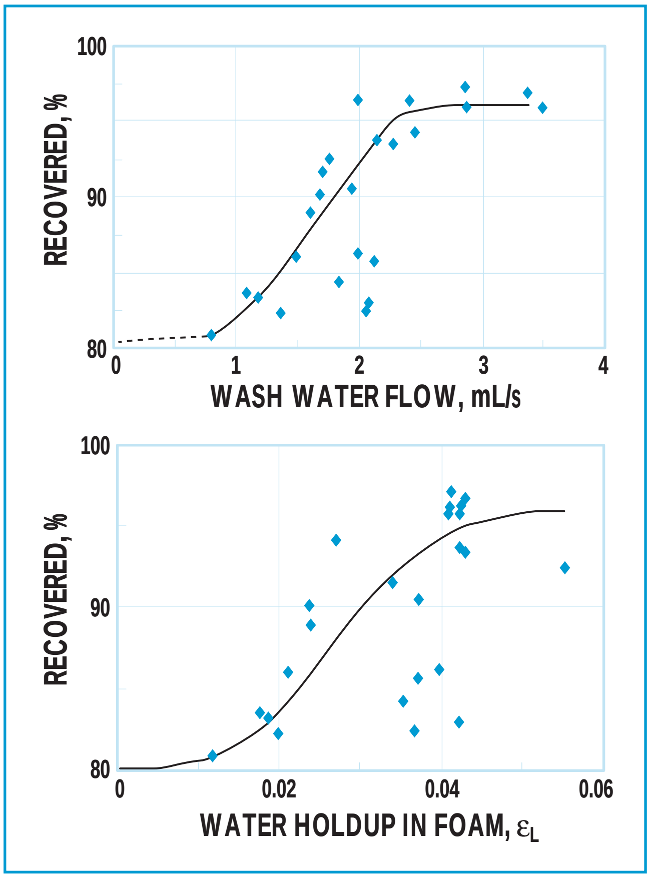
The experiment was initiated by adding dye or latex to the wash water reservoir to give a step change in tracer concentration. Since pulp would block the nozzle, 20 mL of a 1% pulp suspension was poured (the pulse) on top of the foam. Samples were collected from the accepts line as a function of time, and the concentrations of dye, latex, and pulp in the accepts stream were measured by UV-VIS spectroscopy based on calibration curves. At the end of the experiment, the average liquid holdup was determined from both the steady-state foam volume and the increase in liquid volume when the foam was left to collapse after the pump and gas flow were turned off.

Pulp flotation experiments

The same apparatus was used for the flotation experiments. A volume of 500 mL of an aqueous mixture of SDS (0.3 g/L) and pulp (1%) was placed in the cylinder. The gas flow was set to 1600 std. cm³/min, and the wash liquid flow (0.3 g/L SDS) was initiated. The column was operated



3. Curves for bleached kraft pulp injections.



4. Data for mechanical pulp recovery.

in a steady-state flow until the accepts line was free of pulp—that is, when all the pulp was removed from the liquid zone below the froth. Ideally, at this point, all the pulp was either in the accepts reservoir or in the foam. However, typically 4% of the added pulp settled to a quiescent zone at the bottom of the column, where it remained. The quantity of pulp in the accepts reservoir was determined gravimetrically. Typically the elapsed time for an experiment was 15 min.

One run had a wash water flow of zero. In this case, the gas was bubbled through the pulp solution for 15 min without the wash water pump running. The liquid phase was then emptied through the accepts line and was filtered to calculate recovery.

KEYWORDS
Flotation, deinking, yield, recycling, wood fibers, pulps, washing.

RESULTS

Tracer experiments

Initial experiments were conducted using dissolved tracers (dye) and colloidal tracers (latex) to determine the breakthrough characteristics of the foam column. **Figure 1** shows three example breakthrough curves. In the figure, the dimensionless tracer concentration in the liquid phase below the froth is presented as a function of dimensionless time after the step change. For these experiments, the volume of the liquid zone below the froth was 1100 mL, and the wash water flow rates were as given in the diagram. The froth height was 55 cm, the average liquid holdup was approximately 0.04, the SDS concentration was 3.3 g/L initially and 0.15 g/L in wash water, and the superficial gas flow rate was 0.46 cm/s.

The tracer concentrations in the accepts stream are expressed as the dimensionless concentration C_d, which is the actual concentration divided by the tracer concentration in the wash water reservoir. The dimensionless time τ is the total volume of wash water added to the column at time t divided by the volume of liquid holdup in the foam. Because the wash water flow rate was constant, τ is proportional to washing time and to the volume of wash water added.

Within experimental error, the dimensionless breakthrough curves were the same for latex and dye and were not sensitive to the flow rate of the wash water over the range studied. The shape of the curve can be modeled as plug flow through the froth column into an ideal continuously stirred tank—the liquid zone below the froth. The first indication of tracer breakthrough occurred at τ values between 0.8 and 2.2. The volume of the liquid below the froth was approximately twelve times the liquid volume in the froth. Therefore, the shape of the curves in Fig. 1 is dominated by the mixing characteristics in the liquid zone below the froth.

Figure 2 shows breakthrough curves resulting from a pulse injection of wood pulps on top of a froth 55 cm thick. In this figure, the pulp concentration in the liquid zone below the froth is presented as a function of time after a pulse of 7 mL of pulp has been injected to the top of the froth layer. The volume of the liquid zone below the froth was 1100 mL, and the wash water flow rate was 2.66 mL/s. Again, the foam height was 55 cm, the average liquid holdup was approximately 0.04, the SDS concentration was 3.3 g/L initially and 0.15 g/L in wash water, and the superficial gas flow rate was 0.46 cm/s.

The most striking result is that it is possible to wash pulp fibers through the froth. These curves are shaped like typical responses to a pulse injection (*5*). Duplicated runs for the mechanical pulp show reasonable reproducibility in view of both the difficulties of using turbidity as a measure of fiber concentration and the tendency for some fibers to settle to the bottom of the column instead of going into the accepts line. Duplicate runs for the bleached kraft pulp, not shown, displayed the same level of reproducibility.

Based on the average liquid holdup in the froth and on the wash water flow rate, an ideal pulse tracer should exit the froth column in about 37 s. The mechanical pulp took nearly 10 times longer than the ideal case to break through, and the bleached kraft pulp took 3 times longer than the mechanical pulp.

Pulse experiments on bleached kraft pulp were conducted using different rates for wash water flow. **Figure 3** shows the time corresponding to the peak in the breakthrough curve as a function of wash water flow and water holdup in the foam. The gas flow rate was 0.46 cm/s, the liquid volume below the froth was 500 mL, and the froth height was 65 cm. The pulse consisted of 20 mL of 1 wt% of pulp. In this series of experiments, the liquid holdup in the froth increased with the wash water flow rate. The same trend was also observed by Pal and Masliyah (*6*).

The error bars in Fig. 3 reflect our estimates of the accuracy of the measurements of pulp concentration, whereas the error bars along the *x* axis in the lower curve portray the uncertainty in the volume fractions of water in the foams, ε_L. Fibers did not pass through foam when the wash water flow was 2 mL/s, which corresponded to an average liquid holdup, ε_L, of 0.015. The curve labeled "Ideal Plug Flow" was calculated by $(V \cdot \varepsilon_L)/Q$, where V is the froth volume and Q is volumetric flow rate of wash water. At the highest rate of wash water flow, 4 mL/s, the experimental peak time corresponded to the ideal case. At lower rates, however, the experimental peak times were much longer.

Pulp flotation experiments

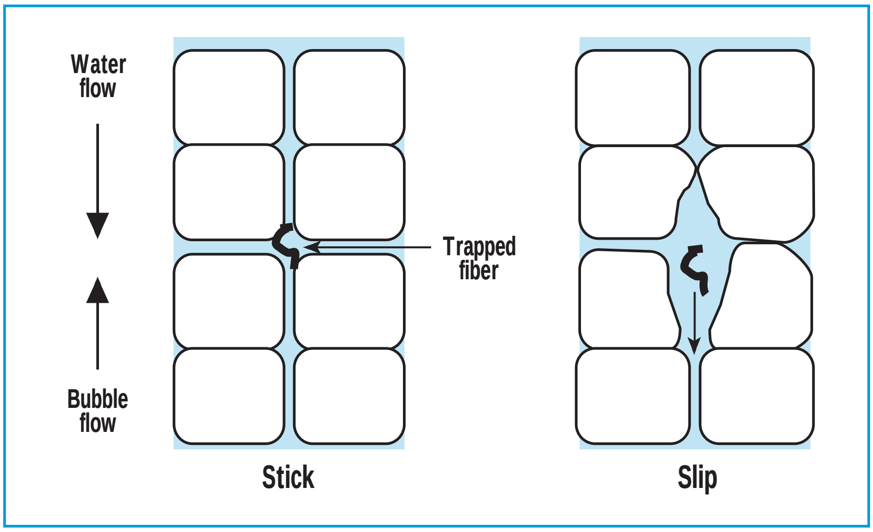
Experiments were run to determine flotation as a function of wash water flow rate. The pulp was initially present in the liquid zone below the froth instead of being added to the top of the froth. The results are summarized in **Fig. 4**, where the percent recovery is the fraction of the added pulp that ended up in the accepts stream. The unrecovered pulp was trapped in the froth.

Each data point represents a separate flotation experiment. At wash water flow rates of less than 1 mL/s (including 0 mL/s), approximately 20% of the added pulp ended up in the foam. Ajersch and Pelton have reported similar results without wash water (*2*). When wash water flows were greater than 2.5 mL/s, nearly all of the added pulp was recovered with the accepts. In all cases, some fibers settled to the bottom of the column, accounting for the 3% unrecoverable material.

The average froth water content, ε_L, corresponding to the onset of froth washing was approximately 0.02, whereas efficient washing corresponded to an ε_L of greater than 0.045. Presumably, the actual water content in the froth near the water–froth interface was much higher than the average values.

PROPOSED MECHANISM

The results in Fig. 3 show that fibers with an average length of 1.2 mm



5. Conceptualized states for a fiber in froth. In the "stick" state, the fiber is trapped in a dry area of froth. In the "slip" state, the fiber moves with a slug of water through the froth.

can be washed through a foam based on 4-mm-diameter bubbles with an average water content of only 0.04. If the froth layer behaves as a uniform porous medium formed by packing spheres, then it is difficult to understand how fibers pass through the foam. As depicted in **Fig. 5**, the liquid spaces between the bubbles are too small for fibers to pass.

Although the foam appeared to be a uniform froth rising in plug flow, there must be domains in the froth that contain enough water to encase the fibers. The fiber breakthrough curves (Fig. 2) displayed narrow peaks and long residence times. Therefore, all the fibers seem to be displaying "slip–stick" behavior. In other words, the fibers become trapped in dry zones in the froth until a slug of water carries them farther down the froth until entrapment occurs again.

The longer fibers require larger water domains to carry them down through the froth. Since it is less probable for larger water domains to form than for smaller ones to form, there will be fewer large slugs. As a result, longer fibers will take longer to be carried through the foam.

The results in Fig. 3 indicate that when the average froth water content approaches 0.15, the residence time (peak time) of the fibers is equal to the theoretical time for a soluble tracer. Thus, under these conditions, the fibers are not trapped between froth bubbles.

CONCLUSIONS

Based on the experimental results, our conclusions are as follows. First, froth washing can prevent fiber loss during flotation deinking. Second, it is possible to wash wood pulp fibers from froths with average liquid holdups as low as 0.04. Third, the fiber transport is by a "slip–stick" behavior impelled by slugs of water passing down through the froth. This mechanism explains why soluble tracers move through the foam faster than do the fibers. Finally, the longer fibers have a greater tendency to be detained in the foam.

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