APPLICATION OF SURFACTANT SPRAY IN FLOTATION DEINKING

Project F00904

Final Report

to the

MEMBER COMPANIES OF THE INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

April 1999

INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY

Atlanta, Georgia

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By

Y. Deng

April 1999

BACKGROUND

The research program to use spray surfactant technology to improve the deinking performance was started in October 1997. The primary results from early small bench-flotation cell experiments indicated that the surfactant spray approach could reduce fiber loss by 50%, water loss by 75%, and surfactant consumption by 95% without sacrificing deinking efficiency. The approach could also prevent fiber from being contaminated by the process surfactant. More importantly, the work developed a simple method to mechanically control froth stability when the physicochemical properties of the pulp source vary. One paper based on the results was published in the Journal of Pulp and Paper Science, and a patent was filed in December 1997.

Although the primary results were exciting, the limited results were with a small bench-flotation cell using toner-printed papers. Therefore, it was not known if this technique could be applied to large flotation cells under high turbulence conditions. Furthermore, how the geometry of flotation cells, the different surfactants, and different wastepaper furnishes would affect the application of this technology had not been answered.

PROJECT OBJECTIVE:

The overall objective of the research is to improve the deinking efficiency, control the fiber loss, and reduce the consumption of surfactant in flotation deinking using a surfactant spray technique. The research included the following tasks:

(1) Design laboratory pilot flotation deinking cells;

(2) Examine the difference in the total deinking performances (ink removal, fiber loss, and water loss) between conventional and surfactant spray techniques under different turbulence;

(3) Study the major factors affecting the total deinking performances in surfactant spray flotation deinking.

ABSTRACT

This research is concerned with increasing utilization of recycled fiber and, more specifically, with improving the ink removal efficiency, reducing the fiber loss, the water loss, the chemical consumption, and the contamination of fibers by processing chemicals.

The research conducted in FY98-99 was focused on the feasibility study of surfactant spray technique in a large-scale pilot flotation cell. Because of the increase in total volume and surface area, the turbulence on the top of the flotation cell was increased. The high turbulence makes it more difficult to stabilize a foam layer on the top of the flotation cell. This influence is being studied.

Several pilot-scale flotation cells with different geometry were installed. The experiments were conducted at different flotation conditions such as varying the nozzle numbers and location, changing the surfactant spray rate and amount, using different flotation cells and wastepaper, etc. The results indicate that although the turbulence of pulp suspension in a flotation cell makes it more difficult to stabilize foam at the top of the flotation cell, a foam layer can still be generated by sprayed surfactant with some modification of spraying method. The results obtained from our pilot tests, using toner-printed papers, indicated that the surfactant spray technique could significantly reduce the total surfactant consumption, fiber loss, water loss, and fiber contamination without sacrificing deinking efficiency. However, these results strongly depend on the flotation conditions and cell geometry. Furthermore, results obtained using mixed office waste (MOW) indicate that our flotation cell and conditions need to be optimized. Based on our laboratory and pilot tests, we believe this technique can be transferred to the paper recycle industry.

INTRODUCTION

Flotation deinking is a common practice for removing ink from wastepaper, and it is becoming a key process in many recycling paper mills. The application of flotation was successfully introduced to the paper recycling industry in the 1980s, and its applications in wax removal, sticky control, and fiber fractionation have attracted great research interest. The chemistry of the flotation process has been reviewed [1-3]. The deinking chemistry and the physicochemical interactions among air bubbles, fibers, and ink particles are very complex. Existing technologies and process designs of flotation deinking are based on the experiences obtained from mineral flotation processes. Limited process control mechanisms are available. Many problems remain unsolved such as high fiber and water losses [4-9], fiber contamination by deinking chemicals, adverse chemistry modification due to surfactant [1,2,10,11], low efficiency in removal of small ink particles [12-14], etc. Therefore, innovative technologies based on the mechanistic understanding of flotation processes are greatly needed to solve or alleviate the above problems. Because of the significant variability in the supply of secondary fibers in recycling practices, process control in flotation deinking is very important to improve recycling operations.

The roles of surfactants have been discussed in detail by Ferguson [1,2]. In general, surfactants play three roles in flotation deinking: as a dispersant to separate the ink particles from the fiber surface and prevent the redeposition of separated particles on fibers, as a collector to agglomerate small particles to large ones and change the surface of particles from hydrophilic to hydrophobic, and as a frother to generate a foam layer at the top of the flotation cell for ink removal. Although surfactants play important roles, they will also cause some adverse effects on ink removal, fiber quality, and water reuse. For example, both hydrophobicity and ink removal efficiency will decrease by the adsorption of dispersant and frother [10,11]. The remaining surfactant in recycled fibers is another problem that may cause a decrease in fiber-fiber bonding, an increase in foams during the papermaking process, an adverse effect on printing, etc. Because surfactants have both positive and negative effects, it is of interest whether dispersant, collector, and frother can be separately controlled.

The surfactants used in mineral flotation may not be necessary in flotation deinking. For instance, some ink particles, such as xerox toner, are hydrophobic in nature and no collector is necessary. The dispersant may also be unnecessary if the ink particles can be removed from fibers by other chemicals, such as sodium silicate, sodium hydroxide, and, enzyme, or by mechanical actions, such as magnetic and electrical fields, and ultrasonic irradiation. Traditionally, the frother and other surfactants are added into the pulp suspension during repulping. However, the surfactant present in pulp slurry will not only contribute to the foam stabilization, but also adsorb onto ink particle surfaces and cause a decrease in the hydrophobicity of ink particles.

Furthermore, the mechanical control of froth stability is very difficult if the surfactant is directly added into the pulp slurry because the properties of wastepaper may vary significantly.

Because the foams are stabilized by surfactant only on the top of the flotation cell, it is of interest to develop a feasible method to directly add the frother to the top of the flotation cell rather than in the pulp suspension. As a result, a separate control of the addition of various surfactants to improve the performance of deinking processes can be achieved.

Ink removal efficiency depends on several factors such as the ability to separate the ink particles from the fibers, the collision probability between ink particles and air bubbles, the interfacial energy between ink particles and the air bubble surface, the specific contact surface area between ink particles and air bubbles, the stability of the froth for final ink removal, etc. It is well-known that surface chemistry plays a key role in flotation deinking. It has also been identified that froth stability is critical for ink removal. Ink removal efficiency increases with an increase in froth stability, so that there is an increase in surfactant concentration in conventional flotation systems. Unfortunately, the increase in surfactant concentration in the pulp suspension will increase the adsorption of surfactant onto ink particles, resulting in a reduction of the surface hydrophobicity of ink particles and ink removal [10]. Therefore, there must be an optimum surfactant concentration and ink removal efficiency. Practically, it is difficult to optimize the surfactant concentration in a paper recycle mill because of the variability in the secondary fiber sources. This indicates that good control of surfactant concentration and its distribution within a flotation column can significantly improve the flotation deinking operation.

Based on the above fundamental understandings of flotation deinking, it is clear that effective controls of the key process variables can increase ink removal and reduce fiber and water losses. Surfactant spray technology that was proposed and tested in our early research can separately control foam properties (the foam stability and the height of foam layer in a flotation cell) from other variables. As a result, the surfactant consumption, concentration and its distribution, froth structure and stability, and fluidynamics in the froth can be controlled.

The mechanism of surfactant spray is easy to be understood. Figure 1 Illustrates that the ink particle adheres to an air bubble and move to the liquid surface in conventional flotation. The surfactant molecules presented in the bulk solution adsorb onto the bubble and ink surfaces and stabilize the bubble in a foam layer. Figure 2 illustrates that the ink particle adheres to an air bubble in the absence of surfactant. The ink particle moves with the air bubble up to the surface of liquid. Because there is no surfactant in this system, the bubble breaks at the surface and the ink particle returns back to the bulk phase. Figure 3 illustrates that the ink particle adheres to an air bubble in the absence of surfactant in the bulk phase. When the bubble and ink move to the surfactant layer that is generated by sprayed surfactant, the surfactant molecules adsorb onto the bubble and ink particle surfaces. As a result, the bubble is stabilized and a foam layer is generated similar to a conventional flotation process. It is clear that, under this idea condition, the following advantages can be achieved using the surfactant spray approach:

1. Hydrophobicity will not decrease because there is no adsorption of surfactant on ink particle and air bubbles before they reach the surface;

2. Fiber will not be contaminated by surfactant because surfactant does not come in contact with fibers in the bulk phase;

3. Surfactant consumption can be significantly reduced because only a thin layer of surfactant is needed;

4. Foaming ability and stability can be controlled by sprayed surfactant.

The research conducted in FY98-99 was focused a feasibility study at high turbulence conditions using different pilot flotation cells. The total performance (ink removal, fiber loss, water loss, surfactant consumption, etc.) of the surfactant spray technology was compared with that of a conventional flotation deinking process.

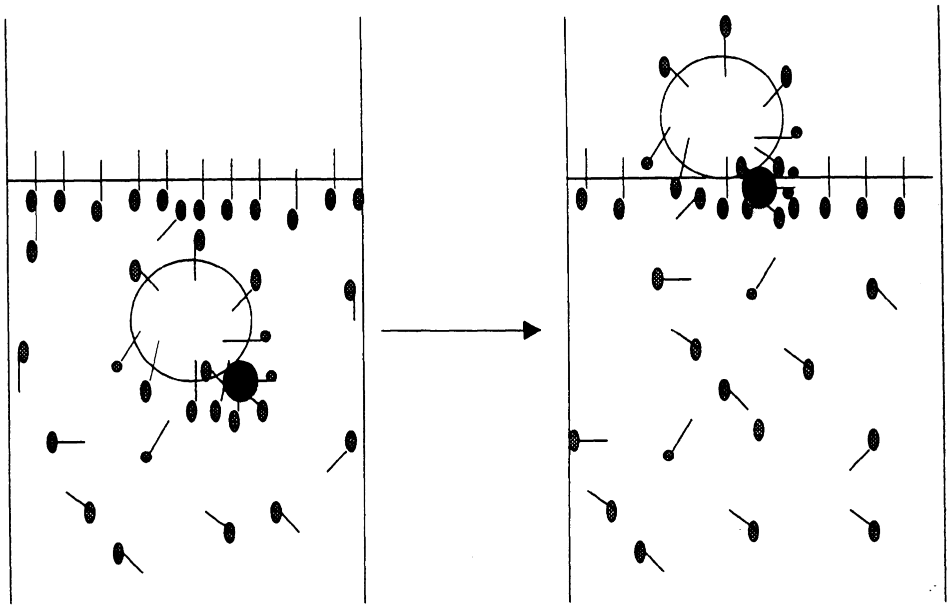

Figure 1: Ink-bubble interaction in a conventional flotation deinking process. The ink particles can be transferred to the foam layer if multiple bubbles are present in the system.

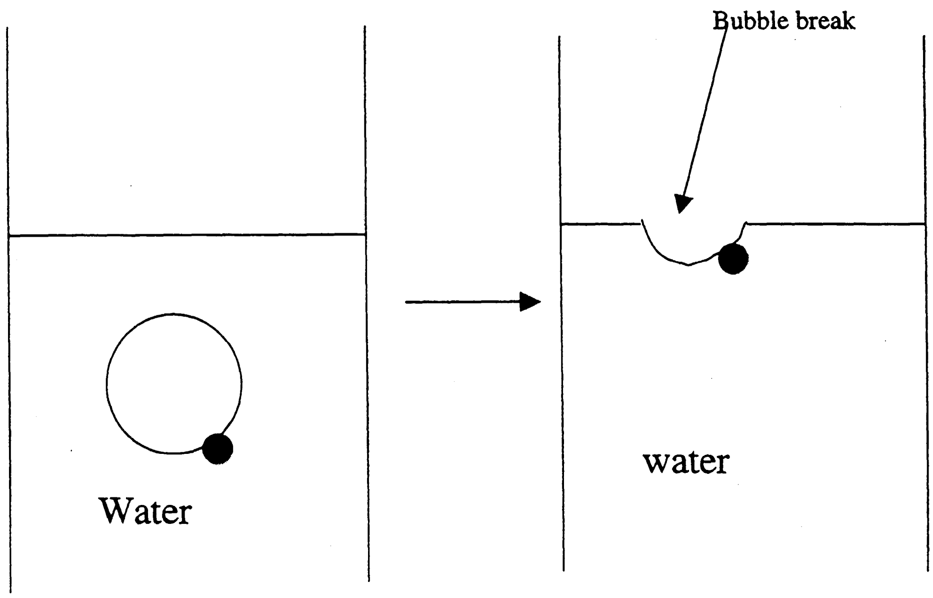

Figure 2: Ink adheres to an air bubble in the absence of surfactant. The bubble breaks when it moves to the surface. As a result, no foam can be generated and the ink particle will return to the bulk phase.

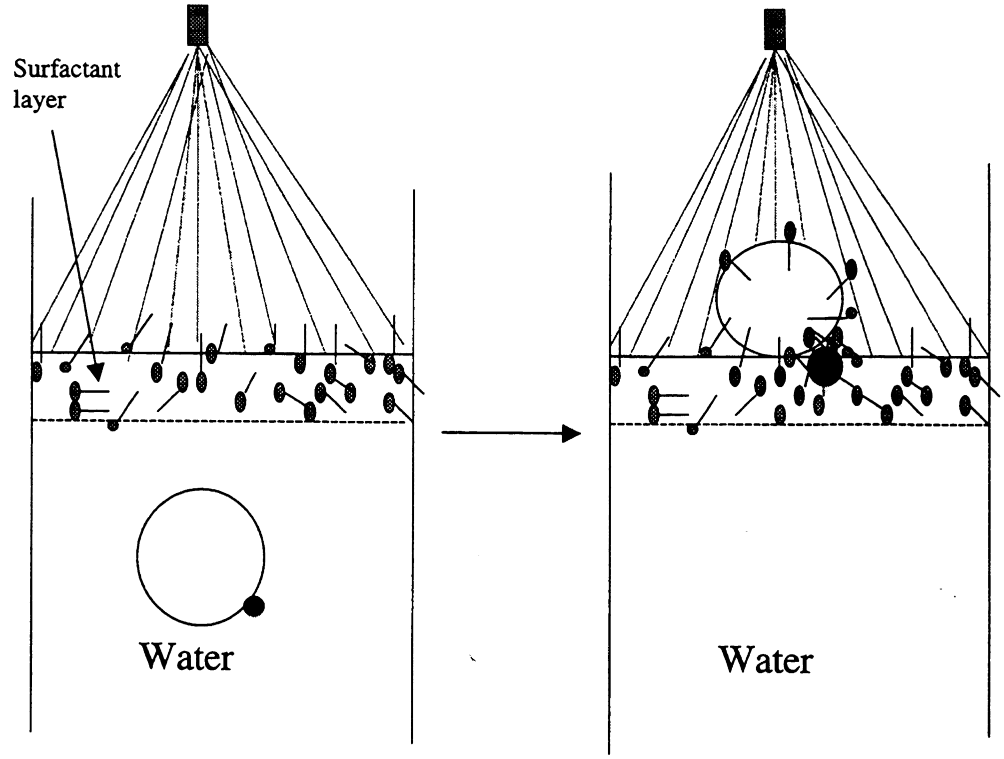

Figure 3: Surfactant spray can create a surfactant layer on the top of the flotation cell. When an air bubble passes through this layer, the surfactant adsorbs onto the bubble surface. As a result, the bubble can be stabilized and a foam layer can be generated. Thereby, the ink particle can be transferred to the foam layer as in a conventional flotation process.

EXPERIMENTAL

Flotation cell and nozzle installation

Two types of flotation cells, i.e., column and Voith flotation cells, were installed. Column flotation cells with different geometries were designed. The column flotation cell-1 was built with a rectangular plastic tank with a volume of 80 liters (0.35m×0.35m×0.6m). The air was injected from the bottom of the flotation cell through spargers with an average pore size of ~0.1 mm. The column flotation cell-2 was built with a cylindrical plastic tank with a volume of 450 liters (0.4 m in diameter and 1 m in height). Five disk spargers with an average pore size of 2 μm were installed at the bottom of the flotation cells. The foam on the surface of flotation cell was removed by a vacuum system.

The Voith flotation cell was on loan from Champion International Corp. The optimum flowrate of the cell is 764 litters/minute. However, because the maximum flowrate of the pumps used in this study is only 90 liters/minute, the cell cannot operates at recommended conditions. Therefore, the original inlet pipe (7.62 cm in radius) of this cell was replaced by a small inlet pipe (2.54 cm in radius) in this study. The foam was removed by a vacuum system.

The pressure spray nozzles were mounted at the top of the deinking cells. Different locations of nozzles were first tested for each flotation cells before the flotation experiments. The distance between nozzle and pulp surface was varied from 2 to 20 cm above the pulp suspension surface. It was found that this distance was not critical for generating a required foam layer. The number of nozzles was varied according to the surface area of the flotation cell. On average, 0.04 m^2 of surface contains one nozzle. The nozzle orifice diameter is about 0.46 mm. The nozzle is operated at a gage pressure of 0.5 atm. The mean spray droplet size (Sauter mean diameter, SMD) is about 50 mm measured by a laser diffraction instrument (Malvern 2600).

Flotation process design

Column flotation cells with different flow loops were used in this study. Figure 4 shows the closed flow loop flotation design, and Figure 5 shows the photograph of the column flotation cell-2. In a close-loop flotation test, the pulp was injected from the top and left the system from the bottom of the column flotation cell. The residency time of pulp staying in the flotation cell varied from a few seconds to two minutes depending on the experimental conditions. The pulp rejected from the bottom was recycled until a required total flotation time was achieved. This close-loop process can actually be considered a batch flotation process. During the experiments, the samples were collected and analyzed at different total flotation times. If the accept was directly collected rather than recycled, it was an open-loop system. If the accept pipe was closed, it was a batch flotation process. The experimental data present in this report were obtained from all of the three types of flotation processes, i.e., close-loop, open-loop and batch flotation processes. The details are given in the figure captions.

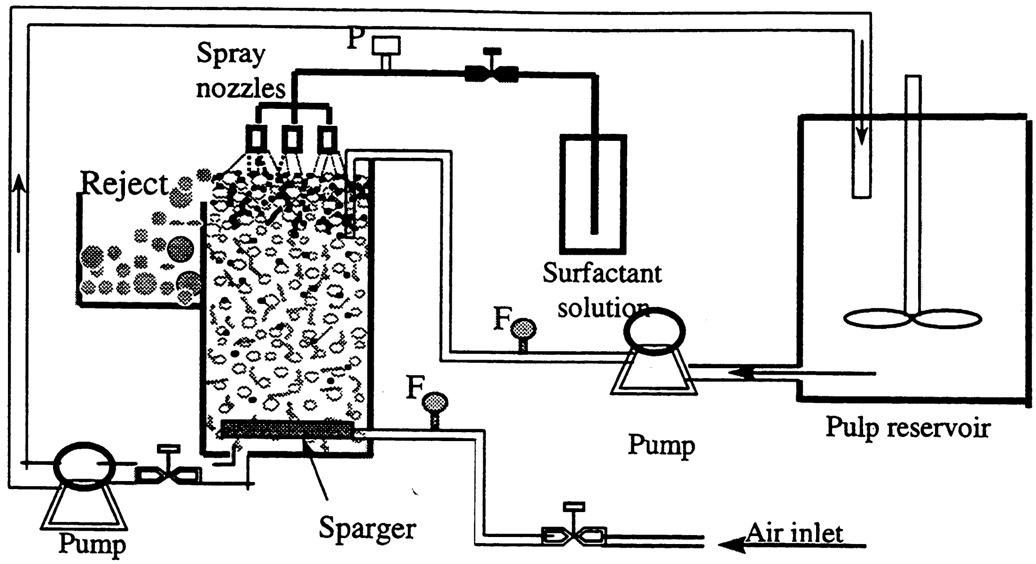

Figure 4. Close-loop flotation cell.

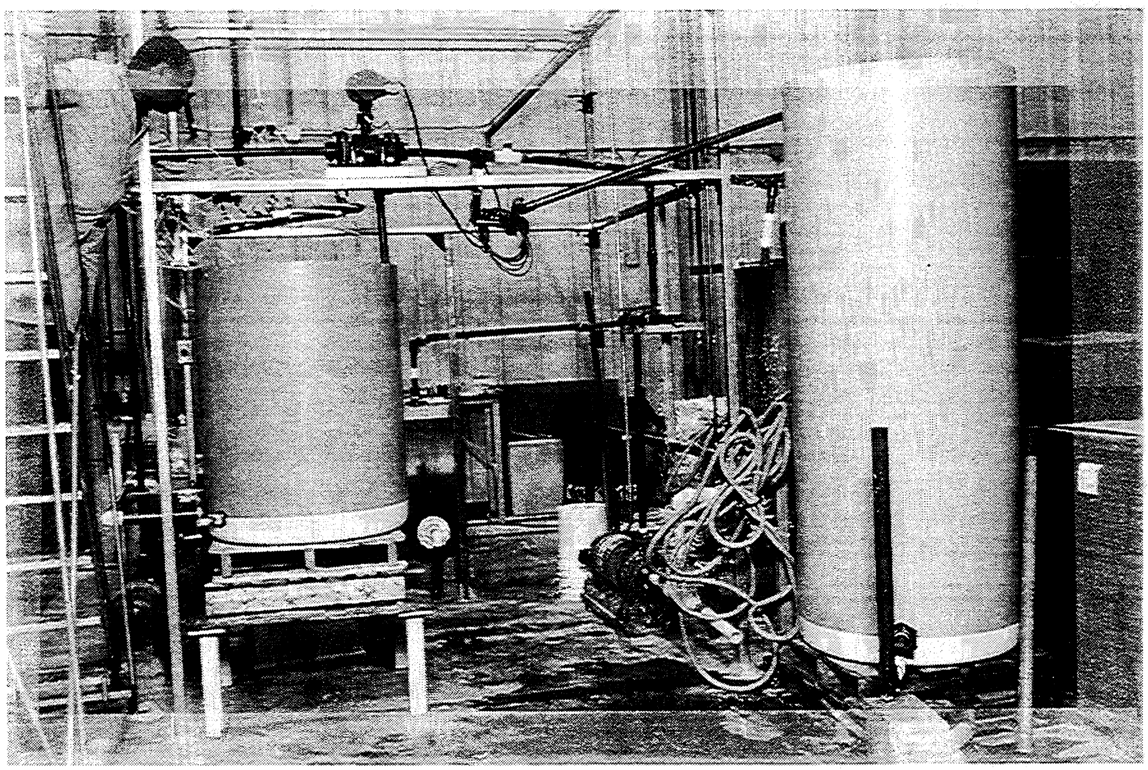

Figure 5. Photograph of the column flotation.

Materials

Both commercial mixed office waste (purchased from Renewal Atlanta Company) furnishes and xerox copied bond papers were used in this study. All papers were repulped at pH of 10 and a consistency of 8% without adding any chemicals except sodium hydroxide. The water and fiber losses were obtained by gravimetric method. The ash contents in the toner printed paper furnish were 16% (original) and 8.2% (after flotation), respectively. The pulp consistency used in the flotation process was 0.8 to 1%. Three different surfactants, alkyl phenol ethoxylate (TX-100, J.T. Backer Inc.), ethoxylate alcohol (Shell Chemical Inc.), and Buckman BRD2340 (a nonionic surfactant from Buckman Inc.) were used as surfactants. The required amount of surfactant was added directly into the pulp in "conventional flotation," but was sprayed through a nozzle from the top of the pulp in "surfactant spray flotation." The handsheets for brightness analysis were made on a 15-cm Büchner funnel according to TAPPI standard method. The brightness of the handsheets was measured using a Shimadzu UV-VIS spectrophotometer (UV-160A). The size of toner particles was analyzed by a laser diffraction instrument (Malvern 2600). Figure 6 shows the particle size distribution of repulped toner printed papers.

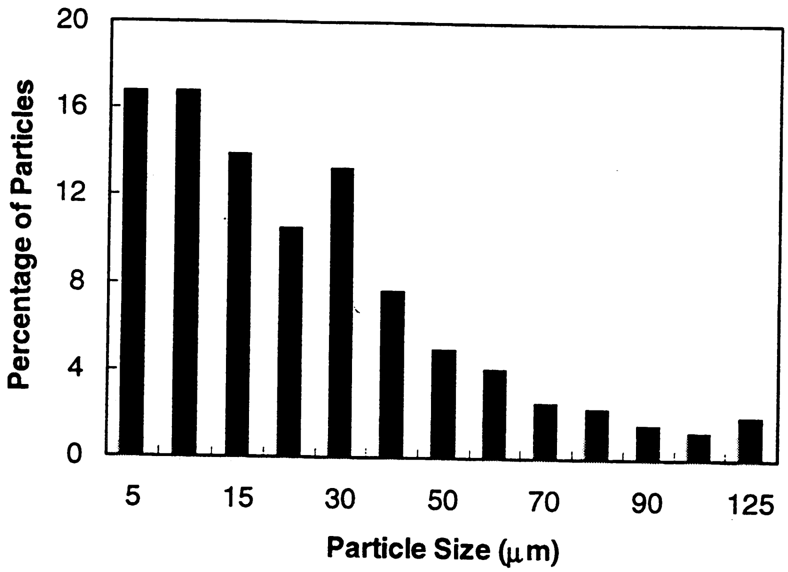

Figure 6. Toner particle size distribution in the pulp made from copied papers.

RESULTS AND DISCUSSION

Froth Establishment by Surfactant Sprays

The froth formation under the application of surfactant spray from the top of a flotation column was first examined in the absence of fibers. No foam layer could be established when air bubbles were injected from the bottom of the flotation column that contains only pure water. However, when a small amount of surfactant solution was sprayed from the top of the flotation cell, a stable foam layer was established on the surface of the pure water phase in a few minutes. The rate of foam formation depends on the spray rate, type of surfactants, surfactant concentration in the spray solution, airflow rate, turbulence of the system, and the geometry of the flotation cell. It was found that the higher the turbulence, the more difficult it was to generate a stable foam layer. Because a column flotation cell has less turbulence compared to Voith cell, the foam is much easier to be stabilized in a column flotation cell.

The concentration and amount of surfactant sprayed on the top of the flotation cell depends on many factors, such as the contents and consistency of pulp furnish, the geometry of the cell, the flotation conditions, the height of the foam, etc. which may vary from mill to mill.

<u>The results from toner-printed papers using a rectangular cell</u>

In these tests, the toner-printed papers were used. **Table 1** shows the results obtained from close-loop flotation tests using column cell-1 (rectangular cell) and toner-printed paper furnish. Alkyl phenol ethoxylate was used as surfactant. It can be seen that at the same flotation time, the surfactant used in spray flotation process is remarkably lower than that used in conventional flotation process. The brightness of the deinking pulps is almost the same or a little higher for surfactant spray as compared to conventional flotation. Other benefits, such as low water loss and fiber loss, can also be seen in **Table 1**.

It was noted that the overall performance using a 165 mg/L surfactant solution in the spray was better than the performance of using a 1000 mg/L surfactant solution. The reason for the enhanced performance was because the hydrophobicity of the toner particle remained higher in low surfactant conditions compared to high-concentration conditions. It was noted that low surfactant concentration created a foam layer with larger bubbles than the high-concentration solution. Because physical entrainment is the major mechanism for water loss and fiber loss in flotation deinking [15,16], the foam containing large bubbles entraps less water and fibers than the foam containing smaller bubbles, resulting in less fiber and water loss. This was supported by our previous results [15,16]. Although low surfactant solution gave better results, we could not further reduce the surfactant concentration because it was very difficult to remove ink particles if the thickness of foam was too low.

Figure 7 shows that, at the same flotation time, the surfactant spray method (165 mg/L alkyl phenol ethoxylate as spray solution) gives better brightness than the conventional flotation method (25 mg/L alkyl phenol ethoxylate). Furthermore, the total surfactant consumption for surfactant spray was much lower than that used in the conventional process. **Figure 8** compares two different methods at different surfactant addition levels. It can be concluded from **Figures 7** and **8** that, for all of the surfactant concentrations tested in this study, the surfactant spray will not sacrifice the deinking efficiency, but can significantly reduce the surfactant consumption, water loss, and fiber loss.

Table 1. Brightness, water loss and fiber loss as a function of surfactant (alkyl phenol ethoxylate) consumption in conventional and surfactant spray flotation. Close-loop flotation cell-1 (rectangular cell) was used. Pulp was made from toner copied papers. Air flowrate: 10 standard cubic feet per hour.

Operation	Time (Min)	TX-100 consumption (kg/T)	Brightness (ISO)	Water loss (L)	Fiber loss (%)
Spray (165 mg/L surfactant in spray solution)	35	0.27	80.76	4.45	1.85
Spray (1000 mg/L surfactant in spray solution)	35	1.665	78.6	5.35	2.56
Conventional (10 mg/L surfactant in pulp furnish)	35	1.32	79.5	8	3.25
Conventional (25 mg/L in pulp furnish)	35	2.75	78.6	13.5	5.92

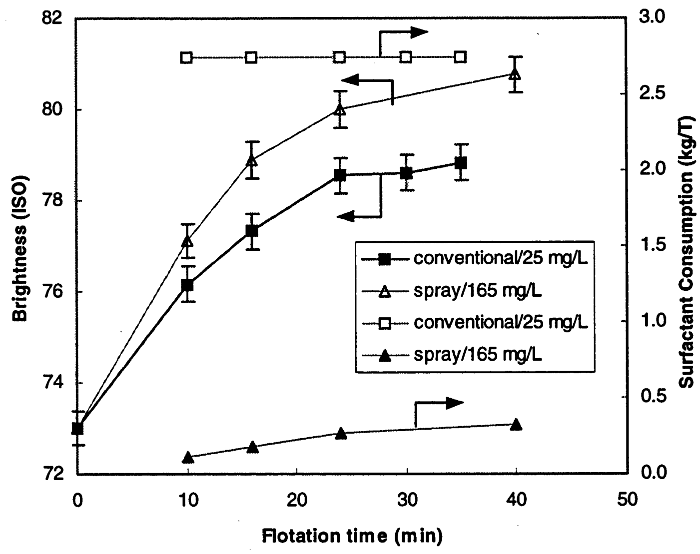

Figure 7. Brightness and surfactant consumption as a function of flotation time. Close-loop flotation cell-1 (rectangular) was used. Toner copied papers; Alkyl phenol ethoxylate as surfactant; Air flowrate: 10 standard cubic feet per hour.

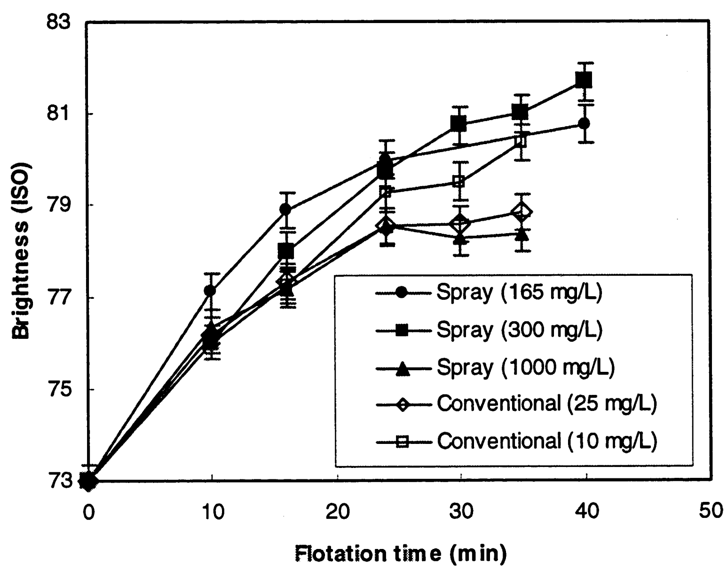

Figure 8. The brightness as a function of flotation time for different surfactant concentrations. Toner copied papers; Alkyl phenol ethoxylate as surfactant; Air flowrate: 10 standard cubic feet per hour.

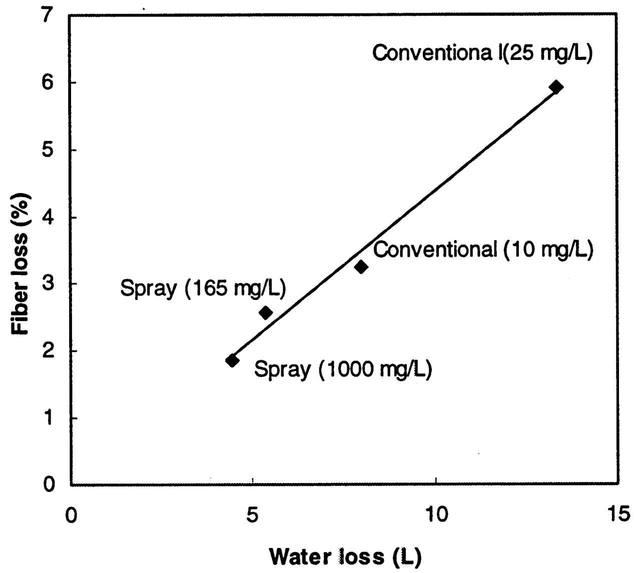

Figure 9. The fiber loss after 30 minutes flotation as a function of water loss obtained from different methods at different surfactant concentrations. Toner copied papers; Alkyl phenol ethoxylate as surfactant; Air flowrate: 10 standard cubic feet per hour.

The fiber loss as a function of water loss is shown in **Figure 9**. It is very interesting to note that the fiber loss has a linear relationship with the water loss, regardless of the methods used (spray and conventional) and surfactant concentrations. Because water loss is only caused by physical entrainment between air bubbles, the linear relationship between fiber loss and water loss suggests that the physical entrainment was the major reason for the fiber loss in flotation deinking. This also suggests that regardless of what chemicals and flotation cells are used, the fiber loss was always closely related to the foam structure and stability. In order to reduce the fiber loss, the foam must be well controlled. This is consistent with our previous mechanism study using a laboratory flotation cell [15,16].

<u>The results from mixed office wastepapers using a cylindrical cell</u>

In order to test the geometry and turbulence effect on the surfactant spray technique, two large-scale flotation cells (cylindrical cells) were built. These two cylindrical cells have the same diameter (0.8 meter) but different heights (1.25 and 2 meters). The volumes are 0.5 m^3 and 1 m^3, respectively. Using these two large-scale flotation cells, the effect of cell height on the total deinking performance can be studied. Unfortunately, only the flotation cell with 1 meter height was tested in FY99 because of the limited time.

For the experiments conducted at this larger scale flotation cell, mixed office waste furnish was used instead of only toner-printed papers. Because of the limitation of time and total budget of the project, only batch flotation tests were conducted this year. The mixed office waste was repulped at pH of 9.5 and 50°C without adding any other chemicals. Two different surfactants, ethoxylate alcohol (flotation deinking surfactant from Shell Chemical Inc.), and Buckman BRD2340 were used in these tests.

Compared with the small-scale flotation cell, a higher turbulence at the surface of the large-scale flotation cell was observed. It was noticed that one of the advantages to use the spray technology was that it creates a concentrate surfactant layer on the surface of the furnish. However, if the flotation cell has a high turbulence and large surface area, it is very difficult to maintain a high concentration surfactant layer on the top of the flotation cell. To reduce the turbulence, a simple device was installed on the top of the flotation cell to reduce the turbulence. It was found that by using this simple device, the turbulence of pulp furnish surface could be reduced without affecting the ink and air bubble transportation. As a result, a high-concentration surfactant layer can be easily created on the surface of the pulp furnish.

It was noted that the mixed office waste we used was very dirty, which includes xerox papers, coated magazines, newsprint, color papers, etc. The tests using this furnish was conducted at a batch flotation cell (cylindrical cell, 0.5 m^3). The deinking efficiency was poor for this furnish. The poor ink removal was seen not only for the surfactant spray flotation test, but also for the conventional flotation test. Several reasons may result in the poor results:

1. This furnish containing many small ink particles, such as the ink from newsprint and computer-printed papers. It is well known that these small ink particles are

very difficult to remove by flotation if no collector, such as calcium soap, was added. Because no any chemicals except the foaming agent was used in our tests, small ink particles could not be removed, which resulted in poor brightness.

2. In a typical deinking plant, the water-soluble components and very small and large particles are usually removed by washing and cleaning before flotation. However, we did not do any washing and cleaning in our experiments. Because flotation can only remove the particles with certain particle size, the final brightness of the handsheets made from our deinked pulp would not be very high.

3. This was the first test using this large-scale laboratory flotation cell. The conditions may not have been optimized. For example, the airflow rate and flotation time were not be adjusted.

More recently, we conducted a series of tests for MOW deinking using surfactant spry in the present a collector. The collectors have been tested are dodecan and calcium soap. It has been found that if a collector was added, the deinking efficiency could be significantly improved. We will give the quantitative results for these tests once the tests are accomplished.

CONCLUSIONS

In summary, the proposed approach of applying process control in flotation deinking using surfactant spray demonstrates several advantages over the conventional flotation deinking process:

1. Spray surfactant at the top of the flotation column can effectively establish a stable froth for good ink removal.

2. Surfactant application through a spray at the top of the column can effectively prevent the fiber from surfactant contamination, and reduce the modification of deinking chemistry through surfactant adsorption, resulting in higher ink removal, lower surfactant consumption, and lower fiber and water losses.

3. Control of surfactant delivery through mechanical devices, such as a spray, is an excellent approach to control froth stability and to improve the performance of the flotation deinking process significantly.

4. Control of surfactant delivery is a potentially effective method to improve the roles of dispersant, collector, and frother in flotation deinking.

5. Control of surfactant delivery has potential advantages in whole process control in flotation deinking, and particularly can be used for stabilizing flotation operations when pulp sources are changed.

6. Laboratory studies demonstrate that without sacrificing deinking efficiency, the proposed approach can significantly reduce fiber loss, water, and surfactant consumption.

7. Reducing the surface turbulence is important for generating a high concentration surfactant layer and stabilizing foam on the top of the flotation cell.

DELIVERABLES

1. Final report will be sent to the Member Companies in March 1999.

2. We will contact recycling mills and equipment manufacturers for mill trials and transfer of the technology.

FUTURE WORK

Although financial support from IPST's member companies will be terminated by the end of FY99, we have submitted a proposal to Agenda 2020 with Voith Corporation as a participant.

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