FINAL REPORT

PROJECT TITLE: SURFACTANT SPRAY: A NOVEL TECHNOLOGY TO IMPROVE FLOTATION DEINKING PERFORMANCE

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Executive Summary

Based on the fundamental understanding of ink removal and fiber loss mechanism in flotation deinking process, we developed this innovative technology using surfactant spray to improve the ink removal efficiency, reduce the water and fiber loss, reduce the chemical consumption and carry over in the flotation deinking. The innovative flotation deinking process uses a spray to deliver the frothing agent during flotation deinking to control several key process variables. The spray can control the foam stability and structure and modify the fluid dynamics to reduce the fibers entrapped in the froth layer. The froth formed at the top part of the flotation column will act as a physical filter to prevent the penetration of frothing agent into the pulp suspension to eliminate fiber contamination and unfavorable deinking surface chemistry modification due to surfactant adsorption on the fiber surface. Because of the filter effect, frothing agents will be better utilized.

Under the sponsorships of the US Dept. of Energy (DOE) and the member companies of the Institute of Paper Science and Technology, we studied the chemmechanical mechanism of surfactant spray for flotation deinking using different furnishes, chemicals, and flotation devices in the past four years. In the final year of the project, we successfully conducted mill trials at Abitibi-Consolidated, Inc., Snowflake paper recycling operation of 100% mixture of ONP/OMG. Results from laboratory, pilot-plant and mill trials indicated that surfactant spray technology can significantly reduce fiber loss in flotation deinking. It can be concluded that paper industry can profit greatly when this technology is commercialized in flotation deinking mills.

For reading convenience, this executive summary was divided into the four parts.

1. Labtororal and Pilot Study of ONP/OMG Deinking Using Surfactant Spray

The initial demonstration of the foaming agent spray concept was carried out in a column flotation cell using an ideal furnish, i.e., pure toner-printed papers with relatively large and hydrophobic particles that are floated easily without adding collectors. It was found from the previous investigation that fiber loss was reduced by 50% while the application of foaming agent was reduced by 95% when spray was applied through a spray on top of a column flotation cell in deinking toner-printed papers in a laboratory study.

Although the results from our previous study using toner copied paper were exciting, it was unclear how this concept can be applied to other deinking systems in that a collector must be used. For example, offset-ink particles in old newsprint (ONP) are different from toner particles in copy papers. Copy-toner particles can be effectively floated by flotation without a collector, but the offset-ink particles are difficult to float if no collector is applied. Therefore we verified the viability of the foaming agent spray concept in the deinking of old newsprint (ONP) printed with offset ink and its mixture with old magazine paper (OMG) in the presence of an effective collector using laboratory and pilot-scale flotation deinking cells.

It was demonstrated in this study the foaming agent spray could achieve separate control of the application of deinking chemicals, i.e., foaming agent and collector, in flotation deinking of waste paper. The foaming agent spray concept was first demonstrated in batch flotation experiments using conventional deinking chemistry (foaming agent and collector blended) to deink old newsprint paper (ONP) and a mixture of ONP with old magazine paper (OMG), respectively, in a Voith Sulzer flotation cell with 18-liter capacity (E-18). A defoamer was used to suppress the foam produced by the blended deinking chemicals applied during pulping, then regenerated at the top layer of the flotation pulp suspension using a foaming agent spray at the top of the flotation cell. The concept was also demonstrated without the application of a defoamer when a low foamability collector was applied during pulping. The results indicate that water spray washing of foam can wash down some fibers entrapped in the bubble network of foam and reduce fiber loss in flotation, but

there is an optimum water spray loading; spraying too much water can cause too fast an overflow of foams, resulting in high fiber loss due to fiber entrainment. The results also indicate that froth produced by spraying a foaming agent from the top of the flotation cell reduced fiber entrainment and therefore fiber yield loss under equivalent ink removal. Typical fiber loss reduction of 50% was achieved at a brightness gain around 9 ISO% in deinking ONP and OMG mixture in an E-18 cell which can be seen in table 1 and in deinking an ONP and OMG mixture in scale-up experiments using Voith Sulzer flotation cell E-250 of capacity of 250-liter which can be seen in Fig. 1. Equivalent dirt counts on handsheets made of deinked fibers were achieved between frothing agent spray deinking of a mixture of ONP and OMG with low foaming agent TDA-32 and conventional deinking of the same furnish with fatty acid, but the former gave much lower fiber loss. By comparing the experiments in the E-18 cell and E-250 cell in flotation deinking a mixture of ONP and OMG, scale-up of the frothing agent spray concept was successfully demonstrated using large scale of flotation cell (250 cell).

technique in the E-18 cell with a low loaming collector TDA-32.							
Experimental	Deinking	Brightness	Fiber Loss	Brightness/F	Water		
Process	Conditions	Gain (ISO%)	(%)	iber Loss	Loss (%)		
				(Selectivity)			
Conventional	Fatty Acid @	9.9	18.9	0.52	18.5		
	30 mg/L						
Conventional	Vinings A @	10.1	19.0	0.53	17.6		
	30 mg/L						
Foaming	TDA-32 @ 30	7.8	11.9	0.66	9.8		
Agent Spray	mg/L + Spray						
••••	72 mg TX-100						
	@ 100 mg/L						

Table 1. Comparisons of the performance of flotation deinking of a mixture of OMG and ONP using a conventional flotation process with collectors Vinings A and fatty acids and the frothing agent spray technique in the E-18 cell with a low foaming collector TDA-32



Fig. 1. Comparisons of flotation deinking efficiencies in deinking a mixture of OMG and ONP using conventional flotation with collectors Vinings A and fatty acids and the surfactant spray technique with a low foaming collector TDA-32 in the E-250 cell.

2. Reducing Fiber Loss in Mill-scale Flotation Deinking ONP/OMG Furnish Using Surfactant Spray Technology

The ability of surfactant spray technology to reduce yield loss without detriment to pulp brightness gains has also been successful transferred to a single flotation unit within the deinking line of a mill producing newsprint from 100% mixture of ONP/OMG (Fig. 2). Initial results suggest that the loss of fiber across the unit may be reduced by more than 50% without obvious detriment to final pulp quality which can be seen in Fig. 3. During mill-scale trials, surfactant spray deinking technology was found to improve yield across a single flotation unit. Although the brightness gains/ERIC reductions within the post-flotation pulps were somewhat lower than those floated under conventional deinking conditions, the difference was viewed as tolerable in light of the remarkably diminished yield losses. In addition, the technology has demonstrated an ability to impart some amount of process control to the flotation

process. Both benefits, reduced yield loss and improved process control, were realized with minimal capital expenditure and equipment modification.



Fig. 2. The GSC flotation cell was fitted with a bank of 14 nozzles for delivery of the surfactant spray onto the surface of the aerated pulp.



Note: Conventional results were obtained during 2 months (15 trials);

SST (surfactant spray technology) results were obtained during 1 day (9 trials).

Fig. 3. Average fiber yield loss as a function of average brightness gain values is presented for both conventional and surfactant spray flotation deinking.

It must be stressed that this initial mill-scale trial was conducted under conditions found optimal during pilot trials. To this end, the system is expected to be far from optimized. Possible variations in future trials may involve modifying the nozzle-bank design (i.e. spray delivery rate, nozzle number, spray pattern, etc.) and/or the composition of the spray itself (e.g. use of a more environmentally benign frothing agent).

3. 100% Flexographic ONP Deinking Using Surfactant Spray In the presence of siloxane-based defoamer

The intransigence of dispersed flexographic ink to conventional alkaline flotation deinking operations is well-documented within the fiber recovery industry. In addition to diminutive sizes that preclude adhesion at the air bubble surface, dispersed flexographic ink particles are characterized by remarkable electrosteric colloidal stabilities that resist the formation of more floatable aggregates.

Although with furnishes composed of oil-based offset ONP and OMG, surfactant spray flotation has been shown to significantly reduce fiber loss, water loss and frother consumption while maintaining brightness gain levels in a laboratory-, pilotand mill-scale flotation cells comparable to those obtained through conventional flotation. This part documents our attempt to extend the surfactant spray technology to flexographic ink deinking. Unfortunately, like conventional flotation, pulp brightness using surfactant spray technology is adversely affected when the percentage of flexo ONP within the feed exceeds 5%. However, a unique combination of siloxane-based defoaming agent and calcium chloride was found to boost flotation deinking efficiency of furnishes comprised of 100% flexo ONP (Fig. 4). Moreover, yield losses were minimal compared to those associated with calcium-fatty acid soap deinking chemistry. Such technology may enable flotation deinking operations to increase the proportion of flexographically-printed material within their feed without sacrificing brightness or yield.



Fig. 4. Comparison of surfactant spray flotation efficiency between TDA-32/calcium system and commercial deinking additives (\Box =200 ppm TDA-32/CaCl₂ curve, \blacktriangle =50 ppm Sansink[®], \bullet =25 ppm Vinings A).

Mechanism study regarding the flexographic ink removal improvement through the surfactant spray flotation deinking incorporating siloxane-based defoamer shows the ability of defoamer to increase the brightness of a pulp consisting of 100% flexographic ONP is a direct function of calcium chloride concentration during flotation. Initial investigation into the fundamental mechanism of this electrolyte-dependent phenomenon indicates that the collection of dispersed flexo ink is not the result of specific chemical interactions between calcium and defoamer or between calcium and the ink particle. Rather, both components must be present in order to initiate flexo ink flotation. Since the defoamer in the pulp precludes formation of a stable froth, surfactant spray technology represents an indispensable means to generate and maintain a foam layer for retention of floated ink. Ongoing investigation is anticipated to reveal the fundamental mechanism of flexo ink flotation in the presence of defoamer and calcium chloride.

4. Deinking Selectivity (Z-Factor): A New Parameter to Evaluate the Performance of Flotation Deinking Process

This study also proposed the deinking selectivity concept that takes both ink removal and fiber yield into consideration in determining the performance of deinking operations. A Z-factor, which can be used for analyze the ratio of ink removal to fiber loss in a flotation was developed. Typical brightness Z-factor is on the order of unit value, while ERIC Z-factor is on the order of 10 units, for most flotation processes. Therefore, the Z-factor weighted brightness gain and ERIC reduction has relevance to the ISO brightness and ordinary ERIC reduction. This study demonstrated that the Z-factor weighted brightness gain and ERIC reduction are good indicators of deinking process performance through conducting pilot scale flotation deinking experiments. The period or stage Z-factors are good indicators of the efficiency of the period or stage of a deinking process. A simple criterion was developed using the stage Zfactor concept and applied to both pilot scale experiments and an industrial recycling mill operation for the determination of the economics of a given period/stage in flotation deinking operations. It was found that surfactant spray technique has the largest Z-factor, which suggests high effectiveness of surfactant spray technology in flotation deinking. Therefore, the deinking selectivity concept defined in this study is useful and has economic importance in deinking operations.

We applied the stage Z-factor concept to a mill flotation deinking operation to determine the efficiency of each flotation stage at the mill. To conduct this exercise,

we sampled the feed and the accept stock of different stages of a production line that has seven stages in series. Determination the reject flow rate of each stage was not possible and was not attempted. Therefore, fiber loss was estimated from the consistency of the stock in each stage. The results indicate that the consistency decreases linearly across the seven stages. Therefore, constant fiber loss of 1/7 of the total fiber loss determined from the consistencies of the feed and final accept stock was used to determine the fiber loss of each stage. Handsheets from then sampled pulps were prepared to measured the brightness and ERIC of then deinked fibers. From the brightness and ERIC data along with the estimated fiber loss through each stage, we determined the deinking selectivity, or Z-factors of each stage. As listed in Table 2, both the brightness and ERIC Z-factors decrease exponentially across the seven stages due to exponential decay of ink removal through the stages downstream. We then calculated the required pulp price gain for economical operation of each stage. The results indicate that the last two stages are not economically justified according to this sampling exercise. The required pulp price gain per unit brightness gain was over 10%, while the required pulp price gain for each percent ERIC reduction for the last stage to be economical is 27%. This exercise demonstrates the practical importance of the deinking selectivity or Z-factors defined in this study.

	Brightness	ERIC	Consistency	Z _{Bi}	Z _{Ei}	P/P (%) per unit ISO GB	P/P (%) per percent RE
Feed	44.17	1177	0.0087				
Stage 1	51.27	574.2		6.179	44.574	0.16	0.02
Stage 2	53.32	428.6		1.784	10.766	0.56	0.09
Stage 3	54.68	363.1		1.184	4.843	0.84	0.21
Stage 4	55.51	303.4		0.722	4.414	1.39	0.23
Stage 5	56.11	273.3		0.522	2.225	1.92	0.45
Stage 6	56.19	266.1		0.070	0.533	14.29	1.88
Stage 7	56.29	265.6	0.0080	0.087	0.037	11.49	27.03
Process	56.29	265.6		1.506	9.624		

Table 2. Deinking performance of an industrial flotation operation with 7 stages

Project Summary

This study demonstrated the foaming agent spray concept to achieve separate control of the application of deinking chemicals, i.e., foaming agent and collector, in flotation deinking of waste paper. The foaming agent spray concept was first demonstrated in batch flotation experiments using conventional deinking chemistry (foaming agent and collector blended) to deink old newsprint paper (ONP) and a mixture of ONP with old magazine paper (OMG), respectively, in a Voith Sulzer flotation cell with 18-liter capacity (E-18). . A defoamer was used to suppress the foam produced by the blended deinking chemicals applied during pulping, then regenerated at the top layer of the flotation pulp suspension using a foaming agent spray at the top of the flotation cell. The concept was also demonstrated without the application of a defoamer when an low foamability collector was applied during pulping The results indicate that water spray can wash down some fibers entrapped in the bubble network of foam and reduce fiber loss in flotation, but there is an optimum water spray loading; spraying too much water can cause too fast an overflow of foams, resulting in high fiber loss due to fiber entrainment. The results also indicate that froth produced by spraying a foaming agent from the top of the flotation cell reduced fiber entrainment and therefore fiber yield loss under equivalent ink removal. Typical fiber loss reduction of 50% was achieved at a brightness gain around 9 ISO% in deinking ONP in an E-18 cell and in deinking an ONP and OMG mixture in scale-up experiments using Voith Sulzer flotation cell E-250 of capacity of 250-liter. Equivalent dirt counts on handsheets made of deinked fibers were achieved between frothing agent spray deinking of a mixture of ONP and OMG with low foaming agent TDA-32 and conventional deinking of the same furnish with fatty acid, but the former gave much lower fiber loss. By comparing the experiments in the E-18 cell and E-250 cell in flotation deinking a mixture of ONP and OMG, scale-up of the frothing agent spray concept was successfully demonstrated in the laboratory.

The ability of surfactant spray technology to reduce yield loss without detriment to pulp brightness gains has also been successful transferred to a single flotation unit within the deinking line of a mill producing newsprint from 100% secondary fiber.

Initial results suggest that the loss of fiber across the unit may be reduced by more than 50% without obvious detriment to final pulp quality.

This study also proposed the deinking selectivity concept that takes both ink removal and fiber yield into consideration in determining the performance of deinking operations. A Z-factor, which can be used for analyze the ratio of ink removal to fiber loss in a flotation was developed. Typical brightness Z-factor is on the order of unit value, while ERIC Z-factor is on the order of 10 units, for most flotation processes. Therefore, the Z-factor weighted brightness gain and ERIC reduction has relevance to the ISO brightness and ordinary ERIC reduction. This study demonstrated that the Z-factor weighted brightness gain and ERIC reduction are good indicators of deinking process performance through conducting pilot scale flotation deinking experiments. The period or stage Z-factors are good indicators of the efficiency of the period or stage of a deinking process. A simple criterion was developed using the stage Zfactor concept and applied to both pilot scale experiments and an industrial recycling mill operation for the determination of the economics of a given period or stage in flotation deinking operations. It was found that surfactant spray technique has the largest Z-factor, which suggests high effectiveness of surfactant spray technology in flotation deinking.

This study also demonstrated the feasibility of using foaming agent spray technique for deinking waste flexographic papers. An effective collector chmistry was developed for deinking flexographic papers. Excellent ink removal were achieved in laboratory studies.

Project Objective

The following objectives were proposed in our original proposal. After four-year study, we successfully accomplished all objectives.

- 1. Conduct pilot scale demonstration of the proposed technology for flotation deinking of mixed office wastepaper.
- 2. Develop collector chemistry for flotation deinking of newsprint, flexographic printings.
- 3. Develop improved understanding of froth fluidynamics in relation to fiber entrapment, water drainage, and ink removal to optimize the performance of the proposed technology for deinking of various grades of paper.
- 4. Conduct pilot scale demonstration of the proposed technology for flotation deinking of newsprint and flexographic printing papers.
- 5. Conduct mill-site demonstration and seek commercialization of the technology with vendors.

General Background

In conventional flotation deinking, all the chemicals including frothing agent, collector, and dispersant are directly added to pulp suspension during stock preparation. There is little control on chemical application once the stock is prepared, which could affect the process runnability. It is well known that frother must be used in flotation deinking to stabilize the froth for ink removal. The proposed innovative flotation deinking process uses a spray to deliver the frothing agent during flotation deinking to control several key process variables. The spray can control the froth structure and modify the fluid dynamics to reduce the fibers entrapped in the froth layer. The froth formed at the top part of the flotation column will act as a physical filter to prevent the penetration of frothing agent into the pulp suspension to eliminate fiber contamination and unfavorable deinking surface chemistry modification due to surfactant adsorption on the fiber surface. Because of the filter effect, frothing agents will be better utilized.

Based on the fundamental understanding of ink removal and fiber loss mechanism in flotation deinking process, we patented a technology of surfactant spray in 1998 to improve the ink removal efficiency, reduce the water and fiber loss, reduce the chemical consumption and carry over in the flotation deinking. The foam stability and structure in the flotation deinking process can also be easily controlled by this innovative technology.

Under the sponsorships of the US Dept. of Energy (DOE) and the member companies of the Institute of Paper Science and Technology, we studied the chemmechanical mechanism of surfactant spray for flotation deinking using different furnishes, chemicals, and flotation devices in the past four years. In the final year of the project, we successfully conducted mill trials at Abitibi-Consolidated, Inc., Snowflake paper recycling operation of 100% mixture of ONP/OMG. Results from laboratory, pilot-plant and mill trials indicated that surfactant spray technology can

significantly reduce fiber loss in flotation deinking. It can be concluded that paper industry can profit greatly when this technology is commercialized in flotation deinking mills.

This final report was divided into the following four parts. Each part has it own's introduction, experimental, results and discussion as well as conclusions.

Part I: Labtororal and Pilot Study of ONP and OMG Deinking Using Surfactant Spray

Part II: Reducing Fiber Loss in Laboratory- and Mill-scale Flotation Deinking Using Surfactant Spray Technology

Part III: Deinking Selectivity (Z-Factor): A New Parameter to Evaluate the Performance of Flotation Deinking Process

Part IV: 100% Flexographic ONP Deinking Using Surfactant Spray In the presence of siloxane-based defoamer

Part I: Laboratory and Pilot Study of ONP and OMG Deinking Using Surfactant Spray

The initial demonstration of the foaming agent spray concept was carried out in a column flotation cell using an ideal furnish, i.e., pure toner-printed papers with relatively large and hydrophobic particles that are floated easily without adding collectors. It was found from the previous investigation that fiber loss was reduced by 50% while the application of foaming agent was reduced by 95% when spray was applied through a spray on top of a column flotation cell in deinking toner-printed papers in a laboratory study. However, it is unclear how this concept can be applied to other deinking systems in that a collector must be used. For example, offset-ink particles in old newsprint (ONP) are different from toner particles in copy papers. Copy-toner particles can be effectively floated by flotation without a collector, but the offset-ink particles are difficult to float if no collector is applied.

The objective of the present study is to verify the viability of the foaming agent spray concept in the deinking of old newsprint (ONP) printed with offset ink and its mixture with old magazine paper (OMG) in the presence of an effective collector using laboratory and pilot-scale flotation deinking cells.

INTRODUCTION

Paper recycling is becoming increasingly important due to the shortage of fiber sources and restrictive environmental regulations on paper landfills. Effective and innovative separation technologies in paper recycling can help to improve the quantity and quality of secondary fibers in the marketplace. Currently, the quality of recycled fibers is not comparable to virgin fibers, and the cost of secondary fibers is high because of the lack of new technologies. Flotation deinking is a common practice to remove ink from waste paper in many paper recycling mills. Despite the modest success achieved in using the flotation technique for ink removal in paper recycling since its introduction in the 1980s, existing technologies and process designs of flotation deinking are based on experiences obtained from mineral flotation processes and have not been optimized for deinking operations. For example, high secondary fiber loss of 8-20% and fiber contamination that causes poor fiber bonding and paper-machine foaming problems through direct contact with surfactants in flotation deinking are typical in the practice of flotation deinking. Furthermore, flotation is still not effective in removing inks with very small ink particles (~1 μm), such as flexographic inks.

The chemistry of the flotation process has been reviewed in the literature [1-3]. A foaming agent is used to generate a stable foam for ink removal, a collector may be applied to agglomerate small ink particles for flotation removal, and a dispersant is used to avoid ink particles being redeposited onto the fiber surface. The foaming agent plays a detrimental role in the flotation deinking process. It is understood that a foaming agent (manually added or contained in waste papers) must be present in flotation deinking to stabilize the foam for ink removal. Furthermore, there is an optimum foaming agent concentration in fiber suspension for ink removal as

observed by Epple et al. [4] and in our previous study [5, 6]. Moreover, foam produced by foaming agents in flotation deinking is the major cause of fiber loss through the "fiber entrapment (in the foam)" mechanism, as indicated by recent studies [7-10]; therefore, foam stability is directly related to the fiber loss. In general, if the foam structure and other conditions are the same, when more foam is generated, more fibers are entrapped in the bubble network of the foam. In a conventional flotation deinking process, the deinking chemicals, including foaming agent, are directly added into the pulp suspension during stock preparation. There is little control of chemical application once the stock is prepared, which will result in chemical carry-over to the paper machine.

There are some conflicting requirements in optimizing the flotation deinking process in terms of high ink removal efficiency and less fiber loss, e.g., (1) ink particles should be hydrophobic, but the fiber surface should be hydrophilic and (2) more foam is needed to increase the ink removal rate, but less foam is needed to reduce fiber loss. To solve these problems, an effective collector for ink particles should be used, and the foam should be controlled in a way that does not affect ink removal but can reduce the fiber entrapment. It is imperative to separately control ink hydrophobicity, ink removal efficiency, and fiber entrapment in the froth.

In surfactant spray flotation deinking, the foaming agent is sprayed from the top of the flotation cell, rather than directly added into the pulp suspension during stock preparation. The rationale of the foaming agent spray concept is that the foaming

agent is only used to stabilize froth. Therefore, it would be much more effective to apply it where foam needs to be stabilized. This also avoids the dilution of the foaming agent by the bulk volume of the pulp suspension and the reduction in the hydrophobicity of the ink particles due to the adsorption of foaming agent on the ink particle surface. It was found that fiber loss was reduced by 50% while the application of foaming agent was reduced by 95% when spray was applied through a spray on top of a column flotation cell in deinking toner-printed papers [6] in a laboratory study. It was believed that the reduction of fiber loss was due to better control of foam stability and the spray washing effect that washed down the fibers entrapped in the foam, as evidenced by Robertson et al. [11] in their experiments on foam washing during flotation.

The objective of the present study is to verify the viability of the foaming agent spray concept in the deinking of old newsprint (ONP) printed with offset ink and its mixture with old magazine paper (OMG) in the presence of an effective collector using laboratory and pilot-scale flotation deinking cells.

In case there was too much foam when commercially available deinking chemicals were used, a two-step strategy for controlling foaming without affecting ink removal was developed. In the first step, a defoamer is added to the system to break the foam. In the second step, a foaming agent is sprayed into the system to regenerate a controlled foam layer again. The concept for this two-step process is that by adding defoamer to reduce the foam stability and foaming ability, the fiber entrapment and fiber loss should be reduced. However, adding defoamer will reduce

the ink removal efficiency. Therefore, a frothing agent is sprayed from the top of the flotation cell to retain enough foam for ink removal. It should be noted that the foam created in the pulp suspension is different from the foam created on the top using surfactant spray. The foam generated in the pulp suspension will entrap fibers when they float to the surface, causing a high fiber loss. However, the foam generated at the top of the flotation cell by spraying froth solution will be concentrated on the top only, and there is much less probability that it will entrap fibers. Therefore, the fiber loss can be reduced, and ink removal efficiency can be improved. This new concept will be tested in this study. Several commercially available deinking chemicals were used in the study. The goal of this research is to bring the foaming agent spray concept a step closer to practical application.

EXPERIMENTAL

Chemicals

Triton X-100 (analytical grade, C8Ph(EO) 10; Ph = phenyl), Triton X-15 [C8C6(EO)15, analytical grade, J. T. Baker Inc.], TDA-32 (Taylor Chemical Company, a polydimethylsiloxane-based deformer, 65% solid), Vinings A (Vinings Industries, mixture of nonionic surfactant and fatty acids, Marietta, GA), oleic acid (Aldrich Tech, 90%), and calcium chloride (Aldrich Tech) were used as received.

Pulping

All the pulping experiments were conducted using a LAMORT pulper. ONP of The Atlanta Journal and Constitution and OMG (collected from an office) were used in this study. For pure ONP pulping, ONP was soaked in hot water (50oC) for 10 min before pulping. For ONP/OMG, OMG was soaked in hot water (50oC) for 2 hours,

and then ONP was mixed with OMG and soaked for 10 min before pulping. Pulping consistency was 8%. After pulping 1 min, 0.8% sodium silicon and 1% sodium hydroxide (both on dry paper weight) were added to the pulp, and then pulping continued for another 9 min. The pulping temperature was 50oC. Deinking chemicals other than the foaming agent were added at the beginning of the pulping process.

Flotation Cells

Two flotation cells, E-18 and E-250, were used to conduct deinking experiments. Both flotation cells were designed by Voith-Sulzer (Appleton, WI). The E-18 flotation cell was made in house according to the manufacture design. The cell has a capacity of 18 L and was operated in a closed circuit. The E-250 cell with a capacity of 250 L was purchased from Voith-Sulzer. Both cells are geometric scaled-down versions of Voith-Sulzer's commercial flotation cells. However, the E-18 cell does contain a control device for air entrainment. The E-250 cell more resembles the commercial cell than the E-18 cell. Pure ONP flotation deinking experiments were conducted in the E-18 cell only. ONP/OMG (70:30) flotation deinking experiments were conducted using both the E-18 and E-250 flotation cells.

Flotation Conditions

Conditions for flotation in the E-18 flotation cell were: pulp consistency: 1%, time: 10 min, temperature: 43°C, air flow rate: 30 SCFH (standard cubic feet per hour). The volume fraction of air was 2.9%. For flotation in the E-250 flotation cell, conditions

were: consistency: 1%, time: 10 min, temperature: 43°C, air flow rate: 60 SCFM (standard cubic feet per minute). The volume fraction of air was 4.0%.

Preparation of Handsheets

4.0-g filter pads were made using related Buchner funnel methods (PAPTAC Standard C.4U). For pure ONP pads, 2.5 mg cationic polyacrylamide was added prior to pad formation to reduce the sidedness of the pad.

Surfactant Spray Conditions

Bottled compressed air was used to drive the pressure swirl atomizers. The design flow rate of the atomizers (Delavan, Inc., Des Moines, IA) is 1.5 gallon per hour (GPH). A TX-100 solution, concentration 100 mg/L, was used as the surfactant spray solution. Two atomizers were used in the E-18 cell. The atomizers were separated by 97 mm and formed a row 50 mm away from the aeration tube and perpendicular to the reject flow direction. Nine atomizers were used in the E-250 cell in three rows separated by 102 mm between rows. The first row was about 270 mm away from the aeration tube. Each spray covers an area of about 20 cm², or a diameter of 5 cm. The atomizers were mounted 8 cm above the suspension surface. Two parameters were used to measure the amount of spray applied in this study. The spray flow loading fraction is the total amount of liquid spray to the flotation cell in a given flotation period (10 min in this study) to the total volume of the flotation cell. Spray loading is the volume of spray applied per unit time on a unit surface area of the flotation cell open surface.

Measurement of Brightness, Fiber Loss, Water Loss, and Dirt Counts

Brightness was analyzed by TAPPI standard method T452 om-92 [12]. Fiber loss was calculated using the ratio of fiber (oven-dried) in the rejects to the amount of fiber at the beginning of flotation. Water loss was calculated using the ratio of water in the rejects to the water in the flotation cell before flotation.

The level of visible dirt on the handsheet made of recycled fibers was measured by dirt count by TAPPI standard method T563 om-96 [13]. The equivalent black area (EBA) as the fraction (in parts per million, ppm) of the sample size is reported in this paper.

RESULTS AND DISCUSSION

The Effect of Water Spray Washing of Foam on Flotation Deinking Performance

Robertson et al. [11] indicated that foam washing by water spray can prevent fiber loss during flotation of pulp suspension. Our early flotation deinking study [6] using foaming agent spray indicated that fiber loss can be reduced. To further verify the washing effect of a water spray on fiber loss reduction in flotation deinking, we conducted a series of conventional flotation experiments for 100% ONP using an E-18 flotation cell. Vinings A, which is commonly used as an ONP deinking chemical, was used in these tests. The concentration of Vinings A in the pulp suspension was 50 mg/L, and flotation time was 10 min for this set of tests. Flotation experiments were conducted with and without water spray. The water flow loading, defined as a fraction of total water in the flotation cell, was varied.

Unlike the work by Robertson et al. [11] that only measured fiber loss but did not study the effect of ink removal by water spraying, we measured both the fiber loss and ink removal simultaneously in this study. Figure 1-1 shows the fiber loss and ink removal as a function of water flow loading fraction. For comparison purposes, Figure 1-2 shows the ratio of brightness gain to fiber loss from the same data. As can be seen from Fig. 1-1, fiber loss decreases initially with water spray and then increases with the increase of water flow loading. Brightness gain is increased with the increase of water flow loading. It is believed that the reduction of fiber loss at low water spray flow loading can be attributed to the washing effect of the foam by the spray. However, it was observed that increasing the water spray flow rate also increased the foam removal rate (fast overflow of the foam from the top of the flotation cell). Because overflow is the only mechanism to remove foam in the E-18 cell, the increased foam removal increases the removal of both ink and fibers entrapped in the foam. As can be seen from Fig. 1-2, there is an optimal spray water flow loading fraction at which deinking performance is optimized in terms of brightness gain per percent of fiber loss, or deinking selectivity, which is 0.82. The optimal spray water flow loading fraction was 0.056, or 5.6%, or in terms of spray flow rate, 0.83 mL/s.



Fig. 1-1. The effect of water spray loading on brightness gain and fiber loss in flotation deinking of 100% ONP in an E-18 flotation cell.



Fig. 1-2. The effect of water spray loading on deinking selectivity in flotation deinking of 100% ONP in an E-18 flotation cell.

Demonstration of Separate Applications of Foaming Agent and Collector in Deinking ONP

The optimized water flow loading (5.6%) from the above experiment was used for all the foaming agent spray experiments with E-18 cell deinking of ONP. TX-100 solution with a concentration of 100 mg/L was again used as the spray solution. We varied the concentration of Vinings A in the pulp suspension from 5-50 mg/L. Figure 1-3 shows the deinking results of foaming agent spray, water spray, and conventional (without any spray) flotation deinking. A linear logarithmic function fits to the data of brightness gain with fiber loss very well. The results indicate that water spray improved deinking efficiency when compared to those results obtained without any spray (conventional). The brightness gains of pulp from water spray deinking are

higher than those obtained from conventional deinking under the same fiber loss for all the experiments conducted. However, the results obtained from foaming agent spray experiments showed no improvement over those from conventional flotation deinking in a wide range of Vinings A dosages. This is because Vinings A is a deinking chemical that acts as a collector and foaming agent as well. When it was added to the pulp slurry, it generated enough foam for flotation ink removal, so there is no need to spray extra foaming agent into the top of the flotation cell. However, foam that is too stable will increase the fiber entrapment, so any benefit from foaming spray was offset by the increase in fiber loss caused by the increased stability of the foam.



Fig. 1-3. Comparisons of flotation deinking efficiencies between conventional, water spray, and surfactant spray method using Vinings A as a collector in deinking of 100% ONP in an E-18 cell.

As we discussed previously, the rationale behind the foaming agent spray concept is to realize separate application and control of various deinking chemicals. In deinking of toner-printed papers, only the foaming agent and no other deinking chemicals are needed; therefore, the foaming agent spray concept can be easily demonstrated [6]. Most commercial chemicals play several roles; for example, Vinings A is a blend of different surfactants with different HLB numbers, which can act as collector and frother. To improve the ink removal efficiency, an effective collector should be used. Because Vinings A is widely used as an ONP deinking chemical in deinking mill practice and it also contains an effective collector, this chemical was used in this study for ONP deinking. However, in order to reduce the fiber entrainment in the foam network, the role of foaming agent played by Vinings A must be suppressed, and a relatively stable foam layer should be stabilized by a foaming agent sprayed from the top of the flotation cell. It should be emphasized that the foam generated at the surface of the pulp slurry is different from that generated inside of the pulp slurry, i.e., the fibers are more easily entrapped in the foam generated inside of the pulp slurry, but less likely to be entrapped by the foam stabilized at the surface.

In order to depress the foam generated but keep the collector function of Vinings A, a defoamer (TX-15) was added to the pulp slurry to depress the foaming ability of Vinings A before flotation. It was found that the foam was reduced with the increased amount of TX-15. However, complete suppression of foam was not obtained by using TX-15 as a defoamer. Figure 1-4 shows the effect of the amount of TX-15 applied on ink removal in terms of brightness gain and fiber loss at a fixed amount of Vinings A

(50 mg/L). The results indicate that with the increase of TX-15 the fiber loss decreased rapidly and then leveled out, while brightness gain increased slightly (possibly due to increased foam removal rate initially by the spray) and then decreased due to a reduced foam removal rate when the foam was less stable at high TX-15 dosage applications. The total reduction of fiber loss is more than 50% or 6 percentage points at a TX-15 application of 1 mg/L. There is also an optimal TX-15 dosage of 1 mg/L at which deinking selectivity (brightness gain per percent fiber loss) is maximized (1.67) as shown in Fig. 1-5, which is a 100% increase over that of water spray flotation deinking. It can be concluded that the application of defoamer TX-15 suppresses foaming by Vinings A, and the foam is restabilized by a foaming agent spray of TX-100, causing deinking selectivity to more than double. In the meantime, brightness gain is increased by 10% compared to that obtained in conventional flotation deinking shown in Fig. 1-3, demonstrating the foaming agent spray concept for flotation deinking of ONP.



Fig. 1-4. The effect of the application of defoamer T-15 on flotation deinking efficiency in surfactant (TX-100) spray deinking of 100% ONP using Vinings A as a collector.



Fig. 1-5. The effect of the application of defoamer T-15 on flotation deinking selectivity in surfactant (TX-100) spray deinking of 100% ONP using Vinings A as a collector

Demonstration of the Foaming Agent Spray Concept in Deinking an ONP and OMG Mixture

An ONP and OMG mixture is one of the most widely processed furnishes in paper recycling mills. The mixture ratio of ONP over OMG varies from 0.6:0.4 to 0.95:0.05. However, most mills use an ONP and OMG mixture ratio around 0.7:0.3. In this study, an ONP and OMG mixture ratio of 0.7:0.3 was used. Because Vinings A is a commercially blended mixture of collector and frother, it is not suitable for realizing true frothing spray deinking. A low foaming agent TDA-32 (polysiloxane oil in water emulsion) was used as collector and directly applied in the pulp suspension. A foam layer was generated at the top of the flotation cell by spraying TX-100 solution of concentration 100 mg/L. Flotation deinking experiments were first conducted in the E-18 cell. After the successful demonstration of the frother spray concept in the E-18 cell. Therefore, scale-up of the flotation experiments were conducted in the E-250 cell. Therefore, scale-up of the frother spray concept can be observed in laboratory studies. Conventional flotation deinking experiments using commercial chemicals of Vinings A and fatty acid soap were also conducted in both flotation cells for comparison.

Table 1-1 lists a typical comparison of deinking performance between the frothing agent spray concept and conventional flotation using two commercially blended chemicals of Vinings A and fatty acid soap. The concentration of the deinking chemicals, i.e., Vinings A, fatty acid soap, TDA-32, directly applied in the pulp suspension was 30 mg/L. A total of 72 mg of frothing agent, TX-100, or 0.72 liter of the solution (4% of the total suspension volume in the flotation cell), was applied

through the atomizers on top of the flotation cell in 10-min flotation. The results indicate that fiber loss was reduced by about 36% in frothing agent spray flotation deinking when compared to conventional flotation deinking. The brightness gain of the deinked paper in the frothing agent spray case was slightly lower than that obtained in conventional flotation. However, deinking selectivity of frothing agent spray flotation deinking. This set of experiments demonstrates the success of the frothing agent spray concept for deinking of a typical furnish, i.e., a mixture of ONP and OMG, used in industry practice.

Table 1-1. Comparisons of the performance of flotation deinking of a mixture of OMG and ONP using a conventional flotation process with collectors Vinings A and fatty acids and the frothing agent spray technique in the E-18 cell with a low foaming collector TDA-32.

Experimental	Deinking	Brightness	Fiber Loss	Brightness/Fi	Water Loss
Process	Conditions	Gain (ISO%)	(%)	ber Loss	(%)
				(Selectivity)	
Conventional	Fatty Acid @	9.9	18.9	0.52	18.5
	30 mg/L				
Conventional	Vinings A @	10.1	19.0	0.53	17.6
	30 mg/L				
Foaming	TDA-32 @ 30	7.8	11.9	0.66	9.8
Agent Spray	mg/L + Spray				
	72 mg TX-				
	100 @ 100				
	mg/L				

The results presented so far were obtained in an E-18 cell with a capacity of 18 L where the turbulence level and flow rate are significantly lower than those in industrial flotation cells. To observe the performance of the frothing agent spray deinking concept in scale-up experiments in the laboratory, flotation deinking experiments of an OMG and ONP mixture were also conducted using a Voith-Sulzer E-250 cell. The E-250 cell is a geometric scale-up of the E-18 cell by design and resembles Voith-

Sulzer's commercial flotation cells. The concentration of the deinking chemicals, i.e., Vinings A, fatty acid soap, TDA-32, directly applied in the pulp suspension, was 50 mg/L. A total of 450 mg of frothing agent, TX-100, or 4.5 liter of the solution (1.8% of the total suspension volume in the flotation cell), was applied through the atomizers on top of the flotation cell in 10-min flotation. Figure 1-6 shows the comparison of deinking performance between frothing agent spray flotation deinking with low foaming collector TDA-32 and conventional flotation deinking using commercial chemicals Vinings A or fatty acid soap. The results indicate that the frothing agent spray deinking process shows substantial improvement over conventional deinking in terms of absolute brightness gain under the same fiber loss and deinking selectivity over a wide range of dosages of deinking chemicals.



Fig. 1-6. Comparisons of flotation deinking efficiencies in deinking a mixture of OMG and ONP using conventional flotation with collectors Vinings A and fatty acids and the surfactant spray technique with a low foaming collector TDA-32 in the E-250 cell.
Table 1-2 shows the detailed comparisons of deinking performance of three experiments. These experiments were chosen because they give equivalent brightness gains for comparison purposes. The result indicates that fiber loss and water loss (or rejects) are significantly lower in foaming agent spray flotation deinking than those found in conventional flotation deinking. The deinking selectivity for the foaming agent spray flotation deinking using fatty acid and Vinings A, respectively. Furthermore, the dirt counts on the handsheet made from deinking fibers through the foaming agent spray process are equivalent to those obtained through conventional flotation using fatty acid soap. The dirt counts of the handsheet made from deinking fibers through the fibers through Vinings A are much higher than the handsheet made from deinking there are the foaming agent spray process.

Table 1-2. Comparisons of the performance of selected flotation deinking experiments in deinking a mixture of OMG and ONP using a conventional flotation process with collectors Vinings A and fatty acids and the frothing agent spray technique in the E-250 cell with a low foaming collector TDA-32.

Experimental	Deinking	Brightness	Fiber	Brightness/	Water	Dirt
Process	Conditions	Gain	Loss (%)	Fiber Loss	Loss (%)	Counts
		(ISO%)		(Selectivity)		$(/m^2)$
Conventional	Fatty Acid @	11.19	21.57	0.52	27.43	16.9
	50 mg/L					
Conventional	Vinings A @	9.94	21.28	0.47	26.68	185.3
	50 mg/L					
Foaming	TDA-32 @ 30	9.64	11.11	0.87	8.27	18.9
Agent Spray	mg/L + Spray					
	450 mg TX-					
	100 @ 100					
	mg/L					

The results shown in Table 1-1 and 1-2 demonstrate the successful scale-up of the frothing agent spray flotation deinking concept in deinking of an ONP and OMP mixture in the laboratory. The experiments also indicate that less spray is required on the basis of spray application per unit volume of suspension in the scale-up experiment using the E-250 cell.

CONCLUSIONS

Separate control of the application of deinking chemicals, foaming agent and collector, was achieved using the frothing agent spray concept in two commercial flotation cells in deinking ONP and a mixture of ONP and OMG. The results indicate that water spray washing of foam can wash down some fibers entrapped in the bubble network of foam and reduce fiber loss in flotation, but there is an optimum water spray loading; spraying too much water can cause too fast an overflow of foams, resulting in high fiber loss due to fiber entrainment. The results also indicate that the combination of using an effective collector, adding a defoaming agent in the furnish to destroy the foam produced by commercial deinking chemicals inside of the slurry to reduce the fiber entrainment, and spraying a foaming agent from the top of the flotation cell to regenerate a foam layer for ink particle removal can significantly improve flotation deinking performance and reduce the fiber loss. Typical fiber loss reduction of 50% was achieved at a brightness gain around 9 ISO% in deinking ONP in an E-18 cell and in deinking an ONP and OMG mixture in an E-250 cell. Equivalent dirt counts on handsheets made of deinked fibers were achieved between frothing agent spray deinking of a mixture of ONP and OMG with defoaming agent TDA-32 and conventional deinking of the same furnish with fatty acid, but the former gave much lower fiber loss. By comparing the experiments in the E-18 cell and E-250 cell in flotation deinking a mixture of ONP and OMG, scale-up of the frothing agent spray concept was successfully demonstrated in the laboratory.

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PART II: 100% FLEXOGRAPHIC ONP DEINKING USING SURFACTANT SPRAY IN THE PRESENCE OF SILOXANE-BASED DEFOAMER

Although it was demonstrated in our previous studies that fiber loss was reduced by up to 50% without affecting the ink removal efficiency when surfactant was sprayed on top of a flotation cell in deinking toner-printed papers in a bench scale column flotation cell and in deinking offset old newspapers (ONP) and old magazine (OMG) in a laboratory- and pilot-scale commercial flotation cells, this part documents our attempt to extend the surfactant spray technology to flexographic ink deinking.

INTRODUCTION

The intransigence of dispersed flexographic ink to conventional alkaline flotation deinking operations is well-documented within the fiber recovery industry. In addition to diminutive sizes that preclude adhesion at the air bubble surface, dispersed flexographic ink particles are characterized by remarkable electrosteric colloidal stabilities that resist the formation of more floatable aggregates (1).

In an effort to maintain required brightness levels in recovered pulp, recycling operations must exercise vigilance to ensure that the feed stream contains extremely small percentages of flexo printed material (≤5%) (2). Currently, flexographic printing of newspapers is primarily confined to North America, Italy and England (3). However, within these countries, conversion to flexo printing is anticipated to increase as flexographic polymer plates become more available and affordable. In fact, in late 2002, News Corporation, the UK-based publisher of such wide circulation dailies as the Sun, News International and Times, was assessing the feasibility of flexography within its pressrooms (paperloop.com).

Numerous attempts to improve the floatability of flexo ink with varying degrees of success are described within current literature. One particularly promising approach involves neutral or slightly acidic flotation of the pulp followed by a traditional alkaline flotation stage (4, 5). Unionized flexo ink is effectively removed during the former stage while oil-based offset inks are preferentially floated during the latter. Unfortunately, this two-pronged approach would appear to necessitate significant modification to existing operations.

Revision of traditional flotation chemistry appears to be a more popular means to augment flexo flotation. Addition of polymeric flexo "collectors" (6, 7), flexo adsorbing organoclays (8, 9, 10) and lignosulphonates (11) to the pulper and/or flotation cell have been purported to enhance ink removal. A hybrid technology employing both equipment modification and novel chemistry may exploit the benefits afforded by both.

Surfactant spray deinking technology was recently patented as an economical means to moderate the substantial loss of fiber and water during conventional operations (12). In brief, the technique involves spraying the frothing agent onto the surface of the active cell rather than mixing with the pulp prior to flotation. Concentrating the frother at the surface of the cell minimizes instances in which fiber hydrophilicity and contaminant hydrophobicity are compromised through nonspecific adsorption of amphiphilic surfactant molecules. Although not as detrimental as physical entrainment, such interactions contribute to the amount of floated fiber within reject streams (13, 14). With furnishes composed of oil-based offset ONP and OMG, surfactant spray flotation has been shown to significantly reduce fiber loss, water loss and frother consumption while maintaining brightness gain levels comparable to those obtained through conventional flotation.

Unfortunately, like conventional flotation, pulp brightness using surfactant spray technology is adversely affected when the percentage of flexo ONP within the feed exceeds 5%. However, a unique combination of siloxane-based defoaming agent

and calcium chloride was found to boost flotation deinking efficiency of furnishes comprised of 100% flexo ONP. Moreover, yield losses were minimal compared to those associated with calcium-fatty acid soap deinking chemistry. Such technology may enable flotation deinking operations to increase the proportion of flexographically-printed material within their feed without sacrificing brightness or yield.

EXPERIMENTAL

Materials

Triton X-100, sodium hydroxide, sodium silicate, anhydrous calcium chloride (Sigma-Aldrich) and sodium oleate (J.T. Baker) were used as received. Samples of commercial flotation deinking additives included: Sansink[®] (BASF Corp.) and Vinings A (Vinings Industries, Inc., now Kemira). TDA-32 (Taylor Chemical Company) was selected as a representative siloxane-based defoaming agent while ECCO P-048 (Eastern Color and Chemical) would serve as a non-siloxane defoaming agent. All commercial additives were acquired directly from the manufacturer and used without modification.

All pulps were prepared from 100% flexographic-printed ONP furnish (The Knoxville News-Sentinel, Knoxville, TN). Sufficient 50°C water was added to an air dry sample of the ONP to ensure an 8% consistency within the pulper. The paper was allowed to soak for 10 minutes prior to pulping. After this time, sodium hydroxide and sodium silicate were added to the soaking paper (respective charges of 1% and 0.8% based on dry paper weight). The mixture was then pulped for 10 minutes within a helical Lamort pulper (France). At the end of the pulping period, the pH and temperature of the pulp were approximately 9.5 and 40°C respectively.

Flotation

Flotations were conducted within an E-18 flotation cell (an 18 L scale model of a Voith-Sulzer flotation cell) (Figure 2-1). Twin nozzles (1.10 gph) positioned immediately above the high water mark within the cell deliver pressurized surfactant spray onto the surface of the aerated pulp. When applicable, deinking additives, defoaming agents and fatty acid were directly mixed into the 8% consistency pulp. All pulps were then diluted to 1% consistency with 45°C deionized H₂O within the 18 L cell. Calcium chloride was subsequently metered into the furnish to establish predetermined concentrations. Using the closed flow circuit within the cell, the pulp was agitated for 5 minutes prior to aeration. During this time, the pH of the slurry was adjusted to 8.5 with 1% NaOH.

At the end of the mixing period, air was introduced into the cell at a flow rate of 30 SCFH. Simultaneously, a frothing solution of 100 ppm TX-100 was sprayed onto the top of the slurry at a pressure of 80 PSI (92 ml/min flow rate). In trials employing traditional Na-oleate/CaCl₂ chemistry, the surfactant spray was not used. Flotation was conducted for 6 minutes and the reject stream collected for subsequent yield loss analysis.



Figure. 2-1. Side view of the E-18 flotation cell equipped with dual nozzles for surfactant spray delivery.

Preparation of Brightness Pads

Using a 15 cm Buchner funnel and Whatman 4 filter paper (150 mm), 2 brightness pads were prepared from each floated pulp. Aliquots of the floated pulp containing 4.0 o.d.g. of solids were adjusted to pH 3.0 with 0.5 N HCl. Lowering the pH of the pulp serves to minimize sidedness during brightness pad formation and facilitate separation of the wet pad from the filter paper. A low vacuum pressure (~25 psi) during pad formation further reduced sidedness. Two additional control pads were prepared from the unfloated pulp. The pads were pressed at 70 psi for 10 minutes

and then allowed to dry for an additional 10 minutes in an Emerson Speed Dryer (Emerson Apparatus).

Brightness Measurement and Total Yield Loss

Pad brightness was determined using TAPPI method T452 om-98 with a Technibrite Micro TB-1C (Technidyne Corp.). Brightness measurements were taken from 5 separate fields on both sides of each pad and the average of the 10 values recorded. Brightness values from pads characterized by a standard deviation of 1 brightness point between the top and bottom were not recorded. Total yield losses were calculated by dividing the oven dry weight of pads made by filtering the reject stream across VWR 415 filter paper by the 180 o.d.g. of preflotation solids (i.e. the initial solids in the full E-18 cell).

Fiber-free Filtrate Preparation

For zeta-potential and particle size analyses, a fiber and fine-free fraction of the 1% slurry of 100% flexo ONP was prepared. Two filtration steps were required to separate the long fiber and the >20 micron fine fraction from the dispersed flexo ink. Suspensions of flexo ink and fines were obtained by fractionating unfloated slurry with a dynamic drainage jar equipped with a 76 m mesh. The filtrate was then vacuum-filtered across a VWR 415 filter and the resultant filtrate stored at 4°C until use.

Zeta Potential and Particle Size Measurement

Particle size and zeta potential measurements were conducted at 45°C on a Malvern Zetasizer 3000 (Malvern Instruments, Malvern, UK). Fiber-free filtrate from the 1% slurry was used without dilution during measurements.

Surface Tension Measurement

Surface tensions of solutions of 2, 5, and 200 ppm TDA-32 at various concentrations of CaCl₂ were obtained via the Wilhelmy plate technique using a dynamic contact angle analyzer (DCA-312, Cahn Instruments, Inc.). Surface tensions were used to calculate the amount of TDA-32 adsorbed at the air-water surface as a function of [CaCl₂] by the Gibbs adsorption isotherm:

$$\Gamma = -\left(\frac{C}{RT}\right)\left(\frac{d\gamma}{dC}\right)$$

where:

 Γ is the surface excess concentration of TDA-32, C is the concentration of TDA-32 (g/m³), R is the gas constant (8.314 J/mol·K), T (K) and γ is the surface tension of the solution (N/m).

RESULTS AND DISCUSSION

Flotation of 100% Flexo ONP

Deng *et al* have provided a detailed account of the merits of surfactant spray flotation deinking and the underlying principles upon which the technology is based (12). The current investigation was initiated to determine whether the benefits afforded by the surfactant spray technology apply to furnishes composed of 100% flexographic ONP. Unfortunately, flotation trials conducted under the optimized conditions of previous trials failed to elicit similar gains with furnishes containing water-based flexo news inks. In fact, surface active compounds (e.g. binder resins) released from flexo ink printed material stabilized the foam and, thereby, contributed to yield loss. The level of TDA-32 defoamer emulsion determined to prevent foaming in 70:30 offset ONP/OMG pulps in the absence of surfactant spray application was insufficient for 100% flexo ONP pulps. Raising the addition level of TDA-32 to 200 ppm was found to effectively prevent foam generation. Yield losses returned to levels obtainable with ONP/OMG furnishes although brightness gains remained negligible.

Calcium addition to fiber-free suspensions of flexo ink has been shown to stimulate the formation of floatable aggregates (15, 16). Bearing in mind that fiber is purported to prevent formation of these species within calcium-soap flotation regimes (15, 16), we wanted to observe the impact, if any, of the electrolyte during surfactant spray deinking trials. Interestingly, the results, presented in figure 2-2, reveal an unexpected consequence of calcium addition. As the calcium chloride concentration increases from 0 to 100 ppm, the floated pulp experiences a linear brightness gain of up to 7 ISO points. Controls floated using conventional calcium-fatty acid soap technology could not provide comparable brightness gains. In addition, the yield loss was roughly twice that of the surfactant spray approach after 6 minutes of flotation. However, it is apparent from the figure that brightness gain exhibits a maximum at 100 ppm CaCl₂. Beyond this level brightness decreases while yield losses continue to mount. As noted from initial flotations using conditions adopted from offset ONP/OMG trials, TDA-32, by itself, could not brightne the floated pulp.



Fig. 2-2. The flotation efficiency of 100% flexo ONP containing 200 ppm TDA-32 as a function of [CaCl₂] employing surfactant spray technology. (■=0 ppm CaCl₂, ▲=25 ppm

CaCl₂, ●=50 ppm CaCl₂, □=100 ppm CaCl₂, \triangle =200 ppm CaCl₂, \bigcirc =200 ppm CaCl₂/100 ppm Na-oleate).

To determine the significance of the observed brightness increase, results obtained through the surfactant spray approach were compared to those generated using manufacturer recommended dosages of commercial deinking additives. Like flotations conducted with calcium soap, the commercial additives tend to indiscriminately float both ink and fiber (Figure 2-3).



Fig. 2-3. Comparison of surfactant spray flotation efficiency between TDA-32/calcium system and commercial deinking additives (\Box =200 ppm TDA-32/CaCl₂ curve, \blacktriangle =50 ppm Sansink[®], \blacksquare =25 ppm Vinings A). Refer to figure 2 for CaCl₂ concentrations.

Flotations conducted without TDA-32 emphasize the requisite that both calcium and defoamer be present when floating flexo (Figure 2-4). Ionized fatty acids and binder resins liberated from the fiber during the alkaline pulping stage probably complex with excess calcium ion to form foam stabilizing agents. Not only does this contribute to yield loss in our experiments, this residue tends to accumulate as paper machine deposits and leads to quality issues within products containing recycled fiber.



Fig. 2-4. When TDA-32 is not present in the pulp, increasing the calcium chloride concentration does not increase brightness (\Box =TDA-32/CaCl₂ curve, \blacksquare =0 ppm, \blacktriangle =25 ppm, \blacklozenge =50 ppm, \blacklozenge =100 ppm and \triangle =200 ppm CaCl₂).

The ability of TDA-32, a siloxane-based defoaming agent to float flexo ink at certain calcium concentrations has been established. To determine whether this capacity is exclusive to polydimethylsiloxane emulsions, a second defoaming agent was substituted for TDA-32 (Figure 2-5). Flotation trials conducted with ECCO P-048, a nonsiloxane-based defoamer emulsion, provided similar results to those obtained with TDA-32. Both siloxane and non-siloxane-based emulsions appeared to brighten pulps in a calcium-dependent manner. To this end, the interaction between the oil droplets and flexo ink particles did not appear to stem from chemical modification of the oil phase. To generate support for this conclusion, trials designed to detect potential calcium-ink and calcium-defoamer interactions were conducted.



Fig. 2-5. Flotation trials conducted with 200 ppm of either TDA-32 (\blacksquare) or 200 ppm ECCO P-048 at 0 ppm CaCl₂ (\Box), 25 ppm CaCl₂ (\triangle), 50 ppm CaCl₂ (\times), 100 ppm CaCl₂ (\bigcirc) or 200 ppm CaCl₂ (\diamondsuit).

Flexo Ink Interactions with Calcium Chloride

Compression of the electrostatic double layer of dispersed flexo ink particles is a simple matter of increasing concentration and/or valence of the counterion within the bulk suspension. At a specific cationic strength, the stabilizing character of the anionic acrylic resins adsorbed at the ink particle surface will be effectively neutralized. Although this electrostatic component to flexo stability is tempered in the presence of calcium ions, the flexo ink particles may continue to resist aggregation due to steric repulsion between the loops of the adsorbed polymeric resins (1). Electrosterically stabilized particles tend to require higher than average electrolyte concentrations to suppress these dual components of dispersion and drive aggregate formation. To determine if the calcium concentrations during flotations were sufficient to aggregate the dispersed flexo, zeta potentials and particle sizes of flexo ink within samples of calcium chloride conditioned fiber-free filtrate were measured.

Figure 2-6 indicates that aggregation within the fiber-free filtrate does not occur within the range of calcium chloride concentrations used during flotation trials. Moreover, when this filtrate contains 200 ppm TDA-32, additional calcium is required to promote aggregation. Apparently, the emulsion droplets actively compete with flexo particles for available calcium ion. Nonpolar oil-in-water ternary systems, even those stabilized with nonionic amphiphilic surfactants, are capable of acquiring a significant anionic surface charge under alkaline conditions (17). A 200 ppm suspension of TDA-32 in deionized water was found to possess a slight surface charge at pH 8.5 (Figure 2-7). Accordingly, the defoamer is expected to combine with the fiber fraction and reduce the amount of calcium ion available to interact with ink particles. Previous research has indicated that the long fiber fraction may also physically impede flocculation of flexo ink particles regardless of calcium concentration (15, 16). To this end, the 200 ppm CaCl₂ concentration found to promote ink particle aggregation within the fiber-free, defoamer-free filtrate is not likely to achieve the same result when added to a whole pulp during actual flotations.



Fig. 2-6. Flexo particle size as a function of $[CaCl_2]$ (45°C, 8.5 pH, 0 ppm TDA-32 (\blacksquare), 200 ppm TDA-32 (\Box)).



Fig. 2-7. Zeta potential of TDA-32 defoamer emulsion droplets as a function of pH. Measurements were taken with a 200 ppm suspension of TDA-32 in millipore water at 45°C and 0 ppm CaCl₂ (counterions provided within the continuous phase of TDA-32 stock).

Figure 2-8 reveals the anionic nature of dispersed flexo ink particles. Ionized flexo ink particles suspended in a fiber-free environment maintain a significant charge at all calcium concentrations employed during actual flotation trials. The trend appears to indicate that the surface charge may resist suppression irrespective of the calcium chloride concentration employed.



Fig. 2-8. Zeta potential of dispersed flexo ink in the filtrate as a function of $[CaCl_2]$ (8.5 pH, 45°C, 0 ppm TDA-32).

From the zeta potential and particle size measurements, calcium-mediated formation of flexo ink aggregates with surface chemistries and/or dimensions amenable to flotation is not occurring within the aqueous bulk phase of the aerated pulp during the trials.

Defoamer Interactions with Calcium Chloride

Concluding that flexo ink-calcium chloride interactions were not solely responsible for the observed brightness gains, the focus shifted to possible calcium-mediated modification of the TDA-32 emulsion. The utility of a defoaming agent depends upon its ability to adsorb at the air-water interface of a bubble within the bulk phase of a solution. Any modification to the chemistry of the emulsion droplet surface should influence this interfacial adsorption of the defoamer. Surface excess concentrations of TDA-32, , as a function of calcium chloride concentration are presented in figure 2-9. Solutions containing 200 ppm TDA-32 did not exhibit diminished adsorption behavior as calcium chloride concentrations increased. In the event that the variation in adsorption was too slight to be observed in 200 ppm defoamer solutions, surface

excess concentration values were derived for solutions of 2 and 5 ppm TDA-32. All results imply that calcium does not affect the ability of the defoamer to adsorb and spread at the air-water interface. The brightness gains obtained during the flotation trials are neither an exclusive function of chemical interactions between ink and calcium nor defoamer and calcium.



Fig. 2-9. Surface excess concentration () calculated for solutions of 2 (\blacksquare), 5 (\blacktriangle) and 200 (\bigcirc) ppm TDA-32 as a function of [CaCl₂].

CONCLUSIONS

Surfactant spray flotation deinking incorporating siloxane-based defoamer has been shown to be capable of enhancing the removal of flexographic news ink. The ability of defoamer to increase the brightness of a pulp consisting of 100% flexographic ONP is a direct function of calcium chloride concentration during flotation. Initial investigation into the fundamental mechanism of this electrolyte-dependent phenomenon indicates that the collection of dispersed flexo ink is not the result of specific chemical interactions between calcium and defoamer or between calcium and the ink particle. Rather, both components must be present in order to initiate flexo ink flotation. Since the defoamer in the pulp precludes formation of a stable froth, surfactant spray technology represents an indispensable means to generate and maintain a foam layer for retention of floated ink. Ongoing investigation is anticipated to reveal the fundamental mechanism of flexo ink flotation in the presence of defoamer and calcium chloride.

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Part III: Deinking Selectivity (Z-Factor): A New Parameter to Evaluate the Performance of Flotation Deinking Process

This part of the study proposes a deinking selectivity concept that considers both ink removal and fiber yield in determining the performance of deinking operations. The defined deinking selectivity, or Z-factor, is expressed by the ratio of ink removal expressed by the International Standards Organization (ISO) brightness gain or the reduction in relative effective residual ink concentration (ERIC) and the relative fiber (oven-dry basis) rejection loss. For most flotation processes, typical brightness Zfactor is on the order of unit value and ERIC Z-factor is on the order of 10 units. Therefore, the Z-factor weighted brightness gain and ERIC reduction have relevance to ISO brightness and ordinary ERIC reduction. Pilot-scale flotation deinking experiments showed that Z-factor weighted brightness gain and ERIC reduction are good indicators of deinking process efficiency. The period or stage Z-factors are good indicators of the efficiency of the periods or stages of a deinking process. A simple criterion developed using the stage Z-factor concept was applied to both pilot-scale experiments and an industrial recycling mill operation for determining the economics of a given period or stage in a flotation deinking operation.

INTRODUCTION

Since its introduction in the 1980s, flotation deinking has been adopted as a standard practice for removing ink from wastepaper in paper recycling operations. Inks are detached from fibers through the pulping process before flotation. The objective of the flotation process is to remove the detached inks from the fiber suspension by injecting air bubbles, with the assumption that the hydrophobic ink particles will stick to air bubbles on collision. Ink is removed when the ink-attached bubble froth floats to the top of a flotation cell and is rejected. An increase in froth rejection rate results in an increase in ink removal. Unfortunately, the bubble froth rejection process also rejects fibers, primarily as a result of the entrainment of fiber into the bubble network [1–5]. Furthermore, fiber rejection loss is increased with an increase of froth rejection [3]. It is apparent that increased ink removal and fiber yield are two contradictory requirements in flotation operations, which makes flotation deinking so different from mineral flotation.

Because the primary concerns in most paper recycling mill operations are machine or process runnability and meeting the ink removal specifications of mill customers without additional processing (e.g., bleaching or washing), most studies on flotation deinking have primarily focused on removal of contaminants. These studies include understanding pulping chemistry and process [6–10] to achieve good ink separation from the fibers for removing ink, removal of wax or stickies through flotation [11,12], and flotation chemistry to improve ink removal [13–19]. Typical gains of paper brightness around 10% ISO standard [20] through flotation are common in laboratory or mill operations. Little attention has been paid to the improvement of fiber yield through flotation. After studying the fiber entrapment mechanism of fiber loss in flotation deinking [5,21], water spray was used to reduce the fiber trapped in the bubble network and thereby increase yield in a laboratory study [21].

A frothing agent spray concept was proposed to obtain separate control of froth stability to increase fiber yield and optimize ink removal in deinking of toner printed papers in a laboratory column flotation cell [4]. This concept was later successfully demonstrated using a mixture of old newsprint (ONP) and old magazine (OMG) furnish in a pilot-scale commercial flotation cell [22]. However, a limited number of commercial trials of the frothing agent spray concept have been conducted. Typical yield losses in recycling mill operations are about 10% to 25%, which contributes to the higher cost of recycled fibers compared with that of virgin fibers. Because loss in fiber yield is mainly caused by the same process used for removing ink in flotation, i.e., the froth rejection process, it is logical to take an integrated approach to study flotation deinking and to evaluate flotation deinking process performance. That is, the flotation process has to be optimized in terms of both high ink removal rate and fiber yield.

The objective of our study was to define a deinking selectivity concept that takes into consideration both ink removal and fiber yield loss in a deinking process. The deinking selectivity concept was then applied to a set of flotation deinking experiments conducted in a laboratory pilot-scale facility to demonstrate its usefulness in evaluating the performance of the flotation process under various experimental conditions. Note that deinking selectivity is completely different from the flotation selectivity used in mineral flotation where selectivity is related to mineral grade selection. The goal of the present study was to develop a balanced evaluation technique to assess the performance of industrial deinking operations.

DEFINITIONS

In a previous study [22], the ratio of deinking brightness gain and percentage of relative fiber loss was used to describe flotation deinking selectivity. We found that selectivity was effective in differentiating the overall performance of several flotation experiments under various conditions. Table 3-1 lists the experimental data presented in figures 4 and 5 of our previous study [22] to illustrate the effectiveness of selectivity. The data clearly show that experiment 2 was optimal in terms of low fiber loss and high brightness gain. While experiment 3 resulted in the highest brightness gain, it suffered from very high fiber loss, 40% more than that incurred in experiment 2. Experiment 1 showed the lowest fiber loss, but brightness gain was also lowest among the three experiments. The selectivity data clearly show that the best results were obtained with experiment 2. The initial success of the term "deinking selectivity" in evaluating deinking performance led us to define deinking selectivity in general terms.

Table 3-1	А	comparison	of	ISO	brightness	gain	and	fiber	loss	of	four	flotation	deinking
experiments													

Exp. No.	Fiber Loss (%)	Brightness Gain (ISO %)	Selectivity
1	5.3	8.75	1.64
2	5.9	9.80	1.67
3	10.0	9.97	1.00
4	12.1	8.52	0.70

Instantaneous Deinking Selectivity

Instantaneous deinking selectivity, Z(t), is defined as

$$Z(t) = \frac{dG}{dFrj} \tag{1}$$

where *G* is the relative percentage of change of any ink removal parameter, e.g., ISO brightness gain, relative effective residual ink concentration (ERIC) reduction, etc., and *Frj* is the percentage of fiber rejection loss. Therefore, instantaneous brightness selectivity is defined as

$$Z_{\rm B}(t) = \frac{dGB}{dFrj} \tag{2a}$$

where *GB* is the gain in brightness in ISO (%) of a sheet made from deinked fibers. Similarly, ERIC selectivity can be defined as

$$Z_{\rm E}(t) = \frac{dRE}{dFrj}$$
(2b)

where *dRE* is percentage of ERIC reduction.

Time-Averaged Period Selectivity (Stage Selectivity or Z-Factor)

In practical deinking operations, it is not possible to evaluate the operation in an infinitesimal period. The definitions in Equations (1) and (2) do not apply in practical situations. Rather, time-averaged deinking selectivities over a short period, t_i , are often used:

$$Z(\Delta t_i) = \frac{\Delta G_i}{\Delta F r j_i}$$
(3)

$$Z_{\rm B}(\Delta t_i) = \frac{\Delta GB_i}{\Delta Frj_i} \tag{4a}$$

$$Z_{\rm E}(\Delta t_i) = \frac{\Delta R E_i}{\Delta F r j_i}$$
(4b)

The importance of time-averaged period deinking selectivities is their application in various stages of flotation in mill operations. When the relative changes of ink removal parameters, i.e., brightness gain or ERIC reduction, and relative fiber loss are evaluated for an individual flotation stage (with typical residence time of 1 to 2 min), then the calculated time-averaged period selectivities are the selectivities of the individual stage. For this reason, the time-averaged period deinking selectivities can be called stage deinking selectivities, or simply stage Z-factors. The stage Z-factor can be used to evaluate the efficiency of a flotation stage.

Accumulative Deinking Selectivity (Process Selectivity or Z-Factor)

Accumulative deinking selectivity or Z-factor is used to evaluate the overall performance of the deinking process. It is the extension of time-averaged selectivity from a short period to the whole process. Therefore, it is the time-averaged deinking selectivity over the whole process. As a result of the nonlinear nature of deinking processes, the accumulative deinking selectivities or Z-factors are not equal to the mean of the stage selectivities or Z-factors. The accumulative selectivities or Z-factors can be calculated from the relative percentage of change of ink removal parameters and relative percentage of fiber loss of the individual stages using the following relations:

$$Z(T) = \frac{\sum_{i} \Delta G_{i}}{\sum_{i} \Delta Frj_{i}} = \frac{G(T)}{Frj(T)}$$
(5)

$$Z_{\rm B}(T) = \frac{\sum_{i} \Delta GB_{i}}{\sum_{i} \Delta Frj_{i}} = \frac{GB(T)}{Frj(T)}$$
(6a)

$$Z_{\rm E}(T) = \frac{\sum_{i} \Delta RE_{i}}{\sum_{i} \Delta Frj_{i}} = \frac{RE(T)}{Frj(T)}$$
(6b)

where T is pulp suspension residence time in whole flotation process of various stages.

Selectivity or Z-Factor Weighted Brightness Gain and ERIC

Selectivity or Z-factor is a measure of the relative percentage of gain in paper brightness or reduction of ERIC per unit percentage of fiber rejection loss. It is a measure of the efficiency of the flotation process or the efficiency of a particular flotation stage. Selectivity or Z-factor does not give the absolute value in brightness gain or ERIC reduction through the process or a particular stage. It is logical to define selectivity or Z-factor weighted brightness gain and ERIC (Eq. (7)) for absolute or quantitative comparison of ink removal through various processes or stages. The Zfactor weighted brightness gain and ERIC are simply equal to the brightness Z-factor times the ISO brightness gain and the ERIC Z-factor times the ordinary ERIC reduction, respectively. In most flotation processes, typical brightness Z-factor is on the order of unit value. Therefore, a brightness Z-factor weighted brightness gain is on the same order of magnitude of ordinary brightness gain and has relevance to the brightness gain used in current industrial practice. While typical ERIC Z-factor is on the order of 10 units, the ERIC Z-factor weighted ERIC reduction will be only an order of magnitude greater than the ordinary ERIC reduction:

$$BG_{\rm ZB} = Z_{\rm B} \cdot BG \tag{7a}$$

$$RE_{\rm ZE} = Z_{\rm E} \cdot RE \tag{7b}$$

where BG_{ZB} and RE_{ZE} are the brightness and ERIC Z-factor weighted brightness and ERIC reduction, respectively. Note that there is always a finite value of fiber loss in practical unit operations. It is not possible to have infinitively large Z-factors, which would distort the intended meaning of the Z-factor weighted brightness gain and ERIC reduction. As will be discussed, large Z-factor values are possible as a result of the rejection of a very small amount of fibers, which only occurs during the start-up of the system. However, small stage, process, or accumulative Z-factors are possible in the later stages of a unit operation as a result of the typical kinetic behavior of ink removal (i.e., exponential decay in ink removal) and constant fiber rejection. Small Zfactors can lessen weighted brightness gain and ERIC reduction, which is the intended purpose of the two parameters defined by Equation (7).

Economic Significance of Deinking Selectivity or Z-Factor

An advantage of the Z-factors is that these parameters can be put into an economic perspective to determine if an additional stage or additional processing is economically justifiable. Assuming that the pulp price gain for an additional unit of ink removal (e.g., one unit of brightness gain or ERIC reduction) is P_G over the initial pulp price *P* and that the additional percentage of fiber loss to achieve the additional

ink removal *G* (either in terms of ISO brightness gain or percentage of ERIC reduction) is *Frj* through the flotation stage or processing under evaluation, then the economic gain of unit ton pulp can be calculated as expressed by the left-hand side of inequality in Equation (8). The economic gain has to be positive to justify the additional flotation stage or any further processing; i.e., the following expression must be held:

$$(P + \Delta P_{\rm G} \cdot \Delta G) \cdot (1 - \Delta Frj/100) - P > 0 \tag{8}$$

Recalling the definition of stage Z-factor in Equation (3), the following criterion can be obtained from Equation (8):

$$Z > \frac{1}{\frac{\Delta P_{\rm G}}{P} \times 100} + \frac{\Delta G}{100} \tag{9}$$

where \tilde{G}_{i} is often an higher order term for any stage or period and can be ignored. Therefore, Equation (9) can be simplified as

$$Z > \frac{1}{\frac{\Delta P_{\rm G}}{P} \times 100} \tag{10}$$

Equation (10) clearly indicates that the stage Z-factor must be greater than the inverse of the percentage of pulp sale price gain from the additional unit of ink removal to justify the additional flotation stage or process. Note that this criterion does not take into account the lost production output resulting from increased residence time in the additional stage and ignores the positive high order term, a hundredth of ink removal (G) in Equation (9). A similar criterion holds for eliminating

a flotation stage or process. Therefore, the Z-factor has significant practical importance in industrial applications.

EXPERIMENTAL

Experiments were conducted in the pilot plant flotation deinking facility at the USDA Forest Service, Forest Products Laboratory, to illustrate the practicality of the Z-factors and their associated deinking parameters defined in the previous section. The facility consists of a two-stage Lamort (Kadant Lamort, France) vertical flotation cell having a capacity of 2,000 L. The flotation cell has concentric inner and outer chambers, each about 1,000 L, as the two stages. The incoming flow rate of pulp suspension is approximately 180 L/min, giving a typical residence time of 11 min; however, all the experiments conducted were run in batch mode.

Fiber suspension feedstock was injected into the flotation cell through eight tangential jets in the inner chamber. Pressurized air was pumped by venturi devices through the jets into the inner and outer chambers of the flotation cell. The flotation air flow rate was set at 10 standard cubic feet/minute (scfm) for most experiments and 15 scfm for one experiment. After entering the bottom of the inner chamber, the fiber suspension feedstock swirled upward, carrying entrained air and ink particles. The feedstock spilled into the outer chamber. At the top of the fiber suspension interface a vacuum manifold suctioned off the top layer of foam to which was attached ink particles produced by air flotation.

To obtain good mixing, suspension stock was drawn from the bottom of the outer chamber, then recirculated tangentially through three jets to the bottom of the outer chamber. Air was also injected through the three recirculating jets using venturi devices. The air recirculation pressure was maintained at 62 kPa (9 lbf/in²) in all experiments. The flotation accept stream was removed from the bottom of the outer cell. One suction shoe was used to extract froth for ink removal. The typical distance between the suction shoe and the top suspension interface was maintained at about 2 cm in most operations.

Old newsprint (ONP) was obtained from London, England (*Daily Mail*, August–September 2002). Before experimentation, the newspapers were sorted to remove inserts. Old magazine papers (OMG) were obtained from Quad Graphics (Sussex, Wisconsin). The exact ratio of ONP and OMG in the wastepaper for pulping was 9:1. The ash content of the feedstock from the pulping of the ONP and OMG mixture was about 5.6%. Commercial deinking chemical Vinings A (Lionsurf 5140, Kemira Chemicals, Kennesaw, Georgia) was used for all experiments. The chemical charge on oven-dry (OD) weight of paper was varied from 0.2 to 0.8, which gave a range of chemical concentration in the suspension of 13.6 to 54.4 mg/L. To obtain time-dependent data from the flotation processes, reject and accept samples were collected every 3 to 5 min, depending on the duration of the batch flotation. Feedstock samples were also collected for each experiment.

Handsheets made from wet samples (TAPPI method T205 sp-95) were used for ERIC measurements, using TAPPI method T567 pm-97 [20]. TAPPI method T218

om–91 [20] was used to make a pad from wet samples to determine the consistency of solid (ash and fiber) and ash content in the feedstock and reject stream. The same method was used to prepare a pad from wet samples for diffuse brightness measurements based on TAPPI standard T525 om–92 [20]. Two pads for measuring brightness were prepared from each sample, and three readings were taken from each pad. A total of six readings were used to calculate the mean and standard deviation of pulp brightness. The pads made from the reject stream were combusted at 525°C to determine ash and fiber (OD basis) content in the reject stream. ERIC measurements of the handsheets were analyzed by Technidyne Corp. (New Albany, Indiana). Five readings were used to calculate the mean and standard deviation of ERIC.

RESULTS AND DISCUSSION

Accumulative (Process) Z-Factors—Effect of Deinking Chemical Charge

It is well known that increasing the deinking surfactant charge initially increases ink removal as a result of the increase in froth stability [4,5,15,16,19]. Further increasing surfactant charge in the flotation stock reduces ink removal as a result of the reduction of the hydrophobicity of ink particle surfaces caused by the adsorption of surfactant. In our pilot-scale study, this effect of deinking surfactant on ink removal was also observed with the commercially blended deinking chemical Lionsurf 5140 (or Vinings A) (Fig. 3-1). The data shown in Figure 3-1 were obtained from four separate pilot-scale batch experiments after 15 min of flotation. The results indicate that the best ink removal in terms of brightness gain and ERIC could be obtained in a

chemical charge around 0.4% on oven-dry fibers. (The 0.35% chemical charge caused by chemical pump malfunction was designed to repeat the 0.4% experiment.) For the two experiments conducted at chemical charges of 0.4% and 0.8%, the 0.8% charge resulted in slightly more ink removal, based on the brightness and ERIC data. This does not rule out the adoption of a 0.8% chemical charge, assuming the difference in chemical cost between it and a 0.4% charge were insignificant. However, evaluation of the Z-factors, Z-factor weighted brightness gain, and ERIC using Equations (6a) and (6b) led to the conclusion that the 0.4% chemical charge is optimal. Moreover, deinking performance was much better under the 0.4% charge when both ink removal and fiber rejection loss are taken into consideration by comparing the deinking Z-factors, Z-factor weighted brightness gain, and ERIC (Figs. 3-2 and 3-3). Both brightness and ERIC Z-factor obtained at 0.4% chemical charge were about 70% greater than values obtained at 0.8% chemical charge, as a result of the significant increase in fiber rejection loss under the 0.8% deinking chemical charge, which, in turn, was caused by the increase in froth stability and consequent entrapment of fibers [5,21]. Fiber (OD basis) rejection loss linearly increased with the increase in deinking chemical charge (Fig. 3-4). The y-intercept at zero chemical charge can be considered as the fiber loss resulting from true flotation [5], which was only 2% for the 15-min flotation conducted in this study. The results shown in Figure 3-4 indicate that an appropriate frothing agent charge not only reduces chemical cost but also increases fiber yield. We conclude that control of froth stability is the key to reducing fiber rejection loss in flotation deinking operations.



Fig. 3-1 Effect of deinking chemical charge on fiber ISO brightness gain and ERIC reduction after 15 min flotation.





Fig. 3-2 Effect of deinking chemical charge on deinking process selectivity after 15 min flotation.

Fig. 3-3 Effect of deinking chemical charge on Z-factor weighted brightness gain and ERIC reduction after 15 min flotation.


Fig. 3-4 Effect of deinking chemical charge on fiber rejection loss after 15 min flotation.

To further investigate the performance of the two flotation processes conducted at chemical charges of 0.4% and 0.8%, we plotted time-dependent ISO brightness gain and ERIC reduction. As shown in Figure 3-5, the two processes were essentially identical if simply judged on the basis of ink removal. However, it becomes evident that the process using 0.4% chemical charge is preferable when the time-dependent fiber loss data are plotted as shown in Figure 3-6. Fiber rejection loss was linearly dependent on flotation time in both runs, but the slope was lower for the 0.4% charge. The negative intercepts of the linear regression results of the fiber rejection loss data were due to the unsteady behavior of the two batch processes during the start-up period. The comparison of these two processes can be easily illustrated by using the cumulative or process deinking selectivities or Z-factors defined by

Equation (6). As shown in Figure 3-7, both the cumulative brightness and ERIC selectivities or Z-factors of the 0.4% chemical process were consistently higher than those of the 0.8% process at any given flotation time.



Fig. 3-5 Time-dependent ISO brightness gain and ERIC reduction under two deinking chemical charges.



Fig. 3-6 Time-dependent fiber rejection loss under two deinking chemical charges.



Fig. 3-7 Time-dependent cumulative deinking selectivity, or Z-factors, of two flotation deinking process

This discussion indicates that it is insufficient to judge deinking performance from ink removal data only and that deinking Z-factors are able to take into consideration both ink removal and fiber yield in determining deinking selectivity. The Z-factor weighted brightness gain and relative ERIC reduction shown in Figure 3-3 illustrate how these two parameters can be used to determine the overall performance of a deinking operation or process without losing the traditional meaning of ordinary brightness gain and ERIC reduction. (Recall that the brightness and ERIC Z-factors are on the order of 1 and 10, respectively, for typical deinking operations.)

Period Z-Factors—Effect of Flotation Residence Time

To illustrate the period Z-factor concept, the time-dependent deinking data, i.e., brightness gain, ERIC, and fiber loss, collected at 5-min intervals at chemical charge 0.35% on OD fiber were used to calculate period Z-factors. Brightness gain, ERIC reduction, and fiber loss over different periods were first calculated from the time-dependent data; period Z-factors were then evaluated according to Equations (4a) and (4b). As shown in Figure 3-8, the period Z-factor follows the law of deminishing return due to the kinetic behavior of ink removal and continued near-constant rate of fiber loss. The period brightness Z-factor was about 4 in the first 5 min of flotation and decreased to about 0.2 after another 20 min of flotation, whereas the ERIC Z-factor was decreased from 56 to about 0.6 in the same 20-min flotation period. The data clearly indicate that the last 5 min of flotation were very inefficient. A pulp sale price gain of more than 5% for unit brightness gain and/or a 1.6% price gain for an ERIC

reduction of 1% is required to make the last period of flotation economical according to Equation (10).

Stage Z-Factors—Performance of Flotation Stage in Industrial Operation

We applied the stage Z-factor concept to a mill flotation deinking operation to determine the efficiency of each flotation stage at the mill. To conduct this exercise, we sampled the feed and accept stock of different stages of a production line with seven stages in series. Determination of the reject flow rate of each stage was not possible and was not attempted. Therefore, fiber loss was estimated from the consistency of the stock in each stage. The results indicate that consistency decreases linearly across the seven stages. Therefore, a constant fiber loss of 1/7 of total fiber loss determined from the consistency of the fiber loss at each stage.



Fig. 3-8 Time-dependent period brightness gain, ERIC reduction, fiber loss, and deinking selectivity of a flotation process under deinking chemical charge of 0.35%.

Handsheets were prepared from the sampled pulps to measure the brightness and ERIC of the deinked fibers. From the brightness and ERIC data along with the estimated fiber loss through each stage, we determined the deinking selectivity or Z-factor of each stage. As listed in Table 3-2 both the brightness and ERIC Z-factors decreased exponentially across the seven stages as a result of exponential decay of ink removal through the stages downstream. We then calculated the required pulp price gain for economical operation of each stage according to Equation (10). The results indicate that the last two stages are not economically justified according to this sampling exercise. The required pulp price gain per unit brightness gain was over 10%, while the pulp price gain for each percentage of ERIC reduction required for the last stage to be economical was 27%. This exercise demonstrates the practical importance of the deinking selectivities or Z-factors defined in this study.

	Brightnes s	ERIC	Consistenc y	Z _{Bi}	Ζ _{Εi}	P/P (%) per unit ISO GB	P/P (%) per percent RE
Feed	44.17	1177	0.0087				
Stage	51.27	574.2		6.179	44.5	0.16	0.02
Stage	53.32	428.6		1.784	10.7	0.56	0.09
Stage	54.68	363.1		1.184	4.84	0.84	0.21
Stage	55.51	303.4		0.722	4.41	1.39	0.23
Stage	56.11	273.3		0.522	2.22	1.92	0.45
Stage	56.19	266.1		0.070	0.53	14.29	1.88
Stage	56.29	265.6	0.0080	0.087	0.03	11.49	27.03
Proce	56.29	265.6		1.506	9.62		
SS					4		

Table 3-2 Deinking performance of an industrial flotation operation with 7 stages

Z-Factor Weighted Brightness Gain and ERIC Reduction—Comparison of Flotation Processes Under Various Operation Conditions

To further illustrate how Z-factor weighted brightness gain and ERIC reduction can be used to determine deinking performance, batch flotation experiments were also conducted under various experimental conditions; i.e., various fiber rejection rates made by adjusting the distance between the suction shoe and the suspension top surface, different chemical charges, and different recirculation air flow rates.

Figure 3-9 shows the brightness Z-factor and ERIC Z-factor weighted brightness gains and ERIC reductions calculated from the measured ISO brightness and ERIC data of the feedstock and final stock after 15 min flotation for the 15 experiments conducted. Because the Z-factor weighted brightness gain and ERIC reduction take fiber loss into consideration, we can easily determine that experiments 7 and 14 gave the best deinking performance (Fig. 3-9) without examining the fiber loss data. Recall that the final brightness Z-factor is about unit value and the ERIC Z-factor is about 10 units, so the ISO brightness for experiment 9 may be on the low side even though it gave good ERIC reduction.

To validate the determined best flotation (experiments 7 and 14), we plotted the corresponding ISO brightness gain, ordinary ERIC reduction, and fiber loss after 15 min flotation for the 15 experiments reported in Figure 3-9. As shown in Figure 3-10, experiments 7 and 14 indeed gave the best performance in terms of both ink removal

and fiber yield. Some experiments were not considered optimal because of low brightness gain (experiments 5 and 9), whereas others resulted in high fiber loss (experiments 6, 10, 11, and 15). In summary, Z-factor weighted brightness gain and ERIC reduction are good indicators of deinking performance that also take fiber yield into consideration.



Fig. 3-9 Z-factor weighted brightness gain and ERIC reduction of 15 batch flotation deinking experiments.



Fig. 3-10 ISO brightness gain, ERIC reduction, and fiber rejection loss of the 15 batch experiments reported in Fig. 3-9.

CONCLUSIONS

This study defined a new parameter for determining deinking performance: deinking selectivity. The study demonstrates that the defined deinking selectivity, the ratio of pulp ISO brightness gain or relative ERIC reduction and relative fiber loss, also called the Z-factor, can be used to determine the efficiency of a deinking stage or process. The pilot-scale flotation deinking experiments indicate that the Z-factor weighted brightness gain and ERIC reduction have relevance to ISO brightness and ordinary ERIC reduction and are good indicators of deinking process performance. The period or stage Z-factors are good indicators of the efficiency of the periods or stages of a deinking process.

developed in this study and applied to both pilot-scale experiments and an industrial recycling mill operation for determining the economics of a given period or stage in flotation deinking operations. Therefore, the deinking selectivity concept defined in this study is useful and has economic importance in deinking operations.

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PART IV: REDUCING FIBER LOSS IN LABORATORY- AND MILL-SCALE FLOTATION DEINKING USING SURFACTANT SPRAY TECHNOLOGY

Although the ability of surfactant spray technology to reduce yield loss, water loss and chemicals consumption without detriment to pulp brightness gains has been demonstrated during both laboratory- and pilot-scale flotation deinking investigations, this part documents the successful transfer of this technology to a single flotation unit within the deinking line of a mill producing newsprint from 100% secondary fiber. Initial results suggest that the loss of fiber across the unit may be reduced by more than 50% without obvious detriment to final pulp quality.

INTRODUCTION

In addition to qualitative properties such as final pulp brightness and residual ink concentration, yield loss during conventional flotation deinking is recognized as one of the core concerns of recovery operations today. As the cost of quality waste paper continues to escalate in response to an increased global demand for the finite resource, loss of saleable fiber within flotation rejects becomes both environmentally and economically irresponsible. Unfortunately, an inverse relationship often exists between qualitative and quantitative gains within mills employing flotation deinking technology. Extreme flotation conditions and/or chemistries leading to enhanced final brightness values often come at the expense of final yield. Inarguably, a means to maintain or even improve the ability of flotation operations to contend with an increasingly heterogenous furnish while significantly reducing the associated fiber yield loss will be of immediate relevance within the recovery industry.

Based on the fundamental understandings of flotation deinking, it is believed the mechanical entrapment of fiber and water in the froth is the major reason for fiber and water losses. Therefore, an effective method to mechanically control the stability, structure, and fluid dynamics of froth is critical for reducing fiber and water losses.

Traditionally, the frother and other surfactants are added into the pulp suspension during pulping. It is difficult to optimize the surfactant concentration in a paper recycle mill because of the variability in the secondary fiber sources. Because the foaming agent only functions when bubbles arise to 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, separate control of the application of a foaming agent, dispersion agent and collector can be achieved.

Surfactant spray technology represents a cost-effective means to impart some amount of process control to conventional flotation deinking operations. As the name implies, an aqueous frothing agent spray is applied onto the surface of an active flotation unit. In addition to displacing entrapped fiber from lamellae of the foam layer, the spray is exploited as a convenient channel for introduction of a frothing agent onto the aerated pulp. The rationale of the foaming agent spray concept is that the foaming agent is used only to stabilize froth. By restricting the surface active frothing agent to the upper layer of the flotation unit rather than mixing it with the incoming furnish, this technology can avoid the dilution of the foaming agent by the bulk volume of the pulp suspension and the reduction in the hydrophobicity of the ink particles due to the adsorption of foaming agent onto the ink particle surface (Figure 4-1). By simply adjusting spray composition and/or delivery rate, the technology may be customized to offset extremes in foam stabilities associated with sudden variations in the composition of the feed furnish. Furthermore, surfactant spray technology also makes it possible for separately controlling the foams across a series of flotation cells by spraying different amounts of frothing agent to them. Therefore, it would be much more effective to apply it where foam needs to be stabilized.



Fig. 4-1. By applying frothing agent as a spray to the top of a conventional flotation cell, unfavorable interactions within the bulk pulp suspension may be avoided.

It was demonstrated in our previous studies that fiber loss was reduced by up to 50% without affecting the ink removal efficiency when surfactant was sprayed on top of a flotation cell in deinking toner-printed papers in a bench scale column flotation cell (1) and in deinking offset old newspapers (ONP) and old magazine (OMG) in a laboratory-scale commercial flotation cells (2). It was believed that the reduction of fiber loss was due to better control of foam stability, foam structure and the spray washing effect that returned the fibers entrapped in the foam to the pulp suspension, as evidenced by Robertson et al. (3) in their experiments on foam washing during flotation.

Although the value of surfactant spray flotation deinking has been demonstrated at the laboratory and pilot-level (1, 2, 4), this paper documents the first attempt to extend the technology to full-scale operations.

EXPERIMTENTAL

Materials

Triton X-100, a nonionic octyl phenyl ethoxylate frother (Sigma-Aldrich, analytical grade, $C_8Ph(EO)_{10}$; Ph = phenyl), and TDA-32, a polydimethylsiloxane-based defoamer emulsion (Taylor Chemicals, 65% solid) were used as received.

Laboratory-scale trials

In preparation for the mill-scale trial, laboratory-scale flotation trials were conducted with the pulp obtained directly from the feed to the cell selected for application of the surfactant spray technology. Since the pulp included the deinking chemistry that had been added at the mill repulping process, , it was necessary to add defoamer into the pulp for the spray surfactant technology to counteract the formation of the froth from the components of the whole furnish. This phase of the investigation was of particular relevance since the chemistry would also be present within the flotation unit feed during the actual mill trial.

Flotation procedures: The flotation trials were carried out using Voith Sulzer flotation cell with 18-liter capacity (E-18) working in circuit operation. The flotation process was conducted at the following conditions: 1% pulp consistency, 5 min flotation duration, 43 °C, and 30 SCFH air flow rate. During flotation, rejects that overflowed from the top of the flotation cell were collected for quantification of yield loss. Certain amount of defoamer was added to the slurry to counteract the frothing agent contained in the mill pulp slurry before the surfactant spray flotation.

Surfactant spray device and conditions: A surfactant spray system is located on the top of the flotation chamber of E-18 cell. Bottled compressed air was used to drive the pressure swirl atomizers. The designed flow rate of the atomizers (Delavan, Inc., Des Moines, IA) is 1.5 gallon per hour (GPH). A TX-100 solution was used as the

surfactant spray solution. Two atomizers were used in the E-18 cell. The atomizers were separated by 97 mm and formed a row 50 mm away from the aeration tube and perpendicular to the reject flow direction.

Measurement of brightness, fiber loss: 4.0g filter pads were made using related Buchner funnel methods (PAPTAC Standard C.4U). Brightness was analyzed by TAPPI method T452 om-98. Yield loss was calculated using the ratio of the weight of the rejects (oven dry) to the weight of the waste paper (oven dry) at the beginning of flotation.

Mill trial

Mill selection: The mill selected for this study utilizes a 100% post-consumer furnish for newsprint production. Ink removal from the incoming feed is accomplished via two separate deinking lines incorporating conventional flotation equipment.

Flotation unit selection: Following an empirical analysis of the deinking operations and equipment employed by the mill, a gas sparged cyclone-type (GSC) (volume: 11000 gallons) flotation cell (AhlFloat, Ahlstrom, Glen Falls, NY), was selected for the surfactant spray trial. The preference for this particular unit stemmed from the ease with which representative pulp samples may be taken from the feed, accept and reject streams. In addition, background analytical data equated this unit with considerable fiber loss.

Nozzle bank construction: The nozzle bank consisted of a central manifold 4" PVC pipe from which 14 "L-shaped" 2" PVC pipe "arms" extended out over the cell surface (Figure 4-2). This design placed the nozzles, mounted at the terminus of each arm, as close as possible to the cell surface without interfering with the regular rotation of the foam scraper. The lengths of the arms were varied to ensure that the nozzles, each capable of delivering 5.05 gallons per min (GPM) when operated at 20 psi (McMaster-Carr catalog number: 32885K221), would provide maximum, uniform coverage with minimum overlap of the full-cone spray patterns. The pressure within

the nozzle bank was maintained at 20 psi by means of a PVC ball valve positioned at the mouth of the bank.

Surfactant spray delivery: In lieu of preparing a pre-mixed surfactant solution prior to the trial (necessitating a container with a volume greater than 12,000 gallons for a three-hour trial), a system was adopted to generate the solution *en route* to the nozzle bank. Fresh water, drawn from a 350-gallon tote by a centrifugal pump, was sent to the nozzle bank at a flow rate of 70.7 GPM. The tote volume was kept constant throughout the trial with fresh water. Undiluted Triton X-100 was metered into the suction side of the pump, across which adequate mixing of the miscible liquids was believed to occur. The dosing rate was adjusted to ensure that a 100ppm TX-100 surfactant solution was pumped into the nozzle bank for delivery onto the aerated pulp.



Fig. 4-2. The GSC flotation cell was fitted with a bank of 14 nozzles for delivery of the surfactant spray onto the surface of the aerated pulp.

Defoamer addition: Since the surfactant spray technology was incorporated within only one of the two deinking lines present at the mill, the deinking chemistry, introduced directly into the drum pulper (providing furnish to both lines) could not be suspended during the trial. To this end, the chemistry, complete with frothing agent, was present within the GSC feed. To counteract the foaming capacity of the whole furnish and ensure that any foam generated during the trial was a consequence of surfactant spray addition, the siloxane-based defoamer, TDA-32, was metered into the suction side of the GSC feed pump. To determine the minimal amount of defoamer needed to completely neutralize the foam, an expectedly high initial dosing rate of 200 ppm within the feed stream was established. This concentration was gradually reduced to the point at which foam could be neutralized with minimum defoamer consumption.

Surfactant spray trial: The trial commenced upon the establishment of dosing rates of defoamer and TX-100 required for a stable, uniform froth on the cell surface. Over the course of 3 hours, samples were taken from the GSC feed, accept and reject streams every 30 minutes. After determining consistencies of all three streams, brightness pads were prepared from the feed and accept streams according to TAPPI Standard Method T 218 sp-97. Brightness and ERIC measurements were taken from 3 separate fields on both sides of each pad using a Technibrite TB-1C equipped with an ERIC 950 module (Technidyne Corp.). Total ash and carbonate contents were determined for samples taken from each stream according to TAPPI Standard Methods T 211 om-93 and T 413 om-93, respectively. For convenience, all oven-dry mass lost during combustion at 525°C was presumed to represent the cellulosic (i.e. fiber) fraction of the total solids. This allowed the consistencies of all streams to be reported as total solids and percent "fiber". Quantification of yield loss during the trial involved performing a simple consistency-dependent mass balance around the GSC at each time point.

RESULTS AND DISCUSSION

Laboratory-scale trials

As we discussed previously, the rationale behind the foaming agent spray concept is to separately apply and control various deinking chemicals. However, the commercial deinking chemical currently used at the mill is a blend of different surfactants, which can act as both collector and frother. In order to reduce the fiber entrainment in the foam network, the role of foaming agent played by the commercial deinking chemicals must be suppressed, and required foams should be recreated and controlled by spraying a foaming agent to the top of the flotation cell. Therefore, defoamer was added into the pulp slurry to counteract the froth generated by the surfactant that was already added into the pulper at the mill. Three different loading levels of 50, 100 and 200 mg/L of defoamer TDA 32 (based on the total pulp slurry) were used. Two concentrations, 100 and 200 mg/L, of TX-100 solution were used as the surfactant spray solutions, and the total spraying amounts during flotation were 4 and 8 mg/L based on the whole pulp furnish respectively.

It was found during the spray flotation process that the froth generated by the deinking chemicals was suppressed by the addition of defoamer TDA-32. Foam volume and stability were reduced as the loading amounts of the defoamer increased. It was also observed that the foam stability and structure responded to the amount of spraying frother TX-100 very quickly, i.e. the amount of foam quickly increased as spraying amount increased and quickly decreased as spraying amount decreased. The response time was about a 10-20 seconds. Furthermore, ink removal efficiency and the final brightness of the recycled pulp were improved by the surfactant spray technology. Figure 4-3 shows the brightness gains obtained from the pads prepared from the feed and accepts during the conventional flotation deinking with and without using surfactant spray technology at the laboratory-scale trials. It can be seen that brightness gain of the pulp produced by the surfactant spray technology is about 0.3-0.5 ISO lower than that produced by the conventional flotation. However, significant reduction in fiber loss was also observed as shown in Figure 4-4.



Note: D: Defoamer TDA 32; Number (50, 100) after D: 50ppm or 100ppm TDA 32 based on the whole furnish; SST: Surfactant Spray Technology; Number (4, 8) after SST: 4 or 8 ppm TX-100 based on the whole furnish.

Fig. 4-3. Comparisons of brightness gains obtained from the pads prepared from the feed and accept during conventional flotation deinking and during the incorporation of surfactant spray technology.

It should be noted that neither ink removal nor fiber loss can be used as a single measure to evaluate the performance of the deinking process because recycling mills need both high ink removal and low fiber loss. Figure 4-5 shows brightness as a function of fiber loss obtained by both conventional and spray technology. Although the ~0.3-0.5 ISO brightness gain reduction was observed by surfactant spray technique, this small deduction was considered tolerable in view of the remarkably reduced yield losses. D100-SST8 (100ppm defoamer TDA-32 and 8ppm spraying amount of frother TX-100) is the optimized condition compared with the other conditions of this set of experiments as far as the ration of yield loss vs. brightness

gain is concerned. Yield loss was reduced by 45.6% (from 16.9% to 9.2%) without obvious detriment to the pulp brightness gain (-0.3).



Note: D: Defoamer TDA 32; Number (50, 100) after D: 50ppm or 100ppm TDA 32 based on the whole furnish; SST: Surfactant Spray Technology; Number (4, 8) after SST: 4 or 8 ppm TX-100 based on the whole furnish.

Fig. 4-4. Comparisons of yield losses accumulated across the flotation cell during conventional flotation deinking and during the trial incorporating surfactant spray technology.



Note: D: Defoamer TDA 32; Number (50, 100) after D: 50ppm or 100ppm TDA 32 based on the whole furnish; SST: Surfactant Spray Technology; Number (4, 8) after SST: 4 or 8 ppm TX-100 based on the whole furnish.

Fig. 4-5. Yield loss as a function of brightness gain values is presented for both conventional and surfactant spray technology.

It should be emphasized here that the foam generated at the surface of the pulp slurry is different from that generated inside of the pulp slurry, i.e., the fibers are more easily entrapped in the foam generated inside of the pulp slurry, but less likely to be entrapped by the foam stabilized at the surface. Therefore, the much lower fiber loss using surfactant spray technology compared with the conventional method at similar brightness gain is not surprising.

Mill-scale trials

Based on flow volume and weight fraction, the amount of defoamer metered into the feed to effectively prevent foam formation was determined to be 7.3 ppm or ~1 lb per

ton of oven-dry solids. When operated at 20psi, frother delivered onto the pulp amounted to 0.083 grams per kilogram of oven-dry pulp (0.17 lb/ton).

The brightness and ERIC values obtained from pads prepared from each stream are presented in Figure 4-6 and Figure 4-7. The slight drop in pulp brightness (~0.5) correlates with results observed during the laboratory-scale trials. This minor detriment may be readily managed within mills employing a series of flotation cells or through optimization of the surfactant spray concentration, delivery rate and/or spray pattern.



Note: SST: Surfactant Spray Technology.

Fig. 4-6. Brightness values obtained from pads prepared from the GSC feed and accept streams during conventional flotation deinking and during the incorporation of surfactant spray technology.



Note: SST: Surfactant Spray Technology.

Fig. 4-7. ERIC values obtained from pads prepared from the GSC feed and accept streams during conventional flotation deinking and during the incorporation of surfactant spray technology.

Figure 4-8 shows the yield losses obtained during the conventional flotation deinking and during surfactant spray deinking at mill-scale trials. The ability of the surfactant spray technology to dramatically reduce both values is immediately evident. In fact, the average yield loss during the trial was less than half of the average yield loss occurred during conventional operations. Analysis of the fundamental furnish solids (i.e. fiber/fines and the ash fractions of both carbonate and non-carbonate nature) indicated that surfactant spray technology did not significantly alter the weightfractional composition of total solids within the accept or reject streams (Table 4-1). Hence, this technology uniformly reduces the yield loss of these fundamental components of the furnish without affecting flotation removal rates of ink.



Note: Conventional results were obtained during 2 months (15 trials);

SST (surfactant spray technology) results were obtained during 1 day (9 trials).

Fig. 4-8. Average fiber yield loss as a function of average brightness gain values is presented for both conventional and surfactant spray flotation deinking.

Table 4-1: Percentile composition of the total solids isolated from the feed, accept and reject streams during both conventional and surfactant spray (SST) flotation.

		Conventiona	1	SST		
	Feed	Accept	Reject	Feed	Accept	Reject
Fibers/fines (%)	86.2	87.5	45.4	86.5	87.8	40.7
Carbonate (%)	1.6	1.4	9.9	1.7	1.4	11.2
Non-carbonate (%)	12.2	11.2	44.6	11.8	10.8	48.1

Total Solids Composition

Yield loss, plotted as a function of brightness gain (Figure 4-8), underscores the quantitative gains afforded by the surfactant spray approach to flotation deinking. Interestingly, the breadth of the results obtained during conventional operations suggests that brightness gain has limited correlation with yield loss. Conversely, the relatively low scatter obtained during surfactant spray trials confirms the technology as an effective means of process control.

CONCLUSIONS

During laboratory- and mill-scale trials, surfactant spray deinking technology was found to improve yield across a single flotation unit. Although the brightness gains/ERIC reductions within the post-flotation pulps were slightly lower than those floated under conventional deinking conditions, the difference was viewed as tolerable in light of the remarkably reduced yield losses. In addition, the technology has demonstrated the ability to better control the flotation process, particularly the foam characteristics across a series of flotation cells. Both benefits, reduced yield loss and improved process control, were realized with minimal capital expenditure and equipment modification. It must be stressed that this initial mill-scale trial was conducted under conditions found optimal during laboratory – not mill - conditions. To this end, the system is expected to be far from optimized. Possible variations in future trials may involve modifying the nozzle-bank design (i.e. spray delivery rate, nozzle number, spray pattern, etc.) and/or the composition of the spray itself (e.g. replacing Triton X-100 with a more environmentally benign frothing agent).

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Potential For Commercialization of the Project

It was demonstrated in our prior laboratory, pilot, and single cell commercial runs that the Surfactant Spray Flotation could significantly improve fiber yield and ink removal efficiency, and decrease water and chemical consumption. Results from single-cell commercial runs indicate (450ton/day deinked pulp):

- 1. An estimated 3.5% yield increase during air flotation deinking;
- 2. An estimated 100gpm water savings;
- 3. An estimated 10% chemical savings;

Based on the fiber yield enhancement, water and chemical consumption reduction, in additional to energy benefits and environmental benefits, the economic benefits were calculated based on a mill that recycles 157500 ton/year (based on 450 ton/day, 350 day/year deinking line) waste paper:

- Fiber yield could be increased by 3.5% which is 5512.5 ton/year valued at \$1,102,500/year based on \$200/ton fiber cost.
- Water consumption could be saved at 202778 ton/year or 100gpm.
- Chemical consumption could be saved at \$500,000/year based on 10% savings in total chemical cost.

It should be stressed here that the Spray Flotation Deinking technology can be easily retrofitted to an existing flotation facility. It does not require significant additional capital cost to replace existing equipment. It is estimated the capital investment only need \$50,000 (\$10,000 for one cell, totally 5 cells) for the whole deinking line. The capital investment could be reimbursed in less than 10 days.

Therefore, the successful development of the Spray Flotation Deinking technology can significantly benefit the snowflake newsprint mill, even the paper industry through improving fiber yield and quality, reducing water and chemicals consumptions, minimizing manufacturing impact, improving capital effectiveness and deducting the energy consumption and environmental pollution.