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### Flotation Deinking Chemistry: The Current Research Program at IPST

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### FLOTATION DEINKING CHEMISTRY: THE CURRENT RESEARCH PROGRAM AT IPST

### Yulin Deng<sup>1</sup>

### ABSTRACT

A research program on flotation deinking chemistry has been conducted at IPST for more than 15 years. This review paper focuses only on the progress over the past three years.

In this paper, the collectors that may be used to improve toner flotation efficiency are first discussed. The study related to the fiber surface chemistry in flotation deinking solutions is then reviewed. Finally, the fiber loss mechanism in the flotation process is described.

#### KEYWORDS

Fibers, Deinking, Flotation, Contact angle, True flotation, Entrainment, Collector, Surfactant, Particle size, Toners, Ink, Cationic, Kerosene, Fatty acid

#### 1. INTRODUCTION

The use of deinked fibers in the paper industry has increased dramatically in recent years, and with current environmental awareness and legislation, this trend is expected to continue. To be used as a highvalue product, recycled paper must be thoroughly deinked. Three deinking processes, flotation, washing, and screening, are mainly employed currently in the paper industry. Although flotation deinking is considered to be more effective and economical, there are still many remaining problems such as low ink removal efficiency of water-based inks, high fiber loss during flotation, contamination of fibers by flotation deinking chemicals, etc. Concerns with deinking efficiency and yield during flotation deinking have prompted an investigation of ink removal chemistry and fiber loss mechanisms.

The flotation deinking chemistry-related research program conducted at IPST has been focused on both the fundamental understanding and new technique development. The fundamental study includes the fiber loss mechanism, the filler flotation, the effect of old magazines on the flotation deinking of old newsprint, and the collector chemistry in flotation deinking. Based on the fundamental understanding, a new technique that can be used to control froth stability, fiber and water losses, and surfactant consumption, has been developed at IPST. Some results, such as the effect of old magazines on this deinking of old newsprint and the filler removal during the flotation deinking, and the new technique developed recently at IPST are not presented in this review article because these projects are confidential.

The people who have contributed to this program are: Dr. Guohua Wu, (Assistant Scientist), Mr. Marcos Abazeri (Assistant Engineer), Mr. Kyoung-Yuong Lim (Special Student), and Dr. Junyong Zhu (Assistant Professor). Dr. Peter Pfromm (Assistant Professor) and Mr. Joel Panek (Ph.D. candidate) have studied the surface chemistry of toner flotation deinking. Their work has been published [1] and is not included in this review paper.

### 2. COLLECTORS FOR THE FLOTATION DEINKING OF TONER PRINTED PAPERS

#### 2.1. Background

Flotation as a separation technique has been practiced in the mineral processing industry since early in this century. The process has been thoroughly studied, and excellent reviews on the science and technology are available [2]. The basic principle of mineral flotation is to add a collector (surfactant), which will preferentially adsorb onto one of the minerals present. This lowers the surface energy of the mineral particle and allows it to selectively attach to the air bubbles in the flotation cell. A froth of bubbles with attached mineral particles is formed at the top of the cell and can be readily separated.

The flotation technique has been practiced as an effective deinking technique for many years. The chemistry of flotation deinking has been reviewed [3-The separation of ink particles from fibers 5]. includes three main steps: ink detaches from fiber surface; ink attaches to the air bubbles; and ink floats and is removed from the pulp suspension [6]. In order to generate a relatively stable foam layer, a frothing agent (surfactant) is usually added. However, the addition of a frothing agent will cause a decrease in the hydrophobicity of hydrophobic ink particles [1,7-10]. To restore the ink hydrophobicity, a collector is commonly used. Although fatty acid/calcium collector has been traditionally used as an effective collector for

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flotation deinking of old newsprint and old magazines, the deposition of calcium fatty acid particles on the flotation equipment and the paper machine is a serious problem. Therefore, it will be beneficial to the paper industry if a more effective collector that does not have negative effects on the papermaking process can be developed.

The deinking of mixed office paper by flotation technique was found to be more difficult than other wastepapers because a) toner is fused into the pores of paper and strongly adheres to the fiber surfaces, resulting in a poor toner-fiber separation during repulping [11,12]; and b) the broad particle size distribution and the disk-like shape of toner particles results in a poor attachment between toner particles bubbles [11,13]. Based on and air these understandings, it is believed that the toner particles must be kept hydrophobic in the flotation cell and the optimum particle size must be achieved to improve the flotation deinking efficiency of toner printed papers. One possible method to achieve this is to use an effective collector. In a recently published paper, Dorris and Page [7] indicated that a fatty acid plus calcium ions can be used as a toner flotation collector. Miller et al. [14] also found that the toner flotation deinking efficiency could be significantly improved by the addition of 5% (based on dry-paper) tetrahydrofuran into the pulp slurry. However, it is not clear if this improvement is caused by "collector" effect or others.

Part of the study conducted at IPST is to develop a fundamental understanding of the collector chemistry involved in flotation deinking. The relationship between the collector and toner surface hydrophobicity was studied. The agglomeration of toner particles in different collector solutions were examined. Finally, the influence of toner hydrophobicity and toner particle size on the flotation efficiency was investigated.

The collector that can be used for flotation deinking strongly depends on the ink particle surface chemistry, the frothing agent present in the flotation cell, the fiber and solution properties etc. The study conducted at IPST was primarily focused on the collectors that can be used for toner deinking. The collector chemistry for old newsprint paper is one of our ongoing research projects.

### 2.2. Contact angle of toner film in different collector solutions

In the flotation deinking of mixed office waste, a nonionic surfactant is widely used as a dispersant and frothing agent. However, the addition of a nonionic surfactant, such as TX-100, will usually reduce the hydrophobicity and the removal efficiency of toner particles in a flotation cell. Therefore, it is interesting to know if there are some collectors that can restore the reduction of contact angle of toner particles in a surfactant solution. Among the possible collectors that have been studied at IPST during last three years, the calcium fatty acid, kerosene, and cationic surfactant are most interesting.

The contact angle of toner film in different aqueous solutions as a function of chemical concentration is summarized in Fig. 1. Some conclusions from Fig. 1 are: a) The TX-100, which is a typical dispersant and frothing agent used in flotation deinking, could significantly reduce the contact angle of toner in water (curve A); b) kerosene (curve E) and oleic acid/calcium (curve D) could also reduce the contact angle of toner in aqueous solution, but the advancing contact angle was still higher than 110 degrees; c) the reduction of toner contact angle in TX-100 solution could be restored if kerosene (curve C) or oleic acid plus calcium chloride (curve B) was used.

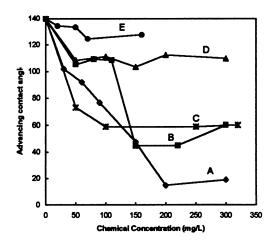


Fig. 1. Advancing contact angle of toner film in aqueous solutions as a function of A: TX-100 concentration without any other chemicals; B: TX-100 concentration in the presence of 50 mg/L oleic acid and 500 mg/L calcium chloride; C: TX-100 concentration in the presence of 20 mg/L kerosene; C: oleic acid concentration in the presence of 500 mg/L calcium chloride; E: kerosene concentration.

Cationic surfactants have been traditionally used in mineral flotation. One of the advantages of using cationic surfactant is that these chemicals can function as both a collector and a frothing agent for some ores. The effect of various functional groups of cationic surfactant on the mineral flotation was studied [15-17]. Although cationic surfactants are very effective flotation chemicals for some mineral particles, limited research [4,5,18,19] has been done on using cationic surfactants as a flotation deinking agent, and none of them has used cationic surfactant to deink toner printed wastepapers.

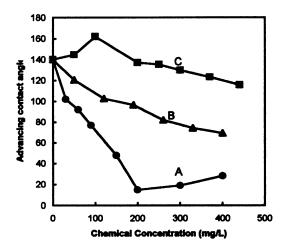


Fig. 2. Advancing contact angle of toner film in aqueous solutions as a function of A: TX-100 concentration; B: concentration of dodecyltrimethylammonium bromide (DTMAB); C: concentration of cetyltrimethylammonium bromide (CTMAB).

Fig. 2 shows the advancing contact angle of toner film in different cationic surfactant solutions. It can been seen that the contact angles of toner film obtained in two different cationic surfactants are much higher than that in TX-100 solution. More interesting is that the contact angle increased initially with the concentration of CTMAB increases, then decreased as further increases in CTMAB concentration. The reason for the contact angle increase is because cationic surfactant molecules have different configurations on the toner surface at low and high surfactant concentrations.

2.3. The agglomeration of toner particles in the presence of collectors

One of important factors affecting the flotation ability is the size of suspended particles. As an effective collector, it will not only increase the hydrophobicity of particles, but also agglomerate the floated particles into an optimum size. The particle size of toner powder in a pulp filtrate as a function of chemical concentration is shown in Fig. 3.

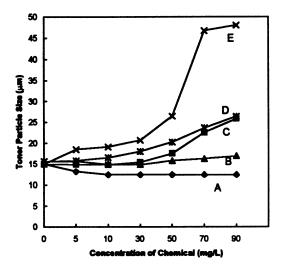


Fig. 3. Toner particle size as a function of A: TX-100 TX-100 concentration alone; B: TX-100 concentration in the presence of 50 mg/L oleic acid and 250 mg/L calcium chloride; C: TX-100 concentration in the presence of 20 mg/L kerosene; D: concentration of dodecyltrimethylammonium bromide (DTMAB); E: concentration of cetyltrimethylammonium bromide (CTMAB).

A significant agglomeration effect of toner particles in the pulp filtrate by kerosene (curve C) and two cationic surfactants (curves D and E) can be seen from Fig. 3. The reason for toner particle agglomeration in the presence of cationic surfactants may be due to the hydrophobic attraction force or charge neutralization of toner particle surface, or both. It can be seen that, when TX-100 was used alone, the toner particle size slightly decreased as the concentration of TX-100 was increased. The slight decrease in the toner particle size is not surprising because although the toner particles can be dispersed in a pulp filtrate and stabilized by adsorbed anionic trash (they will aggregate and float in pure water), there must be some small toner aggregates because anionic trash in the pulp filtrate is not a very effective stabilizer. When TX-100 is added in the suspension, the hydrophobicity and the aggregated toner particles can be redispersed resulting in a decrease in the average particle size.

# 2.4. The effect of collectors on the flotation deinking efficiency of xerographic papers

The effect of hydrophobicity and particle size of toner in pulp slurry on the flotation deinking efficiency was investigated. It can be seen from Fig. 4 that TX-100, which has been used as a surfactant in many flotation deinking mills (the trade name and purity may be different from TX-100), can lead to a brightness gain of 8.5 ISO at a concentration of 120 mg/L. However, in the presence of 20 mg/L kerosene, only 20 mg/L TX-100 is needed to obtain a similar brightness gain as using TX-100 alone. Surprisingly, although calcium oleic acid can restore some loss of the hydrophobicity of toner particles caused by the TX-100, the addition of this collector decreased rather than increased the toner flotation deinking efficiency when TX-100 was used as a frothing agent. Our results are inconsistent with theses previous reported [7]. There is no clean explanation on the reduction of the flotation deinking efficiency of toner particles in this system. However, the effect of calcium fatty acid on the foam stability and structure must be accounted. It was observed during flotation experiments that the foam was much less stable in the presence of calcium fatty acid compared with using TX-100 alone.

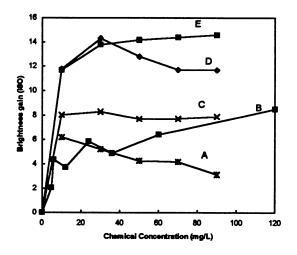


Fig. 4. The brightness gain of handsheet made from flotation deinked fibers as a function of A: concentration of TX-100 in the presence of 50 mg/L oleic acid and 250 mg/L calcium chloride; B: concentration of TX-100 alone; C: concentration of TX-100 in the presence of 20 mg/L kerosene; D: concentration of cetyltri-methylammonium bromide (CTMAB). E: concentration of dodecyltrimethylammonium bromide (DTMAB).

Fig. 4 clearly shows that cationic surfactants gave much higher brightness than other systems, including TX-100 that has been widely used in flotation deinking process. The high ink removal by cationic surfactant is consistent with the contact angle and particle size measurements, which suggests that cationic surfactant is an effective collector and frothing agent for xerographic papers. Further study of the application of cationic surfactants in flotation deinking of old newsprint and other wastepapers is one of the ongoing projects at IPST.

### 3. THE SURFACE CHEMISTRY OF FIBERS IN FLOTATION DEINKING

### 3.1. Background

One of research projects conducted at IPST is the fundamental study of the fiber loss mechanism in flotation deinking. In order to fully understand the mechanism of fiber loss in the flotation deinking process, the wettability of fibers in flotation solution must be studied first.

Although the influence of adsorption and orientation of surfactant molecules on the wettability of wood fiber is very important, it has not been well-studied because of the lack of an effective method to measure the contact angle of liquid on individual fibers. The contact angle measured from a liquid solution against a paper sheet may lead to a misunderstanding because of the pore structure of paper surfaces. The optical measurement of the contact angle of liquid against a single fiber has been attempted by Foote [20], Jones and Porter [21], and Grindstaff [22]. However, these methods are not satisfactory because (1) it is difficult to accurately measure the contact angle of liquid on very small wood fiber using an optical technique; (2) opposing curvatures between the fiber surface and observed meniscus lead to ambiguities in measuring the contact angle from the meniscus profile shape; and (3) they yield only a single  $\theta$ -value, which may be highly unrepresentative of the average properties of the heterogeneous fiber. A more satisfactory approach to the determination of fiber-liquid contact angles employs the Wilhelmy principle in which the downward force upon a fiber suspended vertically through the liquid surface is measured [23-25]. However, a poor reproducibility was observed when a single fiber was used because (1) the heterogeneity of wood fibers is high and the data obtained using a single fiber may be significantly different from the

average value of a pulp furnish, and (2) the wetting force is too small, which results in a large uncertainty.

In order to study the fiber loss mechanism and fiber surface chemistry of individual fibers in a flotation deinking process solution, a new approach for the contact angle measurement of individual fibers in aqueous solution was developed at IPST. In this method, a group of separated fibers was immersed and then removed from a liquid, and the advancing and receding wetting forces were measured, respectively. For the details regarding to the instrument and experiments used for the contact angle measurement, please see reference [26].

# 3.2. Advancing contact angle of contaminated fibers in different flotation deinking process surfactant solutions

In flotation deinking, the fibers and ink particles commonly are dispersed in a surfactant solution. there is no report regarding the However, hydrophobicity change of individual wood fibers in surfactant solutions. It is also interesting to identify how the fiber surface hydrophobicity change as a function of the concentration of a flotation process surfactant if the fiber surface has been contaminated by hydrophobic materials such as sizing agents, stickies, and polymers. In order to compare the contact angle of clean and contaminated fibers in different solutions, AKD (alkyl ketene dimer) sized softwood kraft fibers were used as model contaminated fibers, and the contact angle of these sized fibers as a function of TX-100 concentration was examined. It can be seen from Fig. 5 that, in the absence of surfactant, the advancing contact angle of AKD-sized fibers (hydrophobic) is much higher than unsized fibers (hydrophilic). However, the advancing contact angle of wood fibers decreases as the TX-100 concentration increases regardless if they are sized or unsized. Other interesting results found in our study [26] include that although the advancing contact angle of different wood fibers in different solutions can be significantly different from each other, the receding contact angle of wood fibers in all aqueous solutions, including pure water and surfactant and polymer solutions, is zero. This finding is consistent with previously reported results [23-25].

Although the receding contact angle is zero or close to zero for all wood fibers in different solutions, the advancing contact angle strongly depends on the concentration and chemical composition of soluble

surface active agents. The polymers, including nonionic polyethylene oxide and cationic poly(dially dimethyl-ammonium chloride) (polyDADMAC), have no significant effect on the advancing contact angle in water. The surfactants, including anionic sodium dodecylsulphate (SDS), anionic triton X-100, and cationic cetyltrimethylammonium bromide (CTMAB) can significantly reduce the advancing contact angle of wood fibers in water. The effect of different surfactants on the advancing contact angles of 0.6% AKD-sized softwood kraft fibers in aqueous solutions is shown in Fig. 6.

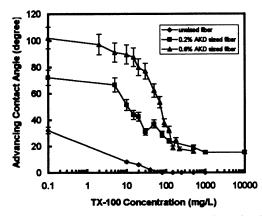


Fig. 5. Advancing contact angle of unsized, 0.2% and 0.6% AKD-sized bleached softwood kraft fibers as a function of TX-100 concentration.

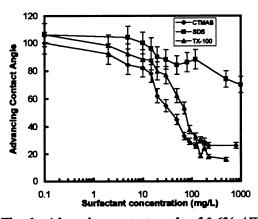


Fig. 6. Advancing contact angle of 0.6% AKD-sized fibers in different surfactant solutions as a function of surfactant concentration at neutral pH and room temperature.

The results of a fiber surface chemistry study conducted at IPST over the past three years strongly suggests that a) all wood fibers have a zero receding contact angle in flotation deinking pulp solution regardless if the fibers are contaminated by the chemicals present in the solution; b) the addition of surfactant in the flotation process will significantly reduce the advancing contact angle of wood fibers, which makes fibers adhering to air bubbles more difficult.

### 4. FIBER LOSS MECHANISM IN FLOTATION DEINKING

### 4.1. Background

Although the deinking efficiency is closely related to the brightness and cleanliness of the fibers, brightness figures without any information on the yield of the deinked fibers have only limited value because the achieved brightness strongly depends on the yield. Low yield caused by high fiber loss is one of the biggest problems in flotation deinking. Reported fiber loss is in the range of 4-24 wt% depending on the processes, equipment, and chemicals used in flotation deinking. For highly sized or waxed fibers, such as old corrugating containers, fiber loss is even higher, and the flotation technique cannot be used for these paper products unless a new technique is established.

Most authors [27-29] have postulated that the fiber loss in flotation deinking is caused by air bubbles routinely adhering to the fibers during the flotation process. In a series of studies on fiber loss in flotation deinking, Turvey [27,28], and Schwinger and Dobias [30] indicated that 1) unprinted fibers do not float; 2) calcium ions can significantly increase fiber loss for printed fibers; 3) nonionic fatty alcohol ethoxylate surfactants cause high fiber loss; and 4) pH plays an insignificant role in fiber loss. From these studies, Turvey [27,29] further concluded that fiber loss is due to the fact that part of the fiber becomes hydrophobic and adheres to air bubbles. However, no direct experimental measurement of fiber surface chemistry can support this assumption. Furthermore, some of the conclusions from Turvey [27] have been argued by other researchers [7,29,31,32] who indicated that unprinted fibers, even very clean bleached fibers, can still float during the flotation deinking process. In contrast to Turvey's study, some recent papers [7,32] indicated that the fiber loss in flotation deinking is solely caused by physical entrainment rather than true flotation. The conflicting experimental results obtained by different researchers suggest that the mechanism of fiber loss in flotation deinking has not be well understood.

It was reported that long fibers float easier than wood fines [29,31]. To explain this phenomenon, Li and Muvundamina [29] assumed that surfactant molecules have different orientations on the fine and fiber surfaces. It is well known that the surface of fines-is more hydrophobic than long fibers because of its high lignin content. When surfactants adsorb onto fines, the hydrophobic tails of surfactant molecules anchor onto the hydrophobic sites of fines and leave the charged heads (or hydrophilic parts) toward the water phase. This leads to an increase of hydrophilicity of the surface of wood fines and prevents them from adhering onto air bubble surfaces. For long fibers, on the other hand, the surface is very hydrophilic, and surfactant adsorption is through the interaction between the hydroxyl groups of fiber surfaces and the charged heads of surfactant molecules (or hydrophilic parts of nonionic surfactant), resulting in an increase of hydrophobicity. Li and Muvundamina [29] believed that the increase in hydrophobicity of fiber surfaces was the main reason for fiber removal. Once again, this is only an assumption, and there is no direct experimental measurement to support it.

It is clear that there are many arguments on the fiber loss mechanism. In order to study the effect of fiber surface chemistry on the fiber loss, both unsized and AKD-sized fibers from the same bleached softwood kraft pulp were used in IPST's study. The average fiber length was 2.83 mm. Because the sized fibers have the same geometric property as unsized fibers but totally different of surface chemistry, the contribution of fiber surface chemistry and fiber length to the total fiber loss can be separately studied.

The flotation cell used in this study includes a polyacrylate column (12 cm in diameter; variable in height) and a gas inlet filter. Nitrogen was injected from the bottom of the flotation cell through a air inlet filter. Two types of inlet filters with pore sizes of 10 or 2µm were used. Both the fibers that were removed from the cell (fiber loss) and those that remained in the cell were filtered, oven dried, and weighed. The technique used at IPST is different from previous studies [7,32]. In our study, the water loss was controlled by varying the froth height (adding extra columns to the top of the flotation cell) at a nitrogen flow rate and flotation time, but in previous studies it was controlled by varying the flotation time at fixed froth height. We believe that our method is more scientific for fiber loss mechanism study than the method used in previous studies. For more details

about the difference between the two methods, please see reference [31].

### 4.2. Effect of fiber hydrophobicity on the fiber loss

The flotation of solid materials can be divided into "true flotation" and "entrainment." True flotation occurs when solids attach to air bubbles and are floated with them. A basic requirement for true flotation is that the solid particles must be hydrophobic enough so that they can strongly adhere onto the bubble surface. Entrainment occurs when particles enter the froth with the water and occupy the spaces between the bubbles. When froth raises up, part of the water and particles entrained in the froth will drain back to the pulp suspension, but the remainder is carried upwards and scraped off. As a result, fiber removal by physical entrainment should be a function of water removal, and at ideal conditions, a linear relationship between fiber removal and water removal is expected. The contributions of true flotation and entrainment in mineral flotation have been discussed, and the following equation has been suggested [33]:

$$R = A + CV_{\text{water}} \tag{1}$$

where R is the total recovery of the given solid suspension at experimental conditions; A is the recovery of the solid by true flotation; C is the concentration of entrained solids in removed water; and  $V_{\text{water}}$  is the volume of removed water. At ideal conditions, the true flotation A and the concentration C of entrained solids in removed water can be obtained from the intercept and the slope of removed solids versus removed water, respectively.

In order to separately study the true flotation and entrainment of fibers in flotation deinking, a bleached softwood kraft pulp was used first in this study. Direct measurement of wettability using a separated fiber group technique indicated that the receding and advancing contact angles of these bleached softwood fibers in a 100-mg/L TX-100 solution are zero and <5degrees, respectively (see Fig. 5). This suggests that these bleached softwood kraft fibers are very hydrophilic in this solution and they cannot adhere to air bubbles by hydrophobic force in the flotation process. In other words, the loss of these hydrophilic fibers in the flotation cell could be caused solely by the physical entrainment.

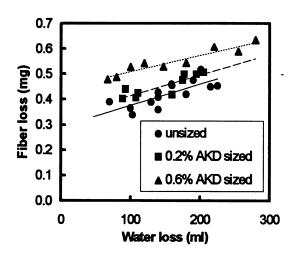


Fig. 7. Fiber loss as a function of water loss for unsized and AKD-sized bleached softwood fibers. The water and fiber losses were measured at different froth heights. Pore size of air inlet filter: 10  $\mu$ m; fiber consistency: 0.52%; concentration of TX-100: 100 mg/L; nitrogen flow rate: 1800 cm<sup>3</sup>/min; flotation time: 2 minutes.

Fig. 7 shows fiber loss as a function of water loss for different fibers after a 2-minute flotation. According to Equation (1) and the contact angle results shown in Fig. 5, a zero intercept of fiber loss against water loss should be expected for hydrophilic unsized fibers because all floated fibers in this system are solely caused by physical entrainment. However, the results of Fig. 7 clearly show that both the slope and intercept for bleached softwood kraft fibers (unsized) are not zero, although the fibers have a zero receding contact angle and <5 degrees of advancing contact angle in this solution. This strongly indicates that although Equation (1) has been used for the mineral flotation system, it cannot be directly used to describe the fiber loss in flotation deinking. It should be noted that Equation (1) is based on the assumption that the concentration of entrained (excluding adhered) solids in the froth phase is a constant during the water drainage, and all entrained solid particles will be washed back to the pulp phase if the water is fully drained from the froth. This may be true if the size of entrained particles is small and the density of the particles is high, such as mineral particles, but it is not the case for wood fibers because some entrained fibers cannot pass through the microchannels between bubbles during the drainage of water in the froth phase due to their large size and small density. As a result, a nonzero intercept of fiber loss plotting water loss must be obtained even though the flotation of wood fibers is

solely caused by entrainment. Because some of entrained fibers cannot be washed away during water drainage, a correction for these "unwashable" entrapped fibers must be made in order to use Equation (1) for wood fibers. It should be noted that unwashable entrapped fibers are different from true flotation fibers, although both of them cannot be washed away during water drainage. Obviously, the consistency of these "unwashable" entrapped fibers in the froth should be a function of foam structure, fluid dynamics of water in the froth microchannels, the fiber length and orientation, etc. If all experimental conditions remain constant during the foam raising up in the flotation cell, it can be approximately assumed that the weight of unwashable entrained fibers is a constant in the froth. Therefore, Equation (1) should be modified to

$$R = A + B + CV_{water} \tag{2}$$

where B is the weight of unwashable entrained fibers.

Equation (2) indicates that the intercept of the plot of total fiber loss R versus water removal should equal the sum of the fiber loss caused by true flotation A and unwashable entrapped fiber B rather than true flotation A alone, and this intercept is independent of total removed water  $V_{water}$  at a fixed flotation time. Equation (2) also indicates that although the true flotation A is zero for hydrophilic wood fibers, the intercept of the plot of total fiber loss versus water loss should equal B rather than zero. By plotting total fiber loss against water removal for unsized fibers shown in Fig. 7, the weight of "unwashable" entrapped fiber B and the consistency of "washable" entrapped fiber C were 0.29g and 0.8 g/L, respectively.

Fig. 7 also gives the comparison of fiber losses for unsized and AKD-sized fibers. It can be determined from Fig. 5 that the advancing contact angles for 0.2 and 0.6% AKD-sized fibers in a 100mg/L TX-100 surfactant solution are 28 and 39 degrees, respectively. which are much higher than that of unsized fibers (<5 degrees) in the same solution. From Fig. 7 it can be determined that the fiber losses of sized fibers are consistently higher than unsized fibers. This result indicates that the fiber surface chemistry will also contribute to the fiber loss. Because both the sized and unsized fibers used in this study have the same length, it is reasonable to assume that the entrainment factors B and C in Equation (2) are the same for all of the sized and unsized fibers. Therefore, the difference in the fiber loss between unsized and sized fibers is

attributed solely to the true flotation A. Although this assumption may be too simple, the experimental results of Fig. 7 indicate that the slope C is almost constant for all three fibers.

In terms of the above assumption, by applying B = 0.29 g and C = 0.8 g/L to Equation (2), the weights of true flotation A for 0.2 and 0.6% AKD-sized fibers were obtained and they were 0.025 and 0.15 g, respectively. Comparing the value of true flotation A with total entrained fibers  $B + CV_{water}$ , it can be found that true flotation A is smaller than total entrained fibers even for highly sized fibers.

The true flotation fraction F is defined as

$$F = A/R \tag{3}$$

The plots of F as a function of water loss fiber loss are shown in Fig. 8.

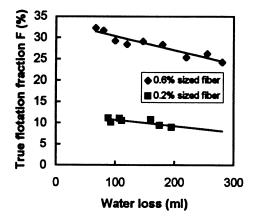


Fig. 8. True flotation fraction F as a function of water loss. The true flotation fraction for unsized bleached softwood fiber is assumed as zero. Pore size of air inlet filter: 10  $\mu$ m; fiber consistency: 0.52%; concentration of TX-100: 100 mg/L; nitrogen flow rate: 1800 cm<sup>3</sup>/min; flotation time: 2 minutes.

It can be seen from Fig. 8 that the true flotation fraction F for 0.2 and 0.6% AKD-sized fibers is in the range of 8-12 and 25-33%, respectively, depending on the froth heights. The results suggest that although true flotation is one of the mechanisms of fiber loss, most lost fibers (>88% for 0.2% AKD-sized and >33% for 0.6% AKD-sized fibers) in flotation deinking are mainly attributed to physical entrainment for these systems. Fig. 8 also shows that the true flotation factor F increases as the froth height is increased. This is because the total fiber loss R is decreased, but the true flotation contribution A remains constant when froth height is increased.

Although water loss in flotation deinking has not been considered as a problem in the paper mill, the energy saved by reducing water loss can also benefit the paper industry. Even for a fully closed flotation deinking mill, water loss by froth removal can still be as high as 10% of the total water in the flotation cell, which corresponds to a water loss of ~10 tons/(ton pulp). Because the discharged water contains many deinking chemicals, such as the surfactant and basic materials, reducing water removal is also important for a deinking mill. The results of a study conducted at IPST indicates that properly controlling the froth height and froth stability is a cost-effective method to reduce both water consumption and fiber loss.

## 4.3. Effect of nitrogen bubble size on the water and fiber losses

Many factors, such as surfactant concentration, gas flow rate, pore size of the nitrogen inlet filter, pulp concentration, soluble and colloidal materials in pulp, etc., may affect the bubble size and fiber loss. For example, when a 2-µm filter was used, some very small nitrogen bubbles were adsorbed onto the fiber surface, which resulted in fibers floating to the top of the flotation cell after the flotation experiment. This

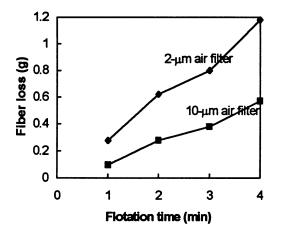


Fig. 9. The effect of pore size of air inlet on the fiber loss. The froth height: 7 cm; TX-100 concentration: 100 mg/L; flow rate: 1500 cm<sup>3</sup>/min.; fibers: 0.45% bleached softwood kraft fibers.

phenomenon was significant only when sized fibers were used. When unsized furnish or a  $10-\mu m$  filter

was used, the fibers sank to the bottom after the flotation experiment.

A typical example of bubble size effect on the total fiber loss is given in Fig. 9. As discussed before, the small bubbles gave higher water loss compared to the large bubbles, which resulted in a higher fiber loss.

### 6. CONCLUDING REMARKS

The study of flotation deinking chemistry conducted at IPST covers many different projects. Many conclusions can be drew from our studies. The remarkable conclusions include:

- a) The cationic surfactant can be used as an effective collector and frothing agent for toner flotation deinking.
- b) The contact angle of wood fibers in liquid can be measured using a group of separated fibers. The receding contact angle of all wood fibers in aqueous solutions is zero regardless of the fiber surface properties. The advancing contact angle of wood fibers decreased as surfactant concentration increased.

Both true flotation and physical entrainment contribute to the fiber loss, but entrainment is a dominating factor.

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#### REFERENCES

- Panek, J.; Pfromm, P. Interfacial Properties of Toner in Flotation Deinking, Progress in Paper Recycling, 49, 1996.
- Ramakrishnan, N.; Balaraman, C. Mineral Processing, Recent Trends, J. Mines, Metals & Fuels, 37 (10) 437 (1989).
- 3. Schulze, H. Fundamentals of flotation deinking in comparison to mineral flotation, 1st Research Forum on Recycling, Montreal, Canada, 161 (1991).
- 4. Ferguson, L. D. Deinking Chemistry: Part 1, Tappi J. 75 (7), 75 (1992).

- 5. Ferguson, L. D. Deinking Chemistry: Part 2, Tappi J. 75 (8), 49 (1992).
- Amand, F. J. S. Hydrodynamics of Flotation: Experimental Studies and Theoretical Analysis. TAPPI Recycling Symp., TAPPI Press, Atlanta, GA, 219 (1997).
- 7. Dorris, G. M.; Page, M. Deinking of Toner-Printed Papers. Part 1: Flotation Kinetics, Froth Stability and Fiber Entrainment. J. Pulp Paper Sci., 23 (5), 206 (1997).
- Drelich, J.; Azevedo, M.; Miller, J.; Dryden, P. Hydrophobicity and Elemental Composition of Laser-printed Toner films. Prog. Paper Recycl. 5 (4), 31 (1996).
- 9. Wu, G.; Deng, Y; Zhu, J. Collector Chemistry in Flotation Deinking of Xerographic Papers, Prog. Paper Recyclng, Submitted, 1998.
- Epple, M.; Schmidt, D.C.; Berg, J.C. The Effect of Froth Stability and Wettability on the Flotation of a Xerographic Toner, Colloid Polymer Sci., 272, 1264 (1994).
- Johnson, D. A.; Thompson, E.V. Fiber/toner Detachment Studies: Repulping and Flotation of Laser Printed Paper. Part I., Proc. TAPPI Pulping Conf., TAPPI Press, Atlanta, GA, 1291 (1995).
- Paulsen, F. G.; Berg, S. R.; Vidotti, R. M.; Johnson, D. A.; Thompson, E. V. Measurement of Long-Range Hydrophobic Attraction Forces and Their Relationship to Deinking Flotation. Part 2, TAPPI Recycling Symp., TAPPI Press, Atlanta, G.A. 41 (1997).
- Snyder, B. A.; Berg, J. C. Liquid Bridge Agglomeration: A Fundamental Approach to Toner Deinking. TAPPI Recycling Symp., TAPPI Press, Atlanta, GA, 277 (1994).
- Miller, J. D.; Azevedo M. A. D.; Xie, X.; Drelich J. Recycling Office Waste - Recovered Paper Deinking Research at the University of Utah, In Paper Recycling Challenge Vol. 2 -Deinking & Bleaching, Doshi M. R. and Dyer J. M. Eds., Doshi & Associates Inc., Appleton, WI, 167, (1997).
- 15. Shergold, H. L. Cationic Surfactants in Mineral Processing. Industrial Application of Surfactants, the Proceedings of a Symposium Organized by the North West Region of the Industrial Division of Royal Society of Chemistry. University of Salford, UK, 269 (1986).
- 16. James, A. D.; Ogden, P. H.; Wates, J. M. Cationic Surfactants. Industrial Application of Surfactants,

the Proceedings of a Symposium Organized by the North West Region of the Industrial Division of Royal Society of Chemistry. University of Salford, 251 (1986).

- 17. Kenny, F. J. Use of Surfactant in Mineral Flotation. Industrial Application of Surfactants 2, the Proceedings of a Symposium Organized by the North West Region of the Industrial Division of Royal Society of Chemistry. University of Salford, UK, 367 (1989).
- Borchardt, J. K. An Introduction to Deinking Chemistry. In Paper Recycling Challenge Vol. 2: Deinking & Bleaching, Doshi M. R. and Dyer J. M. Eds., Doshi & Associates Inc., Appleton, WI, (1997).
- 19. Putz, H. J.; Schaffrath, H. J.; Gottsching, L. Deinking of Oil- and Water-norne Printing Inks: A New Flotation Deinking Model, Pulp Paper Canada, 94 (7), 193 (1993).
- 20. Foote, J. A. Method for the Measurement of the Angle of Contact Formed Between a Liquid Surface and a Fiber, and the Application of This and Swelling Data to Pore Diameter Measurements, Paper Trade J. 10, 40 (1939).
- Jones, W.; Porter, M. A. Method for Measuring Contact Angles on Fibers, J. Colloid Interface Sci. 24, 1 (1967).
- 22. Grindstaff, T. A. Simple Apparatus and Technique for Contact-angle Measurements on Small-denier Single Fibers, Textile Res. J. 39, 958 (1969).
- Hodgson, K.; Berg, J. Dynamic Wettability Properties of Single Wood Pulp Fibers and Their Relationship to Absorbency, Wood Fiber Sci. 20 (1), 3 (1988).
- Krueger, J.; Hodgson, K. The Relationship Between Single Fiber Contact Angle and Sizing Performance, Tappi J. 78 (2),154 (1995).
- 25. Krueger, J.; Hodgson, K. Single-fiber Wettability of High Sized Pulp Fibers, Tappi J. 77 (7), 83 (1994).
- Deng, Y.; Marcos, A. Contact Angle Measurement of Wood Fibers in Surfactant and Polymer Solutions, Wood Fiber Sci., In press.
- Turvey, R.W. Why do fibers float, J. Pulp Paper Sci., 19, 52 (1993).
- Turvey, R.W. Stock loss as a function of water hardness in flotation deinking, Paper Techn. Ind., 366 (1987).

- 29. Li, M.; Muvundamina, M. Fractionation of Fiber Slurries via Froth Flotation and Sedimentation, Prog. Paper Recycling, 4 (3), 32 (1995).
- Schwinger, K.; Dobias B. The Influence of Calcium Ions on the Loss of Fiber in the Flotation Deinking Process, 1st Research Forum on Recycling, Montreal, Canada, 1 (1991).
- 31. Deng, Y.; Abazeri, M. True Flotation and Physical Entrainment: the Mechanisms of Fiber Loss in Flotation Deinking, Nordic Pulp Paper Res. J., in press.
- 32. Ajersch, M.; Pelton, R. Mechanisms of Pulp Loss in Flotation Deinking, J. Pulp Paper Sci., 22 (9), 338 (1996).
- 33. Warren, L. Determination of the contributions of true flotation and entrainment in batch flotation tests, Int. J. Min. Proc. 14, 34 (1985).

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