FROTH CONDUCTIVITY FOR IN SITU MONITORING OF FIBER (SOLID) AND WET REJECTS IN FLOTATION DEINKING

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ABSTRACT

Reduced fiber rejection in flotation deinking is very important to reduce the cost of secondary fibers in paper recycling and to conserve natural resources. Online monitoring of fiber rejection is a prerequisite to achieving process control for the reduction of fiber rejection in flotation deinking. It also can improve understanding of the effects of various operating parameters on fiber rejection in flotation for process optimization. This study demonstrates in situ monitoring of wet (liquid) and solid (mainly fiber) rejections during froth flotation of waste pulp suspensions by means of conductivity measurements of the rejected froth. Laboratory flotation deinking experiments were conducted using mixed office wastepaper (MOW) supplied by a recycling paper mill. It was found that both the total wet rejection and the solid consistency in the rejection stream correlate well to the measured relative conductivity of the rejected froth. The immediate application of the conductivity technique described is to monitor wet and fiber/solid rejection in industrial flotation deinking operations.

INTRODUCTION

Flotation deinking has been adopted as a standard industrial process for removing ink from recycled papers in secondary fiber recovery operations. In this process, ink particles attach to air bubbles because of their hydrophobic behavior and are removed with froth. Unfortunately, the froth removal process also rejects fibers, primarily as a result of the entrainment of fiber into the bubble network [1-3]. Fiber rejection through froth removal is a major contributor to fiber yield loss in secondary fiber recovery. A 4% fiber loss can typically be attributed to froth rejection in industrial operations, which negatively impacts the economics of secondary fiber recovery operations. Therefore, reducing fiber rejection in flotation deinking is very important to reduce the cost of secondary fibers, to increase secondary fiber recovery, and to conserve natural resources.

The primary concerns in most paper recycling mill operations are machine or process runnability and meeting contaminant removal specifications of the market without additional processing (e.g., bleaching or washing). Therefore, limited efforts have been made toward fiber yield improvement in laboratory and mill operations. Early studies on the subject include the work by Turvey [4], who incorrectly identified fiber adhesion to air bubbles as the mechanism of fiber loss. The fiber loss mechanism of fiber entrainment into the bubble network was later identified by Ajersch and Pelton [1-2] and confirmed by Deng and Abazeri [3]. In one of our recent studies [5], the problem of fiber rejection in flotation deinking was delineated into the two aspects of the problem: fiber consistency in the rejection stream (through entrainment) and rate of wet froth rejection. Both parameters were found to be dependent upon froth stability or froth production rate. We also characterized dynamic froth drainage with fibers [6] and fiber fractionation [7] in flotation to provide guidance in developing technologies to reduce fiber loss. From the mechanistic understanding of fiber loss in flotation deinking, the concept of using water spray to wash out the fibers entrapped in froth was proposed by Robertson et al. [8]. The effect of water spray washing can be confirmed using our froth drainage study [6]. A surfactant spray technique developed by Zhu et al. [9] applies frothing agent during flotation rather than during pulping (conventional and current industry practice). The technique can effectively control (when and where) the delivery of frothing agent and therefore froth stability, the key to controlling fiber loss [5]. Significant fiber loss reductions were achieved both in laboratory studies [9,10] and during recycling mill trials [11]. The reduction in fiber loss is attributed to favorable flotation chemistry and froth stability that reduce fiber entrainment in addition to the effect of spray washing.

The surfactant spray technique [9] provides the possibility for real-time control of froth stability to optimize flotation deinking performance, leading to increased contaminant removal with minimal fiber loss. However, a sensor that can monitor solid (fiber) or wet rejections in real time during flotation deinking is required to achieve real-time process control using the surfactant spray technique. In a previous study, we demonstrated the feasibility of using conductivity to monitor solid (fiber) and liquid content in flotation experiments of nylon and wood fiber suspensions [12]. However, most of the experiments were conducted in a column flotation cell using nylon fibers. Furthermore, the wood fibers used were from a deinked pulp and did not contain significant amount of inks. The objective of the present study is to demonstrate the applicability of the froth conductivity technique for fiber and wet rejection loss monitoring in real deinking-pulp suspensions. If demonstrated, a conductivity probe could be ultimately developed to integrate with the surfactant spray technique to reduce fiber loss in recycling operations.

THEORY

The froth conductivity technique for monitoring wet and solid rejections in flotation is based on the Lemlich's classic one-third relation (Eq. (1)) between liquid volumetric fraction (ϕ_l) and froth relative conductivity (σ_{froth}) [13]. The froth relative conductivity is defined as the ratio of froth conductivity to the conductivity of the liquid solution, which in this case is the pulp suspension. The relation was derived theoretically using the Plateau border (PB) structure of an aqueous froth.

$$\sigma_{froth} = \phi_l / 3 \tag{1}$$

We experimentally verified this relation for fiber suspension systems [12]. Our previous study [5] delineated fiber (solid) rejection loss in flotation into two aspects of the problem: wet froth rejection rate and fiber (solid) consistency in the rejection stream, both of which are dependent on froth stability. This suggests that froth relative conductivity, which is a measure of froth liquid volumetric fraction, can be used to measure froth stability and therefore fiber (solid) rejection loss in flotation. Solid rejection is rejection of total solid (e.g., fibers, ink, and clay particles). Fiber is the main component of solid in most flotation deinking systems. Wet rejection is the sum of the rejection of liquid and total solid (e.g., fiber, ink). The amount of solid rejection is negligible compared with liquid rejection in flotation deinking operations. Therefore, we use liquid rejection interchangeably with wet, and solid rejection interchangeably with fiber. By applying Lemlich's theory to deinking-pulp froth, wet rejection (R_W) can be expressed as the product of froth relative conductivity and volumetric rate of froth removal (proportional or equal to the rate of froth production to maintain a constant layer of froth):

$$R_W = \phi_l \cdot R_{froth} = 3 \cdot \sigma_{froth} \cdot R_{froth} \tag{2}$$

In the previous study [12], we experimentally verified the hypothesis that the froth volumetric production rate (R_{froth}), monotonically dependent on operating conditions (e.g., aeration rate and surfactant concentration), has a monotonic relation to froth relative conductivity:

$$\begin{aligned} R_{froth}&(\text{aeration, surfactant, etc.}) \\ &\propto R_{froth}[f_a(\sigma_{froth}), g_s(\sigma_{froth}), \text{etc.}] \end{aligned} \tag{3}$$

where functions f_a and g_s are monotonic and unique. The monotonic relations can provide one-to-one correspondence between froth conductivity and flotation conditions, and therefore froth volumetric production. Furthermore, we revealed that fiber consistency in the reject stream was mainly affected by drainage of liquid (water) [5,6], and drainage of fibers to some extent (fiber drainage is caused by water carryover [6]), through the Plateau border channels. We also experimentally verified that fiber (solid) consistency can be measured by froth conductivity; for example,

$$\chi_{fib/solid} \propto j(\sigma_{froth}) \tag{4}$$

Therefore, we can establish the following relation for the froth conductivity probe:

$$\begin{aligned} R_{fib} = \chi_{fib/solid} \cdot R_W &\propto \sigma_{froth} \cdot j(\sigma_{froth}) \\ &\cdot R_{froth}[f_a(\sigma_{froth}), g_s(\sigma_{froth}), \text{etc.}] \end{aligned} \tag{5}$$

EXPERIMENTAL

Conductivity Probe

We constructed a conductivity probe with two platinum wires 0.36 mm in diameter and 3.2 cm long as the sensing electrodes. The two wires, with a center distance of 7 cm, were supported by two acrylic drop legs and an acrylic frame (Fig. 1). The drop legs of the frame were designed to allow froth to fall over the reject weir without restriction. The acrylic frame was supported and aligned by resting on the walls of the upper section of the flotation cell (described later in the Flotation Cells section). The probe was connected to a conductance meter (Model 35, Yellow Springs Instrument Co., Yellow Springs, Ohio). A model IQ Vma data logger (Measurement Computing, Middleboro, Massachusetts) was connected to the analog output of the conductance meter to record readings during experiments. The probe measured the conductance of the medium with a length of 3.2 cm (the wire length) and depth of 7 cm (the wire separation distance) between the two wires when the probe was submerged into a medium. When the probe was submerged in froth, measured conductivity was the average conductivity of the medium within the described dimensions. The volume of the dimensions correlates to the volumetric liquid fraction of the froth according to Lemlich [13]. The conductivity probe was calibrated using a standard aqueous electrolytic

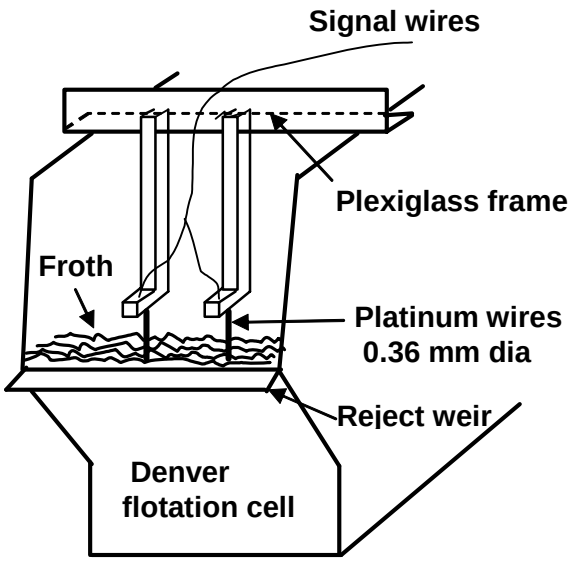

Fig. 1 Schematic diagram of flotation cell and conductivity probe.

solution of potassium chloride, KCL 7447-40-7 (Cole–Parmer Instrument Company, Vernon Hills, Illinois). Excellent linear response was obtained.

Flotation Cells

A stainless steel bench-scale commercial Denver flotation cell model 53-3000 (Denver Equipment Company, Colorado Springs, Colorado) was used in this study. The capacity of the cell was 8 L. The lower section of the cell is a rectangular-shaped tank; one wall of the upper section angled outward to form the froth-reject weir (Fig. 1). A vertical axis motor-driven rotor was centrally located at the bottom of the tank to mix the pulp suspension. The spinning of the rotor also aerated the suspension through entrainment. Ambient air was ported to the bottom of the rotor through the hollow channel of the rotor axis and flowed through the four holes into the suspension.

Chemicals

A pulping chemical was used during pulping of waste paper (discussed in detail in Waste Paper Pulping section). Triton X-100 (TX-100), an analytical grade nonionic octyl phenyl oxyethylenic surfactant (Advance Scientific and Chemical, Inc., Fort Lauderdale, Florida), and DI-700 (Kao Specialties Americas LLC, High Point, North Carolina) were used separately as frothing agents to stabilize froth in the flotation experiments. The TX-100 concentration varied from 5 to 40 mg/L in the suspension. The DI-700 concentration varied from 10 to 25 mg/L in the suspension.

Pulping of Waste Paper

The fibrous raw material used was a mixed office waste (MOW) blend supplied by a North American recycling paper mill. The MOW was pulped at 12.5% consistency in a Voith 50L laboratory hydropulper (model HD.05, Voith, Inc., Appleton, Wisconsin) with a helical rotor for 20 min, at 46°C, and at pH 10. The pulping chemical (PolyDADMAC) was supplied by the same recycling paper mill and was used by the mill in commercial production. The pulping chemical dosage was 0.0625% based on ovendry weight of the wastepaper.

Measurements during Flotation Deinking

The pulp was diluted to 1.0% consistency for flotation deinking experiments in batch mode. The conductivity probe was used to measure conductivity of the pulp suspension after the frothing agent was added into the pulp suspension before flotation. The conductivity probe was secured horizontally on the sidewalls of the flotation cell so that the platinum wires were vertically oriented into the froth during flotation (Fig. 1). The tips of the two platinum wires were about 1 cm above the suspension level to keep them from directly contacting the suspension as water level rose by aeration and flow turbulence during flotation. Variations in suspension level or in the active length of the platinum wire in contact with froth changed the volume of the froth probed, as discussed in the Conductivity Probe section, and therefore the measured froth conductivity. However, this affects only the calibration between froth conductivity and the amount of wet and solid rejects as long as the platinum wire does not directly contact the pulp suspension. With the application of a frothing agent, froth rose to completely submerge the conductivity probe. Water level rose initially by aeration and then decreased because of froth rejection. Durations of flotation runs were 240 s with TX-100 and 300 s when frothing agent DI-700 was used. Conductivity of the froth was recorded in real time through a laptop computer at a frequency of 0.2 Hz. Time-averaged conductivity was used to correlate with wet and fiber (solid) rejections. The reject stream was collected periodically during a flotation experiment to obtain time-dependent wet and solid rejections.

Determination of Fiber in the Reject Streams

A gravimetric method was used to determine wet and fiber (solid, to be specific) rejection in froth flotation. Wet rejects were first collected in a reject container and weighed, then dewatered using a buchner funnel and filter pad before being placed in an oven set at 105°C to dry. The wet rejects collected were used to make a paper pad according to TAPPI method T218 om-91 [14] for the determination of fiber rejection and consistency in the rejection stream.

Determination of Optical Properties of Deinked Pulp

Following flotation deinking of the MOW pulp suspension using the TX-100 frothing agent, both diffuse (ISO) brightness and effect residual ink concentratyion (ERIC) measurements of paper samples made from deinked pulps (collected from the accept stream) were conducted. Five handsheets per pulp sample were made according to TAPPI method T272 om-92 [14] for the determination of optical properties. Five ISO brightness measurements were made per handsheet using Technibryte model TB–1 (Technidyne Corporation, New Albany, Indiana) according to TAPPI method T525 om-92 [14]. In addition, measurements of reflectance at 700 nm from the paper samples were conducted using a spectrophotometer (Model U-3010, Hitachi High Technologies America, Inc., San Jose, California). Reflectance data were used to calculate five ERIC values for each sample according to Jordan and Popson [15].

RESULTS AND DISCUSISONS

Wet or solid rejection data presented below are the amount of wet and solid rejects divided by the mass of the liquid and ovendry pulp in the suspension, respectively. Froth conductivity refers to froth relative conductivity.

Correlation between Froth Conductivity and Flotation Conditions

One premise for using froth conductivity as a means to monitor wet and fiber (solid) rejections is that froth rejection or production is correlated to froth conductivity. It is well known that froth production is proportional to flotation conditions, especially frothing agent concentration and aeration rate. As we experimentally demonstrated in a previous study [12], flotation conditions (i.e., surfactant concentration and aeration rate) were linearly (monotonic) correlated to froth conductivity. The premise was validated as a result. Figure 2 shows that excellent linear (monotonic) correlations exist between measured froth relative conductivity and concentration of frothing agent employed in this study when a fixed aeration rate was used. Therefore, the hypothesis that froth production is monotonically related to froth conductivity is verified in this study.

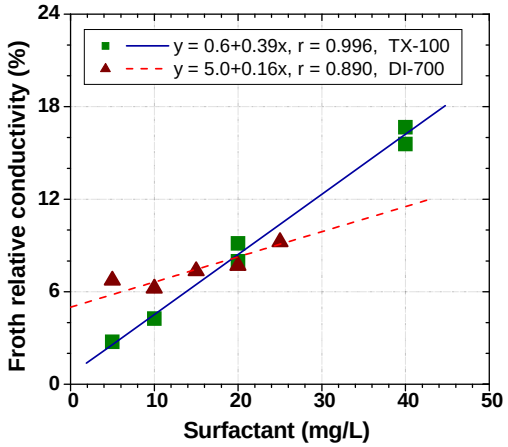

Fig. 2 Experimentally measured monotonic correlations between froth relative conductivity and applied frothing agent concentration.

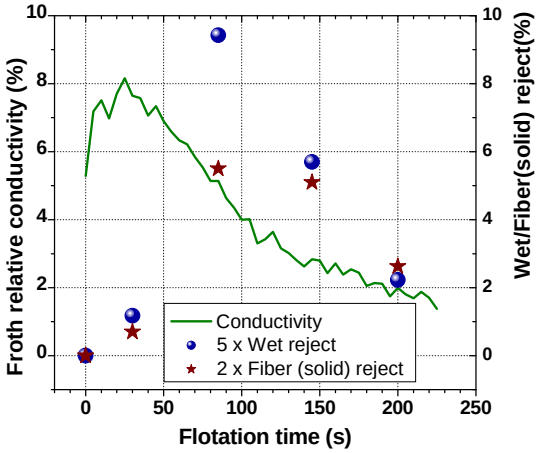

Fig. 3 Temporal response of the conductivity probe to changes in wet and solid rejection in a flotation deinking experiment.

Temporal Response of the Froth Conductivity Probe

Figure 3 shows time-dependent froth relative conductivity along with time-dependent wet and solid rejections. Froth conductivity increased to a maximum level as a result of froth production and the rising of the water level by aeration, then decreased gradually due to decreases in froth stability due to loss of frothing agent and suspension level through froth rejection. Time-dependent wet and solid rejections follow the trend of time-dependent froth relative conductivity very well but with a delay. The time lag was caused by the delay of froth rejection in relation to the rising of froth and the fact that the so-called time-dependent wet and solid rejections are averaged over a short period of time. Results shown in Figure 3 demonstrate that the froth conductivity probe qualitatively responded to temporal changes in wet and fiber rejection during flotation deinking.

Froth Conductivity to Monitor Solid and Wet Rejection

Equation (4) was proposed based on our previous fiber-laden froth drainage studies [5,6]. We identified the exponential functionality (monotonic) for the equation for some of the experiments conducted [12]. Figure 4 shows that monotonic functions were obtained between solid consistency and froth relative conductivity for the two sets of flotation deinking experiments conducted. Exponential functions can be used to fit the data obtained (Fig. 4).

Figure 5 shows the relations between measured wet and solid rejections and froth relative conductivity for various frothing agent concentrations, recalling from Figure 2 that froth conductivity is linearly proportional to frothing agent concentration. The data show that both wet and solid rejections increase with increase in froth relative conductivity in general.

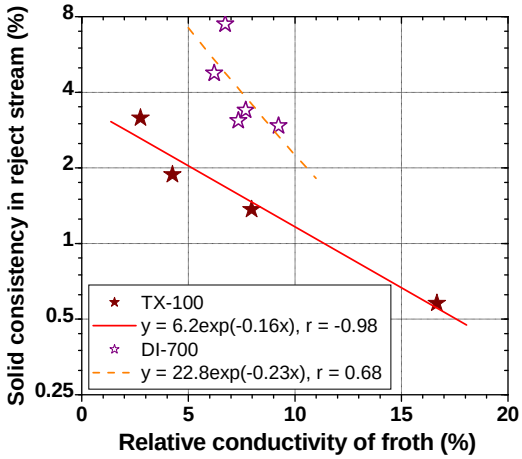

Fig. 4 Experimentally measured monotonic correlations between froth relative conductivity and solid consistency in the reject stream.

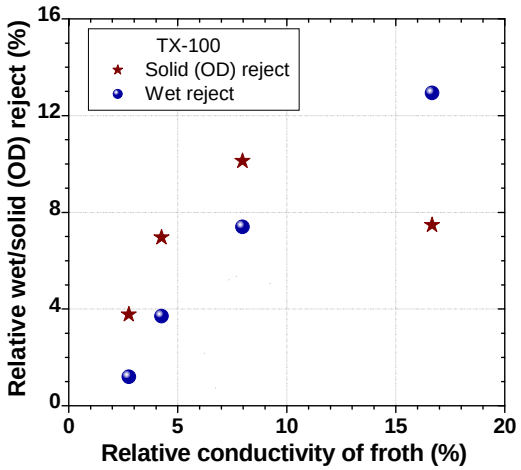

Fig. 5 Effects of frothing agent concentration represented by froth relative conductivity on wet and solid rejections in a set of flotation deinking experiments. Overdosage of frothing agent at 40 mg/L resulted in reduced fiber loss.

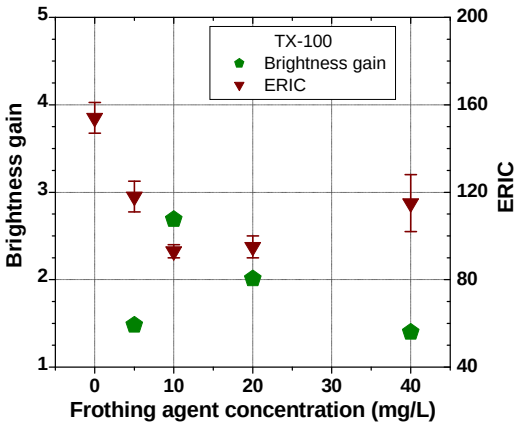

Fig. 6 Effects of frothing agent concentration on ink removal represented by brightness and ERIC of paper made from deinked pulp.

However, fiber rejection decreased slightly at high froth conductivities (corresponding to a frothing agent concentration in the pulp suspension of 40 mg/L). Similar data were obtained for deinking experiments conducted using DI-700. It is noted that high froth conductivity corresponds to a high frothing agent concentration in the pulp suspension. Over dosage of frothing agent concentrations were used in this study (e.g., 20 and 40 mg/L for TX-100) to obtain more data points in a wide range of operating conditions. Sizes of air bubbles in the froth were reduced significantly at very high frothing agent concentrations (e.g., bubble sizes of around 1 mm were observed at TX-100 concentration of 40 mg/L). Observed bubble sizes were about 5 to 10 mm at TX-100 concentrations of 5–10 mg/L. We believe that the froth structure deviated significantly from those of typical laboratory and commercial flotation deinking operations. According to Ajersch and Pelton [2], bubble size can affect fiber entrainment significantly. They found that froth structure can cause fiber loss data to deviate from (below) the entrainment line that they defined based on data from many flotation experiments [2]. The findings of Ajersch and Pelton [2] can be used to explain the reduced fiber loss at TX-100 concentration of 40 mg/L in Figure 5.

Brightness and effective residual ink concentration (ERIC) of paper made from deinked pulp clearly shows the effect of over dosage of a frothing agent (Fig. 6). Ink removal, as represented by the ISO brightness and ERIC of the paper, increased initially to a maximum value with the increase of frothing agent due to increased removal of froth with ink attached. Further increases in application of the frothing agent decreased ink removal due to reduced hydrophobicity of the ink particles by the frothing agent [16]. Therefore, when frothing agent concentration is greater than 20 mg/L in the pulp suspension, over dosage occurs for the experiments conducted. Despite the over dosage, the conductivity probe can continue to provide good measurements of wet reject and solid reject with a reduced chemical dosage range (to 20 mg/L), sufficient for flotation deinking operations.

CONCLUSIONS

This study demonstrated that froth conductivity can be used to monitor wet and solid (mainly fiber) rejections in flotation deinking operations. The constructed conductivity probe responded correctly to temporal changes in wet and solid rejection during flotation experiments. Furthermore, the wet and solid rejections correlate to measured froth conductivity monotonically in a wide range of flotation deinking conditions.

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