

# IMPROVE DEINKING FLOTATION OF WATER-BASED INKS FROM FUNDAMENTAL STUDIES

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## Abstract

Deinking flotation plays a critical role in paper recycling, where small ink particles are removed from paper pulp by air bubbles. While oil-based inks can be easily eliminated, water-based inks remain a significant challenge due to their inherent hydrophilicity and small particle size. This limitation has become a major barrier to increasing the paper recycling rate in the U.S. In the present work, we performed a series of fundamental studies to address the challenges of deinking flotation of water-based inks. *First*, pigment particles (carbon black) from water-based inks were characterized using different methods. Surface hydrophobicity and surface charge of the pigment were determined using contact angle measurement and zeta potential measurement, respectively. The surface free energy of pigment was also determined using van Oss-Chaudhury-Good (vOCG) equation. *Second*, surface force measurements were conducted between a model-ink surface and an air bubble in water using a Force Apparatus for Deformable Surfaces (FADS). The role of different surface forces in deinking flotation was investigated. *Finally*, various hydrophobicity enhancing reagents and surfactants were tested to improve the performance of deinking flotation of water-based inks. The findings in this work provide new insights into the fundamental mechanisms governing the poor flotation response of water-based inks and demonstrate promising strategies to enhance their removal efficiency.

## Introduction and Motivation

Flotation is regarded as the best available technology for deinking used paper. This process, which is widely employed in the mining industry, uses small air bubbles to selectively collect hydrophobic particles, transporting them to a froth phase while leaving hydrophilic particles in the aqueous phase. Although this method is effective for separating hydrophobic oil-based ink from used paper fibers, the technology cannot be used to remove the pigments in water-based inks as they are inherently hydrophilic (Sardjeva & Koeva, 2015). To mitigate the detrimental impact of water-based inks, paper recyclers blend oil-based and water-based feedstocks at levels that allow them to meet their overall brightness specification (Hanecker et al., 2000). At present, paper mills can blend only small amounts of recycled papers coated with water-based inks to minimize the detrimental effect (Heise et al., 1999). Given this limitation, significant portions of papers coated with water-based inks are considered unrecyclable, which is a contributing factor to the low paper recycling rates in the US as compared to those in many European countries.

There are two fundamental issues with removing water-based inks by flotation: i) poor pigment liberation, and ii) the hydrophilic nature of pigments. Liberation is a prerequisite for any physical separation process. Particles that are not adequately liberated from neighboring materials cannot be separated, regardless of the efficacy of the downstream separation process. For water-based inks, poor pigment liberation is due to the strong attractive forces holding the pigments onto fibers (Larsson et al., 1985). Furthermore, the pigments in water-based inks are extremely small, often  $< 1 \mu\text{m}$ , making it difficult to remove them from the aqueous phase by flotation. In the mining industry, flotation is most efficient in a relatively narrow particle size range of 20-150  $\mu\text{m}$ . Therefore, it will be a challenge to remove small amounts of extremely small hydrophilic pigments from used paper fibers by flotation.

In the present work, we addressed two major issues associated with recycling papers printed with water-based inks. These included i) improving pigment liberation and ii) rendering the liberated pigments hydrophobic so that air bubbles can collect them by hydrophobic interaction during flotation. To address the first issue, we increased the  $\zeta$ -potentials of the pigments and fibers using a polyelectrolyte so that the disjoining pressure ( $\Pi$ ) in the thin liquid films (TLFs) of water confined between them becomes high enough to separate them from each other. To address the second issue, we rendered the liberated pigments hydrophobic using a cationic surfactant. We also used hydrophobicity-enhancing reagents, known as Super Collectors, that can increase the contact angles of the pigment surfaces beyond the level obtainable using a cationic surfactant alone. These new approaches greatly improved the brightness of the recycled fibers at higher recoveries.

## Review of Related Work

Recognizing the inefficiencies associated with removing water-based inks from recycle fibers by flotation, the paper recycling industry resorts to adding a post-flotation water-washing step to improve pigments and pulp brightness (Masamizu et al., 1997). Although the water washing step may remove small pigments from recycled fibers, the large water consumption rate is a major barrier to making the process economically viable (Jarrehuit et al., 1997; Putz, 1989; Rangamannar et al., 1992). Furthermore, significant amounts of suspended pigments are reported to the system during the washing process. It is, therefore, necessary to have a water clarification step to remove pigments from recycled wash water (Hodgson, 1996; Masamizu et al., 1997; Schriver & Friel, 1992).

## Technology Approach

In the present work, several different approaches have been taken to address the challenges of removing water-based inks (or pigments) from spent paper fibers. Figure 1a shows a pigment adhering to a fiber with a thin liquid film (TLF) of water formed in between. Thermodynamically, adhesion should occur under conditions of  $\Pi < 0$ . Conversely, a pigment can be liberated from fiber by creating a positive disjoining pressure, i.e.,  $\Pi > 0$ , as shown in the figure, which can be achieved by increasing the repulsive electrical double-layer (EDL) force. The EDL force can be readily increased by adding polyelectrolytes, e.g., sodium silicate. This concept has been validated using a method developed by the authors of this communication (Huang *et al.*, 2022). It should be noted also that liberation by control of disjoining pressure has been in use in the kaolin industry in Georgia and South Carolina to liberate anatase ( $\text{TiO}_2$ ) from kaolin clay and remove them as an impurity by flotation and selective flocculation (US Patent 4,629,556). The authors also developed a novel approach of increasing pigment contact angles from the range of 60-70° to well above 90° to create strongly negative disjoining pressures so that  $\Pi < 0$  and hence, air bubbles can more readily attach to pigments, as illustrated in Figure 1b. PIs developed a two-step hydrophobization process that can increase  $\theta > 150^\circ$ . The novel flotation reagents have been tested successfully for mineral flotation under the trade name Super Collector. Some of the reagents have also been tested for deinking flotation in laboratory-scale experiments. In addition, the authors developed a flotation model that can study the effects of contact angles on flotation performance (Huang et al, 2022).

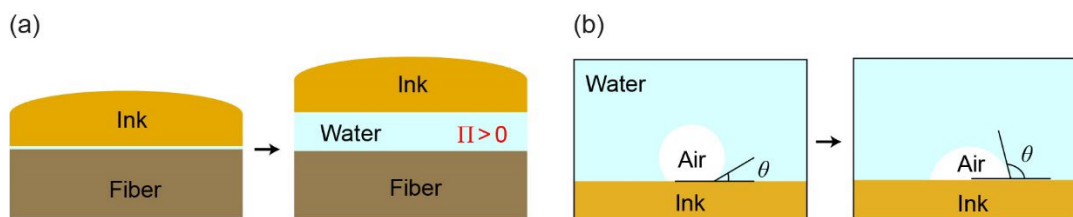


Figure 1. a) An ink particle can be liberated by increasing  $\Pi$  in the TLF formed between fiber and ink; b) an air bubble attaches to the surface of liberated ink particle by increasing its  $\theta > 90^\circ$  and decrease  $\Pi < 0$ .

## $\zeta$ -Potential Measurements

Water-based inkjet ink was obtained from Hewlett-Packard Inc. for use in the  $\zeta$ -potential measurement. The HP32XL was a black, pigment-based ink with a proprietary chemical formulation. While the chemical makeup of the ink was unknown, it may have contained a polymer resin (<12%), defoamers, waxes, and/or dispersants that made up less than

11%, a carbon black pigment (~10%), and water (~66%) (Borchardt, 1999). Basic 20-pound weight, 92 brightness office paper was obtained from Staples. The office paper was used as received for any test requiring unprinted paper. For printed paper samples, a Brother® MFC-J1205W inkjet printer was used to print a black and white standard page document. Ultrapure water with resistivities over 18.2 MΩ.cm was obtained from a Direct-Q water purification system. The Malvern Zetasizer Nano ZS (ZEN3600) was used for measuring the ζ-potential and its distributions. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) from Fisher Scientific were used to control the pH in the measurements.

An ink particle suspension for the ζ-potential measurement was prepared using the following procedure: 0.1 mL of inkjet ink and 50 ml of ultrapure water were added into the plastic vial to form a suspension. To achieve a homogenous suspension of particles, the vial was agitated by a vortex mixer for 2 minutes, followed by hand shaking for 5 minutes. 1 mL of the prepared particle suspension was taken using a syringe pump and transferred into a U-shaped cell (DTS1070, Malvern) for ζ-potential measurements. The ζ-potential measurements were also performed using the diluted paper pulp to study the degree of pigment liberation from fibers. The pulp for the paper, both printed and blank, was prepared by taking 105 g of dry paper fiber and shredding it using a standard cross-cut office shredder. The shredded paper was then mixed down with water to 15% solids by weight. The mixture was placed in a Hobart countertop stand mixer and pulped for 10 minutes. The wet pulp was then weighed out to make the ~0.01% by weight suspension needed for the ζ-potential tests.

## Disjoining Pressure

To better understand the role of the dispersant in the liberation of water-based inks from paper fiber, we constructed disjoining pressure isotherms at different experimental conditions using DLVO theory,

$$\begin{aligned}\Pi &= \Pi_d + \Pi_e \\ &= -\frac{A_{132}}{6\pi h^3} - \frac{\epsilon\epsilon_0\kappa^2}{2\sinh(\kappa h)} [(\Psi_1^2 + \Psi_2^2) \csc(\kappa h) - 2\Psi_1\Psi_2 \coth(\kappa h)] \quad (1)\end{aligned}$$

in which  $\Pi$  is the disjoining pressure in the thin liquid film (TLF) formed between ink and fiber surfaces.  $\Pi$  changes with the film thickness  $h$  and has a unit of N/m<sup>2</sup>. If  $\Pi$  is negative ( $\Pi < 0$ ), the TLF will rupture, and the ink and fiber will hetero-coagulate. If  $\Pi > 0$ , the ink will not attach onto the fiber surface. According to Eq. (1), two surface forces contribute to  $\Pi$ , *i.e.*, van der Waals (vdW) force ( $\Pi_d$ ) and electrical double layer (EDL) force ( $\Pi_e$ ).  $\Pi_d$  can be readily calculated if the Hamaker constant ( $A_{132}$ ) between ink and fiber is known.  $\Pi_e$  is a function of Debye length ( $\kappa^{-1}$ ), surface potentials of ink ( $\Psi_1$ ) and fiber ( $\Psi_2$ ), and the dielectric constant of water ( $\epsilon$ ). In the present work,  $A_{132}$  has been determined using the combining rule (Israelachvili, 2011),

$$A_{132} = (\sqrt{A_{11}} - \sqrt{A_{33}})(\sqrt{A_{22}} - \sqrt{A_{33}}) \quad (2)$$

in which  $A_{11}$ ,  $A_{22}$ , and  $A_{33}$  are the Hamaker constants for ink, fiber, and water, respectively. The values for  $A_{11}$ ,  $A_{22}$ , and  $A_{33}$  were obtained from Israelachvili (2011).  $\Psi_1$  and  $\Psi_2$  were approximated using the ζ-potential measured using the Malvern Zeta meter described in the foregoing section.

## Contact Angle Measurement

Contact angle plays an important role in deinking flotation. In general, the higher the contact angles of pigment particles are, the more efficient the deinking flotation becomes. In the present work, a series of contact angle measurements have been conducted to validate the improvement of pigment surface hydrophobicity achieved by cationic surfactants and super collectors. The measurements were conducted using either glass surfaces coated with water-based ink or a model ink surface, *i.e.*, hydrophilic quartz surfaces. The black water-based ink sample (HP 32XL) had a solid content of 22% by weight. Prior to the coating process, the HP ink was diluted in DI water to form a stock solution for coating. In some tests, different collectors (hydrophobizing agents) were also added in the stock solution to increase the hydrophobicity of the pigment particles, such as a series of cationic surfactants, and two commercially available deinking reagent (DR1) and deinking reagent (DR2). The coating test was conducted by transporting a desired amount of the stock solution onto a flat glass slice (25x100x1 mm) using a pipette. The slice was subsequently dried in an oven for 24 hours at 70 °C so that a homogeneous coating of water-based ink was formed on the slice. A drop of DI water was then placed onto the ink-coated slices to determine the contact angles using the sessile drop technique *via* a Rame-Hart goniometer. For a given surface, eight different measurements were conducted and averaged.

## Flotation

After completing the  $\zeta$ -potential measurements and constructing disjoining pressure isotherms, a candidate reagent package was identified to achieve better liberation and increased hydrophobicity of water-based inks. Laboratory-scale batch flotation tests were then conducted as a means to compare the deinking performance of the novel reagents against that of an industry stand. The flotation tests were conducted using a modified INGEDE method (International Association of the Deinking Industry, 2011). Initially, a pulp feedstock was generated by mixing inkjet-printed paper (similar to those described above) in a Hobart mixer for a fixed period. During the pulping process, collectors and pH modifiers were added directly into the Hobart mixer. The collectors used in this work include a non-ionic industrial standard collector for deinking flotation, a novel cationic surfactant (Strickland et al., 2022), as well as a “super collector.” The full pulping time for flotation was 10 minutes with the dispersant and pH modifying chemicals being added at the beginning, the collector being added after the first five minutes of pulping, and the “super collector” added just prior to flotation. Once the pulping process was complete, a 20-gram pulp was taken and made into a paper pad for brightness measurements to obtain a pre-flotation brightness. Another 150-gram pulp was added to a 2-liter flotation cell and mixed down with warm tap water to a 1% by dry fiber weight solution. A 5-minute flotation test was performed in a Denver cell at 1,200 rpm and an airflow rate of 2 liters per minute. The floated ink was discarded while a representative sample of the remaining pulp was taken, diluted, and made into a paper pad for brightness measurements similar to previous tests. The remaining pulp was dried and weighed to determine the fiber yield of the flotation process.

The primary indicators of process performance included the calculated fiber yield and the brightness gain. For these tests, brightness gain was calculated by the difference in final pad brightness and the control pre-flotation pad brightness. Due to the effect of the dispersant on the pre-flotation brightness, all flotation tests use the pre-flotation brightness of the non-dispersant tests.

## Discussion

Figure 2 shows the particle size distribution of the carbon black pigment presented in the oil- and water-based inks. The oil-based ink exhibited an average particle size ( $d_{50}$ ) of 2428 nm, while the  $d_{50}$  of the water-based ink was only 157 nm. Compared with mineral flotation, particle sizes in deinking flotation are significantly smaller, resulting in a low probability of bubble-ink particle collisions, and consequently, reduced flotation efficiency. Notably, the  $d_{50}$  of the water-based ink is less than one tenth that of oil-based ink, suggesting that removal of water-based ink is considerably more challenging than that of oil-based inks.

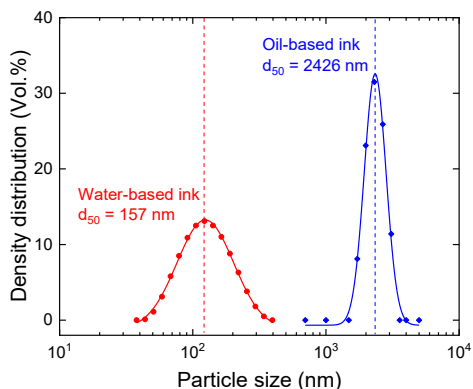


Figure 2. Comparison of the pigment sizes of water- and oil-based inks.

Figure 3 shows the results of the  $\zeta$ -potential measurements conducted as a function of pH. The samples used for the measurements included portions of unprinted office paper pulped with no chemical additives and a sample of water-based ink. As shown, the  $\zeta$ -potentials of unprinted office paper became more negative as the pH increased. On the other hand, the  $\zeta$ -potentials of water-based ink were substantially more negative than those of office paper, which was probably due to the fact that a small amount of dispersant with high-charge density was used during the manufacturing of water-based ink (Chabot et al., 1993).

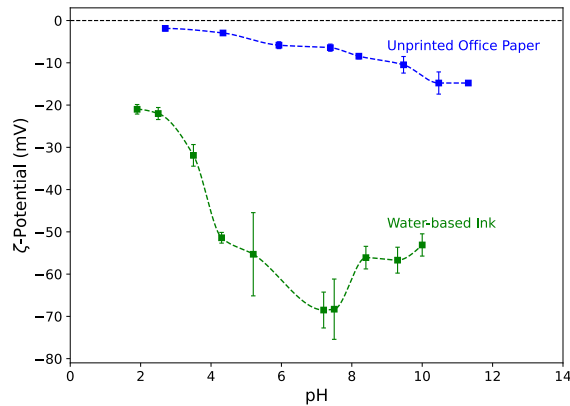


Figure 3.  $\zeta$ -potential vs. pH for unprinted office paper and water-based ink used in inkjet printers.

Due to the marked differences in the  $\zeta$ -potentials between ink and paper, it was easy to determine if the two different materials, *i.e.*, ink and fiber, existed as dispersed (liberated) particles or as an aggregate. The authors of this communication developed a method of determining the degree of liberation of oil-based ink particles from fibers by analyzing the  $\zeta$ -potential distributions (Huang *et al.*, 2022).

Figure 4a shows the  $\zeta$ -potential distributions of a printed office paper sample that has been pulped without reagents at a pH of 9.7, while Figure 4b shows the same after pulping with a dispersant at pH 9.8. The ordinates of the two figures represent the number of densities of particles with different  $\zeta$ -potentials. As can be seen in Figure 4a, there is one distinct peak in the frequency vs.  $\zeta$ -potential plot, indicating that ink particles interacted with fibers to form aggregates despite the fact that both were negatively charged. This observation indicated poor liberation of ink particles from fibers. In the presence of a dispersant, however, a bimodal distribution of frequency was observed, as can be seen in Figure 4b, with one peak observed at -12.5 mV and another at -22 mV. According to the  $\zeta$ -potential measurements shown in Figure 4, it would be reasonable to assume that the -12.5 mV peak represents the unprinted office paper, while the -22 mV peak represents the partially liberated water-based ink. The results shown in Figure 4 suggest that ink liberation can be improved by adding a polyelectrolyte during pulping.

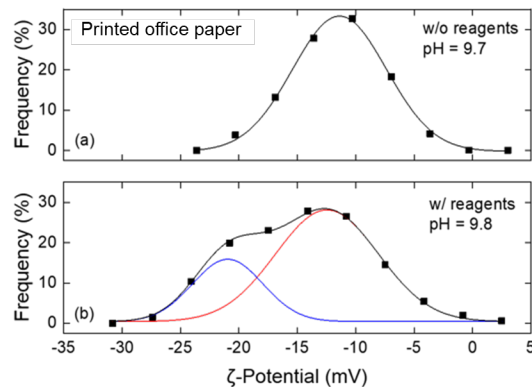


Figure 4.  $\zeta$ -potential distribution curves obtained in the absence (a) and presence (b) of a dispersant. IN the presence of dispersant, ink particles are separated (or liberated) from fibers.

To obtain a better understanding of the mechanisms involved in the liberation of water-based ink pigment, DLVO calculations have been carried out using Eq. (1) and the disjoining pressure isotherms ( $\Pi(h)$ ) in the presence of dispersant are plotted in Figure 5. As shown, the van der Waals force ( $\Pi_d$ , blue) between water-based ink and office paper is attractive (negative), which is due to the positive Hamaker constant ( $A_{132}$ ) for the interaction between water-based ink and office paper in water. As a result, the pigment particles in the water-based ink are difficult to be detached/liberated from the paper fibers. Note that the attractive van der Waals force between ink and fiber can also lead to ink redeposition, which is a process that the pigment particles dispersed in the paper pulp re-attach onto the fiber surfaces. Also shown in Figure 5 is the EDL force ( $\Pi_e$ , green) calculated using the Hogg-Healy-Fuerstenau (HHF)

model (1966). The Debye length ( $\kappa^{-1}$ ) used in the calculation was estimated at 1.1 nm based on the dispersant concentration in the system. The  $\zeta$ -potentials for water-based ink ( $\Psi_1$ ) and office paper ( $\Psi_2$ ) were estimated from Figure 3 with at  $\Psi_1 = -68$  mV and  $\Psi_2 = -12.5$  mV. Since both water-based ink and office paper were negatively charged,  $\Pi_e$  was repulsive (positive), which reached maximum at 2.75 nm with a value of  $\sim 45,000$  N/m<sup>2</sup>. The total disjoining pressure ( $\Pi$ , black) was obtained by adding  $\Pi_d$  and  $\Pi_e$ . Due to the repulsive  $\Pi_e$ , a huge force (pressure) barrier was obtained at 3.2 nm with a value of  $\sim 28,000$  N/m<sup>2</sup>. This repulsive force barrier plays an important role in improving ink pigment liberation and decreasing the degree of ink redeposition.

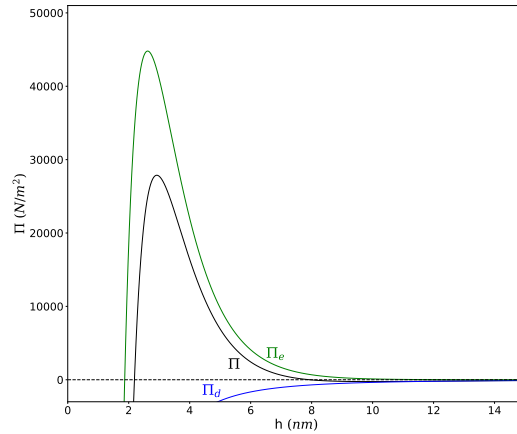


Figure 5. Disjoining isotherm between water-based ink and office paper when using dispersant. Combining method was used to calculate  $A_{132} = 6.75 \times 10^{-21}$  J with a Debye length ( $\kappa^{-1}$ ) of 1.1 nm and  $\Psi_1 = -68$  mV and  $\Psi_2 = -12.5$  mV.

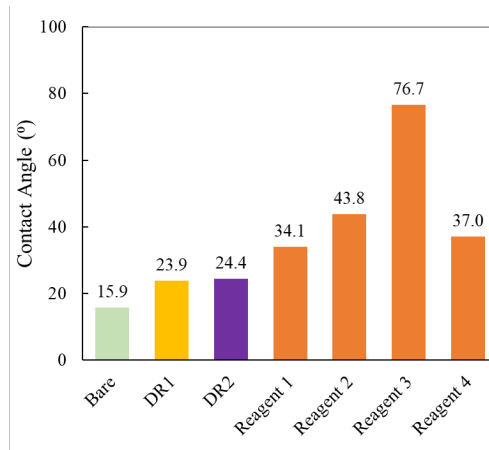


Figure 6. Summary of the contact angle measurement results obtained with standard deinking reagents (DR1 and DR2) and cationic surfactants (Reagents 1-4).

Improving the hydrophobicity of the water-based ink pigment was done by measuring the contact angles of several different types of reagents. A higher contact angle indicates a more hydrophobic ink pigment particle. Figure 6 shows a summary of the contact angle measurements obtained with the glass surfaces coated with water-based HP ink. As shown, the ink-coated surfaces without any collector (hydrophilizing agent) treatment showed a contact angle of 15.9°. The collectors commonly used in the paper recycling industry, *e.g.*, DR1 and DR2, increased the contact angles of water-based ink to  $\sim 25^\circ$ . The contact angles obtained using the four different cationic surfactants were substantially higher as shown in Figure 6. The improvements were in the range of 10-50°.

Note that all contact angles reported in Figure 6 were below 90°. To further increase the contact angles above 90°, a series of hydrophobizing reagents known as “Super Collectors” (SC) have been developed. Figure 7 shows a summary of the contact angle measurements conducted using various Super Collectors. The measurements were conducted using a model ink surface (hydrophilic quartz). The base contact angle of 85° was obtained using dodecylamine hydrochloride (DAH) alone. The measurements conducted using both DAH and Super Collectors increased the contact

angles well over 90°. The highest contact angle obtained was as high as 150°, which was achieved using a combination of DAH and SC4. The large increase in contact angle can lead to a substantial increase in the kinetics of bubble-ink interactions and deinking flotation.

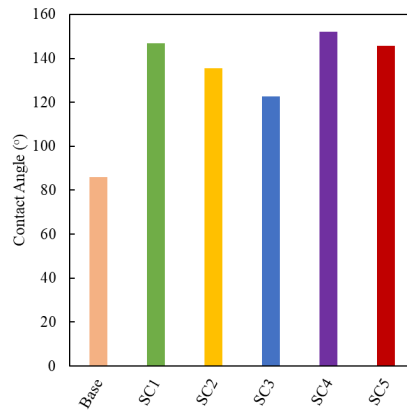


Figure 7. Summary of the contact angle measurement results obtained with different super collectors (SC).

A series of deinking flotation tests were carried out to validate the benefits of improving ink liberation and increasing the hydrophobicity of pigments. The flotation tests were conducted with the pulp of office papers printed with water-based inks. Two different collectors (hydrophobizing agents) were studied, *i.e.*, an industry standard collector and a novel collector (cationic surfactant), with the results presented in Figure 8. The term ‘delta’, which represents the brightness difference between the product and the feed pulp, may be a good indicator for improved flotation efficiency. Without dispersant, the cationic surfactant was able to achieve a brightness delta of 9.7. Use of the dispersant in the pulping stage increased the brightness delta to 13.1, a 35% improvement. Note here that a combined use of the dispersant and a cationic surfactant resulted in a three-fold increase in brightness over that of the case of using an industry standard.

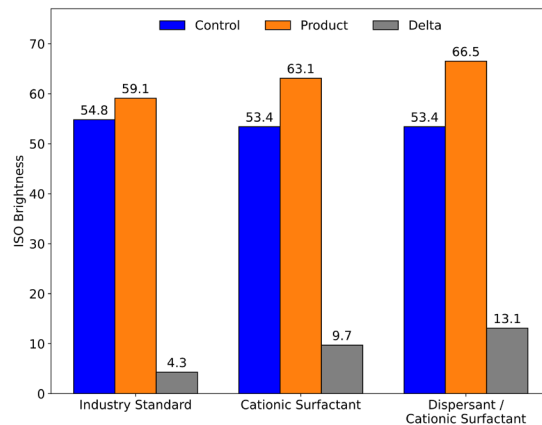


Figure 8. Flotation results from using improved chemistries with a dispersant to improve liberation and flotation performance.

Figure 9 shows a plot of the brightness improvements (Delta) vs. fiber yields using various reagents tested in the present work. Use of the industry standard (DR1) increased the brightness by 4 points at approximately a 50% fiber yield. Reagent 1 was tested at two different dosages. At the higher dosage, different amounts of a Super Collector 1 (SC-1) were also used to produce substantially high brightness fibers. Using Super Collector 2 (SC-2), the brightness was further increased by 10.5 points but at lower fiber yields. Thus, there is a tradeoff between brightness improvement and fiber yields. As shown in Figure 9, Reagent 1 in combination with SC-1 probably provides to the optimum deinking results, achieving both relatively high fiber yield and high brightness. Note also that the fiber yields obtained with Reagent 1 and Super Collectors are substantially higher than the same obtained with DR1, which may be due to that the novel hydrophobizing reagents can lead to much higher contact angles of pigment particles than the standard

reagents. In general, the higher the contact angles of the pigment particles, the higher the deinking flotation efficiencies.

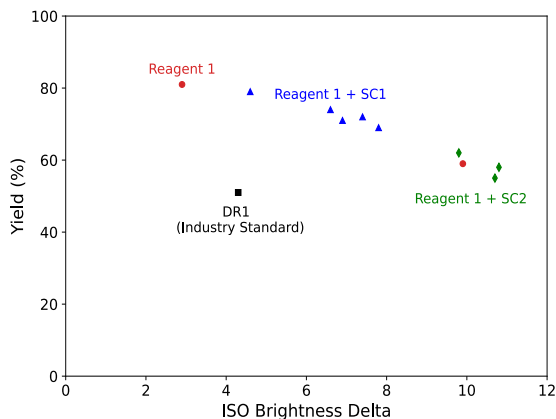


Figure 9. Bench scale flotation results of water-based ink printed fiber where DR1 is an industry-standard non-ionic collector, Reagent 1 is a cationic surfactant, and SC1 and SC2 are formulated “super collectors” to increase hydrophobicity.

## Conclusion and Recommendations

Liberating ink particles from used fibers is a prerequisite for efficient deinking flotation. The results obtained in the present work showed that  $\zeta$ -potential measurements can be useful for studying the liberation of water-based ink from paper fibers. The results of the liberation studies were in good agreement with the ISO brightness measurements. Flotation separation tests were conducted to show the importance of i) improving pigment liberation and ii) increasing pigment hydrophobicity. The use of a polyelectrolyte to improve pigment liberation and a suitable cationic surfactant to render the pigment hydrophobic greatly improve the removal of water-based ink from used paper fibers. The use of Super Collectors to further increase the pigment hydrophobicity further increased the brightness of recycled fibers but at a slight loss of fiber yield. The novel deinking reagents developed in this work, including polyelectrolyte and Super Collectors, can be readily implemented in industrial deinking flotation processes as direct replacements for the current deinking flotation reagents. In the future, in-plant trials will be conducted to confirm the improved performance using these technologies.

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