A new method of studying the fundamental mechanisms involved in pigment liberation from recycle papers

KAIWU HUANG, KRIS STRICKLAND, AARON NOBLE, ROE-HOAN YOON, AND CESAR BASILIO

ABSTRACT: Deinking flotation is the most efficient and widely used method of removing ink particles from printed papers to improve the recyclability. A prerequisite for successful deinking flotation is detachment of pigments from paper fibers, a subprocess known as liberation. The degree of liberation is usually determined via hyperwashing tests, which are costly and time consuming. Furthermore, they provide no information on the fundamental mechanisms controlling liberation.

In the present work, we developed a new method in which ζ-potentials of the particles in a pulp are measured and analyzed. If pigments are not liberated from paper fibers, a frequency distribution plot gives a single peak, while two peaks appear when they are liberated. One can readily determine the degrees of liberation from the peak positions and peak heights. In addition, the ζ-potential data can be used to construct disjoining pressure isotherms using the DLVO theory that are useful to better understand the fundamental mechanisms involved and the roles of different reagents used to improve pigment liberation.

Application: Flotation deinking is widely used to remove ink from paper fibers. A prerequisite to flotation is the liberation of ink particles from fibers. We have developed a new method of determining the degree of liberation. The new method will also be useful for better understanding the mechanisms involved in liberating ink particles from paper fibers.

Recycling of wastepaper is important for improving environmental sustainability and economic returns. A ton of recycled paper can save 17 trees, 250 lb of carbon dioxide (CO_2) per year, 380 gal of oil, 3 yd^3 of landfill space, 4000 kWh of energy, and 7000 gal of water [1]. A critical factor affecting the overall yield of paper recycling is the efficiency of the deinking process [2]. Generally, a deinking process involves various steps, e.g., screening, pulping, flotation, washing, thickening, and bleaching [3].

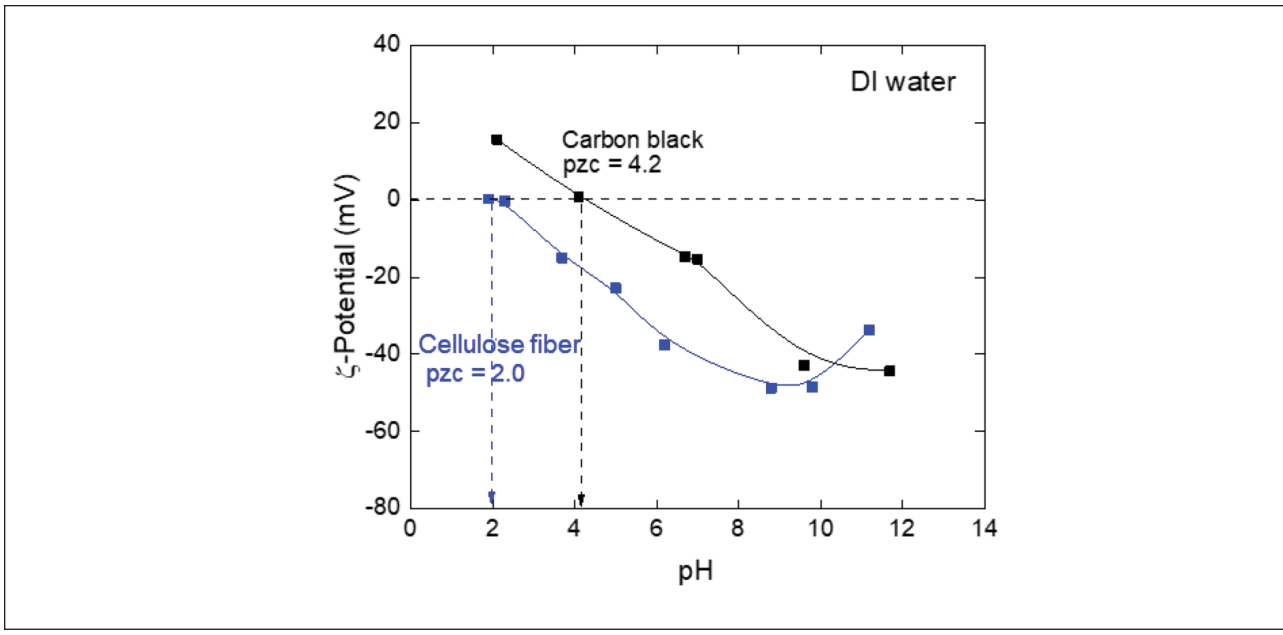
Flotation deinking can effectively remove the ink particles to improve the quality of the recycled fibers [4-5]. The effective particle size range for flotation is usually between 10 and 100 μm [6-7]; therefore, the ink particles outside this range are removed by water washing and screening. Flotation efficiencies are typically less than 60%, which makes it necessary to repeat the process 4 to 5 times. One of the reasons for the poor efficiencies is the difficulty in liberating ink particles from spent paper fibers. It is generally recognized that liberation is a prerequisite for flotation deinking. It is, therefore, important to know the degree of liberation prior to flotation deinking. A conventional way to determine the degree of liberation is to perform a hyperwashing test in which a pulp is washed using a screen for several times while monitoring brightness changes. While the hyperwashing tests are relatively easy to perform and useful for feedstock evaluation, they are also time consuming and require lots of water. Moreover, they do not give fundamental information that can be used to improve the liberation process.

In the present work, a new method of determining the degree of pigment liberation from recycle papers printed with oil-based ink has been developed. In this method, ζ-potential measurements are conducted on a pulp sample, and the resultant data are subsequently used to determine the fraction of the pigments liberated from paper fibers on the basis of the frequency distribution curves. The ζ-potential data can also be used to construct disjoining pressure isotherms that can be used to better understand the mechanisms involved in liberation and to identify the reagents that can improve liberation and thereby increase the recyclability of postconsumer paper products.

EXPERIMENTAL

Materials

An artificial pulp and an old newsprint (ONP) pulp were prepared to study the mechanisms of pigment liberation from the papers printed with oil-based ink. The artificial pulp was prepared by mixing carbon black (Orion Engineered Carbon LLC; Senningerberg, Luxembourg) and clean cellulose fibers (Thiele Kaolin Company; Sandersville, GA, USA) in water. The ONP printed with oil-based ink was obtained from *The Roanoke Times* in Roanoke, VA, USA. Hydrochloric acid (HCl, Thermo Fisher Scientific; Hampton, NH, USA) and sodium hydroxide (NaOH, Fisher Scientific) were used for pH



1. The ζ-potentials of carbon black particles and cellulose fiber as a function of pH (DI = deionized; pzc = point of zero charge).

adjustment. In some pulping tests, hydrogen peroxide (H_2O_2, 30% solution, Fisher Scientific) and sodium silicate (Na_2SiO_3, >97%, Fisher Scientific) were used as additives. For ζ-potential measurements, Ultrapure water with a resistivity of >18.2 MΩ•cm was obtained from a Direct-Q 3 UV water purification system (Merck; Kenilworth, NJ, USA).

Methods

The ζ-potentials of the carbon black, cellulose fiber, and ONP pulp were measured using a Malvern Zetasizer ZEN3600 (Malvern Panalytical; Malvern, UK). For the case of single component systems, e.g., carbon black and cellulose, particle suspensions were prepared by mixing 0.05 g sample in 50 ml Ultrapure water. A particle suspension was agitated by means of a vortex mixer for 5 min, followed by 10-min conditioning in an ultrasonic bath. All the ζ-potential measurements were conducted at room temperature. For the case of binary systems, e.g., carbon black and cellulose fiber mixture (artificial pulp) or the ONP pulp, the particle suspensions were diluted to ~0.01% solids by weight.

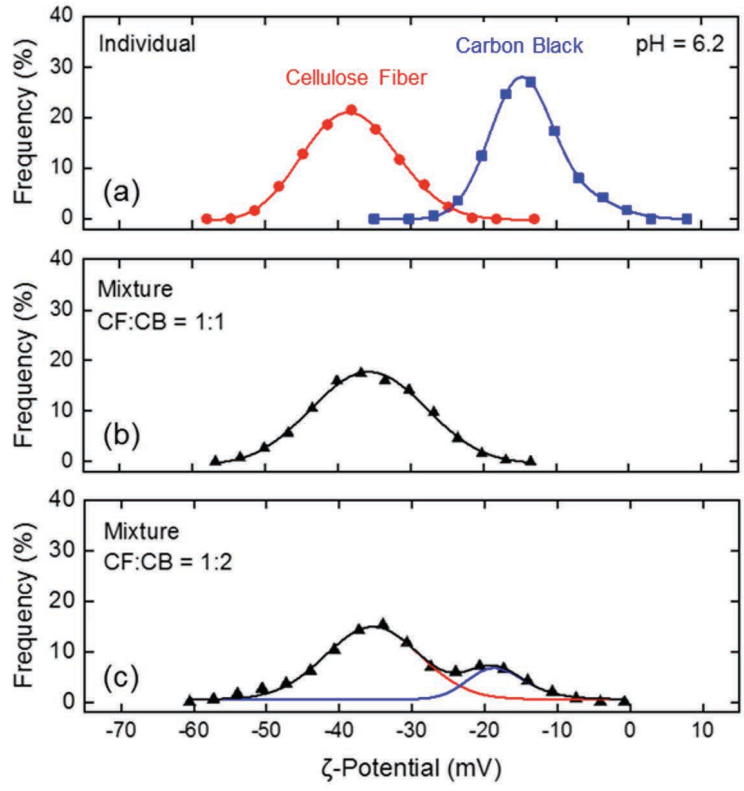
A series of pulping and hyperwashing tests were conducted on a sample of ONP printed with oil-based ink. The ONP sample was shredded first and mixed with water at 15% solids by means of a Hobart mixer (Hobart; Troy, OH, USA) for 20 min at a medium agitation setting. In a given test, 20 g of pulp (3 g dry equivalent) were diluted to 0.5% solids by adding 580 g of water. The sample was then agitated for 2 min at 250 rpm and subjected to screening at 50 mesh. The screen overflow was collected and then mixed with water again to bring the total weight back to 600 g at ~0.5% solids. The hyperwashing step was repeated once more. After the second washing cycle, the screen overflow was collected and then brought back to 600 g total with water to obtain a final suspension of ~0.5% solids. The sample was then agitated at 250 rpm for 30 s. Finally, the sample was filtered and dried at 60°C.

Washed samples were dried and subjected to ISO brightness measurements using the standard procedure. The brightness measurements were made in four different places on a pad and averaged. The scanning electron microscope (SEM) images of the pad were also taken using a JEOL IT-500 SEM (JEOL Ltd.; Tokyo, Japan).

RESULTS AND DISCUSSION

A series of ζ-potential measurements were conducted as a means to determine the number densities of particles with different surface charge characteristics suspended in water. In the present work, carbon black (CB) was used as a surrogate for the black pigment in oil-based ink, while cellulose fiber (CF) was used as a surrogate paper fiber. **Figure 1** shows the ζ-potentials of CB and CF particles suspended in Nanopure water (Thermo Fisher Scientific) as a function of pH. As shown, the CB samples exhibited positive ζ-potentials at pH < 4.2, while they became negative at pH > 4.2. Thus, the point of zero charge (pzc) of the CB sample was pH 4.2. On the other hand, the pzc of the CF sample was pH 2.0, at which the CF surface was uncharged.

Note here that the ζ-potentials of the CB and CF particles were substantially different from each other, which makes it easy to determine if the two different materials existed as dispersed particles, representing liberated state, or interact with each other to form coagula, representing interlocked state. If the latter is the case, the ζ-potentials of the coagula should assume a value between the two components at a given pH. This method of studying the interactions between CB and CF particles, as applied for studying



2. a) The ζ-potential distributions of carbon black (CB) and cellulose fiber (CF); b) ζ-potential distribution of a 1:1 mixed CB and CF suspension; and c) ζ-potential distribution of a 1:2 mixture. All measurements were conducted at pH 6.2.

pigment liberation from recycle paper, is similar to the method of studying the interactions between bubbles and particles in flotation, coal and clay particles in coal beneficiation, and bitumen and clay particle interactions in the heavy oil extraction process on the basis of distributed ζ-potentials of the particulate materials involved [8-10].

The data presented in **Fig. 2b** show the results of the ζ-potential measurement conducted with a 1:1 mixture by weight of CB and CF in Nanopure water. The measurements were conducted at 0.5% solids. As shown, the frequency distribution of particles at different ζ-potentials exhibited a single peak at -37.0 mV, indicating that the CB and CF particles interacted with each other to form coagula at pH 6.2, despite the fact that both were negatively charged.

To better understand the mechanisms involved in the coagulation between the CB and CF particles, we carried out DLVO calculations using the ζ-potential data presented in Fig. 2a. Equation 1 represents a form of the DLVO theory [11-12],

$$\Pi = \Pi_d + \Pi_e = -\frac{A_{132}}{6\pi h^3} - \frac{\varepsilon\varepsilon_0\kappa^2}{2\sinh(\kappa h)}\left[\left(\psi_1^2+\psi_2^2\right)\cos ech(\kappa h) - 2\psi_1\psi_2\coth(\kappa h)\right] \quad (1)$$

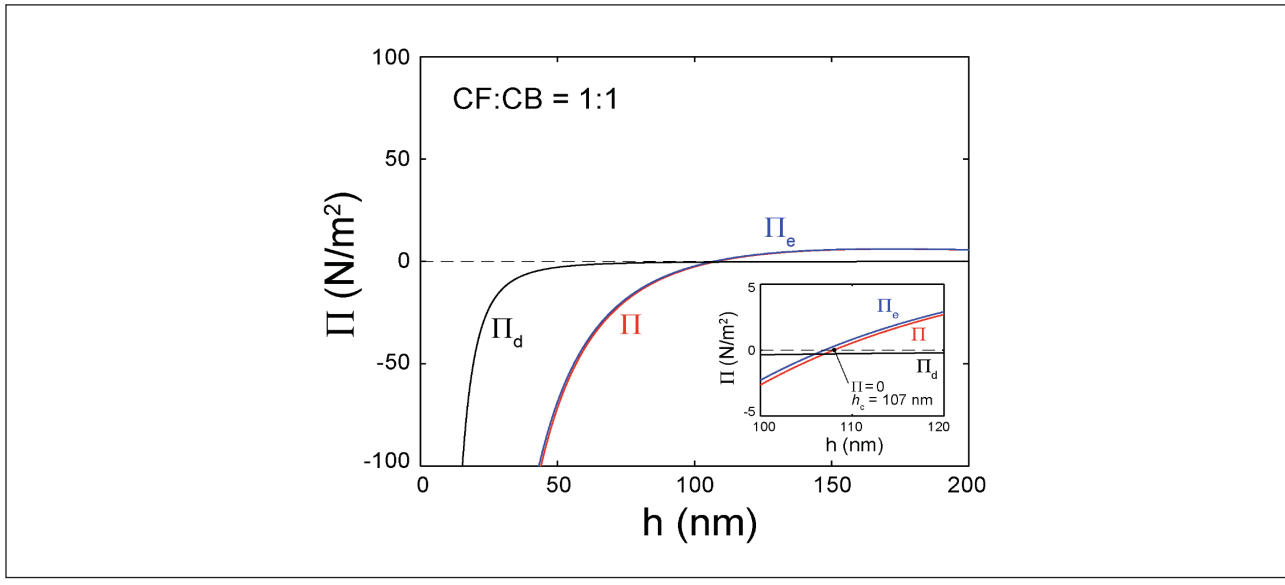
in which Π is the disjoining pressure in the thin liquid film (TLF) formed between CB and CF in aqueous phase. If Π, which is given in units of mN/m², is negative (Π < 0), the TLF will rupture and the CB and CF will heterocoagulate. If Π > 0, the two particles will stay dispersed in water. The Π term consists of the van der Waals-dispersion force component (Π_d) and the electrical double-layer (EDL) force component (Π_e). The disjoining pressures vary with the separation distance (h) between two interacting surfaces in close proximity, and are functions of the permittivity of vacuum (ε_0), dielectric constant of water (ε), reciprocal Debye length (κ^{-1}), double-layer potential (ψ_1) of cellulose fiber, and the same (ψ_2) of CB. Thus, the first term of Eq. 1 represents the pressure in the film due to the van der Waals force, while the second term represents the same due to the EDL force.

The Π_d can be calculated if the Hamaker constant (A_{132}) for the interaction between CF 1 and CB 2 in water 3 are known. In the present work, A_{132} has been calculated using the combining rule [13],

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

in which A_{11} = 5.8x10^{-20} J [14], A_{22} = 1.1x10^{-19} J [15], and A_{33} = 3.7x10^{-20} J [13] represent the Hamaker constants in vacuo. Substituting these values into Eq. 2, one obtains A_{132} of 0.68x10^{-20} J.

Equation 1 has been plotted in **Fig. 3** using the



3. Disjoining pressure isotherm (Π(h)) for the interaction between carbon black (CB) and cellulose fiber (CF) in water: Π_d represents the van der Waals component of Π, and Π_e represents the electrical double-layer (EDL) force component of Π; A_{132} = 6.8x10^{-21} J, ψ_1 = -38.7 mV, ψ_2 = -12.7 mV, and k^{-1} = 96 nm.

ζ-potential data presented in Fig. 2a. The results show that the pressure (or force) barrier to film rupture is very small. Therefore, the CF and CB particles should heterocoagulate readily with each other, as was the case when the CF and CB were mixed at a 1:1 weigh ratio. When the two different materials were mixed in Nanopure water at a 1:2 ratio, a second peak appeared at a lower ζ-potential (-20.8 mV), as shown in Fig. 2c. It appears that the CF particles had a limited capacity to accommodate all of the CB particles in view of the large difference in particle sizes involved. Due to the large particle size (d_{50} = 480 μm) of the CF in the mixture relative to the particle size of CB (d_{50} = 2.4 μm), the surface area of the CF substrate, on which CB particles can adsorb, may be limited. Since the heights of the two peaks in Fig. 2c represent the number densities of the CF and CB particles, one can calculate the fraction of the CB particles that are not associated with CF from the peak heights. The peak heights for CF and CB are 14.9% and 6.7%, respectively, which gives 31.0% as the fraction of the free CB particles. This number may be equivalent to the degree of liberation. In this case, the liberation is driven by the limitation in the surface area of the CF substrate.

The inset of Fig. 3 shows changes in Π, Π_d, and Π_e, with film thickness h at separations close to the critical film thickness (h_c = 107 nm), where Π = 0 and hence the TLF should rupture. A consequence of film rupture is, of course, the heterocoagulation between CF and CB particles, which is not a desired situation for pigment liberation and hence for deinking flotation.

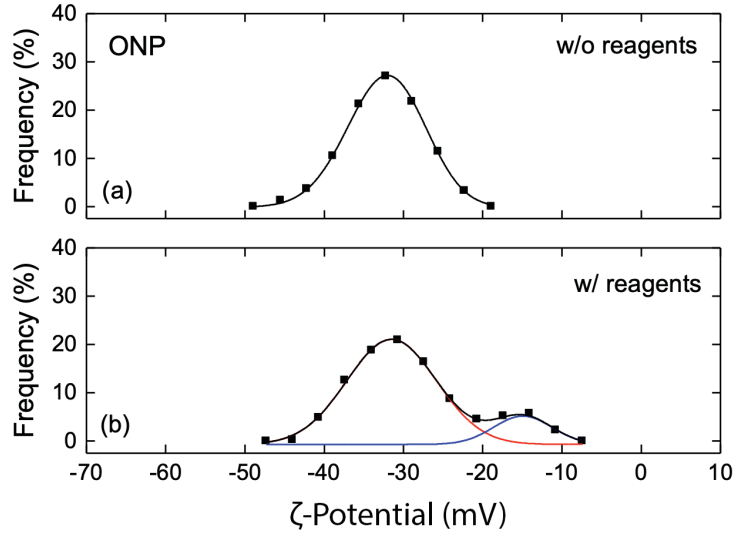
Note also at the inset that Π becomes negative at h below the h_c. In this region, both the EDL and van der Waals forces were negative (or attractive); however,

$$\left|\Pi_e\right| > \left|\Pi_d\right| \quad (3)$$

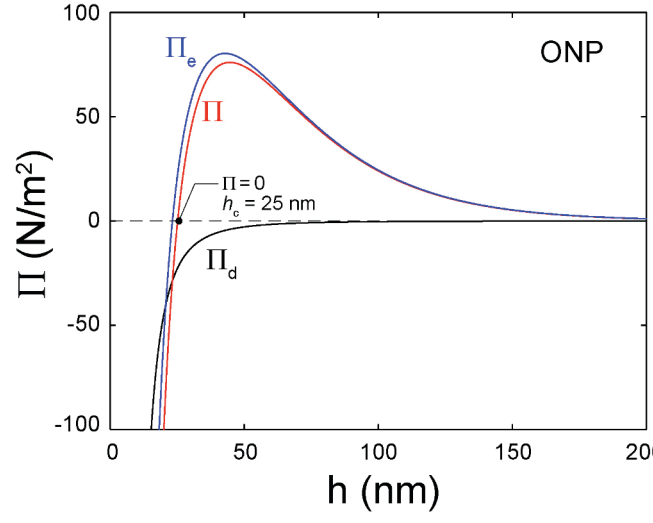
this indicates that the EDL force was more attractive than the van der Waals force, and the driving force for film thinning and rupture was the EDL force. This is contrary to many colloidal systems in which coagulation is induced by the attractive van der Waals force. The unexpected observation can be attributed to the fact that A_{132} was small for the interaction between CF and CB particles in water, which in turn can be attributed to the fact that A_{11} and A_{22} are small, as both are carbon-based materials with small atomic weights.

Figure 4 shows the distributed ζ-potentials measured with an ONP sample (from *The Roanoke Times*) dispersed in the tap water in the absence of any reagent. The paper was printed with an oil-based ink. As shown, the frequency distribution shows a single peak at ζ= -32.6 mV, indicating that the ink particles were heterocoagulated with little or no liberation of CBs from CF substrates.

The same ONP sample was subjected to another set of ζ-potential measurements after conditioning the pulp in the presence of a set of chemicals, which included 1.8% Na_2SiO_3, 0.6% NaOH, and 0.7% H_2O_2 by weight of ONP. These are the standard reagents that are used to determine the recyclability of papers printed with ink. The result in this case shows a bimodal ζ-potential distribution with two peaks, one at -31.5 mV and the other at -14.9 mV, the latter representing the CB pigments liberated from the CF substrates. The percentage distributions represented by the two peak heights are 21.1% and 5.2% for the CF and CB particles, respectively, in the pulp, from which one can estimate the degree of liberation of the CB pigment to be 19.8%. In this case, the liberation is driven by the EDL force,



4. a) The ζ-potential distribution of an old newsprint (ONP) printed with oil-based ink; and b) ζ-potential distribution in the presence of chemical additives: 1.8% sodium silicate, 0.6% sodium hydroxide, and 0.7% hydrogen peroxide.

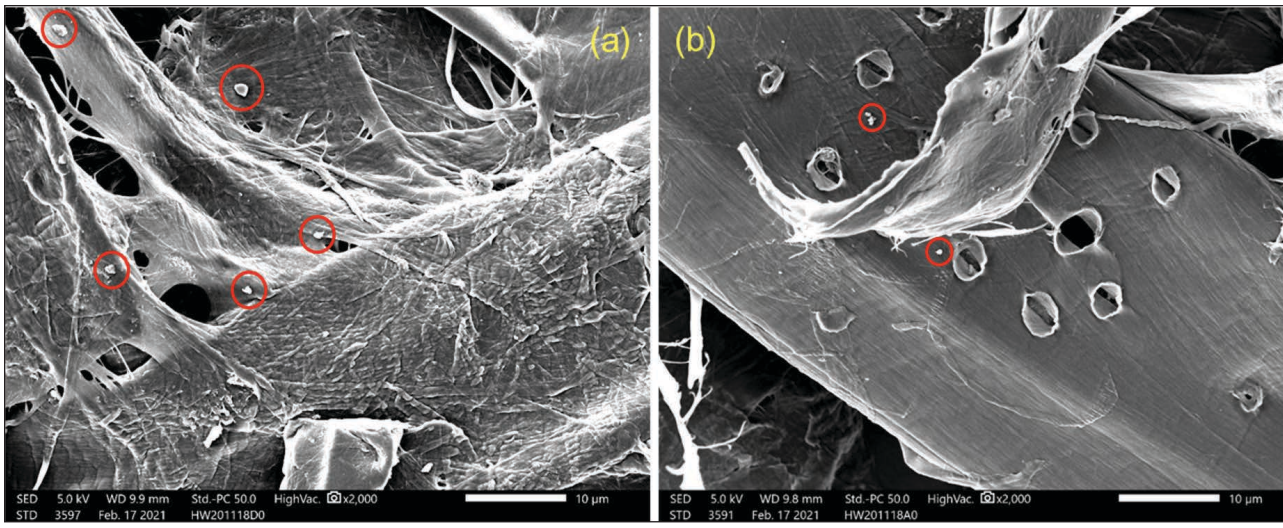


5. Disjoining pressure isotherm (Π(h)) for the interactions between wood fiber and pigment in oil-based ink: Π_d represents the van der Waals component of Π, and Π_e represents the EDL force component of Π_e: $A_{132} = 6.8 \times 10^{-21}$ J, $\psi_1 = -31.5$ mV, $\psi_2 = -14.9$ mV, and $k^{-1} = 31$ nm.

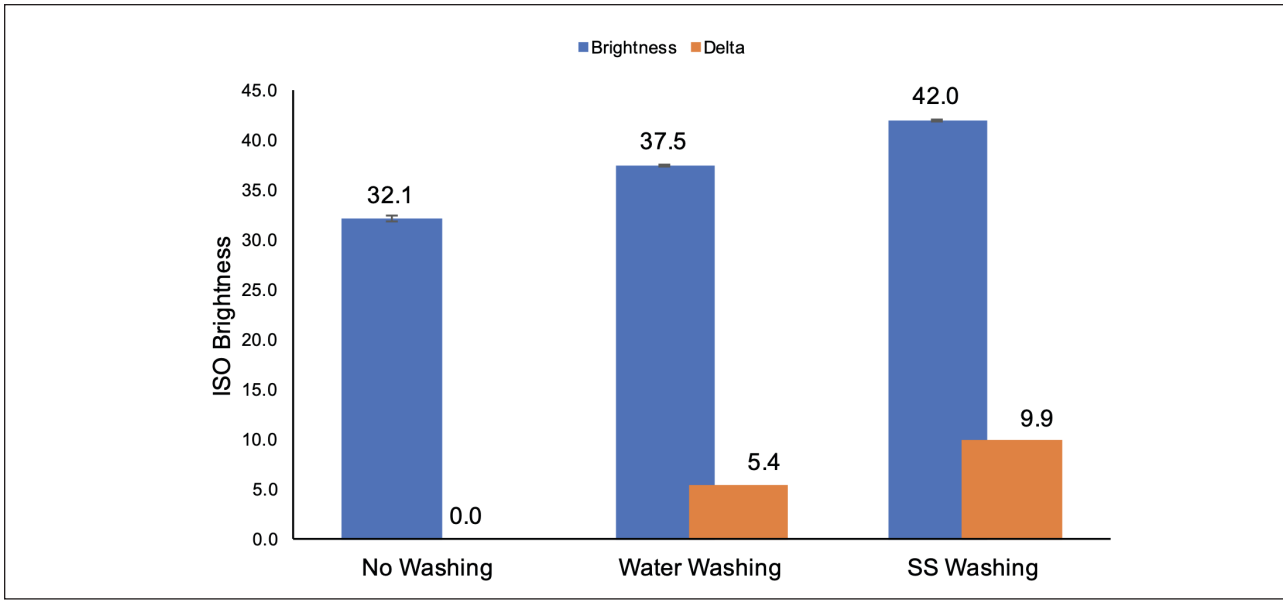
which gave rise to a high force (or disjoining pressure) barrier, as can be seen in **Fig. 5**.

Using the two peak ζ-potentials as the double-layer potentials for CF substrate (ψ_1 = -31.5 mV) and for CB pigments (ψ_2 = -14.9 mV), a DLVO calculation has been carried out using Eq. 1, with the resulting $\Pi(h)$, $\Pi_d(h)$, and $\Pi_e(h)$ isotherms presented in Fig. 5. In this calculation, we used the same Hamaker constant ($A_{132} = 0.68 \times 10^{-20}$ J) as for the isotherms obtained for the artificial mixtures (Fig. 3). A distinct difference between the two different calculations was that the disjoining pressure isotherms for the ONP sample showed orders of magnitude higher force barrier, which may provide a mechanism by which CB pigments stay liberated from the CF substrates. A high force barrier will provide a kinetic stability for the binary mixtures of the CF and CB particles in an aqueous medium and hence keep the CB pigments liberated from the CF substrates.

It may be of interest to discuss the reasons for the difference between the $\Pi(h)$ isotherms observed for the artificial mixtures of CF and CB particles (Fig. 3) and the $\Pi(h)$ isotherms observed for the ONP sample (Fig. 5). According to the Π_e term of Eq. 1, which is known as the Hogg-Healey-Fuerstenau approximation [16], the large difference between ψ_1 = -38.8 mV and ψ_2 = -12.7 mV was responsible for



6. a) Scanning electron microscope (SEM) images of paper pads made from an ONP in tap water; b) SEM images of the ONP samples after a sodium silicate treatment. The pigment's oil-based ink are red circles.



7. Effect of sodium silicate (SS) on the brightness improvement during the pulping and hyperwashing tests.

the attractive force, despite the fact that they were of the same sign, and it was responsible for the heterocoagulation. For the ONP sample, ψ_1 = -31.5 mV and ψ_2 = -14.9 mV, which also created attractive forces but only at very thin films below h < 23 nm. At h values close to that of h_c = 25 nm, the EDL force is repulsive, while the van der Waals force is attractive. Thus, the driving force for the heterocoagulation of the CF and CB particles present in the ONP pulp was the van der Waals force, which was the opposite of the case with the artificial sample and also with the ONP sample in the absence of the chemical additives.

The favorable situation with regard to CB liberation from the ONP slurry was created by the reagents used to promote liberation and hence deinking flotation. Sodium silicate may be responsible for the increase in the negative ζ-potential (or -ψ_2) from -12.7 to -14.9 mV. The use of H_2O_2 may also be responsible for the increase in the negative zeta-potential via oxidation of carbon to carbonyl or carboxylates. The polyelectrolyte may also be responsible for the decrease in ψ_1 of CF from -38.7 to -31.9 mV by way of removing some adherents (e.g., clays, submicronic wood fibers, etc.) from the surface, as shown in the SEM images given in **Fig. 6**. The use of NaOH may also be responsible for the decrease in ψ_1 of fibers in that the ζ-potential of fibers decreased considerably as the pH was increased above 10. In the presence of the reagent additives, the Debye length (κ^{-1}) decreased from 96 nm to 31 nm, which also contributed to the high force barrier observed with the ONP sample.

By virtue of the reagent packages used to improve the recyclability of the ONP sample, liberation of the CB pig-

ments used in the ONP sample was greatly improved, as shown in Fig. 4b and in Fig. 6. The small particle size of the pigments is one of the reasons for the difficulty encountered in deinking flotation.

The results presented in Figs. 4–6 show that Na_2SiO_3 was beneficial for liberating pigments from the ONP sample printed with oil-based ink. As validation of these findings, **Fig. 7** shows the result of the ISO brightness measurements conducted on the same sample after two stages of hyperwashing with and without Na_2SiO_3. Also shown is the result obtained after pulping the sample without hyperwashing. After hyperwashing, the brightness was increased by 5.4 over the result obtained without water washing, while the method of using Na_2SiO_3 increased the ISO brightness by 9.9. These results confirmed that increasing pigment liberation is critically important for improving the brightness of the ONP and hence its recyclability. The brightness improvement would have been more significant, if NaOH and H_2O_2 had also been used in conjunction with Na_2SiO_3.

The brightness increase of 9.9 shown in Fig. 7 is equivalent to a 30.8% increase in ISO brightness over the case of no washing and hence no liberation, which may be related to the 19.8% increase in the degree of liberation of the CB pigment from the CF substrate. This slight deviation is not unexpected, considering that the degree of liberation was a rough estimation based on the peak heights of the CF and CB frequency distribution curves presented in Fig. 4b. It would have been better to use the area under the ζ-potential distribution curves rather than the peak heights. Further work is, therefore, necessary to develop a rigorous mathematic algorithm to determine the degree of liberation more accurately. As has already been noted, pigment liberation is a prerequisite to flotation deinking. In this regard, improving pigment liberation is critically important in increasing the recyclability of printed papers.

Some investigators were skeptical of the role of Na_2SiO_3 in deinking flotation [17-18], while others reported beneficial effects [3,19-21]. The data obtain in the present work support the observation of the latter investigators. The results presented in Fig. 7 actually show that Na_2SiO_3 is an enabling reagent in the sense that, without it, the ζ-potential data show that the degree of CB liberation was practically zero. Also, the disjoining pressure isotherms presented in Fig. 3 and Fig. 5 show the fundamental mechanisms involved in liberation. The new method of determining the degree of pigment liberation from recycle paper is being tested for water-based ink. It can be used to study the liberation of pigments of various colors. The method is also useful for testing different reagents that can improve pigment liberation and thereby increase the recyclability of printed papers. Furthermore, the experimental data obtained in the present work can be used to construct disjoining pressure isotherms that can be useful for better understanding the fundamental mechanisms involved.

CONCLUSION

A new method of studying pigment liberation from recycle papers printed with oil-based ink has been developed. It consists of determining the number densities of pigments and fibers by measuring their characteristic ζ-potentials. Under conditions of favorable liberation, a ζ-potential distribution gives two peaks, with the relative peak heights providing information on pigment liberation. Under conditions of unfavorable liberation, only a single peak appears. Using the characteristic ζ-potentials of the pigments and fibers, one can also construct the disjoining pressure isotherms using the DLVO theory, which can be used to better understand the roles of different reagents that are used to control the surface properties of the materials involved. The results obtained in the present work show that control of EDL forces is critical for controlling pigment liberation and hence improving the recyclability of printed papers. **TJ**

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About this journal

ABOUT THE AUTHORS

This work has been funded by REMADE, which in turn has been funded by the U.S. Department of Energy. The objective of the project is to study ways to improve deinking flotation, which will in turn increase the tonnage of recycle papers in the United States.

We have been actively developing more efficient methods of improving the efficiency of separating mineral fines. A key to improving the separation efficiency is to improve mineral liberation. In the present work, we have developed a new method of determining the degree of ink particle liberation from recycle papers. The method consists of measuring the zeta-potentials of both ink particles and paper fibers. We may be able to apply the same methodology to determine the degree of mineral liberation.

We were able to develop this method because we have expertise in advanced colloid chemistry. We used the DLVO theory to determine the disjoining pressure in the wetting films between ink particles and paper fibers. The theoretical analysis made it possible to find that control of electrical double layer force is the key to improve the liberation of ink particles from fibers. We were surprised that the degree of liberation of ink particles is quite low. Control of surface forces is important for achieving a high degree of liberation of ink particles from recycle papers.

There should be many other reagents aside from those we have used in the present work that can increase the double layer force and thereby improve the liberation and hence the efficiency of deinking flotation. As we noted in our conclusion section, we are in the process of studying the liberation of ink particles from water-based papers and developing more powerful reagents than the one we used in the present work to improve the liberation of ink particles.

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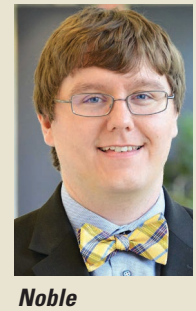
Huang


Strickland


Noble


Yoon


Basilio