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An Examination on the Effect of pH on Flotation Deinking Kinetics

Brian R. Moran Western Michigan University

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AN EXAMINATION ON THE EFFECT OF pH ON FLOTATION DEINKING **KINETICS**

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Brian R. Moran

A Thesis submitted in fulfillment of the course requirements for The Bachelor of Science Degree.

Western Michigan University

Kalamazoo, Michigan

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ABSTRACT

This study was performed to determine if the flotation deinking process is first order with respect to ink particle concentration and to examine the effects that pH may have on the kinetics of ink removal flotation. A Hallimond tube laboratory flotation device was used to examine the process. Image analysis of ink particles was used to obtain quantitative results regarding flotation efficiency.

The examination verified that the flotation process follows the first order rate equation with some degree of experimental error. A trend in the effect of pH on deinking rate was also found. It was determined that the rate constant, k, increased with decreasing pH. An increase in k corresponded to improved flotation rate.

The results follow the theory that decreasing the repulsive forces between negatively charged ink particles and bubbles improves deinking efficiency.

KEY WORDS

Deinking, Flotation, Kinetics, pH, Hallimond Tube, Photo-copy Ink, Surface Chemistry, Image Analysis.

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INTRODUCTION

The high volume of office waste paper being generated has brought a concern in the recycling of paper printed with difficult to remove UV cured and heat set inks. Recently, studies have been made in hopes of optimizing office waste recycling processes. Most of the studies investigate entire washing and flotation recycling systems. Neglected is the examination of the unit process of flotation. The Hallimond tube apparatus allows for such a study in a laboratory operation.

This study attempts to isolate the effect pH may have on the rate at which ink is removed in the flotation process using a nonionic surfactant. In order to simplify the investigation and allow for general conclusions on flotation kinetics, most mechanical considerations, the effects of fibers, fines and contaminants other than ink are not introduced.

The removal of ink from a water suspension through flotation is a first order reaction. This study will verify the order and examine the effect pH has on the rate constant, k. The first order equation describes the reaction rate. The rate constant quantifies the rate in a single numeric value.

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THEORETICAL AND BACKGROUND

DEINKING

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The recycling of printed stock is not a recent phenomenon. The first patent for deinking was developed by Mathias Koops on April 28th, 1800 by the British Patent Office. (1) Even earlier, was the first recorded attempt to reuse printed stock by George Balthasar Illy in Denmark in $1695.(1)$ At first, recycling of waste paper was not economical nor practical due to the great supply of virgin fiber. However, as paper production steadily increased through the 1800's and early 1900's, it became evident that the reuse of waste paper was essential. The technical development of processes both in paper manufacturing and recycling have progressed through time.

The original process for deinking of paper consisted of three principal steps; (1) the defiberization of the stock, (2) dispersion of the ink particles, and (3) the removal of the ink particles from the fiber suspension. These steps still give a general description to the current deinking methods. Developments in how to go about achieving these objectives have contributed to the optimization of the deinking process.

The defiberization of the stock and the dispersion of ink is achieved through mechanical stress and system chemistry modification. This initial step is called pulping. The second step, separation of the ink from the fiber suspension, can be achieved by two methods; washing and deinking. One or both of the processes may be used. The two differ both chemically and mechanically.

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Wash deinking relies on the dispersion of ink particles. $\begin{bmatrix} 1 & p \ q & A \end{bmatrix}$ Surfactant is added to disperse and impart hydrophilic properties onto the ink particles. The ink is subsequently removed with the flow of water by subjecting the fiber suspension to alternating $pg5$ dilution and thickening steps.

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The flotation of ink particles depends on the ability of air bubbles to effectively attract, hold and float ink particles to the surface of a water-filled cell. Surfactants are added to 195 agglomerate and impart hydrophobic properties to the ink (particles so that they are attracted to the air bubbles and not water.

PHOTO-COPYING

The development of photo-copying, or Xerographic reprography is credited to Chester F. Carlson. He developed electrostatic recording in the 1930's in attempts to develop a quick, high quality method for copying printed documents. Carlson obtained his first patent for this work in this area in 1939.(2) Carlson's invention made a significant impact on the office environment and the paper industry as well.

Waste paper generated from photo-copy machines is the primary fiber source along with other non-impact printed paper in mixed office waste recycling. In photo-copying, the ink toner is heat set onto the paper. This fusing of the ink onto the fiber causes difficulty in its removal. The ink also becomes difficult to disperse. Thus, it is more efficient to remove heat set inks through flotation deinking.

FLOTATION DEINKING

Original applications for flotation were developed for separation of metal from ore in mining operations. Pierre Hines was the pioneer for applying this technique to remove ink from waste paper in the mid $1930's.(3)$ The first industrial application for flotation deinking was initiated by J.W. Jelkes in $1950.(4)$

Flotation is now the predominate process for deinking of waste paper in Europe and Japan. This is due to the ability of the process to remove the larger, non-dispersible ink particles found in the recycled furnish mostly utilized in these regions. Flotation is not as sensitive to particle size as washing and can effectively remove medium and larger particle size ink contaminants. In North America, old newsprint (ONP) is a more common furnish for recycling operations. The smaller, dispersible ink particles found in ONP are more easily removed by the wash deinking process. However, the use of flotation deinking is growing with increasing relative office wastes volumes in the United States.

The attachment of ink particles to the rising air bubbles is the most fundamental requirement for successful flotation. (5) This attachment is dependent upon the thinning and collapse of the liquid-film layer between the particle and air bubble. (5) The time for film rupture and subsequent ink-bubble attachment is called the induction time. The induction time depends on the many variables including particle size, bubble size, surface tension, electrostatic forces and the viscosity of the continuous phase. (5) Subsequently, these characteristics all affect the flotation process. Process kinetics, or the rate at which the ink

particles are effectively floated, may also be affected by these variables.

FLOTATION KINETICS

The time required to effectively remove ink particles from the secondary fiber furnish is an economic consideration. Increased flotation rate allows for less turnover time between **flotation cell batches and increased production.**

Sylvester and Byeseda (6) investigated the process kinetics **for the flotation of oil droplets in water. They showed that the rate of flotation 1s approximately first order with respect to the** concentration of droplets at constant air flow and bubble size. (6) The system is similar to the flotation of ink particles. This parallel was proven by Larsson(7) when he studied the effect of particle **size on flotation rate.**

The first order equation is written:

 $\ln C = -kt + \ln C^{\circ}$

where t is the reaction time, k is the rate constant, C is the concentration of ink at time t and C° is the concentration at t=0. Larsson(7) showed that the number of unremoved ink particles **was directly proportional to the ink particle concentration. Thus,** N **can be substituted for** C **and the rate equation becomes:**

$\ln N = \ln N^{\circ} - kt$

where N is the number of unremoved ink particles at time, t and N° is the original amount of ink particles at $t = 0$. The number of **particles can be quantified through image analysis of ink particles deposited onto quantitative filter paper. The rate constant, k, can**

be obtained by a linear least squares fit of the In N against t data where $y = \ln N$ and $x = t$. The y-intercept is equal to $\ln N^{\circ}$ and the rate constant, k, is equal to the negative slope. Consequently, the effects of pH on flotation kinetics can be investigated by comparing k values with pH.

The effects of flotation variables on the kinetics of the process have been investigated by previous research. The study conducted by Li, Fitzpatrick and Slattery(\leq) concluded that the rate constant was affected by the bubble size, particle size and the turbulence of the flotation system. (5) An experiment by Collins and Jameson(8) showed that k decreased as the electrostatic surface potentials increase when the electrostatic forces were repulsive.

All electrostatic forces usually are repulsive in a system consisting solely of ink particles of the same charge. However, if the electrochemistry of the system is altered, this condition would not be true. A change in hydrogen ion concentration, or pH, would probably change the electrochemistry of the dispersed phase, due to the ionization of carboxyl groups on the ink particles. This study will attempt to examine the effects.

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.PROBLEM STATEMENT

This study has two major objectives. One, is to verify the literature statements on the characteristic rate order of one for the flotation process. The second objective is to examine the electrochemical effects that hydrogen ion concentration may have on the rate or kinetics of flotation deinking.

The importance of this study is in the value of increased knowledge of a process which is not fully understood. This study will take a very general look into flotation kinetics. Many variables commonly encountered in industrial applications, such as filler, fiber and water hardness considerations are not considered by this study. These areas, as well as mechanical considerations are topics that could be investigated by further studies. This thesis should lead to a better understanding of flotation kinetics and the effects pH may have on the process.

EXPERIMENTAL DESIGN

There have been a number of studies on the influence of chemical additives during the deinking process as a whole. The results of the experiments have most often been based on the properties of the deinked pulp. McCormick (9) evaluated pH levels during the pulping of secondary fiber. His studies explored variables using a non-ionic surfactant in wash deinking processes.

Investigated by Larsson, (2) is the dependence of flotation kinetics on ink particle size using a cationic surfactant. Larsson also examined the effect of pH on flotation deinking performance, but not on process kinetics. Larsson's studies explored the effects of process variables using a dispersion of mineral oil based news ink and deinking chemicals, without pulp fibers. His studies utilized the Hallimond Tube apparatus.

This investigation is similar to Larsson's in that the ink particles were isolated in the flotation process. However, a nonionic surfactant and photo-copy ink toner were used to determine the effect pH may have on flotation kinetics.

Experimental runs were conducted at pH values of 5, 7, 9 and 11 to examine the effects of hydrogen ion concentration on flotation kinetics. Samples of floated ink were taken from the tube at designated time intervals of 20, 40 and 80 seconds. The samples from each run were evaluated using image analysis.

INK DISPERSION

The study investigated flotation kinetics of photo-copy ink. This ink is heat set onto paper during the photo-copying process. Since this study was an investigation of flotation of ink particles, not the removal of ink from fiber, ink in the absence of fiber was floated.

The ink toner used was a carbon black pigment with a polyester carrier. The specific gravity was 1.2. The Material Safety Data Sheet is included in the appendix.

A thin layer of the powder toner was spread onto a glass plate and melted in a muffle furnace for five minutes at approximately 225° C. The plate and fused ink was then cooled at room temperature until the ink was hardened and the glass cool enough to handle. The ink then was scraped off into a beaker and covered for storage at room temperature. The process was repeated several times to generate a sufficient amount of ink for testing.

The ink was ground with a mortar and pestle to a uniform consistency. 325 and 200 mesh Fischer sieves were used to remove particles larger than 75 microns and smaller than 45 microns. However, ink particles outside of this range certainly could be included in the end sample. Deionized water was used to wash the ink particles through the screens.

Next, about 2 grams of the screened ink was dispersed in a volumetric flask with deionized water to a total volume of 1 liter. Some of the ink floated without the aid of air bubbles. Thus, these particles were skimmed off. Only the well-dispersed ink particles

were used. A pipette was used to extract 5 mL aliquots of the ink dispersion for each run in the Hallimond tube.

SURFACTANT ADDITION

An alcohol ethoxylate from Shell Chemical was used for a flotation aid in the experiment. A non-ionic surfactant was chosen to eliminate electrochemical effects that would be contributed from the use of an ionic surfactant.

The concentrated surfactant was diluted to 0.10 % using a 500 mL volumetric flask. A pipette was used to add 5 mL aliquots of dilute surfactant to the 100 mL sample for each run.

pH ADJUSTMENT

The 10 mL of ink/surfactant suspension was diluted to 80 mL, mixed and adjusted to the designated pH value (5, 7, 9 & 11) using dilute NaOH and dilute H2SO4 for each run using a stir bar and a 150 mL beaker. Once adjusted for pH, the remaining volume was transferred into a volumetric flask and made up to a total volume of 100 mL with deionized water pre-adjusted for pH using dilute NaOH and dilute H2SO4.

FLOTATION

The 100 mL sample was mixed and poured into the Hallimond tube having medium frit size.(see figure 1.) The excess (about 15 mL) was discarded as it spilled from the side tube. Nitrogen gas flow was set at 50 mL/min to the Hallimond tube. Agitation was accomplished using a small magnetic stir bar in the

base of the tube with stir plate speed constant at setting "2". Using a stop-watch, the nitrogen gas flow and agitation was turned off at the prescribed time intervals of 20, 40 and 80 seconds. The floated ink particles were removed from the side tube, rinsing with deionized water into a sample jar. The floated ink samples were poured from the collecting beaker into a filtering apparatus, using Whatman #42 quantitative, ashless filter pads for image analysis. Two samples were made for each experimental condition.

ANALYSIS OF SAMPLES

Image analysis of the filter paper samples was used to determine how much ink was removed for the time interval of each run. From the two sample pads prepared by filtration of the tube rejects for each run, 20 fields were evaluated (10 from each pad). The minimum particle size counted by the analysis had an area of $73.89 \mu m^2$. Assuming that the particles were round, this area would correspond to a diameter of 9.70 microns. This setting was recommended by Matt T. Stoops, image analysis technician at Western Michigan University.

A "blank" sample of the original amount of ink in the suspension was analyzed to represent N° , the amount of ink in the tube at $t = 0$. The number of particles (N) in the tube at each time interval (t) was calculated by:

$N = N^{\circ} - Nc$

where Nc was the number of ink particles counted from the side tube rejects of each run at $t = 20$, 40 and 80 seconds.

From the natural log of the number of unremoved ink particles at each time interval and the 1st order rate equation, the rate constant was determined for each pH. The rate constant, k was plotted against pH to examine the effects of hydrogen ion concentration on the kinetics of flotation deinking.

RESULTS

Experimental data is summarized in appendices I and II. From this data, the graphs showing the effect of pH on the rate constant, k, are constructed. The following tables summarize the experimental data and results.

Table 1. Ink Sample Characteristics and Verification of Sample Repeatability

VALUE	BLANK #1	BLANK #2	
$particles$ counted (No)	3518	3576	
avg. particle area	2900 μ m ²	$2650 \ \mu m^2$	
avg. particle diameter	$61 \mu m$	$58 \mu m$	
maximum diameter	$259 \mu m$	$226 \mu m$	

Table 2. Effect of pH on Rate Constant, k.

рH	5.00	7.00	9.00	11.00
	0.0171	0.0122	0.0113	0.00854
sigma k	0.0020	0.0051	0.0037	0.00080

Table 1. indicates good repeatability between samples. It also shows the ink dispersion characteristics with regard to particles size distribution. Appendix IV contains particle size distribution data for the floated ink from each sample.

Table 2. gives the numeric results from the experiment. This data are illustrated by figures 2 through 6.

Figure 2. pH = 5.00

Figure 3. pH = 7.00

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Figure 5. **pH** = 11.00

Figure 6. Effect of pH on Rate Constant

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CONCLUSIONS

The data presented clearly show a trend of increasing rate constant with lower pH values. A higher value for the rate constant, k indicates improving deinking efficiency as described by the first order rate equation

$\ln N = \ln N^{\circ} - kt$

where N is the number of unremoved ink particles at time, t and N° is the original amount of ink particles at $t = 0$. As k increases, the number of unfloated ink particles decreases. Thus, obtaining a large k is indicative of efficient ink removal.

This trend is consistent with the theory presented. That is, k increases with decreasing electrostatic surface potentials when electrostatic forces are repulsive. For this experiment, the constituents (air bubbles and ink particles) are negative in charge and all forces are repulsive.¹⁰ Thus, a decrease in pH, or increase in hydrogen ion concentration corresponds to lower repulsive forces between negatively charged ink particles and air bubbles. A reduction in repulsive forces corresponds to a higher rate constant due to a higher collision probability and increased deinking efficiency.

RECOMMENDATIONS FOR FURTHER STUDY

This study is an abbreviated analysis of deinking kinetics. It shows that hydrogen ion concentration has a notable impact on flotation rate. An investigation of how zeta-potential changes with the pH of these ink dispersions and deinking efficiency would be good follow-up work on this experiment. The findings could verify the conclusions reached by this study.

Also, one may investigate fiber effects in regards to deinking kinetics. Fiber length and concentration could be included in the study.

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APPENDIX I. DATA FROM IMAGE ANALYSIS

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APPENDIX II. LEAST SQUARES ANALYSIS OF IA-DATA

pH 5 7 9 11 $D = n \Sigma (x^2) - (\Sigma x)^2 = 5600$ 5600 5600 5600 **m** = (nΣxy-ΣxΣy)/D = -0.0171327 -0.0122475 -0.01127062 -0.0085401 $b = [\sum (x^{2})\sum y - \sum x \sum xy]/D = 8.10190757$ 7.74395166 7.901610369 7.74595486 **s:�.� y = [(I{y-mx-b]"2)/(n-2)]"0.5 = 0.08723479 0.22125497 0.161635949 0.04255355 s i i**₁ma m = (sigma y)(n/D)^0.5 = 0.00201909 0.00512106 0.003741145 0.00080419

and b = (sigma y)(Σ (x^2)/D|^0.5 = 0.10684036 0.27098089 0.1979628 0.05211724 $s_{\text{left}} \approx b = (\text{sigma y})[\Sigma(x^2)/D]^2$ **b** = **0.10684036** 0.27098089 0.1979628 **k = -m = 0.01713272 0.01224748 0.011270618 0.00854014 Noo = EXP(b) = 3300.75851 2307.57313 2701.629447 2312.20031**

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IMAGE ANALYSIS OF BEFORE DEINKING

Paper ID : Blank # Ink Particles Material Special Note $\ddot{\mathbf{z}}$ Performed By : Brian Moran Date Fri., Apr. 9, 1993 Threshold Level : 100 Unit of Area : μ m²
Number of Bins : 32 Bin Size : 10.00 Bin Offset \therefore 0.00

TABLE 1. Analysis Results

1) Number of Particles detected 3576 2) Total Areas of Particles (pm2) 9.4830E+ 6 3) Total Field Areas (pm2) $2.8209E + 8$ 3.36 4) Percentage Area 5) Minimum Area detectable (pm²) 73.89 60 Maximum Area detected (pm2) 40198.60 7) Mean Area (μm^2) 2651.85 3) Standard Deviation 0480.40 9) Parts per Million (um²/mm²) 33616.60

 d_{max} : 226/1 \overline{d} = 58.4. $= 2650 \mu m^2$ \overline{A} N = 5576

IMAGE ANALYSIS OF BEFORE DEINKING

$$
\frac{d_{max} = 252.4m}{\sqrt{d_{max} + 2900.4m^2}}
$$
\n
$$
\frac{d_{max}}{d_{max} + 2900.4m^2}
$$
\n
$$
M_{max} = 35.1\%
$$

TABLE 1. Analysis Results

1) Number of Particles detected 1996 2) Total Areas of Particles (μ m²) 5.6512E+ 6 3) Total Field Areas (μm^2) $2.8209E + 8$ 2.00 4) Percentage Area 5) Minimum Area detectable (um²) 73.89 6) Maximum Area detected (pm²) 44706.20 2831.24 7) Mean Area (µm2) 8) Standard Deviation 3057.16 9) Parts per Million (pm² 4mm²) 20032.95

TABLE 1. Analysis Results

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TABLE 1. Analysis Results

1) Number of Particles detected 2748 2) Total Areas of Particles (pm²) 8.1306E+ 6 3) Total Field Areas (pm²) $2.8209E + 8$ 4) Percentage Area 2.88 5) Minimum Area detectable (pm²) 73.89 33178.60 6) Maximum Area detected (pm2) 7) Mean Area (um²) 2958.74 3298.41 3) Standard Deviation 35.35 9) Parts per Million (um²/mm²)

Special Note ~ 10 Special Note

Performed By (: Brian Moran

Date (: Fri., Apr. 9, 1993) Threshold Level # 100 Unit of Area = $\frac{1}{2}$ um²
Number of Bins = 1 32 Bin Size : 10.00 Bin Offset (1 0.00

TABLE 1. Analysis Results

1) Number of Particles detected 2363 2) Total Areas of Particles (µm²) 6.8860E- 6 3) Total Field Areas (μm^2) 2.8209E+ 3 2.44 4) Percentage Area 73.89 5) Minimum Area detectable (pm²) 6) Maximum Area detected (pm²) 37021.20 $\frac{2914.10}{3110.79}$ 7) Mean Area (μm^2) *()* event area (µm*)
8) Standard Deviation 9) Parts per Million (um²/mm²) 124410.45 $\tilde{\epsilon}$

TABLE 1. Analysis Results \sim

1) Number of Particles detected 2620 2) Total Areas of Particles (um²) 8.0244E+ 6 $2.8209E + 8$ 3) Total Field Areas (μm^2) 4) Percentage Area 2.84 12.39 5) Minimum Area detectable (um²) 6) Maximum Area detected (pm) 38277.40 7) Mean Area (um²) 3053.41 8) Standard Deviation 3541.38 9) Parts per Million (µm²/mm²) 28445.80

TABLE 1. Analysis Results

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TABLE 1. Analysis Results

1) Number of Particles detected 1196 2) Total Areas of Particles (µm²) 3.5808E+ 6 3) Total Field Areas (um²) $2.8210E + 8$ 1.27 4) Percentage Area 5) Minimum Area detectable (µm²) 73.89 6) Maximum Area detected (pm²) 31553.00 7) Mean Area (pm2) 2993.96 8) Standard Deviation 2722.92 9) Parts per Million (um²/mm²) 12693.52

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TABLE 1. Analysis Results

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TABLE 1. Analysis Results

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