Improved Deinkability of LEP (Liquid Electrophotography) Digital Prints

Wei

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IMPROVED DEINKABILITY OF LEP (LIQUID ELECTROPHOTOGRAPHY) DIGITAL PRINTS

by

Zheng Wei

A thesis submitted to the Graduate College in partial fulfillment of the requirements for the degree of Master of Science in Paper Engineering, Chemical Engineering and Imaging Western Michigan University August 2013

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IMPROVED DEINKABILITY OF LEP (LIQUID ELECTROPHOTOGRAPHY) DIGITAL PRINTS

Zheng Wei, M.S.
Western Michigan University, 2013

Digital printing plays a very important role in the life of modern society. With the development of digital printing technologies, and the increasing market share of digital printing, the amount of digital prints in waste paper is rapidly increasing. Under “green” manufacturing and environmental sustainability, deinking of digital print inks is an urgent problem that needs to be solved. Indigo is a typical series of HP (Hewlett-Packard) digital presses that use a liquid ink named ElectroInk (Liquid Electrophotography or LEP). The particle size of HP ElectroInk is very small, 1-2 microns, which is smaller than dry toners. But, during flotation, the ink platelets were too big to be removed by foaming.

In this research, we focus on a new way of deinking that uses ultrasound treatment. Ultrasound could break down the ink platelets and then help the ink detachment from fiber. During the deinking processes, we choose both neutral and alkaline environments. For different ultrasound times, we will compare the results using a variety of Indigo certified substrates at 20 kHz. ElectroInks can be detached by ultrasound treatment and then we can remove them by conventional flotation technology.
ACKNOWLEDGMENTS

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Zheng Wei
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CHAPTER I

INTRODUCTION

With the development of digital technology, digital printing has already become a necessary printing method. Compared with the other traditional printing methods, digital printing is easy to control and convenient. Traditional printings have their own advantages and some of these cannot yet be matched by digital printing methods. Giant amounts of printing requirements still depend on traditional printing because of the costs for long runs. But digital printing is the future. People do not need to become experts before they use the printing machine and they do not need to touch the ink and make their hands dirty.

The appearance of “digital” changed the world. People realized that digital technology is powerful and amazing. They focus on the research of “digital” include digital printing. Digital printing plays a very important role in the life of modern society. There is variety of digital printers that were invented by different printing companies. At the same time, with higher requirements of production, different kinds of inks have appeared every day. Thus, it is so difficult to find a uniform method of deinking those digital inks.

Under “green” manufacturing and environmental sustainability, deinking of digital print inks is an urgent problem that needs to be solved. INGEDE (International Association of the deinking Industry) has published 17 methods to establish a specification of deinking [1]. It works very well on traditional printing productions, but for digital printing it is barely satisfactory.
LEP (Liquid Electrophotography) is used by a typical series of HP (Hewlett-Packard) Indigo digital presses. The particle size of Indigo ink is very small, just 1-2 microns [2]. The small particle size provides high resolution, uniform gloss and very thin ink films. At the same time the particle size does not work in the flotation range, which is 10-100 micron [3].

Traditional deinking is under alkaline conditions. Following an alkaline deinking method, people found that, during the flotation process, no foam is generated in that step [4]. So trying some new chemicals is necessary for LEP deinking. Some of the chemicals are easy to choose, some of them are so complex to choose from. The details will be given later.

Neutral chemicals, instead of alkaline chemicals, may solve the foaming problem. But the ink plates are too large to be removed by that foam [5]. In this research, ultrasound treatments are used before flotation to break down the ink plates.

Ultrasound treatment has popular for use in different areas. For deinking, people did research on ultrasound about 15 years ago [6]. Because of the cost, ultrasound was not competitive compared with the other methods to improve deinking. Many people still focus on chemicals to consummate the deinking processes. Nevertheless, it is so difficult to find one chemical to work on all of digital inks and the chemicals also pollute the water and environment. In addition, it is unpractical for paper machines to separate and deink different kinds of digital prints.

Ultrasound treatment does not pollute the environment, which is perfect to match the purpose of green manufacturing. In addition, it could be widely used in deinking with different inks.

In this research, through the Clark Classifier screening machine, it was easy to determine the size of the Ink plates. Some publications have mentioned that Indigo
Ink is very soft; they could deform through the slot, so the screening process does not work well on them [7]. But if we could reverse operation of the machine, it should be an efficient method for deinking.

From the above, the objectives of this thesis are:

1. Solve the foaming problem for LEP deinking. One method to solve this problem is to use different chemicals.

2. Add in ultrasound treatment to determine the effect of the deinking process.

3. Compare the results of the samples under alkaline and neutral conditions.

4. Analyze the influence of the ultrasound treatment and compare the results under different ultrasound times.

5. Use the screening machine to get more information about the ElectroInk.
CHAPTER II

REFERENCES REVIEW

Forest Shortage

The forest is very important for creatures of the world. Forests supplied the essential services and could provide important goods. Forests cover 31% of the world’s land surface [8], which can filter water, protect soil, provide habitat for countless animal species and so on.

Figure 1 is the world forest cover from 1990 to 2010. Those data came from the U.N. Food and Agriculture Organization. From 1990 to 2010, the total world’s forest cover decreased from 4168 million hectares to 4033 million hectares [8]. During these decades, Asia and Europe increased their forest coverage. North and Central America were stable. The deforestation of Africa and South America were serious. Each year the world lost on average 16 million hectares of forest, which is roughly equal to the size of Michigan [8]. Some of the places realize the importance of the forest then started planting and expanded their natural area, but the loss of the forests is still 8.3 million hectares.
From 1990 to 2010, the Forest coverage of Asia changed its trajectory from forest loss to forest expansion. China has contributed much to this increase. The disastrous flooding in 1998 was considered the worst Chinese flood in 40 years [9]. After the flooding, there was $26 billion in economic loss and 15 million homeless. The government realized the importance of forest protection and soil protection. Alone with development of the industry, environmental protection was much more important than before. China’s heavy planting can disguise the trends elsewhere in the region. From 1990 to 2010, the loss of forest in India was 24 million hectares [8].

Recycled Paper

Recycling is the process of re-using a given product. Due the forest forestation and non-renewable energy, the awareness of recycling has greatly increased. Plastic, glass and paper all can be recycled. Paper recycling is one of the most important parts of the recycling effort, which is the future of the papermaking
industry. The papermaking industries need to face the energy crisis and shortage of forest in the next two decades. Nevertheless, paper recycling will bring a turning point to the paper industry. Paper recycling has both economic and ecological value.

Europe, Japan and the United States are the highest recycling rate areas. Europe has a long history of paper recycling and they already have many mature organizations of paper-recycling. In 2009, the paper-recycling rate of Europe was 72.2%. Japan is a poor resource country. Therefore, the government established a series of recycling policies, including paper-recycling. Their paper-recycling rate also could achieve 72% right now [10]. The United States is one of the largest countries of paper manufacturing. Today, over half of the paper used in the US is recycled paper.
Compared with non-recycled paper, recycled paper has many advantages. The process of recycled paper manufacture has less impact on forest resources, less energy consumption and less solid waste. From Figure 3, the paper-recycling rate already achieved 72% in 2009. From 1991 to 2009, in this decade, the paper recycling rate increased 30%.

![Figure 3. Paper Recycling Rate of Europe from 1991 to 2009 [11]](image)

**Figure 3. Paper Recycling Rate of Europe from 1991 to 2009 [11]**

![Figure 4. Paper Manufactures and Paper Recycling Diagram [12]](image)

**Figure 4. Paper Manufactures and Paper Recycling Diagram [12]**
Figure 4 shows the flowcharts of paper manufacture and paper recycling. The recycled paper comes from newspaper, magazines and some other grades. After collection, water and chemicals is added into the waste paper to break it down. Then, they are heated and chopped up to break down further and the resulting suspension is called pulp or slurry [13]. The pulp goes through screens to remove glue and plastic materials that may still be in the mixture. Then cleaned, deinked and bleached and the pulp can be used to make new paper.

Normally, the same fiber can be recycled only 7 times, because every time fibers will get shorter and at last strained out. Recycled pulp could produce different kinds of paper that even some virgin pulp could not produce [14]. Recycled pulp has high opacity, uniform fibrous tissue and could meet the quality requirements of the majority of the paper without beating [15]. Recovered paper accounted for more than 37% of fiber used to make new paper products in the United States in 2007, which was clarified by the American Forest and Paper Association [14].

Deinking

Deinking is the heart of paper recycling. The purpose of deinking is removing those inks, which are 2% on average in waste paper. The ink is considered dirt and the deinking process is similar to a laundering process. Deinking can restore or even exceed the whiteness, the softness and other characteristics of the original fiber. Normally there are three steps of deinking: separate fiber, ink detachment from fiber and remove the ink from the fiber suspension.

Deinking chemicals are used during the re-pulping stage to dislodge the ink
particles from the fiber and disperse them in the stock suspension. Those ink particles can be removed by washing, flotation or the combination of washing and flotation.

Figure 5. Deinking Line [16]

Figure 5 is the diagram of the deinking process in a paper mill. The result of deinking can be judged by the optical properties and ink elimination area. One of the deinking organizations, which is INGEDE (International Association of the Deinking Industry) [1], has already published the standard of each deinking process.

Flotation and Washing

The purpose of flotation and washing is to remove the ink from the fiber suspension. The conventional deinking process includes flotation, washing and combined washing and flotation. Depending on the ink particle size which could help to choose the most efficient method. Figure 6 shows that, depending on particle size, different methods have different ink removal efficiencies. The larger particle size is
removed by cleaning and screening, the smaller ones are removed by washing and flotation. Because of water shortages and the cost, flotation is becoming much more popular than washing. In addition, engineers have invented some new mechanical technologies to combine the advantages of washing and flotation to perfect the deinking process.

![Figure 6. Particle Size Removal Efficiency [17]](image)

The washing method uses a hydraulic force to separate and force pulp suspension through a filter or mesh and the water entrains those ink particles to be removed. When the particle size of ink is under 5 microns, washing should be chosen and when the particle sizes are from 15 to 30 microns, the flotation has the highest ink removal efficiency.
There are two important parts in the flotation operation cell, which are the agitator and air supply instrument. The deinking chemicals added into the flotation cell help the ink be detached from the fiber. Then those air bubbles attach to those inks to move them on the top of the flotation cell. That combination is called froth and a paddle is used to remove the froth before the air bubbles brake down. That’s the reason why the flotation could save more water than washing. Figure 7 shows each part of the flotation cell and the operation principle.

Deinking Chemicals

Chemicals play a very important role in deinking processes. For conventional printing products, there are already efficient chemicals to deink them. However, it is so difficult to find the chemicals that work on all of the digital prints. Each digital printer has its ink to match the printing requirements. Some of them are inkjet, some are dry toner and some are liquid toner, each of them with their own different
characteristics [19].

Some of the chemicals are simple to choose, such as Sodium hydroxide, hydrogen peroxide and oleic acid. The others are difficult to choose. Those normal chemicals added in the sample of this research [4], which is based on LEP (Liquid Electrophotography), is barely satisfactory. There are two reasons LEP inks are difficult to deink. The most important one is the small particle size of digital particle ink and the second one is the big ink plates which were too big to remove in the flotation process.

In the washing method, the chemicals should have strong dispersing ability [20]. For flotation, the deinking chemicals should have good foam generation ability with the function of trapping ink. In this research, some neutral chemicals were considered, instead of conventional alkaline deinking chemicals. They are POES (polyoxyethylene stearate) and SDS (sodium dodecyl sulfate) [21].

POES is a non-ionic cream emulsifying agent. The industrial application is wide [21]. In this research, POES, instead of the other conventional chemicals, was used in the defibration process. The concentration is 1% and the total chemical should be 400 ml. A pH buffer was used to control the pH around 7. After each of the deinking processes, the pH value was measured by a pH Meter. From storage to flotation process, the pulp consistency decreased to 1% from 5%. Before flotation, SDS was added. SDS is an anionic surfactant used in many cleaning products. SDS has good emulsifying, foaming and dispersing performance. SDS is used as a deinking agent, flocculating agent and penetrant in papermaking [21]. In the experiment the concentration of SDS is 0.1%.
Table 1
Chemicals for Deinking Processes

<table>
<thead>
<tr>
<th>pH</th>
<th>Chemicals</th>
<th>Add in position</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.5</td>
<td>0.6% NaOH</td>
<td>All of them are added at the beginning of the defibration process</td>
</tr>
<tr>
<td></td>
<td>1.8% Sodium silicate</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.7% H₂O₂</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.8% Oleic acid</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>1% POES</td>
<td>The POES is added in the same position with the alkaline chemicals. The SDS is added in the beginning of flotation</td>
</tr>
<tr>
<td></td>
<td>0.1% SDS</td>
<td></td>
</tr>
</tbody>
</table>

INGEDE and INGEDE Method 11

INGEDE (International Association of the Deinking Industry) is an international organization, which works in many fields to study the recyclability of printed graphic products and to promote steps for improvements. In many areas, the improvements can be observed [22]. The main objective is gradually developing among the partners of the paper chain: to understand each other’s problems and to discuss new developments in the production of printed products with regard to recyclability besides printing quality.
INGEDE Method 11 [23]

The INGEDE Methods have drawn much attention in the deinking field in recent years. INGEDE supplies the standard of deinkability and tries to unify the instruments and standards. (See Figure 8)

Figure 8. Deinkability Test Method for Printed Products (According to INGEDE Method)
Screening

Screening has been applied to different areas [17]. Screening could perform at low, medium or high consistency. In deinking, the medium and high consistency are normally used. The major types of screens are vibratory, gravity centrifugal and pressure screens. Screening is required to remove oversized and unwanted particles from fibers.

In this research, the Clark Classifier (T233) screening machine was used. There are four screens whose sizes varying from the left side to the right side of Figure 9 are 15, 30, 100 and 200 meshes. Four plugs in each of the cell are shown in Figure 9.

Figure 9. Clark Classifier
Stickies

According to the INGEDE Method, the deinkability and potential of stickies formation influences the recyclability of printed products. INGEDE Method 4 [24] mentions macrostickies and INGEDE Method 6 [25] talks about the potential secondary stickies by cationic precipitation. Stickies do not have an accurate definition in deinking. The general understanding is the tacky substances, which is contained in pulp and water systems and deposits onto the paper machine cylinders or rolls at certain stages of papermaking. There are two different kinds of stickies, primary and secondary stickies. From Figure 10, the micro and macro stickies are primary stickies. Micro stickies could pass a slotted plate screen of 0.10-0.15mm. Micro stickies could be finely dispersed, colloidal or molecularly dissolved [26]. Macro stickies are the materials retained during screening. Macro stickies are much easier to remove by filter compared with micro stickies. The latter ones are easy to get in the water system or paper machine then cause problematic deposits [27]. INGEDE Method 4 describes the macrostickies removal [24].
LEC inks contain plastic polymers, which create the potential source of stickies [29]. LEC does not have primary stickies, because the Indigo digital presses use light sensitive photoreceptor to attract the charged toner. The ink is attached on the paper by heat and pressure. After deinking, we may find that the ink plates from Indigo inks are large. The screening process added here could remove the big ink plates to help to increase the deinkability.

Digital Printing

Digital printing is any printing process that uses a digital file to create an image using a non-impact image transfer method [30]. Common methods include ink jet and electrophotography. There are two categories of digital printing, direct to paper or image carrier. Both of them are non-impact printing.

Digital printing is a high-efficiency and convenient printing method. Compared with the other printing methods, digital printing has a short history, having emerged in the late 1900s [30]. However, digital printing has evolved rapidly and has become one of the most common methods of printing for both business and individuals alike. Digital printing is growing and more digital printing devices are introduced every day and new inks are introduced to increase the quality of digital products. At the same time, it has been difficult to detach these digital inks from fibers with the same deinking processes used for conventional inks [30].
LEP

Electrophotographic printing was invented by Chester Carlson in 1938 [31]. After 70 years of development, electrophotography has become a more mature process in the ranks of digital printing. The printing companies are always seeking better ink, which will increase the printing quality and image fidelity. The particle size of the pigment should be as small as possible, in order to achieve higher print image resolution and smoother gradation level reproduction.

LEP (Liquid Electrophotography) ink combines the advantages of electronic printing with the qualities of liquid ink. The toner particle size is in the 1-2μm range [2]. Because of the small particle size, the ink provides high resolution, uniform gloss, sharp image edges and very thin ink films. Since the basic material of the liquid electrophotographic ink is a novel resin material, rather than oily material, curing is not dependent on the drying time of the ink film on the medium, when it is in contact with the paper or other medium, it can immediately cure and produce a clear printed images, the edges do not blur and bleed [32]. The biggest difference between Indigo ink and conventional ink is HP ElectroInk technology, which directs the ink electronically using charged particles in the ink. Unlike conventional printing that uses a fixed plate, HP indigo digital presses use a light sensitive photoconductor that attracts ink in charged areas. The digital press is shown in Figure.11. The number of consumers of LEP is expected to increase from 1 Billion in 2000 to 100 Billion in 2016, according to HP [33]. Not just for LEP, the percentage of all digital printing has increased in areas, such as packaging, books and labels.
In addition, the INGEDE method does not work very well on LEP inks, which have been described as “unusable” [34]. The particles of LEP inks are thin, soft and flexible. There is little information about the composition of these inks. Nevertheless, it has been mentioned that they contain a mineral oil varnish and pigments similar to offset inks [21]. Bhattacharyya, et al. [21] suggest using near-neutral chemistry (termed as HPES), or an alkaline chemistry (termed as HPMF). However, most of the paper mills still refuse to use Indigo printed products [34]. Therefore, in this research; we will investigate ultrasound treatment to increase the deinkability.

Application of Ultrasound

Ultrasound is an oscillating sound pressure wave with a frequency from 20 kHz to 200MHz, which is greater than the upper limit of human hearing range [35]. Ultrasound is used in many areas, such as medical, petrochemical industry and
Different kinds of digital inks have different characteristics that challenge the deinking process. Normal deinking methods do not work well on them. Ultrasound has become another step to add into the flotation process [36, 37].

Figure 12. The Range of Ultrasound [35]

Although the particle size of LEP is small, the ink plates are too large to remove by foam. Ultrasound treatment on LEP could aid ink removal because after ultrasound treatment, the ink plates became smaller. Ultrasonic energy is well known for detaching particles or platelets from one another and surfaces [38]. The process was very effective at breaking down larger particles for improved flotation. In the present investigation, a frequency of 20 kHz will be used. The purpose of this investigation is to use the INGEDE deinking method 11, with the aid of ultrasound treatment. We will compare the alkaline and neutral deinking environments with and without 20 kHz ultrasound treatments. Using the INGEDE deinking method 11, we will investigate an ultrasound treatment before flotation and compare the neutral and alkaline deinking environments.
Instruments Review

Hobart Pulper

A Hobart type pulper (Kitchen Aid) was used for repulping, which is recommended by INGEDE. The repulping time was 20 minutes. The difficulty of this process is controlling the temperature at 45º. This Kitchen Aid machine does not have a temperature control system. So, in this experiment, we use plastic bags and separate them to add the required chemicals and dilution water to hold the temperature. The plastic bag looks unscientific, but it does work very well.

Figure 13. Hobart Pulper

Figure 14. The Temperature Keeping Bag
Ultrasound Machine

The ultrasound treatment machine is a VCX 130 PB from SONICS & MATERIALS, INC. and consists of piezoelectric lead zirconate titanate crystals (PZT). The peak power output is 130 Watts and the frequency is 20 kHz (the resonance frequency of PZT) [39].

The probe tip diameter is 3 mm and the volume treated is up to 150 ml. Because the flotation cell volume is 2L, which is much larger than 150 ml, it is necessary to treat the pulp for an extended period of time. With good circulation by mixing, the entire volume of the cell is definitely impacted by the ultrasonic energy.
Figure 16. Ultrasound Work in The Cameron Flotation Cell

Handmade Flotation Cell

Figure 17. Cameron Flotation cell

Air Input

Magnetic Stirrer

The flotation time is 12 minutes. Every two minutes, use the paddle to remove
the froth. This highly effected flotation cell was designed and built by Dr. John Cameron. This handmade flotation cell was easy to operate and the function is complete. The aeration was on the left side, which the red arrow shows in Figure 17. The flow rate of air was 1 L/min. Because of the ultrasound treatment machine needs to be added in the flotation cell at the top, a normal mixer could not work with the ultrasound machine. A magnetic stirrer was used to instead of the normal spindle mixer during air flotation.

**Brightness and Luminosity Measurement Machine**

The brightness will be measured by a Technidyne BrightiMeter Micro S-5 (show in Figure 18), which is a C/2° light source and 457nm. (T458) In addition, based on T524, Luminosity (Y) will be measured by this instrument also.
L, a* and b* Value Measurement Machine

X-rite Eyeone Pro [41,42] was used to measure Y (557nm), CIE a* and CIR b* values at D65/2°. All of the optical properties were measured on both sides.

![X-rite Eyeone Pro](image)

Figure 19. X-rite Eyeone Pro [41,42]

Measurement of Dirt Area

The dirt area was measured by an Epson Perfection V750 Pro scanner and Verity IA Light and Dark Dirt v3.4 software [43]. All of the handsheets scanned at 1200 dpi. The warm-up time of the scanner is up to 60 minutes. Through “preview scan” can adjust the position of the handsheets and reposition the measure area and the other parameters. Then do “Normal scan” and “run test on the mode” the data will output in an excel file automatically. The luminance range will be from 0 to 255 and the shape range will be from 12.56 to 300.

The Other Instruments

During this experiment also used some other instruments. The details are shown in Table 2.
Table 2
The Instruments Used in Deinking Process

<table>
<thead>
<tr>
<th>Instrument name</th>
<th>Running step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Oven</td>
<td>Ageing</td>
</tr>
<tr>
<td></td>
<td>ageing</td>
<td>Aging time is 72 hours and the</td>
</tr>
<tr>
<td></td>
<td></td>
<td>temperature is 60 °C</td>
</tr>
<tr>
<td>2</td>
<td>Container</td>
<td>Storage</td>
</tr>
<tr>
<td></td>
<td>Storage</td>
<td>Storage time is 1 hour and the</td>
</tr>
<tr>
<td></td>
<td></td>
<td>temperature is 45 °C</td>
</tr>
</tbody>
</table>
CHAPTER III

STATEMENT OF THE PROBLEM

The competition of printer marketing is very stiff right now. Therefore, finding an efficient deinking method of Indigo prints is an emergent problem. Indigo is a series of HP Company’s digital printers. Under alkaline condition, which means following conventional deinking methods, the deinking results are poor. Through an analysis of the character of liquid electrophotography, we need to find an efficient deinking method.

The ink particle size of indigo is from 1 to 2 micron, which is much smaller than the conventional dry toner inks. In addition, they are too soft to be removed by screening. Normally, the screening process is before or after the deinking process. In this research, the screening machine aids in deinking by yielding more information about Indigo inks. The screening machine normally is used to remove the undersized particles. However, in this research the fibers are considering as the oversized staff and the ink could go through the screening machine. After screening, the fiber stays on the screen and the ink goes through the screen. The difficulty of this experiment is during the screening process the water should flow through the screening machine at the same time. Because of the requirements of INGEDE, the whole process of deinking should use simulated tap water by adding known hardness to DI water, with temperature being kept around 45°C. Considering that the consistency of the next step of flotation is around 1% in this experiment, during the screening process no flow water gets into the chamber. Normally, the screening treatment should have flow
water to wash out the fiber and rejects. Without water in the screening process should be influence the results of deinking.

The objective of this thesis is to compare the results of deinking under alkaline conditions and neutral conditions to determine which one works better for Indigo ink. Need to choose some good neutral chemicals, which can increase the deinkability of Indigo prints. Should add ultrasound treatment in the conventional deinking process, and then compare the results to determine the best ultrasound treatment time. According to the results of screening, this could give information for deinking of Indigo ink.
CHAPTER IV

EXPERIMENTAL

Deinking

Figure 20. Flowchart of Alkaline Deinking Processes

Figure 21. Alkaline Deinking with Ultrasound Treatment

Figure 20 and figure 21 are the flowcharts of alkaline deinking processes. The
The difference of those two experiments is the ultrasound treatment. The ultrasound process is applied before the flotation process and the ultrasound treatment times are 0, 10 and 20 minutes.

In the experiment, each of the substrate samples yields prepared filter pads and handsheets. There are five groups of each sample: unprinted, undeinked, normal deinked ultrasound 10 minutes and ultrasound 20 minutes. (Shown below in table 3)

Table 3
Description of Samples in 5 Groups

| Group 1: U.P                  | Unprinted                                      |
|                              | (With Standard Deinking Process)               |
| Group 2: U.D                 | Undeinked                                       |
|                              | (Without Flotation)                            |
| Group 3: S.D                 | Normal Deinked                                  |
|                              | (with Standard Deinking Process)               |
| Group 4: U.S 10min           | Ultrasound 10min                               |
|                              | (with Standard Deinking Process)               |
| Group 5: U.S 20min           | Ultrasound 20min                               |
|                              | (with Standard Deinking Process)               |

Each group produced 5 handsheets (1.2g OD) and two filter pads (4.0g OD) according to the T272 standard, which are 200 cm² in area (shown in Figures 22 to 26).
From Figures 22 to 26, the handsheets that were treated under alkaline conditions are shown. For Figures 27 to 30, the handsheets were treated under neutral conditions.
Figure 29. Ultrasound 10min

Figure 30. Ultrasound 20min

Figure 31. Flowchart of Neutral Deinking Processes

Figure 32. Neutral Deinking with Ultrasound Treatment

Print product

Ageing

Defibration

Storage

Flotation

1% POES (Neutral Chemicals)

0.1% SDS

Undenked pulp
2 filter pads
5 Handsheets
2 Membrane filters

Deinked pulp
2 filter pads
5 Handsheets
2 Membrane filters

1% POES (Neutral Chemicals)

0.1% SDS

Undenked pulp
2 filter pads
5 Handsheets
Figures 31 and 32 show the deinking processes under neutral conditions. SDS is added before flotation, which could help to generate better foam compared with alkaline chemicals. The difference between Figures 31 and 32 is also the ultrasound treatment process.

Screening

The Clark Classifier has four screens, so in the experiment there are five types of pulp shown in Figure 31. The sizes of the screens are 15 meshes, 30 meshes, 100 meshes and 200 meshes. A, B, C, D and E are those five of chambers which separate by those four screeners.

In this experiment, the Ageing, defibration and storage process are all the same as with the deinking process under neutral condition without ultrasound treatment. When the pulp is full in these four cells, the screening machine starts running for 5 minutes. The screening times are 5 minutes. After screening, A and B pulp could get enough filter pads and handsheets. Position C could get 2 filter pads and 2 handsheets. Pulp D only get 2 handsheets and the E pulp is clean, but could not get any handsheets and filter pads, since consistency was too low. The flotation process is the same as with the neutral deinking processes.
Figure 33. Zones for Clark Classifier
<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2 filter pads and 5 handsheets</td>
</tr>
<tr>
<td>B</td>
<td>2 filter pads and 5 handsheets</td>
</tr>
<tr>
<td>C</td>
<td>2 filter pads and 2 handsheets</td>
</tr>
<tr>
<td>D</td>
<td>2 handsheets</td>
</tr>
<tr>
<td>---</td>
<td>-------------</td>
</tr>
<tr>
<td>E</td>
<td>NA</td>
</tr>
</tbody>
</table>

Figure 34. Chamber Details of Clark Classifier
CHAPTER V

RESULTS AND DISCUSSION

Deinking

All of the samples were supplied by HP. The substrate data were obtained at WMU. The details are in reference 4. In this research, sample number 5 was chosen to finish all of the experiments. To start to design the experiment sample number 1 to number 5 were under different pH, deinking chemicals and ultrasound treatment time. The results of sample number 5 got better results compared with the others. So, all of the results below were obtained from sample number 5.

5.1.1 Brightness (457nm)

![Figure 35. Brightness of Sample 5 (Alkaline)](image1)

![Figure 36. Brightness of Sample 5 (Neutral)](image2)
From Figures 35 and 36, the filter pad brightness was higher than the handsheets. There are two reasons for the difference in brightness. There are five handsheets for each group of the experiments because of the 2L flotation cell. One of the reason is the filter pads are not flat, another reason is the thickness of those five handsheets are thinner than the filter pads when measuring the brightness.

After ultrasound treatment, the samples brightness decreased compared with the samples that were not treated by ultrasound. When the ultrasound instrument breaks down the inks, the fibers were broken down as well. Depending on the requirements of paper brightness, one often needs to add chemicals to increase the brightness.

Under alkaline and neutral conditions, the brightness does show a big difference.

**Luminosity by C/2° (557nm)**

Figure 37. Luminosity of Sample 5 by C/2°(Alkaline)

Figure 38. Luminosity of Sample 5 by C/2°(Neutral)
The Y values measured by C/2° are shown in Figures 37 and 38. Because of the same reason of the thickness of the handsheets, the luminosity of filter pads were higher than the handsheets. The brightness and the luminosity were measured by the same equipment. Under neutral condition, the luminosity was higher than the samples under alkaline condition.

**Luminosity of D65/2° (557nm)**

![Figure 39. Luminosity of Sample 5 by D65/2° (Alkaline)](image1)

![Figure 40. Luminosity of Sample 5 by C/2° (Neutral)](image2)

The luminosity measured by D65/2° is shown in Figures 39 and 40. Under alkaline condition, the trend is similar and the value of the luminosity does not have significant differences. However, under neutral condition the luminosity by D65/2° is much higher than the luminosity measured by C/2° also under neutral condition. The unprinted samples were lower by over 10, compared with the other samples. After ultrasound, the luminosity increased.
From Figures 41 and 42, most of a* values are above the range of a* value from INGEDE. In figure 41, the normal deinked samples and ultrasound treatment by 20min samples, their a* values were under 2, which is in the range of a*.
Comparing Figures 43 and 44, b* values in 43 are better than the b* values in 44 because they approach the unprinted values.

IE\textsubscript{AREA}  (Ink Elimination by Area)

Table 4

Data of Dark Objects

<table>
<thead>
<tr>
<th></th>
<th>Alkaline</th>
<th>Neutral</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Dark Objects</td>
<td>IE\textsubscript{AREA}</td>
</tr>
<tr>
<td></td>
<td>PPM Holey</td>
<td>%</td>
</tr>
<tr>
<td>U.D</td>
<td>107299</td>
<td>99869</td>
</tr>
<tr>
<td>S.D</td>
<td>89829</td>
<td>16.28</td>
</tr>
<tr>
<td>US 10min</td>
<td>53088</td>
<td>50.52</td>
</tr>
<tr>
<td>US 20min</td>
<td>30133</td>
<td>71.92</td>
</tr>
</tbody>
</table>

The data in table 4 are from the Verity IA and Dark Dirt v3.4 software. The
software automatically outputs the results in an Excel file. From the dark objects one can calculate the ink elimination area simply.

\[
IE = \frac{EBA_{UP} - EBA_{DP}}{EBA_{UP}} \times 100
\]

The equation is [44] which is named Gong equation. UP means undeinked pulp and DP means deinked pulp. The dark of objects of undeinked samples value minus the values of “S.D; US 10min and US 20min” then divided the value of undeinked dark objects.

Figure 45 shows the increasing ink elimination area under alkaline and neutral conditions with application of ultrasound. Without any ultrasound treatment, the normal deinked samples ink elimination areas were similar. After 10 minutes ultrasound treatment, the ink elimination area increased by almost 40% under neutral
condition. The ink elimination area also achieved 50% under alkaline conditions. After 20 minutes treatment, the ink elimination area achieved 76.37% under neutral condition, and under alkaline conditions the ink elimination area was 71.92%, which increased by 55% total.

**Ink Speck Size Distribution**

**Table 5**

Data of Ink Speck Size Distribution

<table>
<thead>
<tr>
<th></th>
<th>Alkaline</th>
<th>Neutral</th>
</tr>
</thead>
<tbody>
<tr>
<td>Condition</td>
<td>Mean(mm²)</td>
<td>Std(mm²)</td>
</tr>
<tr>
<td>U.D</td>
<td>1.11</td>
<td>1.96</td>
</tr>
<tr>
<td>S.D</td>
<td>1.00</td>
<td>1.55</td>
</tr>
<tr>
<td>US 10min</td>
<td>0.270</td>
<td>0.361</td>
</tr>
<tr>
<td>US 20 min</td>
<td>0.179</td>
<td>0.127</td>
</tr>
</tbody>
</table>

In table 5, under both neutral and alkaline conditions, the mean value and standard deviation are listed there. Figure 45 shows the ink speck size distribution below.
Figure 46. The Histogram of Ink Spec Size Distribution

Figure 46 showed both alkaline and neutral conditions ink spec size distribution. After 10 minutes ultrasound treatment, the ink specs decreased a lot. And after 20 minutes ultrasound the value are lower than the ultrasound treatment by 10 minutes. The ultrasound treatment definitely could break down the inkplates. The smaller inkplates were easy to remove by the flotation cell which could increase the deinkability.
Screening

The Clark Classifier was adding between the storage and flotation which same position with the ultrasound treatment. The requirement of flotation is the consistency of pulp should above 1%. During screening, simulated pipe water, whose temperature is 45 degree was added in the screening machine instead of the pipe water. After operation for five minutes, stop the machine and recover the pulp on different sides of the screener. Then use those pulps in the flotation process to make handsheets. From Figure 34, there are 2 filter pads and 5 handsheets were made in A, B and C. At D, only 2 handsheets were made and nothing at E.

Brightness (457nm)

![Histogram of Ink Spec Size Distribution](image)

Figure 47. The Histogram of Ink Spec Size Distribution

Each of the position the pulp consistencies were different. The A, B and C
still could obtain enough handsheets and filter pads. From Figure 47, the brightness of the C handsheet had the highest value. Because of the thickness of the handsheets, the brightness and luminosity for D were not measured.

**Luminosity by C/2° (557nm)**

![Figure 48. Y Value by C/2°](image-url)
Luminosity of D65/2° (557nm)

Figure 49. Y Value by D65/2°

From Figures 48 and 49, the luminosity in figure 49 was much higher than the luminosity in figure 48.
In position B, C and D, a* value of handsheets were under 2, which in the standard range of INGEDE.
From A to E the consistency of the pulps decreased and the pulp was much finer than the last screen. The b* value increased for the finer pulp.

**IE\textsubscript{AREA} (Ink Elimination by Area)**

**Table 6**
Data of Dark Objects Only Under Neutral Condition

<table>
<thead>
<tr>
<th>Neutral</th>
<th>Dark Objects PPM</th>
<th>IE\textsubscript{AREA} %</th>
</tr>
</thead>
<tbody>
<tr>
<td>U.D</td>
<td>99869</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>80079</td>
<td>19.82</td>
</tr>
<tr>
<td>B</td>
<td>16439</td>
<td>83.54</td>
</tr>
<tr>
<td>C</td>
<td>5142</td>
<td>94.85</td>
</tr>
</tbody>
</table>
From Figure 52, the ink elimination area achieved 96.3% at position D. At position C the ink elimination area was 94.85% and at position B the ink elimination area was 83.54%. It was easy to see that the most ink platelets are on both sides of the screen that is 15 meshes. Although some of the ink platelets could cross the screen, most of them still remain with the fibers.
CHAPTER VI

CONCLUSION

Under alkaline conditions, the foam in flotation process is a serious problem. POES and SDS, which are neutral chemicals, instead of alkaline chemicals from INGEDE did solve the foam generation problem. However, the optical properties and dirt area count did not yet yielded an optimal result. The ink platelets were too big to remove by foaming. However, the ultrasound does benefit ElectroInk for dirt area.

After ultrasound treatment the ink elimination area increased almost 50%. With the increasing of the ultrasound time, the ink elimination area increased. Under neutral condition, the ink elimination area was a little higher than the ink elimination area under alkaline conditions. The ultrasound treatment could breakdown the ink platelets, then the smaller ink platelets could remove by foaming. However, after 20 minutes ultrasound treatment there still is 25% ink not removed by SDS foaming. Prolonging the ultrasound treatment time might increase the efficiency of ink elimination, but the cost may be too high.

In this research, we also investigated screening instead of the ultrasound treatment. Because the ink platelets of ElectroInk were big, but those were very soft and thin, which could cross the screener. In the experiment, most of the ink platelets still get together with the fibers in the Clark Classifier. Therefore, the screening process did not work efficiently with the LEP prints.
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