Elimination of Pressure Sensitive Label and Deinkability of Water Based Flexographic and NIP Inks in One Recycling Step

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Abstract

Due to environmental concerns, progressively more water-based inks are used in conventional and nonimpact printing (NIP) technologies. Water-based flexo inks as well as inks used in NIP technologies use significantly smaller pigment particles than traditional litho inks [Chovancova et al, 2005; Frimova et al, 2005]. Conventional deinking eliminates significantly larger pigments than particles coming from new ink systems. Therefore, novel water-based inks for flexography and inkjet pose a huge issue to recycling facilities. Pressure sensitive label is a significantly larger contaminant of recycled fibers than inks. It was realized that its chemistry and chemistry of novel inks is very similar. Both types of contaminants are based on acrylic chemistry.

This experimental work focuses on deinking of water-based flexo and NIP inks. Elimination of pressure sensitive adhesive in this experimental work serves as a model for ink elimination procedures due to similar chemistries thus similar behavior. The impact of pH on stickies and ink agglomeration during deinking was studied. Experiments demonstrated that acidic pH causes acrylic based inks and stickies to grow in size, which eases their removal.

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Introduction

Environmental concerns and shortage of various raw materials made consumers aware of recycling needs. Paper and printing industries are following these eco trends as well [Tischner et al, 2003]. Recycled fiber comes from various sources. The criteria for recycled paper are high. They require the fiber to be clean of impurities, since it is further used for production of cardboard, packaging and newsprint paper grades, office paper, and hygiene paper. Consumers goods made out of recycled fiber should be also light colored which require elimination of the previously used ink [Laroche et al, 2001; Retner, 2008].

A recycled paper stream carries a multitude of contaminants. To simplify their characterization, they can be divided into two major groups. First group is represented by macro contaminants such as pressure sensitive adhesive (PSA). As mentioned, recycled pulp has to result in light colored products. But before solving the deinking issue and choosing proper deinking chemistry, macro contaminants have to be eliminated first. Typically, screening techniques are used to separate macro contaminants from the recycled pulp. Removal efficiency of macrostickies is 80% for particle size of 800 µm to 1000 µm. Completely successful removal for stickies is accomplished if the particle size reaches 2000 µm or more [Assessment of printed product recyclability, scorecard for the removability of adhesive applications. Retrieved 2012].

The second group of contaminants that enter the paper recovery system is called micro contaminants. Typical representative of the second contaminant's group would be the colorant. Inks and dyes are significantly smaller in size than stickies. Elimination of both groups of contaminants is based on their size. Larger the size of the contaminant means easier and more efficient removal. Flotation is preferred method used for deinking. Its principle is to collect ink particles on air bubbles and create froth layer. Froth is later removed by scraping. Particle size for effective flotation process has to be in the range of 10 µm to 250 µm [Assessment of printed product recyclability, scorecard for the removability of adhesive applications. Retrieved 2012]. There is wide pallet of chemistries that assure the ink particle agglomeration. In this experimental work, a simple approach is taken. The goal is to demonstrate that a simple change in pH can positively influence the growth of both pressure sensitive label and ink particles.

The chemistry of pressure sensitive label adhesive is based on acrylic polymers [Creton, 2003]. The choice of acrylic resin is abundant and it allows flexibility in PSA's formulation. Furthermore, acrylic based chemistry is cost efficient along with excellent performance [Guo et al, 2000; Venditti et al., 2007]. The chemistry of conventional flexo and NIP water-based ink is very comparable. While pigment sizes are different, resin chemistries are alike, mostly based on acrylic polymers chemistry.

Recycling and pulping condition impact removal of adhesives as well as water-based inks. Due to the complexity of recycling, the focus of the experiment is to eliminate the macro contaminants first, and to comprehend the behavior of acrylic materials in various environments. High pulping temperatures cause fracture of the pressure sensitive adhesives and create numerous small sized stickies within the recycling process. In addition to unfavorable pulping temperatures, the chemicals used during pulping may negatively affect the particle size of the adhesive as well. Therefore, the goal of this experiment is to create an ambient environment for the growth of PSA and ink agglomeration. Further, screening and flotation could benefit from this particle enlargement. In addition, it is possible that this would allow lower amounts or partial omitting of deinking additives.

Experimental

The first part of the experimental work was oriented on re-pulping, screening, handsheet preparation and evaluation of stickies formation. Second part of the experiment was focused on behavior of resin and ink in various pH environments. A MicroMaelstromTM Laboratory Pulper (**Figure 1**) was used for substrate pulping. Duration of pulping was set up to 10 minutes at 650 RPM.

Figure 1: *MicroMaelstrom™ laboratory pulper*

Re-pulping experiments were done with the help of a three factorial statistical design. The weight of variables on re-pulping was monitored. The first set of experiments was performed using unprinted "multipurpose" office paper (20lbs) with varied level of unprinted semi gloss label stock (60lbs). High pulping temperatures cause fracture of the pressure sensitive adhesive and it creates numerous small sized stickies in the recycling process [Cameron et al., 2000]. Therefore pulping temperatures were altered and their impact on stickies removal and deinking was monitored. Nine deinking runs were designed with changing pH and temperature (**Table 1**). In three experiments, pulping pH was adjusted to pH 9, which is commonly used within paper recycling industry [Galland, 2006]. In addition, pulping in the neutral region was performed [Galland et al., 1997]. Finally, pulping pH was shifted into the low pH region. The pH level in all experiments was adjusted with 1N sodium hydroxide or 1N hydrochloric acid.

Each of the nine runs from laboratory pulper was screened. A laboratory vibratory screen with slots of 0.006" was used to screen the recycled pulp. The accept was collected on the 200 mesh round screen. Further, handsheets were prepared from the accept. The aim was to evaluate the amount and size of stickies that passed through the screens into accepts, if any. This screening was done using RCA Specification (2012) for paper labels (Litho, Semi Gloss) that are coated with pressure sensitive adhesive. Protocol RC IAP – evaluates adhesive's presence in paper handsheets via image analysis. RCA Specification employs dyeing of the handsheets with the goal of targeting unwanted residue of pressure sensitive adhesive [RCA Specification, Retrieved 2011].

Further, 20 g of "model" acrylic resin (water-based, commercial) was dissolved in 2 liters of deionized water. A total of three solutions were made and their pH was adjusted to 5, 7 and 9 respectively. Solutions were stirred for 5 minutes and filtered with a Buchner funnel. Filter pads were stained using modified RCA specifications (RCA protocol deals with stickies identification. Since acrylic resin is the building block of PSA, this specification was used for resin dyeing and resin identification within the system).

Next step was to prepare "model" ink. The ink was a magenta water based packaging flexographic ink. 20 g of ink was diluted in 2 liter of deionized water. Similarly to "model" resin, three of such ink solutions were prepared with the same pH adjustments. All ink solutions were stirred for 5 minutes using a laboratory air mixer (Model CM-100, Morehouse-Cowles, Inc.) with a propeller blade. Speed was adjusted to 1000 RPM. Ink solutions were filtered through filter pads.

Results and Discussion

Stickies from each run were examined for length and width (**Table 1**). Based on the qualitative evaluation, it was observed that temperature plays an important role on the stickies formation, which was confirmed by other laboratories [Guo et al., 2000; Venditti et al., 2007; Cameron et al., 2000; Galland, 2006]. In addition to these findings, it was noted that pH value is significant in stickies formation (**Figure 2 - 4**). The lower pH region produces long and thick stickies, formed by agglomeration of multiple stickies (**Figure 2**). In the neutral pH region stickies are becoming more or less individual (**Figure 3**). Their length is somewhat similar to the length of stickies obtained in low pH region. Their width is significantly less than that of the width of blended stickies. Stickies that were produced in the alkaline pulping conditions are individual, very fine and their length is somewhat less than the length of stickies from the neutral region (**Figure 4**). According to the theory, complete stickies removal can be achieved if stickies size is above 2000 µm [Assessment of printed product recyclability, scorecard for the removability of adhesive applications. Retrieved 2012]. In this experiment, length and width of stickies was monitored. In all cases the length correspond with criteria for successful removal. On the other hand, width parameter of 2000 µm was achieved only in acidic re-pulping conditions (**Table 1**).

Experiment	Stickies (%)	Pulping Temperature (°C)	pH	Stickies Width (cm)	Stickies Length (cm)
1	10	35	5	0.2 - 0.4	0.5- 3.0
2	25	40	5	0.2- 0.3	0.5 - 1.5
3	5	45	5	0.1-0.2	0.5 - 1.5
4	5	35	7	0.1	0.3 - 1.0
5	10	40	7	<0.1	0.2-1.0
6	25	45	7	<0.1	0.2 -1.0
7	25	35	9	<0.1	0.3 - 0.8
8	5	40	9	<0.1	0.2 - 0.8
9	10	45	9	<0.1	0.2 - 0.8

Table 1: *Effect of pH and Pulping Temperature on Stickies Formation*

Statistical evaluation of nine pulping experiments was performed. Analysis of variance for stickies length and width was done. The results indicate that both of the morphology parameters (width and length) are greatly influenced by the pH value. The pulping temperature also plays an important role, while the level of stickies in the pulp seems to be unimportant. Due to absence of the stickies within the accepts, a smaller experiment was re-run to confirm the effect of pH on stickies formation. This time pulping conditions and level of stickies were the same. The only condition that was varied was pH. The aim of the additional experiment was to observe the impact of pH on the stickies size and formation. Again, an important effect of pH on size of stickies was confirmed **(Figure 2-4)**.

The second part of the experiment was to implement the above findings on water-based resin and water based inks. In all cases lower pH regions resulted in precipitated matter and thus formation of larger particle size **(Figure 5- 8)**. Lower acidic region caused precipitation of water-based resin while alkaline environment had no impact on resin agglomeration **(Figure 7, 8)**.

Figure 2: Stickies@ pH5, 40°C

Figure 3: Stickies @pH7, 40°C

Figure 4: Stickies @pH9, 40°C

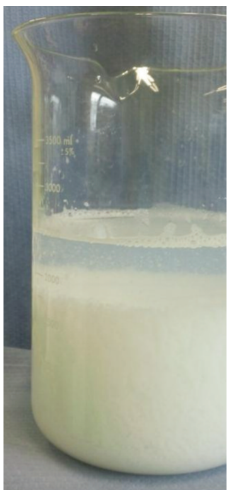

Figure 5: Acrylic resin @ pH 5

Figure 6: Acrylic resin @ pH7

Figure 7: Acrylic resin @ pH 9

Filtration and staining of resin filter pads showed that there is no material left behind the alkaline resin solution. It was also compared to unused but stained filter paper **(Figure 8, 9)**. On the other hand, acidic solution and its impact on precipitation is obvious, when examining the filter pads from acidic resin solution **(Figure 8)**.

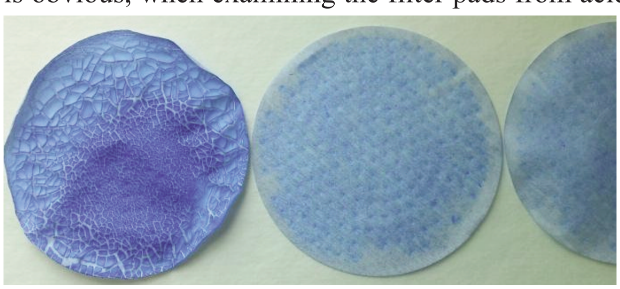

Figure 8: Stained filter pads with acrylic resin residue. Left filter pad comes from acidic resin solution, neutral resin solution pad is in the middle, and filter pad on right is from alkaline resin solution.

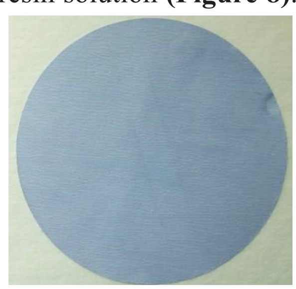

Figure 9: Stained plain filter paper

Water-based inks in general have slightly alkaline pH. Water-based ink used during the experiment had pH of 8.5. It was not a surprise to observe the precipitation of water-based ink in acidic environment. However, for the purpose of this study is was essential to adjust the pH of the water based ink to 5. Within seconds, the ink particles agglomerated and "kicked out" ink could be seen on the sides of the beaker **(Figure 10)**. On the other hand water-based inks in alkaline region stayed unchanged.

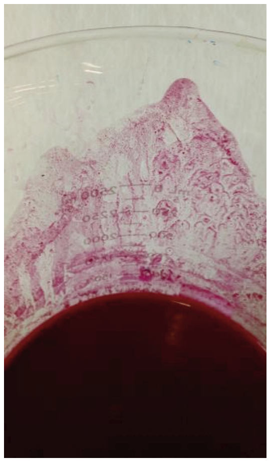

Figure 10: *WB ink @pH 5*

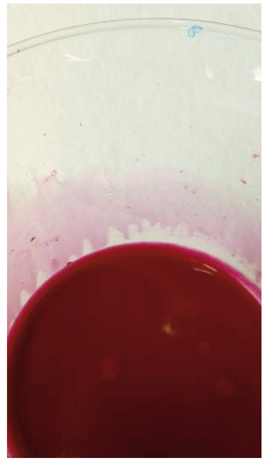

Figure 11: *WB ink @pH 7*

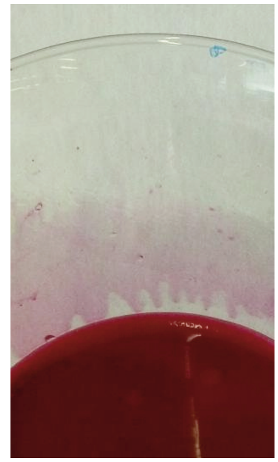

Figure 12: *WB ink @pH 9*

All three inks were filtered with the purpose to collect and quantify the precipitated matter.

Ink that was adjusted to pH 5 was filtered for a duration of 20 minutes and the filter cake showed large amounts of agglomerated ink particles. Collected pigment was dark in color. Ink, which was adjusted to pH 7 showed some precipitated particles. This was due to pH adjustments using 1N hydrochloric acid. Ink that had alkaline pH did not leave behind any residue and filtration was accomplished immediately.

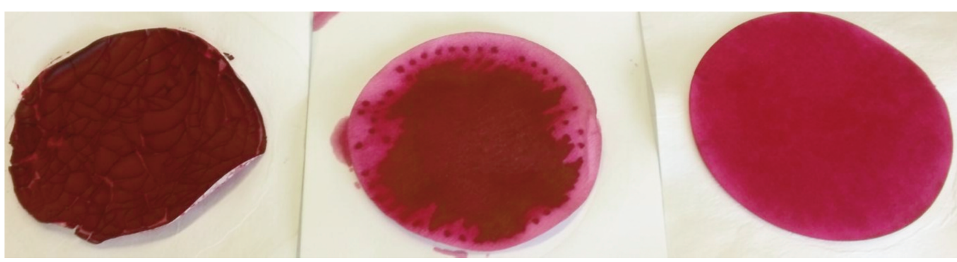

Figure13: *Filter pads with ink residue, left is the pad from acidic ink, center ink adjusted to pH 7, right ink adjusted to pH 9.*

Morphology of stickies in various pH environments was correlated to the morphology of ink particle size. In addition to pulping temperatures, the chemicals used during pulping greatly affect the particles length and width of the adhesive as well. In the acidic regions, carboxylic acid groups should be protonated, which most likely causes the agglomeration of the binder and respectively growth of adhesive particle size. This particle growth is desirable for screening and stickies separation. In theory, a similar mechanism occurs within water-based inks. While inks present submicron contaminants, PSA are viewed as macro contaminants. Also, the deinking concept can be better illustrated with the macro contaminants, which was the aim of this experimental work. Flexo water-based ink's fragmentation was significantly reduced when moving away from alkaline environments towards neutral. It was found that small particles of water-based flexo inks (respectively digital) can be overcome in acidic regions. This behavior was demonstrated using water-based acrylic resin in free acid stage when it becomes insoluble, resulting in large particles.

Traditional recycling is performed in alkaline regions where precipitation of acrylic materials does not occur. It is acknowledged that acidic deinking is not standard but its results are hopeful for further study. It is anticipated to implement the agglomeration concept into ongoing experiments related to true stickies removal and deinking, using flotation.

Conclusion

Current work was focused on deinking of water-based flexo and NIP inks, along with pressure sensitive adhesives all in one step. Experimental study used recycled pulp's macro and micro contaminants. Macro contaminants in this study were stickies, while micro-contaminants were ink particles. It was demonstrated that both types of contaminants are formulated around the same chemistry, which would be acrylic. The behavior of visible macro contaminant in various pH region suggested that particle growth occurs in acidic pH. Similar performance was noted using water-based ink in a low pH region, while the alkaline region had no impact on particle growth. Elimination of both types of contaminants benefited from larger particle sizes. Therefore, application of these findings is desirable, despite the fact that traditional recycling is done in alkaline environment. Recycling efforts are ongoing with aim to implement some of the steps shown here.

Acknowledgement

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