# **Laboratory Scale Two-loop Deinking Trials**

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### **Abstract**

Here we describe our work on the design of a laboratoryscale two-loop deinking process with inclusion of a low-speed high shear kneading step in between two flotation steps. Our approach was then applied to evaluate the deinkability of MOW (mixed office waste) and mixtures of MOW with various amounts of digital print products (where liquid electrophotographic (LEP) prints were used as an example of digital print products). The process conditions at respective stages were optimized to closely mimic that of representative mill-scale processes. The effect of deinking chemistries was also evaluated, with a particular emphasis on nonionic surfactant-based neutral chemistries. The evolutions of dirt area, dirt counts, speck size and optical properties with respect to process stages were monitored to determine the efficacy of our approach. We show that with controlled flotation yields (~ 80% in the pre-flotation, ~ 97% in the post-flotation), the kneading step facilitates noticeable reduction in dirt area. The average speck size was noticed to fall within the perceived deinkable range (i.e. 5-200 microns). It is noteworthy to observe that the two-loop process successfully brings the dirt area for MOW with LEP prints (5% and 20%) to a satisfactory level.

# Introduction

Laboratory-scale single loop deinking process (e.g. INGEDE method 11), which consists of pulping and flotation as two key steps, is performed to simulate mill-scale deinking processes and evaluate the deinkability of print products. With increased amount of digital prints and other difficult-to-deink inks and media in recycling furnish, deinking mills are adopting two-loop or multiloop deinking processes to improve ink detachment and reduce speck contamination. Since the end of the 80's, high-speed disperging or low-speed kneading has become a basic treatment implemented after the conventional pre-flotation in two-loop or multi-loop deinking processes, and is now included in most modern deinking facilities [1].

Disperging or kneading is a major step in two-loop or multi-loop deinking processes. Early work on disperging could be traced back to 1946 by a group of American companies with the intent of recycling kraft bitumen paper [2]. Since then, research has been performed to study the effect of such high-shear process on the detachment of ink particles, bleaching, microbiological decontamination, and changes in fibre properties [3-5]. Today, both high-speed disperging and low-speed kneading are two most commonly used technologies for dispersion processes [1,6].

With the increased complexity of deinking plants and the adoption of multi-loop deinking processes, there is a need to devise relevant laboratory-scale deinking processes to predict and correlate results to that of mill-scale performance. In 2008, PMV (Paper Technology and Mechanical Process Engineering) and PTS (Papiertechnische Stiftung) co-developed presumably the first laboratory-scale two-loop method, in an attempt to correlate results to that of mill-scale processes which employ alkaline chemistry [7]. Besides pulping and flotation steps as in the laboratory-scale single-loop process, three additional steps - dewatering, kneading and/or disperging, and post-flotation were added to mimic industrial processes. Laboratory-scale kneader such as the MKD0.6-H60 was used for kneading while disperger such as the CaviMix was used for disperging. Two different laboratory-scale flotation devices, Voith Delta 25 and PTS flotation cell were used for pre-flotation and post-flotation respectively.

In our work, we have employed a similar process but with conditions at respective stages which closely mimic representative mill's process conditions. In addition, we focused on non-ionic surfactant-based deinking chemistry which has shown encouraging results from our single-loop laboratory-scale experiments to reduce significantly the dirt area for liquid electrophotographic (LEP), and filtrate darkening for inkjet prints [8,9]. The efficacy of our deinking chemistry was also compared to that of non-ionic commercial surfactant.

Representative furnish which includes mixed office waste (MOW), and MOW with varying amounts of digital prints were subjected to our two-loop process. We used liquid electrophotographic (LEP) prints in this particular case. Since the typical challenge with LEP prints is the anticipated larger ink speck size, we expect the performance on dirt area reduction to be significantly improved as a result of implementing the high-shear kneading or disperging process.

## **Experimental**

Figure 1 depicts our two-loop laboratory-scale deinking process which is similar, in terms of the process steps, to that of PMV-PTS approach - pulping, pre-flotation, dewatering, kneading and post-flotation. During pulping, the samples and deinking chemicals were mixed with warm deionized water (~ 55°C) which was pre-treated with 1 gram of calcium chloride so as to achieve the desired water hardness. A pulp consistency (solid content by weight) of 18% was maintained. A Hobart N50 mixer was used to pulp the samples for 15min.

Before the pre-flotation stage, the pulped sample was further diluted with 55°C water to a consistency of 1% in a Voith Delta 25 flotation cell. Pre-flotation was proceeded for 5min, with extra warm water added during the process to ensure overflow. The reject yield was controlled at  $\sim\!20\%$  so as to achieve  $\sim\!80\%$  flotation yield. Next, the pulp was dewatered to 20% consistency. The

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process water from the pre-flotation stage was preserved to be used in the subsequent post-flotation step.

In the kneading step, an IKA HKD 0.6 horizontal kneader was employed to mimic the industrial disperging/kneading stage. The pulp was added to the kneader and preheated for 15min with a rotational speed of 18rpm. The kneader heater was adjusted to achieve an equilibrated temperature of 72°C in the kneading chamber. The pulp was then kneaded for 20min with a higher rotational speed of 40 rpm.

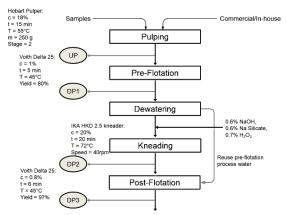


Figure 1. Flowchart of a two-loop laboratory-scale deinking process. UP = Undeinked Pulp; DP = Deinked Pulp.

The kneaded pulp was subsequently transferred to the Voith Delta 25 flotation cell and diluted to 0.8% consistency using the preserved process water from the pre-flotation stage. The post-flotation was proceed for 6min with the reject yield controlled at 3% so as to achieve 97% flotation yield.

Filter pads and handsheets were made after each stage, as shown in Figure 1. Optical measurements of the handsheets and filter pads were performed using an Elrepho spectrophotometer. The dirt areas, total speck counts and speck sizes were determined by an image analysis software.

Table 1 shows the furnish configurations. The two-loop laboratory-scale deinking evaluations were performed using MOW furnish which was acquired from an industrial deinking mill. To this furnish, 5% and 20% LEP prints (by weight) were added. An average single-sided ink coverage of 37% (based on INGEDE color test plot) was adopted for each LEP print page.

Table I. Sample configurations.

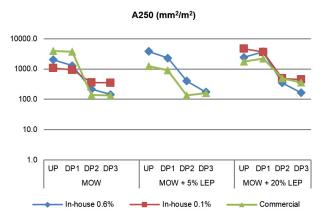
Sample 1	Sample 2	Sample 3
100% MOW	95% MOW + 5%	80% MOW + 20%
	LEP	LEP

Two deinking chemical recipes were used - a commercial non-ionic surfactant chemistry and an in-house developed non-ionic surfactant chemistry [8]. For the latter, two different dosages of the non-ionic surfactant were employed - 0.1% and 0.6%, during the pulping stage. In addition, an anionic surfactant of 0.05% loading was introduced during pre-flotation to maximize the efficiency of our chemistry.

## **Results and Discussion**

We found that the kneading step has a noticeable effect on reducing the dirt speck size and dirt area. The average speck size falls within 200µm after the kneading step, even in the presence of LEP prints, suggesting the effectivness of high-shear process to break ElectroInk specks and other possible large stickies into smaller fragments for efficienct post-flotation.

As shown in Figure 2, regardless of the chemistries and furnish configurations, we notice that, particularly for the commercial and in-house (0.6%) chemistries, the final dirt area  $A_{250}$  (i.e. total area of dirt specks having diameter greater than  $250\mu m$ ) was reduced significantly to  $<600 mm^2/m^2$ , and in some cases to  $<200 mm^2/m^2$ . Similar trend was observed (Figure 3) for T563 (i.e. equivalent black area of dirt specks within the physical area range of 0.02 to 3.0 mm², according to Tappi T563). Within each group, the discrepancy in dirt areas for UP can be attributed to variations in quality of MOW as a result of the smaller amount of furnish (250g) used in each run.



**Figure 2.** Evolution of dirt area  $A_{250}$  wrt deinking stages.

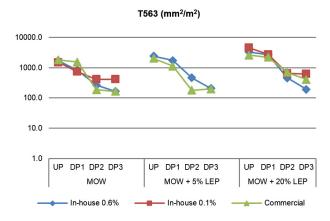


Figure 3. Evolution of dirt area T563 wrt deinking stages.

By calculating the ratio of dirt area of final deinked pulp to that of un-deinked pulp (i.e.  $A_{DP3}/A_{UP}$ ), the extent of dirt area reduction with respect to the different chemistries could be revealed.

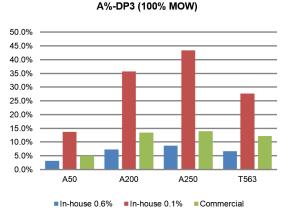


Figure 4A. Percentage of A<sub>DP3</sub>/A<sub>UP</sub> for runs with MOW furnish only.

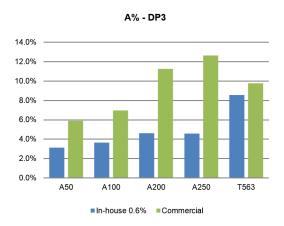


Figure 4B. Percentage of  $A_{DP3}/A_{UP}$  for runs with MOW + 5% LEP furnish.

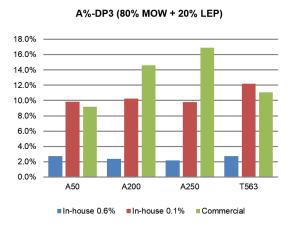


Figure 4C. Percentage of  $A_{DP3}/A_{UP}$  for runs with MOW + 20% LEP furnish.

Figure 4 shows that our in-house developed non-ionic surfactant chemistry (with 0.6% dosage) consistently gives the best dirt area reduction. We believe the chosen surfactant interacts with ink specks and fibers, separating them and preventing the ink specks

from reattaching to the fibers, while also aggregating smaller ink particles into larger specks. The anionic surfactant, added during the pre-flotation, facilitates attachment of the ink specks to the air bubbles which carry them to the top of the flotation cell where they can be skimmed off with the froth, leaving the clean pulp to be recovered [9].

### Conclusion

In summary, we demonstrated the effectiveness of two-loop laboratory-scale deinking process to obtain favorably low dirt areas for both MOW and MOW furnish with 5% and 20% LEP prints. We further show that kneading is a critical step towards enabling the attainment of ink specks with sizes which fall within the deinkable range. The impact of chemistry on the outcome of the process can be clearly observed as well. The implication of the present results to mill-scale processes will be further studied.

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# **Author Biography**

Wenjia Zhang is a Postdoctoral Researcher at Hewlett-Packard Laboratories. Her research at HP Labs is focused on deinking chemistry and process optimization. She received her Bachelor's degree in Chemistry from Nanjing University, China, in 2001. She received her Ph.D. in Chemistry at Missouri University of Science and Technology, where her research includes NMR spectroscopy and Biomass-To-Fuel conversion.