

# Industrial scaleup of enzyme-enhanced deinking of nonimpact printed toners

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**R**ECYCLING WILL BE THE IMPLICIT fate of all paper produced in the future (1). Papermakers are focusing on recycling as an economic necessity (2, 3). New deinking mills established in response to this projected need are already competing for the cleanest and most homogeneous postconsumer paper source, e.g., sorted white ledger (4, 5), and soon will have to dip deeper into the postconsumer paper stream, e.g., unsorted mixed office waste (MOW), to remain competitive.

Current deinking technology is being stretched to accommodate both the hard-to-remove toner inks, stickies, and the colored, dyed, and unbleached fibers present in unsorted MOW. Additional chemicals, multiple flotation steps, and dispersion alleviate some of the limitations of the heterogeneous paper stock. While pulp cleanliness resulting from this sequence is often excellent, the process is capital and energy intensive. Enzyme-enhanced deinking shows promise as a process for improving toner removal (6,7) so that lower quality office postconsumer paper with high liner content can be upgraded.

Our short-term objective was to improve final pulp quality of recycled sorted postconsumer paper so that we can study the recycling of lower quality postconsumer paper. The long-term objective is to establish a simplified deinking process robust enough to handle the variability of MOW. This report focuses on the

short-term objective. Results of enzyme-enhanced, continuous, single-stage flotation pilot plant trials are discussed. Fiber and handsheet properties of the deinked pulp are presented.

All equipment used in this trial was industrial-sized equipment. Since residual toner inks are the primary contaminant remaining in deinked recycled market pulp, a 100% laser pack was chosen as the postconsumer paper stock for these trials. Operating conditions selected for the industrial-scale trials were based on laboratory evaluations of enzyme-enhanced deinking optimized for the removal of toners (8). This paper summarizes the industrial trials and subsequent testing of pulp and handsheet properties and process water quality.

## STRATEGY

Three trials were run at the Voith Sulzer pilot plant in Appleton, WI. To isolate the effect of the enzyme, we used a heat-killed enzyme preparation for the control trial. We used active enzyme as the primary deinking chemical in the other two trials. In all three trials, the surfactant was added into the pulper.

Composite pulp samples were taken at the five designated sampling points: after pulping, before and after flotation, after cleaning and screening, and after pressing (final wet-lap) (Fig. 1). Pulp slurries were gathered every 15 min after the system had been equilibrated and were combined to make the composite sample

## DEINKING

### ABSTRACT

The Mixed office waste (MOW) portion of presently under-utilized mixed recovered paper grade contains more than 70% uncoated free sheet, mainly printed with difficult-to-deink toners. Both the cost and difficulty of removing toners from MOW have limited upgrading MOW into printing and writing grades. This paper presents the results of an industrial-scale investigation exploring the benefits of enzyme-enhanced deinking. Since residual toner inks are the primary contaminant remaining in deinked marked pulp, 100% laser-printed white office postconsumer waste paper was used for these trials. The results of

three deinking trials--two enzyme trials and a control--are presented. The control was similar to the enzyme trials with the exception that a heat-killed enzyme preparation was substituted for the active enzyme. Results showed increased ink removal is achieved at a low level of a commercially available enzyme preparation in combination with a surfactant. The enzyme-enhanced trials also displayed improved drainage and comparable strength when compared with the control. There were no significant differences in the quality and treatability of the process water from the various trials. Effluent samples from these trials were lower in oxygen demand and toxicity than effluents from the control.

for fiber analysis and making handsheet or evaluations of paper properties.

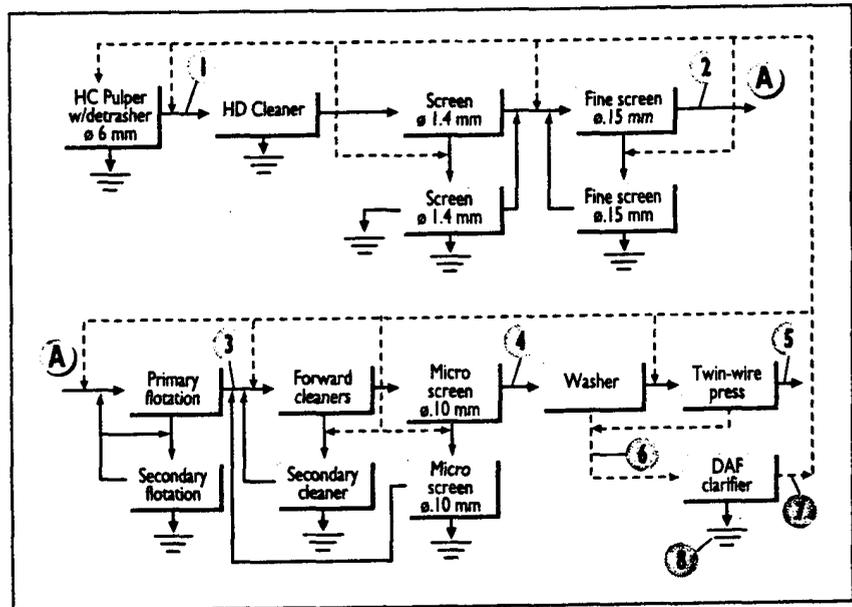
The enzyme preparation used in these trials was a commercially available cellulase mixture, Novozyme 342. It was applied at 0.4 mL concentrate/kilogram paper, 0.04% dry weight basis. The surfactant was a commercial flotation aid. Handsheet were made from the composite samples from each trial and examined for

residual ink. Ink specks were counted within the TAPPI Dirt Range, 0.04 mm<sup>2</sup> (225 μm diameter) to 5.0 mm<sup>2</sup> and 0.02 mm<sup>2</sup> (160 μm diameter) to 5.0 mm<sup>2</sup> (Fig. 2). The near-visible (80-160 μm) and subvisible (10-80 μm) range was also covered. Particles as small as 50 μm in diameter are visible to the naked eye. Handsheets were prepared according to TAPPI Test Method 205 om-88 procedure. We counted residual ink by optical scanner. Brightness was measured according to TAPPI T 525 om-92; brightness pads (TAPPI T 218 om-91) were also used to measure subvisible ink. Burst index was measured by TAPPI 220 om-88 and freeness by TAPPI 277 om-92.

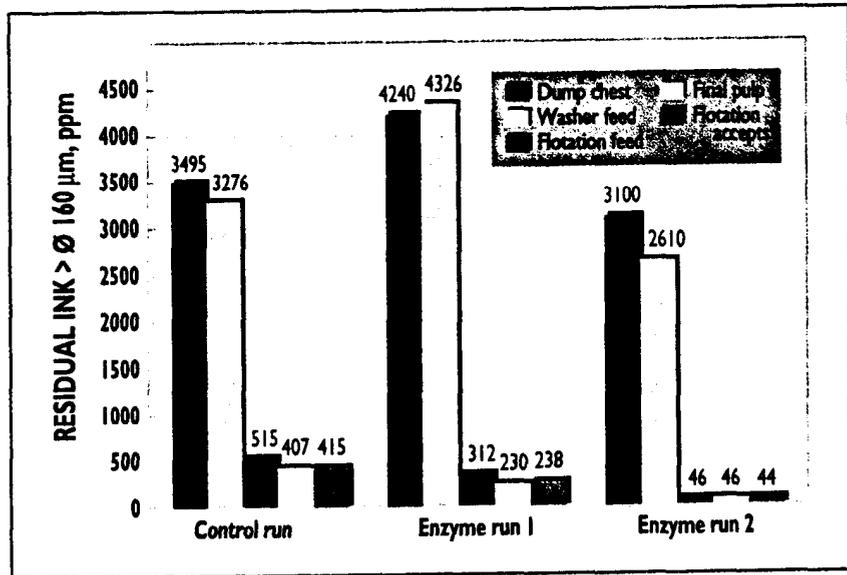
After each trial had been equilibrated, process water was sampled entering and exiting the clarifier (DAF) and analyzed for oxygen demand and toxicity. A composite sampling of the reject effluent stream from each trial was also analyzed for these same parameters. Additionally, a BOD rate study was conducted on the reject effluents. NCASI used an unpublished method for the BOD rate study. This analysis measures oxygen depletion on diluted effluent samples at intervals over a period of several days. Water quality (BOD and COD) was determined at Badger Laboratories in Appleton, WI. Toxicity was measured by Microtox at NCASI in Anacortes, WA, and the BOD rate study was conducted by NCASI in Kalamazoo, MI.

DISCUSSION/RESULTS

The heat-killed enzyme control trial was run using the following conditions: fiberization at 16% consistency, 50°C for 10 min followed by 25 min of pulping at 14% while maintaining 50°C- 55°C. This trial was run without adjusting the pH of the pulped stock (pH 8.3). For the second trial (Enzyme Run 1), we used active enzyme. We intended to use the same target consistencies and temperature



1. Process for enzyme-enhanced deinking



2. Residual ink in handsheets from control and enzyme runs. Dirt count: diameter 160 μm.

as those used for the control trial however, the temperature of the water was 45°C-48°C for the batches in the enzyme trial and the consistencies were on average 2% lower for both fiberization and pulping as a result of the additional moisture content in the paper stock.

Enzyme Run 1 was not as successful in removing ink as we had anticipated. Although this trial was considerably better than the control, it was not as effective as our laboratory experiments.

We reached target conditions in the second enzyme trial (Enzyme

| Dirt count                 | Control | Enzyme Run 1 | Enzyme Run 2 |
|----------------------------|---------|--------------|--------------|
| >225 $\mu\text{m}$ (TAPPI) | 258     | 173          | 26           |
| >160 $\mu\text{m}$         | 417     | 239          | 47           |
| 80 - 160 $\mu\text{m}$     | 167     | 99           | 24           |
| 10 - 80 $\mu\text{m}$      | 310     | 110          | 200          |

### I. Residual ink in control and enzyme trials

| Trial        | CSF, ml | Kajaani, mm | Tensile index, kN·m/g | Burst index, kPa·m <sup>2</sup> /g | Tear index, mN·m <sup>2</sup> /g | Viscosity, (cps,) mPa·s |
|--------------|---------|-------------|-----------------------|------------------------------------|----------------------------------|-------------------------|
| Control run  | 510     | 1.88        | 0.0410                | 2.20                               | 4.28                             | 17.2                    |
| Enzyme Run 1 | 570     | 2.01        | 0.0431                | 2.42                               | 4.39                             | 17.4                    |
| Enzyme Run 2 | 565     | 1.87        | 0.0412                | 2.34                               | 4.25                             | 16.5                    |

### II. Strength properties of pulp

Run 2). In this trial, the pH of the fiberized pulp was lowered to under pH 8—closer to the optimum pH range of this enzyme preparation. Also, fiberization time was decreased by 5 min and pulping time was increased by 5 min. This increased the residence time of the enzyme preparation on the paper fibers without altering the total time in the pulper.

#### Dirt count

As expected, the dirt count dropped most significantly after flotation in all trials. Sheets made from the wet-lap sample 5 from Enzyme Run 2 were within acceptable TAPPI Dirt Count range. Wet-lap from the control run had substantially more residual ink than that from either enzyme run. As **Table I** shows, toner ink removal was excellent when target temperature and consistency conditions were used with this enzyme preparation and surfactant. The five bales for each trial were selected at random

from the 15 bales purchased. Because of the variability of printing within the bales, the trials had different initial pulper ink counts: control, 1600 ppm; Enzyme Run 1, 2100 ppm; and Enzyme Run 2, 1850 ppm. When compared with the respective pulper samples, ink removal efficiency was **98.6%** in Enzyme Run 2 compared with 92% in Enzyme Run 1 and 84% in the control. These efficiency values are based on dirt counts [ppm] larger than 160  $\mu\text{m}$ .

Each trial consisted of five 660-kg air-dried basis (AD) pulper batches. After high consistency pulping with a surfactant and enzyme preparation, the pulp was diluted and pumped into a holding chest for up to 2 h until the system was charged and a continuous flow was possible through the screens, flotation cell, find cleaners. The process diagram (Fig. 1) shows five points where composite pulp samples were taken for handsheet studies: (1) after pulp-

ing. (2) before flowtion, (3) after flotation. (4) after cleaning and screening, and (5) after twin-wire pressing (final wet-lap). Process water samples were taken at the entrance (6) and exit (7) to the dissolved air flotation (DAF) unit and the clarifier sludge (8). After the system had been equilibrated, an effluent reject stream composite was taken at the points indicated by the sewer symbols in the diagram. Clarified process water was recirculated back into the system for dilution purposes as is the case in deinking mills. However, fresh city water was used to make up each pulper batch. Composite effluents from reject streams and from recirculated process water entering and exiting the DAF were analyzed for oxygen demand and toxicity.

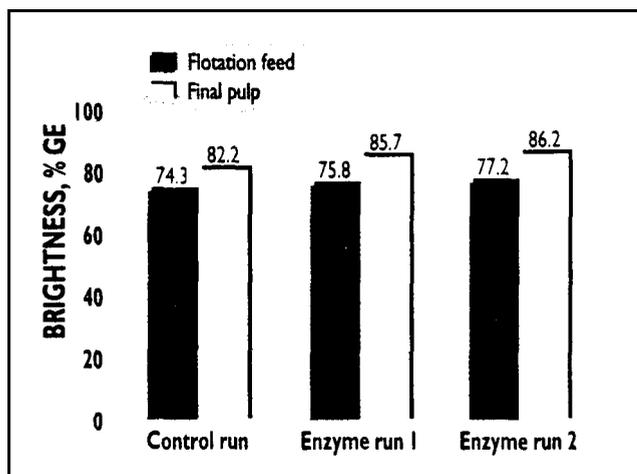
### EXPERIMENTAL

Target pulping conditions used in the trials were as follows:

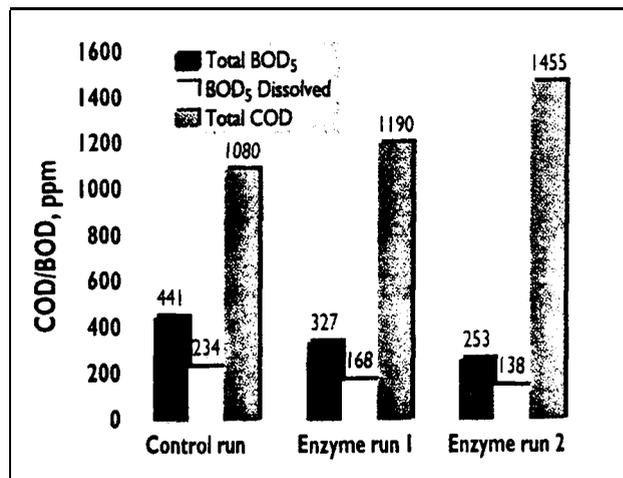
- Add bale of paper into enough 50°C water to make up to 16% solids.
- Fiberize at 16% in a high consistency pulper with 0.125% surfactant (BRD 2340, Buckman Laboratories).
- Add dilute enzyme preparation (Novozyme 342, Novo Nordisk) at a 0.04% level based on oven dry (OD) paper and continue pulping for 30 min at 14% consistency.
- Add 50°C dilution water and pump over to dump chest.

Several pulper batches were combined in the dump chest until enough pulp had been accumulated to charge the system to assure a continuous flow through the subsequent cleaning, screening, and flotation processes.

Industrial trials were followed by laboratory experiments intended to isolate which parameter (consistency, temperature, pH adjustment,



3. GE brightness of pulp from control and enzyme runs



4. Quality of water entering dissolved air flotation clarifier

or increased enzyme residence time) contributed to the improved ink removal in the second enzyme trial. We found that no single parameter was responsible for the improvement observed in the industrial trial, which emphasized the importance of controlling all of the process conditions for successful deinking.

**Physical and optical properties**

Results of burst, tensile, and tear on the final deinked pulp from each trial are summarized in Table II. These strength parameters confirm that fiber bonding was maintained during the enzymatic deinking process.

Optical properties were also measured: brightness is traced through the fiber sampling points in Fig. 3. This graph shows that the enzyme trials produced a pulp with 86% GE brightness compared with 82% for the control. Since the control pulp had an initial brightness about two points lower than that of the pulp in the enzyme trials, the enzyme trials reflect a net gain of two additional brightness points compared with the deactivated enzyme trial. This enhancement might be attributed to the presence

of the hemicellulase in the enzyme preparation.

In all three trials, the pulp had an initial opacity of 81-82%. However, when the final wet-lap was examined, pulp opacity in both enzyme trials was lower (76-77%) than the control (79-80%). Although ash decreased significantly between samples 2 and 5 in all trials (Table III), additional loss of opacity in the enzyme trials might be explained by the improved removal of toners and fines by enzyme action during deinking.

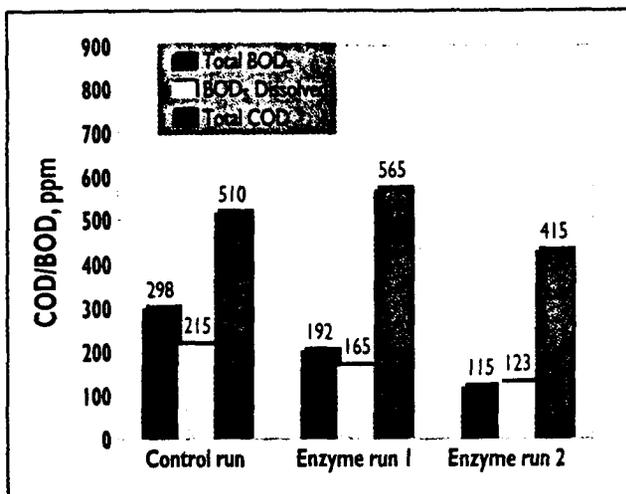
**Pulp analysis**

Pulp from various sampling points in the deinking trials was analyzed for ash, viscosity, fiber length and freeness. Canadian Standard Freeness (CSF) measurements indicated an increase of approximately 60 mL CSF in both enzyme trials. Final wet-lap freeness for the control was 510 mL CSF compared with 570 and 565 mL CSF for the enzyme trials. This increase in pulp freeness observed with the enzyme trials translates into improved pulp drainage and better paper machine runnability (9). Ash content was measured on pulp from each sampling point. Table IV summarizes these results at both high and low temperatures.

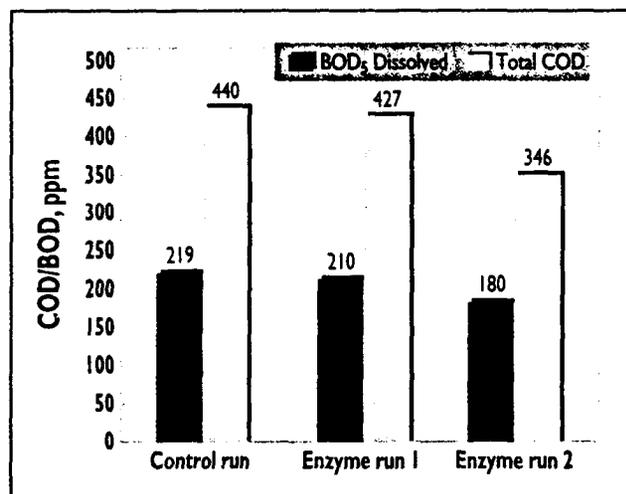
| Trial and pulping stage | Ash, % | Opacity, % |
|-------------------------|--------|------------|
| <b>Control Run</b>      |        |            |
| Flotation feed          | 11.9   | 82         |
| Final pulp              | 2.5    | 79         |
| <b>Enzyme Run 1</b>     |        |            |
| Flotation feed          | 13.5   | 81         |
| Final pulp              | 1.7    | 76         |
| <b>Enzyme Run 2</b>     |        |            |
| Flotation feed          | 12.1   | 81         |
| Final pulp              | 1.4    | 77         |

**III. Relationship of ash content to opacity**

Viscosities of the deinked pulp were 17.2, 17.4, and 16.5 mPa•s, respectively, while the viscosity of the Enzyme Run 2 pulp was slightly lower than that of the other pulps, the strength data do not confirm a loss of fiber integrity as a result of enzymatic treatment. Fiber length measurements were taken on pulp sample 2 (after pulping) and sample 5 (wet-lap pulp). No appreciable difference was noted in the fiber length distribution with or without active enzyme treatment, which is consis-



5. Quality of water exiting dissolved air flotation clarifier



6. Quality of reject stream composite

| Sample Point | Control Run |       | Enzyme Run 1 |       | Enzyme Run 2 |       |
|--------------|-------------|-------|--------------|-------|--------------|-------|
|              | 575°C       | 900°C | 575°C        | 900°C | 575°C        | 900°C |
| 1.           | 12.3        | 9.0   | 13.0         | 9.1   | 11.6         | 9.1   |
| 2.           | 11.9        | 9.3   | 13.5         | 12.1  | 12.1         | 9.7   |
| 4.           | 12.4        | 9.6   | 11.8         | 9.0   | 10.5         | 8.9   |
| 5.           | 2.5         | 1.8   | 1.7          | 1.2   | 1.4          | 1.1   |

IV. Ash content in pulp trials at various sampling points

tent with other measured parameters and which indicates that enzyme treatment did not have a deleterious effect on fiber.

#### Process water quality

Process water entering the clarifier from the enzyme runs contained lower total and dissolved BOD and higher COD than that of the comparable control (Fig. 4). However, the dissolved air flotation (DAF) cell readily clarified the process water, and the water exiting the DAF contained lower BOD and COD than the clarified control water (Fig. 5). The best quality reprocessed water was achieved with Enzyme Run 2, which was also the best trial for ink removal. This trial also contained the lowest BOD and COD of the reject

effluents stream (Fig. 6). There was no detectable difference in the BOD rate study of effluent samples from each trial. Toxicity of reject streams was also comparable. However, if the conventional chemical control were used for comparison, the enzyme runs would undoubtedly be less toxic than the conventional run, as we have observed previously on effluents collected from bench experiments.

#### CONCLUSIONS

Our data led to the following conclusions:

- *Enzyme flotation deinking process*
- *Industrial scale-up confirmed lab-*

oratory results on 100% toner printed paper.

- Residual ink after only primary flotation and limited washing and cleaning was within acceptable TAPPI Dirt Count range of 20 ppm.
- Control of process parameters is critical.

#### Enzyme preparations

- Facilitate toner removal
- Increase pulp brightness
- Improve pulp drainage
- Preserve fiber integrity.

#### RESEARCH PLANS

We plan to move to a more heterogeneous office mix containing at least 60% toner inks for the next scale-up trial. A deinking trial using an enzyme preparation will be compared against a conventional chemical deinking trial. Assuming that ink removal is satisfactory on this paper stock, we will then try a lower grade mixed office postconsumer paper including some mechanical fibers and more highly colored papers. Ultimately, we plan to verify these industrial deinking trials at a deinking mill producing market pulp. TJ

**KEYWORDS**

***Deinking, enzymes, floatation, lasers, mixed waste papers (postconsumer papers), toners.***

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