ENZYMATIC AND CHEMICAL DEINKING OF MIXED OFFICE WASTEPAPER AND OLD NEWSPAPER: PAPER QUALITY AND EFFLUENT CHARACTERISTICS

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Enzymatic and chemical deinking not only significantly influence the optical and mechanical properties of deinked paper, but also influence the pulp properties and wastewater effluent generated. Both enzymatic and chemical deinking of mixed office wastepaper (MOW) and old newspaper (ONP) showed improvement in brightness (1.4-4.7 units), tensile index (1-14%), burst index (1.2-3.8%), freeness (1.9-2.9%), and residual ink removal (31.1-51.2%) but caused loss in opacity (0.1-2.6%) and tear index (0.1-9.6%). Chemical Oxygen Demand (COD) analysis indicated that effluent produced from enzymatic deinking were about 33.9% and 33.8% lower compared to chemical deinking of ONP and MOW, respectively. Meanwhile, Biological Oxygen Demand (BOD_5) obtained from enzymatic deinking of MOW and ONP were 47.1% and 39.3% lower compared to the chemical deinking process, respectively. The results obtained in this work demonstrated that the quality of the pulp and paper obtained from enzymatic deinking process was better than that from the chemical deinking process. This suggests that enzymatic deinking has high potential as an alternative to the chemical method.

Keywords: Enzymatic and chemical deinking; Optical and mechanical properties; Residual ink; BOD_5*; COD*

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INTRODUCTION

The traditional (chemical) deinking process is widely used and is considered to be effective for the removal of ink particles. However, the widespread technology using thermoplastic toner in the printing process presents a special challenge in the conventional deinking process. Furthermore, the process requires the usage of large quantities of chemical agents (Shrinath et al. 1991). This makes the process highly damaging to the environment, and treatment of the effluent to meet environmental regulations is costly (Prasad et al. 1992). On the contrary, a biological process (using enzymes) has been evaluated and proven successful in deinking various types of wastepaper. One of the benefits of using enzymes in the deinking process is the minimum treatment of effluent produced; it is also less harmful to the environment. The effluent from an enzymatic deinking has been reported to be lower in COD content than wastewater from a corresponding chemical deinking process (Putz et al. 1994). Furthermore, enzymatic deinking can avoid the alkaline environment that is commonly employed in the chemical deinking process. This consequently will cut down the cost of chemicals that are used to treat the effluent and also reduce the COD loaded into the wastewater system.

Enzymes used in the deinking process detach ink particles from fiber by partially hydrolyzing the cellulose fibers on the fiber/ink inter-bonding regions, which facilitates ink detachment during the flotation process (Kim et al. 1991). In addition, enzymatic deinking technology has been found to possess advantages in deinking MOW because removal of toner used in xerographic and laser printed processes is very difficult using the alkaline deinking process (Prasad 1993; Jeffries et al. 1994). This is because the toner contains thermoplastic binders that fuse onto the paper fibers during the printing process. They will remain as flat, large, rigid particles when treated with chemical; such particles are poorly separated during the flotation process (Jeffries et al. 1994, 1995; Viesturs et al. 1999). Even a longer pulping process is not very efficient at breaking down the toner particles to a favorable size to be removed by flotation process (Shrinath et al. 1991).

In addition to ink removal, enzymatic deinking may improve the strength properties and increase freeness of the paper sheets, reduce fines content of the recycled fiber, and enhance the brightness and cleanliness of the pulp. On the other hand, ink particles sizes larger than 40 µm are visible to the naked eye, and recycled fiber containing these ink particles size would downgrade the quality of the final product (Carr 1991; Shrinath et al. 1991; Ferguson 1992; Borchardt and Rask 1994). However, this problem can be solved by the enzymatic deinking process. Reduction in ink particles size can be achieved by the enzyme action. Furthermore, small particles are removed more effectively during the flotation process.

Previous studies have found that, based on increment in brightness, deinking of MOW and ONP were more effective using enzymes compared to the chemical method (Lee et al. 2011). However, brightness alone cannot represent the overall quality of the deinked paper. Other pulp and paper properties such as optical properties, mechanical strength, and cleanliness are also very important but have yet to be examined and determined, as the previous studies are limited. Therefore, objective of this present work was to examine and compare the quality of the deinked paper and the effluent after enzymatic and chemical deinking process.

EXPERIMENTAL

Sources of Enzymes

The enzymes used in this study were a mixture of cellulase and xylanase. These enzymes were produced by a local isolate; *A. niger* USM AI 1 *via* solid state fermentation (SSF) process, using a newly developed SSF bioreactor namely FERMSOSTAT. The cellulase to xylanase ratio obtained was 1:5, and this enzymes ratio was used in deinking of MOW and ONP. Cellulase and xylanase activities were determined according to Gessesse and Gashaw (1999) and Gessesse and Gashe (1997), respectively.

Selection and Preparation of MOW and ONP

The wastepaper samples (MOW and ONP) used in this study were obtained within the campus of the university. The MOW mainly consisted of photocopier and computer printout paper. The wastepaper was manually sorted by hand to remove non-paper objects. The sorted wastepaper was kept in a room away from sunlight and high moisture until needed.

Enzymatic and Chemical Deinking Process

Prior to pulping, wastepaper was soaked in tap water for one hour at room temperature and then transferred into the developed bioreactor system for disintegration. The disintegration process was carried out for 60 minutes under room temperature at 6% consistency and 600 rpm. After disintegration, the pulp was recovered by dewatering before being used in enzymatic deinking process (Pala et al. 2004). Pulp (2 kg on air-dry basis) was suspended in water and pulped for 60 minutes at 4% consistency and 400 rpm.

After the pulping process the appropriate volume of water was removed and replaced by the appropriate amount of diluted enzyme solution in order to maintain the pulp slurry at 4% consistency. Diluted enzyme solution was used in order to achieve better dispersion. The reaction of enzymes with pulp occurred at pH 5.5 and 55^{o}C for 45 min with continuous slow mixing, and the total enzyme used was 1.2 U per gram of air-dry pulp (0.2 U of cellulase and 1.0 U of xylanase). A control was run as described above except using thermally inactivated enzyme (Gubitz et al. 1998a). After the enzymatic hydrolysis process, a small volume of the pulp slurry was used to assay for the reducing sugar being produced. The pulp slurry was diluted with water and transferred to flotation vessel using a diaphragm pump.

Water was added to the flotation vessel after the pulp suspension had been transferred into the flotation vessel, and the water addition was controlled by the water level sensor. The flotation process was carried out at 0.24% consistency with 280 L/min of supplied air.

After the flotation process, the pulp suspension was transferred into pulp collecting vessel using a diaphragm pump in order to separate the pulp from pulp water suspension. Thereafter, the deinked pulp was rinsed with water (3X) and handsheets was made in order to determine the efficiency of the deinking process.

The conditions used in enzymatic deinking of MOW and ONP were previously optimized, and the results are summarized in Table 1. Control and sample pulps were processed in a similar manner to that given in the enzymatic hydrolysis described above except heat inactivated and active enzymes were used, respectively.

Meanwhile, similar conditions were used in the chemical deinking process except replacing enzyme with 2% (w/w) NaOH and 2% (w/w) sodium silicate (Pala et al. 2004). The pH of the pulp slurry was adjusted to pH 11.4. The control condition was run as described above with the absence of chemicals. Blank refers to the pulp slurry after pulping without performing the flotation process. Three trials run were carried out for all the experiments.

Table 1. Optimized Conditions of Enzymatic Deinking of MOW and ONP

Pulping Process	**MOW**	**ONP**
Pulping consistency	2%	3%
Pulping time	60 min	45 min
Enzymatic Hydrolysis Process		
Temperature	50°C	50°C
pH	5.5	5.5
Enzyme concentration	4.8 U/g air-dry pulp	2.4 U/g of air dry pulp
Hydrolysis time	60 min	45 min
Flotation Process		
Flotation pH	8.0	8.0
Tween 80 concentration	0.20% (w/w)	0.55% (w/w)
Flotation time	5 min	20 min

Evaluation of Enzymatic and Chemical Deinking Process

Preparation of handsheets for physical tests of pulp was performed using TAPPI test method T205: forming handsheet for physical test of pulp. The prepared handsheet was conditioned under control conditions as described in TAPPI test method TAPPI T402 (Standard conditioning and testing atmosphere for paper, board, pulp handsheets and related products) before the de-inked paper was evaluated. The sample avoided exposure to direct sunlight, extreme temperatures, and relative humidity above 58%.

Determination of De-inked Paper Properties

After enzymatic and chemical deinking processes, the deinked paper was examined for optical and mechanical properties as well as pulp properties. The optical properties of the paper such as brightness and opacity were determined. Meanwhile, mechanical properties of the paper including tensile strength, tear resistance, and burst strength of the paper were determined. Finally, residual ink and freeness of the pulp were analyzed in order to evaluate the properties of the deinked pulp.

Optical properties of paper

The measurement of the paper brightness was carried out as described in TAPPI T452 (Brightness of Pulp, Paper, and Paperboard; Directional Reflectance at 457 nm). Meanwhile, the opacity of the handsheets was measured according to TAPPI T425 os-75 (Opacity of paper).

Mechanical properties of paper

The tensile breaking properties of the handsheets were examined using TAPPI test method T494 but deviating from the standard size of the specimen. Meanwhile, TAPPI T414 procedure was used to measure the internal resistance of the handsheet. The burst strength of the handsheet was measured according to TAPPI T403 procedure (Bursting Strength of Paper).

Pulp properties

The procedure used to measure the freeness of pulp was carried out as described in TAPPI T227 om-94 (Freeness of pulp; Canadian Standard Method). The examination of residual ink of pulp was performed according to TAPPI Standards T213 om-85: Dirt in pulp. The image analysis of the handsheet was carried out using a computer notebook, an Epson Perfection V200 Photo scanner and Spec San 2000 software version V.2.3.30 (Apogee Systems Inc, USA). The scanning condition used was according to TAPPI Dirt Estimation Chart setting. Scanning resolution was 600 dots per inch (dpi), and the area of the handsheet to be analyzed was a 6 inch round handsheet made from TAPPI test method T205: forming handsheet for physical test of pulp. The thresholds used were 90 and 130 for ONP and MOW, respectively (Morkbak and Zimmermann 1998). The lower limit of the ink particle size to be detected was 0.04 mm^2.

Effluent Characteristics after Deinking Process

The effluents obtained from both enzymatic and chemical deinking processes were analyzed in terms of the biochemical oxygen demand (BOD) and chemical oxygen demand (COD) value. This was because effluent with high BOD or COD values needs to be pre-treated before it can be discharged into the environment. The determination of BOD and COD were carried out according to American Public Health Association (APHA) standard method.

Statistical Method

The significance of difference between each test variable were determined using one way ANOVA analysis and Least Significance Test, computed using SPSS version 11.5 software. All tests were done with a confidence interval of 95%. Arrow bars shown in figures indicate means with standard error of three replicates. Means with the same letter indicate no significantly differences. Means with the symbol (+, -) are different in percentage relative to the corresponding blank.

RESULTS AND DISCUSSION

Brightness of Paper

Statistical analysis indicated significant difference ($P<0.05$) in brightness obtained by enzyme and chemically treated MOW and ONP (Fig. 1A and 1B). The brightness values detected from enzymatic and chemical deinking of MOW were about 83.6% and 83.1%, which were 4.7 and 2.3 units higher relative to their respective blank sample. In addition, the final brightness obtained in this work was only about 6.50 units lower compared to unprinted paper. On top of that, the brightness obtained from present work had fulfilled the final minimum brightness of at least 80% for producing writing and printing paper (Schwarz 2000). Different brightness increments in enzymatic deinking of MOW and nonimpact printed wastepaper have been obtained and vary from 2.0 to 7.1 units (Prasad 1993; Yang et al.1995; Heise et al. 1996; Vyas and Lachke 2003). The brightness obtained from enzymatic and chemical deinking of ONP were 41.9% and 41.8%, which were about 2.5 and 1.4 units higher relative to their respective blank sample. Previous studies reported brightness increments after enzymatic deinking of ONP ranging from 0.9 to 5.7 units (Prasad et al. 1992; Morkbak and Zimmermann 1998; Lee 2005). The increase in brightness obtained after enzymatic deinking was attributed to enzymatic action in reduction of residual ink specks. The brightness improvement obtained from present work was comparable with previous literature, which was in a range of 0.0 to 7.1 units.

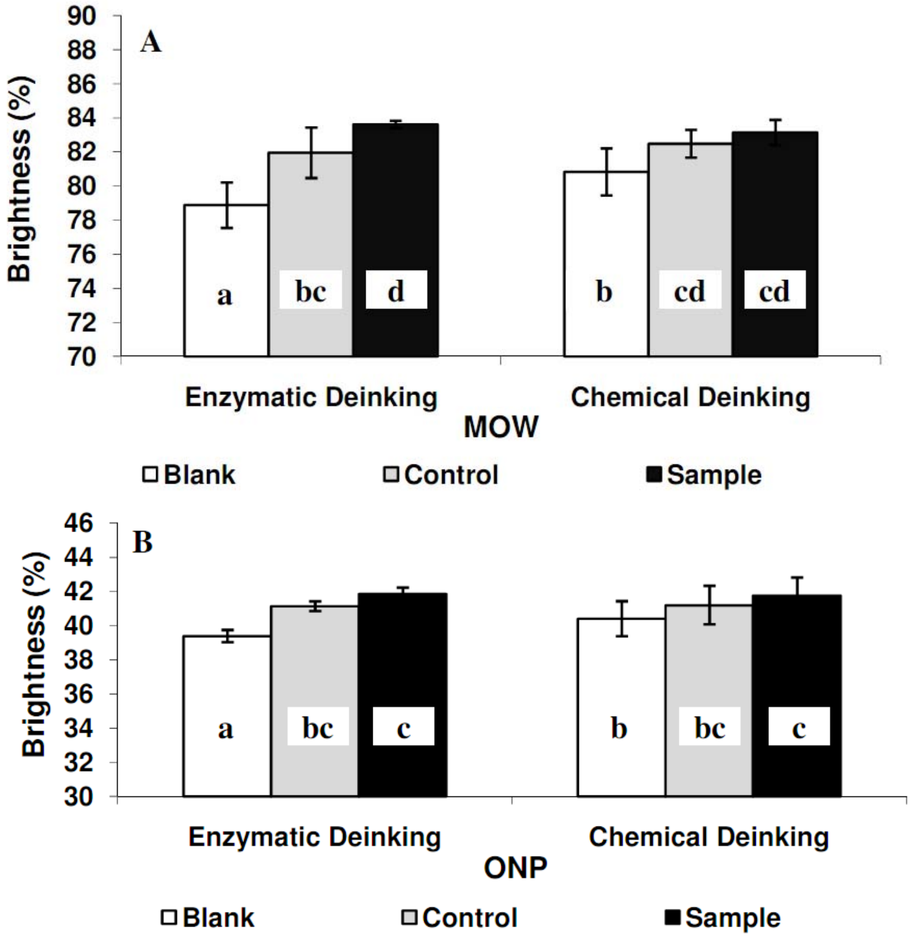

Fig. 1. Brightness of the enzymatic and chemical deinking process of MOW (A) and ONP (B).

Opacity of Paper

Both enzymatic and chemical deinking processes resulted in significant reductions ($P<0.05$) in opacity of MOW (1.4%-2.6%) and ONP (0.1-0.4%) (Fig. 2A and 2B). This is probably due to improvement in removal of toners and fines by enzyme and chemical action during the deinking process. When toner removal was increased, the reflectivity of paper sheet was increased. Thus, opacity of paper was decreased. In addition, the reduction was higher due to enzyme action compared to chemical method. This was attributed to the fact that more toner removal was detected after enzymatic deinking compared to the chemical deinking process. Although the reduction in paper opacity was significant, the final paper opacity still remained high. The opacity of MOW and ONP were 89 to 92% and nearly 100%, respectively. Meanwhile, Rushdan (2003) reported about 98.7% of ONP opacity. The higher opacity of ONP compared to MOW was probably due to the different fiber composition of the paper itself. ONP is commonly produced using a mechanical pulping process and it contained a significant amount of lignin components. These components cause the paper look more "dark" in color and this may result in lower reflectivity of the paper sheet and thus causes an increase in the paper's opacity. Unlike ONP, MOW is generally produced from the chemical (kraft) pulping process, in which almost/all lignin has been separated from cellulose fiber. The lower scattering and lower absorption of light from the kraft fiber, compared to TMP, made the paper brighter and less opaque. Meanwhile, Vyas and Lachke (2003) detected about 1.3% increment in opacity, but Heise et al. (1996) observed about 6.17% reduction in opacity after enzymatic deinking of nonimpact printed toners and MOW, respectively.

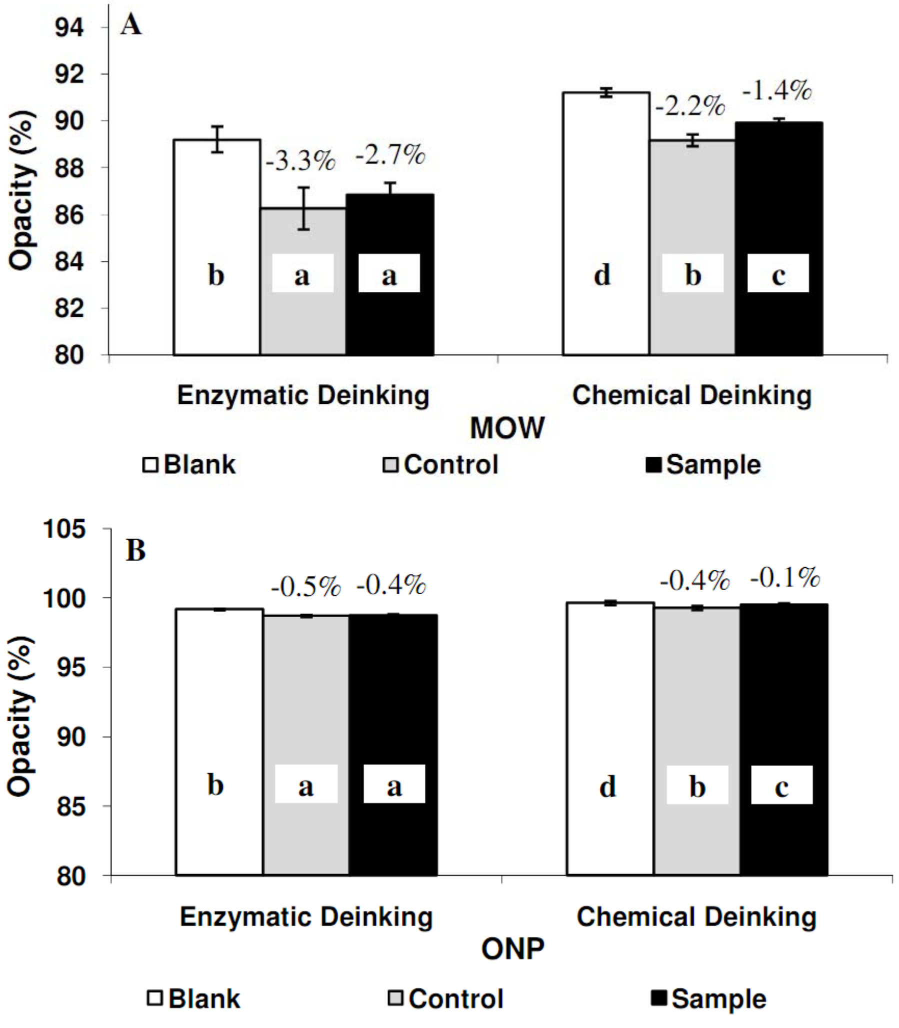

Fig. 2. Opacity of the enzymatic and chemical deinking process of MOW (A) and ONP (B).

Tensile Strength

About 14% and 1% increase in tensile index were detected after enzymatic and chemical deinking of MOW, respectively (Fig. 3A). Statistical analysis indicated significant difference ($P<0.05$) in the result obtained after enzymatic deinking. As reported by previous literature, enzymatic deinking can either bring no effect, enhanced, or deleterious effect to the tensile strength of deinked paper (Pala et al. 2004). The different effects may due to different sources of enzyme preparations and types of wastepaper used in the deinking process, as well as the concentrations and durations used in the enzymatic treatments. About 10% and 7% increase in tensile index were detected after enzymatic and chemical deinking of ONP, respectively (Fig. 3B). In contrast, Lee (2005) detected about 7% drop in tensile index after enzymatic deinking of ONP using commercial available cellulase and hemicellulases. The increment of tensile index after enzymatic deinking process suggested that the activity of cellulase and xylanase were restricted to the surface of the paper fibers. Furthermore, partial hydrolysis of fiber surfaces could contribute to fibrillation and result in an increase in bonding, resulting in stronger paper. Moreover, cellulase acts directly on fines and microfibrils protruding out from the surfaces. This action may remove fines content and improve the interfibrillar bonding, which may result in better paper strength properties such as tensile strength (Jeffries et al. 1994; Gubitz et al. 1998b; Lee and Eom 1999).

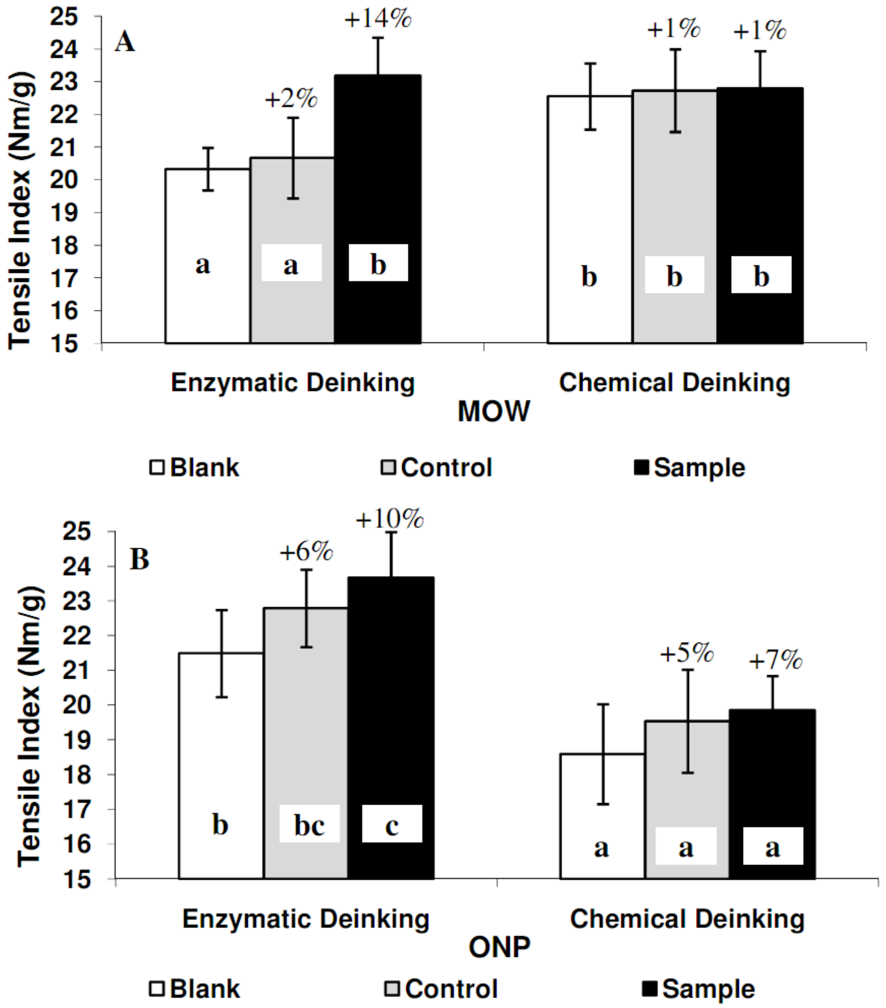

Fig. 3. Tensile index of the enzymatic and chemical deinking process of MOW (A) and ONP (B)

Internal Tearing Resistance

Enzymatic and chemical deinking resulted in adverse effects in the tear strength (Fig. 4A and 4B). The deleterious effect was higher due to enzyme action compared to the chemical process. About 9.6% drop and no effect on tear index were obtained after enzymatic and chemical deinking of MOW, respectively. Meanwhile, previous researchers found that enzyme can either bring no effect or improved or deleterious effect to the tear strength of the paper (Prasad 1993; Morkbak and Zimmermann 1998; Pala et al. 2004). About 3.9% and 1.1% decrease in tear index were obtained after enzymatic and chemical deinking of ONP, respectively (Fig. 4B). A similar finding was reported by Xia et al. (1996), who observed severe damage to tear strength in deinking of ONP. The widely accepted mechanism for enzymatic removal of ink suggested that the surface layers are removed by enzymatic hydrolysis of cellulose in combination with the shear forces of mixing. According to previous findings, most cellulase and xylanase mixtures have been found to remove microfibrils and fines, as indicated by an increase in the freeness of the pulp (Prasad 1993; Jeffries et al. 1994; Heise et al. 1996). However, when fines are removed, the paper sheets become more permeable and less dense, subsequently decreasing the paper strength (Welt and Dinus 1995).

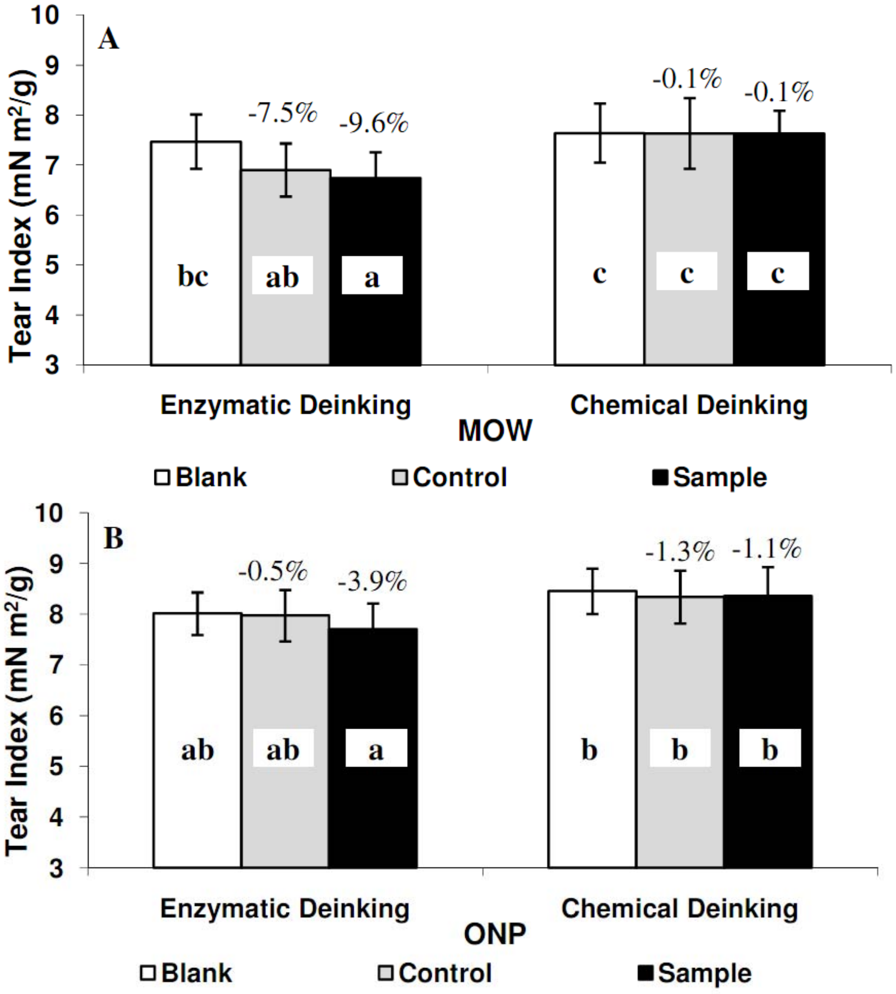

Fig. 4. Tear index of the enzymatic and chemical deinking process of MOW (A) and ONP (B)

Bursting Strength

Both enzymatic and chemical deinking processes were able to enhance the burst strength of the deinked paper (Fig. 5A and 5B). Statistical analysis indicated significant difference ($P<0.05$) in the results obtained, except for chemical deinking of MOW. About 3.4% and 1.2% increase in burst index were detected after enzymatic and chemical deinking of MOW, respectively. However, Jeffries et al. (1994) observed about 8.4% loss in burst index. About 3.8% and 3.0% improvement in burst index were obtained from enzymatic and chemical deinking process of ONP, respectively (Fig. 5B). Xia et al. (1996) reported a nearly 10% drop, but Lee (2005) observed a 1.8% increase in burst index after enzymatic deinking of ONP. In chemical deinking of MOW, alkaline conditions are responsible for fiber swelling, which favors ink detachment at larger particle size and avoids the partial removal of toner. This subsequently decreased the release of fines particles and thus reduced the mechanical action on the fiber surface. In contrast, when impact inks are present (ONP), NaOH (alkali) can act directly on the ink, break down its structure, promote ink dispersion, and thus improve the release of fines content. This probably can explain why burst index improvement from chemical deinking of ONP was higher compared to chemical deinking of MOW.

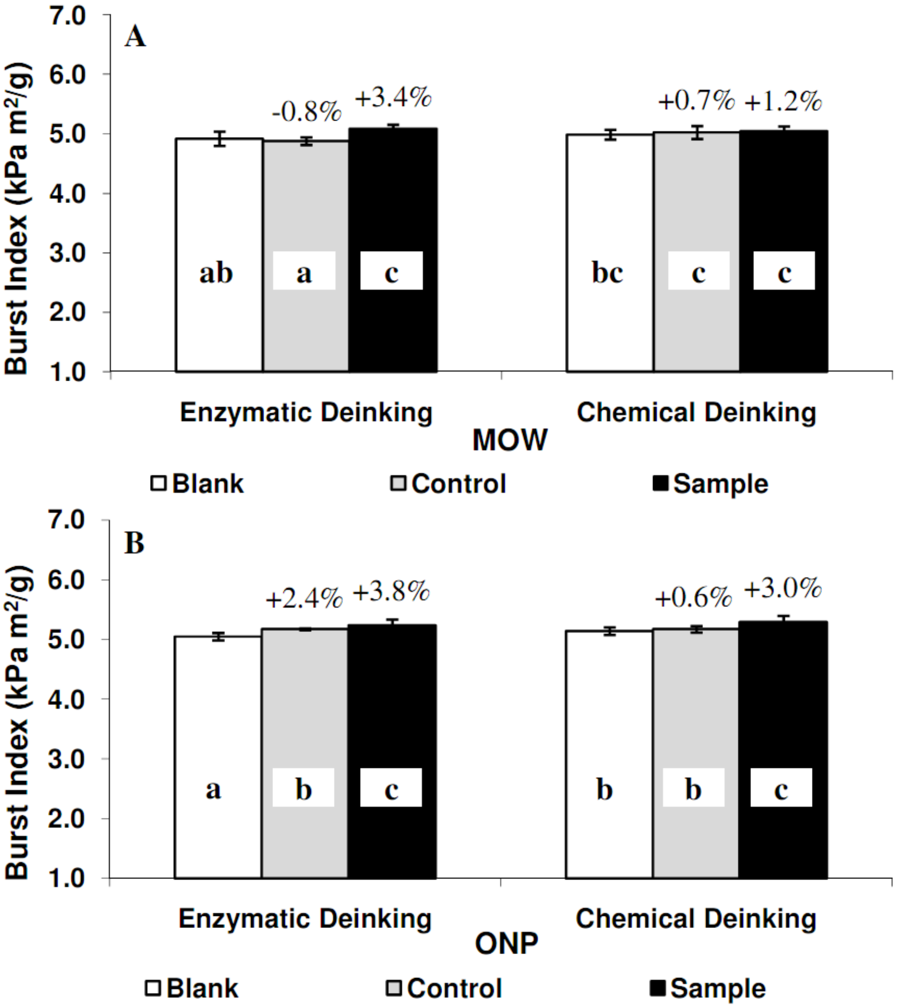

Fig. 5. Burst index of the enzymatic and chemical deinking process of MOW (A) and ONP (B)

Freeness of Pulp

Pulp freeness values obtained from MOW and ONP after the deinking process were in a range of 500 to 550 mL CSF and 200 to 225 mL CSF, respectively (Fig. 6A and 6B). Statistical analysis demonstrated that increase in pulp freeness was significant ($P>0.05$) after enzymatic and chemical deinking of MOW. Similar findings were also observed by previous researchers, who reported increases in pulp freeness after enzymatic deinking of laser and xerographic printed papers as well as MOW (Jefferies et al. 1994; Yang et al. 1995; Heise et al. 1996). About 2.9% and 2.3% increases in pulp freeness were detected after enzymatic and chemical deinking of ONP. Meanwhile, previous literature reported that modification of secondary fiber with cellulase or mixtures of cellulase and hemicellulases enzymes at low concentration can significantly increase pulp freeness but with little or no change in mechanical strength of the pulp (Pommier et al. 1989).

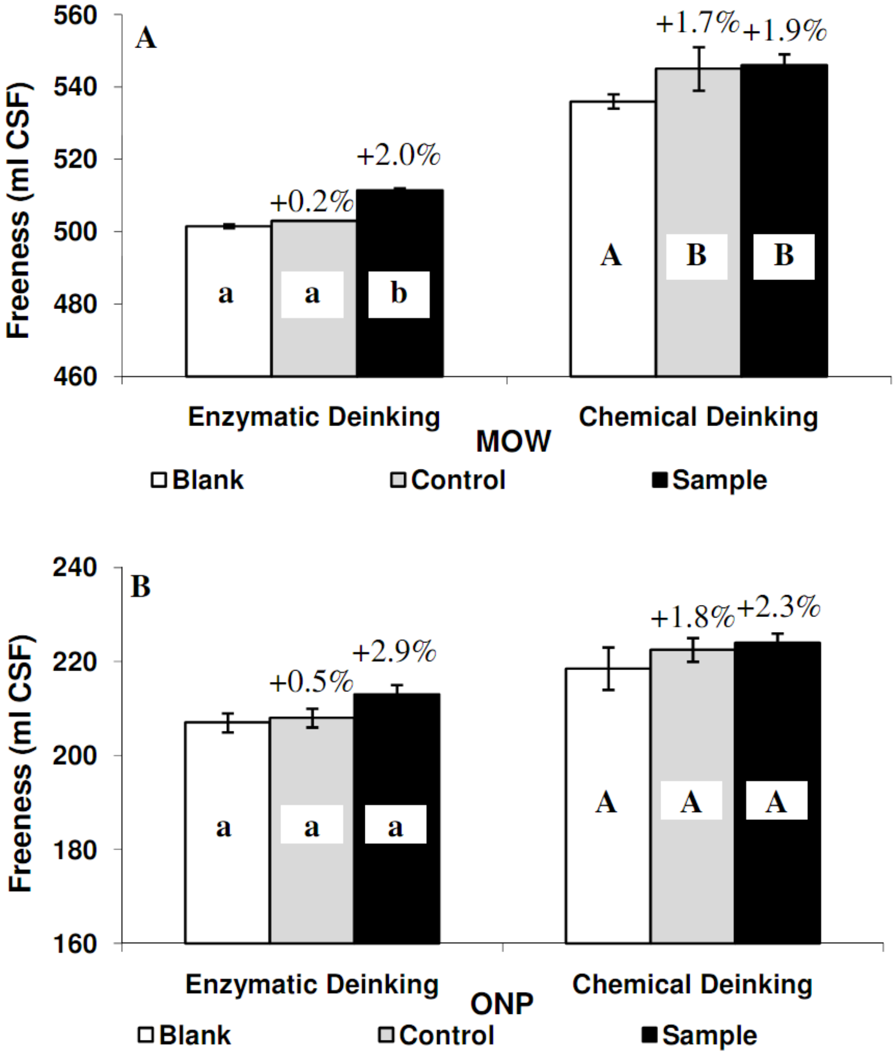

Fig. 6. Freeness of the enzymatic and chemical deinking process of MOW (A) and ONP (B)

Residual Ink of Pulp

Residual ink refers to the total ink particle area (mm^2) per sheet area (m^2) found before and after the deinking process. Significant increases ($P<0.05$) in ink removal were detected after enzymatic and chemical deinking processes (Fig. 7A and 7B). About 44.5% and 31.1% improvement in ink removal was obtained after enzymatic and chemical deinking of MOW, respectively. This was due to the fact that toner was detached at larger particle size during the chemical deinking process, and it subsequently floated poorly and was not efficiently removed by the flotation process (Shrinath et al. 1991). Unlike the chemical deinking process, it is possible that the larger ink particles might have been broken down into small particles due to enzyme action, and smaller particles were effectively removed by flotation process (Vyas and Lachke 2003).

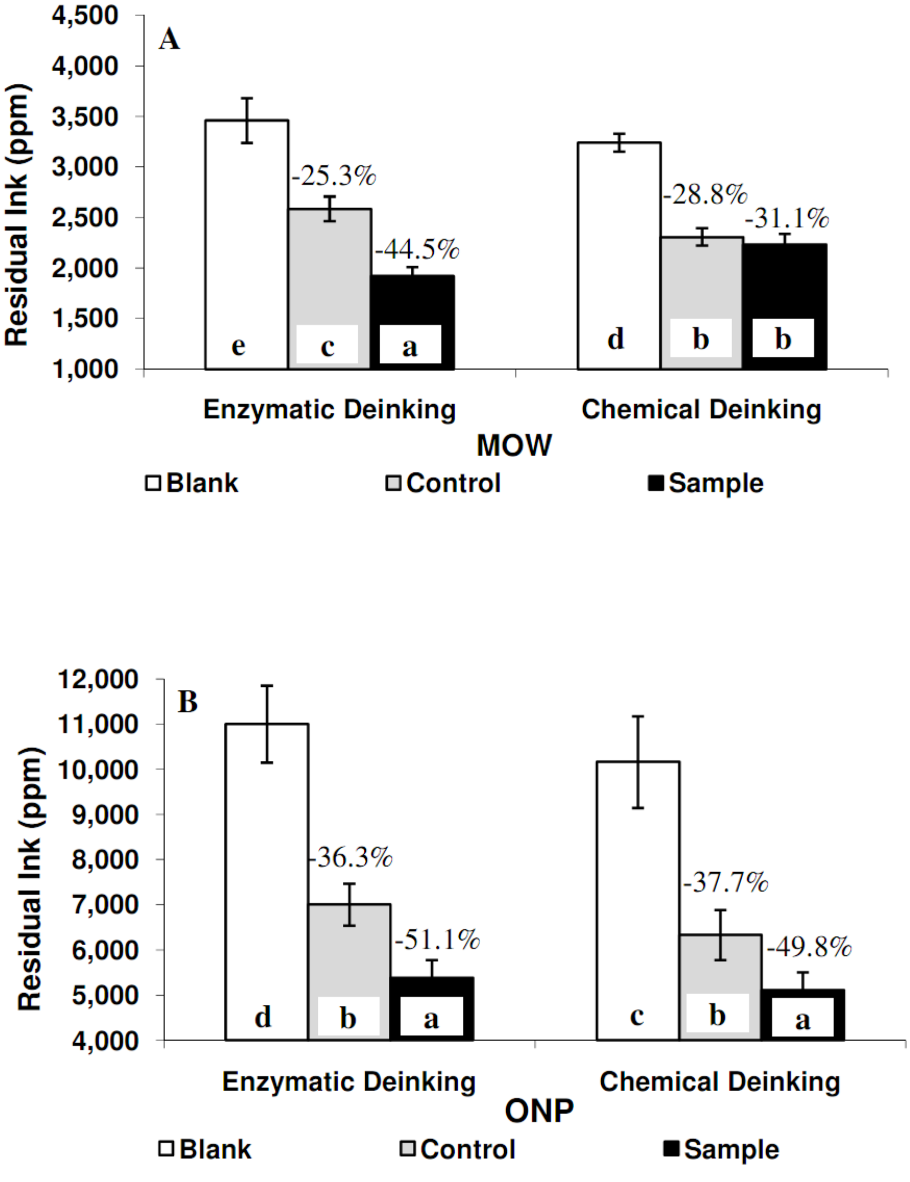

Fig. 7. Residual ink after enzymatic and chemical deinking process of MOW (A) and ONP (B)

Meanwhile, about 50% of ink was removed after enzymatic and chemical deinking of ONP. Impact ink that is commonly used in newspaper printing does not fuse on the paper fiber during the printing process. This makes it easier to deink even by conventional deinking processes (Pala et al. 2004). In contrast, nonimpact inks, which are commonly used in xerographic, laser, and inject printing, are more difficult to deink (Zhu et al. 2005). Furthermore, enzyme or chemical treatment can act directly on the impact ink particles and subsequently break down the ink particles to a size suitable for removal by flotation process (Vyas and Lachke, 2003). This probably can explain the result obtained from present work showing higher ink removal on ONP compared to MOW.

Effluent Characteristics after Deinking Process

BOD_5 and COD

The BOD_5 and COD values obtained from wastewater effluent after enzymatic deinking process were significantly ($P<0.05$) lower compared to chemical deinking processes (Fig. 8A and 8B). About 47.1% and 33. 8% reductions in BOD_5 and COD values were detected after enzymatic deinking compared to chemical deinking of MOW, respectively. Meanwhile, Kim et al. (1991) reported that wastewater effluent from enzymatic deinking process was about 20 to 30% lower in COD value compared to a chemical deinking process. About 39.3% and 33.9% lower BOD_5 and COD values were obtained after enzymatic deinking of ONP compared to the chemical deinking process. The BOD_5 and COD obtained after deinking of ONP was higher compared to deinking of MOW. This may be due to different fiber composition of MOW and ONP. Besides cellulose fiber, ONP contained some lignin, hemicelluloses, and more fines content, which may be extracted out during the deinking process into the wastewater effluent. On the contrary, MOW contained essentially no lignin and created less wastewater effluent after the deinking process. The wastewater effluent obtained from both deinking process were low and can be safely discharged into the environment without any treatment, with the exception of chemical deinking of ONP. Based on the Malaysia Environmental Act 1974, the permitted BOD_5 and COD values for effluent safely discharged into non-catchment areas are equal to or less than 50 ppm and 100 ppm, respectively.

Table 2. Summary of Pulp and Paper Properties after Deinking Process

Pulp/Paper properties	Deinking Method	MOW	ONP
Brightness	Enzymatic	+ 4.7 units	+ 2.5 units
	Chemical	+ 2.3 units	+ 1.4 units
Opacity	Enzymatic	- 2.6%	- 0.4%
	Chemical	- 1.4%	- 0.1%
Tensile Index	Enzymatic	+ 14%	+ 10%
	Chemical	+ 1%	+ 7%
Tear Index	Enzymatic	- 9.6%	- 3.9%
	Chemical	- 0.1%	- 1.1%
Burst Index	Enzymatic	+ 3.4%	+ 3.8%
	Chemical	+ 1.2%	+ 3.0%
Freeness	Enzymatic	+ 2.0%	+ 2.9%
	Chemical	+ 1.9%	+ 2.3%
Residual Inks	Enzymatic	-44.5%	- 51.1%
	Chemical	-31.1%	- 49. 8%

Note: Means with the symbol (+, -) are different (sample) in percentage relative to its blank.

In summary, the pulp and paper properties after enzymatic and chemical deinking processes were characterized. The results obtained are shown in Table 2. Based on the results obtained, it can be concluded that the overall pulp and paper properties obtained from enzymatic deinking process were better compared to the chemical deinking process.

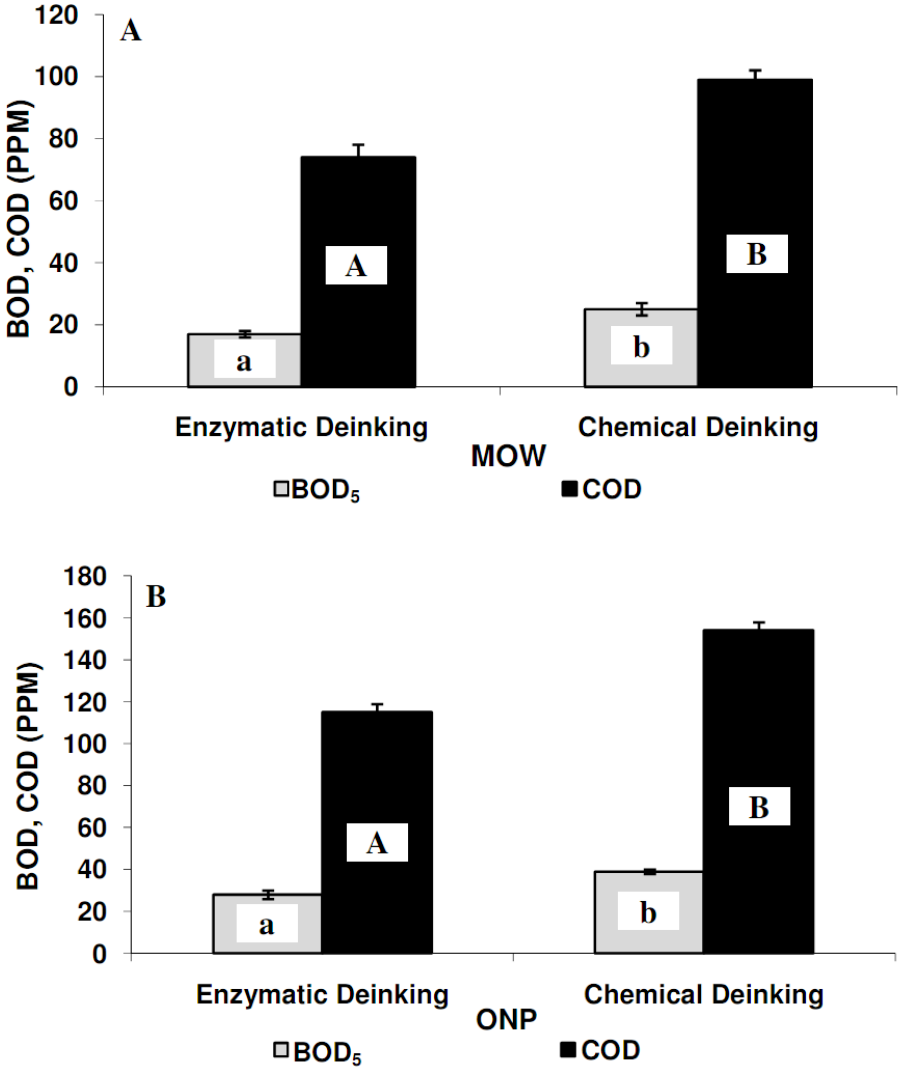

Fig. 8. BOD and COD of the enzymatic and chemical deinking process of MOW (A) and ONP (B)

CONCLUSIONS

1. The quality of the pulp and paper properties obtained from an enzymatic deinking process was better, in terms of brightness, tensile, burst, and freeness, compared to a chemical deinking process.
2. An increase in pulp freeness, as a consequence of deinking, may indirectly improve drainage rate, resulting in faster machine speed and the possibility of significant savings in energy and thus overall operating costs.

3. For cleanliness of deinked paper, the enzyme process was found to be a more effective substitute for chemical deinking.
4. In addition to ink removal, the wastewater effluent produced from the enzymatic deinking process was lower in BOD_5 and COD content than the chemical deinking process.
5. Enzymatic deinking was found to have high potential as an alternative to the chemical deinking process, which showed several problems and disadvantages compared to enzymatic deinking process.

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