PILOT SCALE ENZYMATIC DEINKING OF MIXED OFFICE WASTEPAPER AND OLD NEWSPAPER

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The performance of a newly developed pilot scale continuous enzymatic deinking system has been evaluated using a mixture of cellulase and xylanase enzymes in the deinking of mixed office wastepaper (MOW) and old newspaper (ONP). Optimizations of the enzymatic deinking processes were carried out, and the optimum conditions obtained for MOW and ONP were different. The highest brightness obtained from enzymatic deinking of MOW and ONP under their respective optimum conditions were about 83.6% and 41.9%, respectively. The deinking efficiency of 6.0% and 6.3% were obtained by enzymatic deinking process using MOW and ONP, respectively. On the other hand, the deinking efficiency of 2.9% and 3.5% were obtained by a chemical deinking process using MOW and ONP, respectively. The findings obtained from present work indicated that enzymes have potential to be used in deinking of MOW, which is difficult to deink by conventional chemical deinking methods.

Keywords: Enzymatic deinking; Chemical deinking; Pilot scale deinking system; Cellulase; Xylanase; Mixed office wastepaper, Old newspaper

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INTRODUCTION

Due to the fast exhaustion of forest resources, the paper manufacturing industry has focused on alternative fast-growing wood species as well as the use of secondary fibers. This has led to the increased recycling and reuse of wastepaper all over the world. In addition, the use of wastepaper has become a crucial and environmentally friendly source for papermaking (Gubitz et al. 1998). On top of that, it also helps to preserve forest resources, reduce environmental pollution, and contribute to water and energy conservation (Bhardwaj et al. 1995). However, the major problem in recycled wastepaper is the removal of ink or toners. Paper mills need to remove contaminants, particularly ink, from the wastepaper through deinking processes before the wastepaper pulp can be recycled. The degree of ink removal depends mainly on the printing process, ink, and fiber type. Generally printing can be performed either using impact or nonimpact ink. Impact ink, which is commonly used in newspaper printing, does not fuse on the paper fiber and this make it relatively easy to deink even by conventional deinking processes (Pala et al. 2004).

On the contrary, nonimpact inks that are generally used in photocopy and computer printing are more difficult to deink (Zhu et al. 2005). This is due to the toners that are thermally fused and strongly crosslinked with cellulosic fibers of the paper during the printing process. This make it remain as large, flat and rigid particles that are difficult to disperse and poorly separated from fibers during the washing and flotation processes (Jeffries et al. 1994, 1995; Viesturs et al. 1999). This had led to the suggestion of enzymatic approaches to overcome the problems encountered by the conventional chemical techniques. The potential of using enzymes in deinking of wastepaper has been investigated and proven successful using different types of enzymes. Treatments with cellulases and hemicellulases mixtures have been shown to deink MOW (Prasad 1993; Jeffries et al. 1994) and ONP (Prasad et al. 1992, 1993). In addition, previous studies in this laboratory on deinking of laser printed wastepaper using commercially available cellulases and hemicellulases have shown significant results (Lee et al. 2007).

Therefore, this had led to the development of a pilot scale enzymatic deinking system to continue exploring the possibility of using enzyme in deinking of wastepaper. The aim is to give priority to the recycling of wastepaper as an ecofriendly approach in the paper industries in Malaysia. In addition, this study evaluates the use of a cellulase and xylanase mixture on deinking of MOW and ONP using a newly developed pilot scale enzymatic deinking system.

EXPERIMENTAL

Development of Pilot Scale Enzymatic Deinking System

The developed pilot scale of enzymatic deinking system is made up of stainless steel, which is resistant to corrosion (Fig.1). It basically consists of a water/enzyme recycling tank, enzyme storage vessel, bioreactor for pulp hydrolysis, flotation system, and pulp collecting vessel. The deinking system is equipped with sets of control systems for mixer, vacuum pump, compressor, blower, heater, pH probe, thermocouples, foam scraper, and various pumps.

The uniqueness of this deinking system is that most of the process sequence can be programmed and performed automatically. In addition, a sieve is provided at the bottom of the bioreactor with an outlet located at the center. The function of the sieve is to filter enzyme solution or water from the bioreactor to the enzyme/water recycling vessel after the enzymatic hydrolysis or pulping process with the help of a vacuum pump. On top of that, the pulp collecting vessel is equipped with two stainless steel sieves that are used to retain and separate pulp fiber after the flotation process. The water will be collected in the pulp collecting vessel and can be reused again in next batch of deinking process or discarded into wastewater treatment pond. The developed enzymatic deinking system is a novel design and not an improvement of any existing one. The developed enzymatic deinking system was used to optimize the deinking process of MOW and ONP.


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Selection and Preparation of MOW and ONP

The wastepaper (MOW and ONP) used in this study were obtained within the USM campus. MOW mainly consisted of photocopier and computer printout paper. The wastepaper was manually sorted by hand to remove non-paper objects such as staples, rubber bands, stickers, and others. The sorted wastepaper was kept in a room away from sunlight and high moisture until needed. The storage time of waste paper was about 1 month.

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* a solid state fermentation (SSF), 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.

Enzymatic Deinking Process

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

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

Water was added to the flotation vessel after all the pulp suspension had been transferred into the flotation vessel. The water addition was controlled by the water level sensor. The flotation process was carried out at 0.24% consistency with 280 L/minutes of air supplied. The initial conditions set for the process are summarized in Table 1. After the flotation process, the pulp suspension was transferred into the pulp collecting vessel using a diaphragm pump. After that, the deinked pulp was rinsed with water (3X), and handsheets were made in order to determine the efficiency of the deinking process.

Optimization of Enzymatic Deinking Process

Generally enzymatic deinking processes include a pulping process, enzymatic hydrolysis of wastepaper, and flotation process. In order to improve the efficiency of deinking as a whole, each of the above processes needs to be optimized.

Table 1: Initial Conditions Set for Enzymatic Deinking of MOW and ONP

Deinking Parameters	MOW and ONP
Pulping process	
Pulping consistency	4%
Pulping time	60 min
Enzymatic Hydrolysis process	
Temperature	50^{o}C
pH	5.5
Enzyme concentration	1.2 U/g air-dry pulp
Hydrolysis time	45 min
Flotation Process	
Flotation pH	8.0
Tween 80 Concentration	0.200% (w/w)
Flotation Time	5 min

Evaluation of Enzymatic Deinking Process

Preparation of handsheets for physical tests of pulp was performed using TAPPI test method T205, forming handsheets for physical tests of pulp. The prepared handsheets were conditioned under controlled conditions as described in TAPPI test method T402 (Standard conditioning and testing atmosphere for paper, board, pulp handsheets and related products) before the de-inked paper was evaluated. The sample was kept from exposure to direct sunlight, extremes of temperature, and to relative humidities above 58%. Meanwhile, 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) using brightness and opacity tester; model: Micro S-5 (Technidyne corporation, USA).

Deinking Efficiency

The deinking efficiency (%) was determined based on Eq. 1. Blank refers to the pulp slurry after pulping without performing the flotation process. Control and sample pulps were processed in a similar manner to that given in the enzymatic hydrolysis described above, except that heat-inactivated and active enzymes were used, respectively. Three trial runs were carried out for all the experiments. The enzymatic deinking conditions described above were applied for MOW and ONP. The optimum condition obtained from each experiment was used unless otherwise stated. The average brightness of blank MOW and ONP pulps were 80.10% and 40.10%, respectively. Meanwhile, the average brightness detected by unprinted writing paper and unprinted newspaper were 88.75% and 48.29%, respectively. Based on Eq. 1, the maximum deinking efficiency that could be obtained with the MOW and ONP were 10.80% and 20.42%, respectively.

$$\text{Deinking efficiency} = \frac{Sb - Bb}{Bb} \times 100\% \quad (1)$$

In this equation *Sb* is the sample brightness (%) and *Bb* is the blank brightness.

Comparison with Chemical Deinking Method

In order to compare the efficiency of enzymes in deinking of MOW and ONP as well as the effectiveness of developed enzymatic deinking system, a chemical deinking process was carried out. The chemicals used in the deinking process included 2% (w/w) NaOH and 2% (w/w) sodium silicate. The pH of the pulp slurry was adjusted to pH 11.4. A control was run as described above with the absence of chemicals (Pala et al. 2004). The other conditions used in chemicals deinking process were based on the optimum enzymatic deinking conditions obtained from the studies.

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%.

RESULTS AND DISCUSSION

Pulping Process

There was a very low ink removal obtained with the increase of pulping consistency. In addition, no significant differences (P>0.05) in deinking efficiency were obtained when the pulping process was carried out in the range of 2% to 6% (data not shown). Pulping using MOW at 2% consistencies for 60 minutes and ONP at 3% for 45 minutes gave the highest deinking efficiencies of about 2.3% and 3.1%, respectively (Figs. 2). The results obtained indicated significant differences (P<0.05) for ONP but not a significant difference (P>0.05) for MOW. Similar findings were reported by Viesturs et al. (1999), who observed darkening of the pulp as the pulping time was increased. Meanwhile, Prasad (1993) reported that the deinking efficiency obtained from enzymatic deinking of laser and xerographic office waste was decreased linearly with increased pulping time, reaching a minimum in the range of 30 minutes.

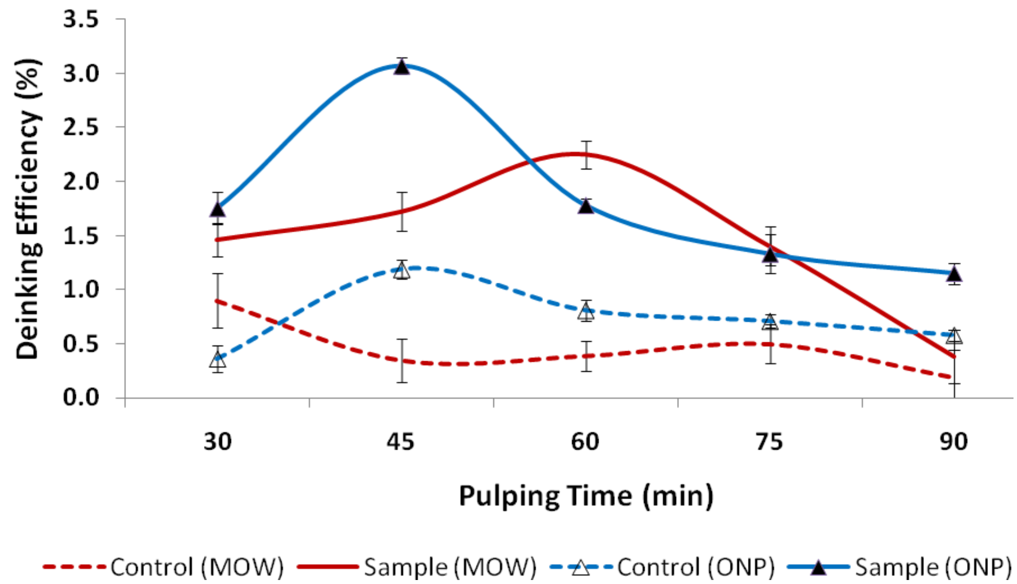
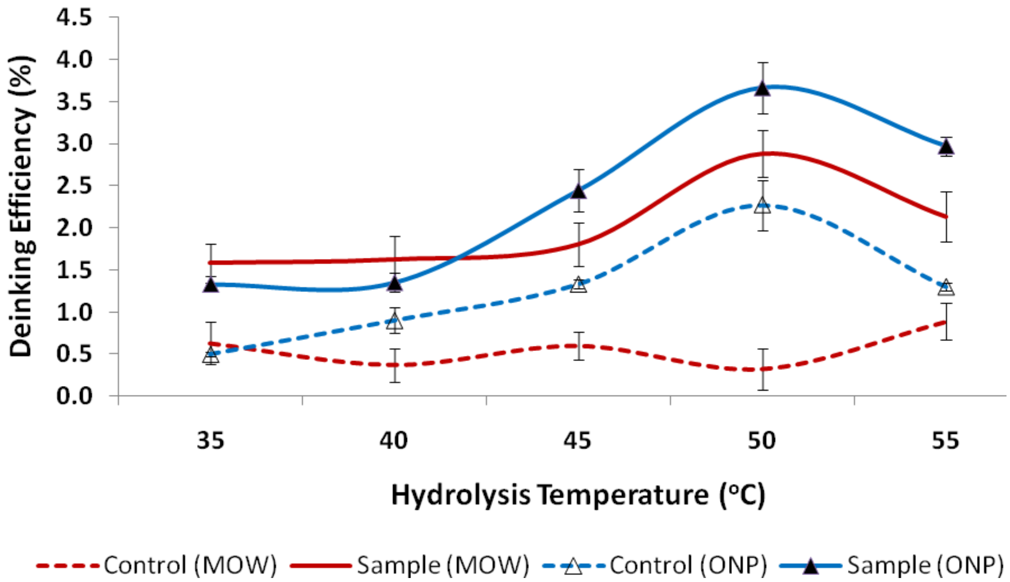


Fig. 2. Effect of different pulping time on ink removal using MOW and ONP.
Note: Total reducing sugar and deinking efficiency arrow bars indicate means with standard error of three replicates.

Effect of Enzymatic Hydrolysis Process

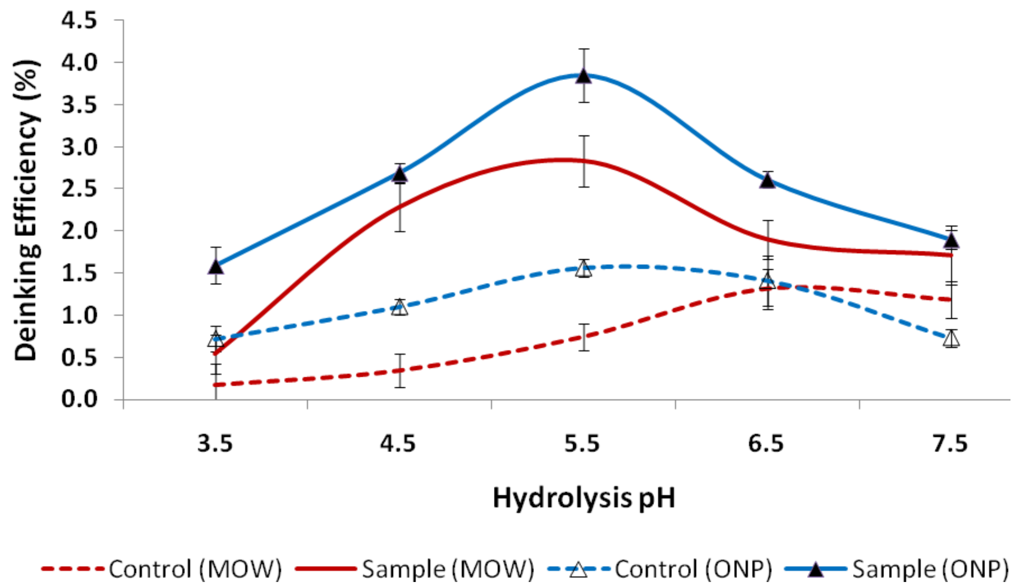
Effect of temperature

With the increase of incubation temperature the deinking efficiency obtained from enzyme treatment increased, reaching a maximum level at 50°C for both MOW and ONP (Fig. 3). Under optimum temperature, the highest deinking efficiency obtained by MOW and ONP were 2.9% and 3.7%, respectively. However, statistical analysis showed no significant differences ($P>0.05$) for deinking of MOW. The results obtained were similar to the optimum temperatures for cellulase and xylanase, which were 55°C and 50°C, respectively. Therefore, the optimum temperature for enzymatic hydrolysis of pulp was expected to be between 50°C and 55°C. Jeffries et al. (1994) performed enzymatic deinking at 55°C, which was similar to the optimum temperature of the enzyme reaction. This suggested that the optimum deinking temperature would mainly depend on the optimum temperature of the particular enzyme used in deinking.



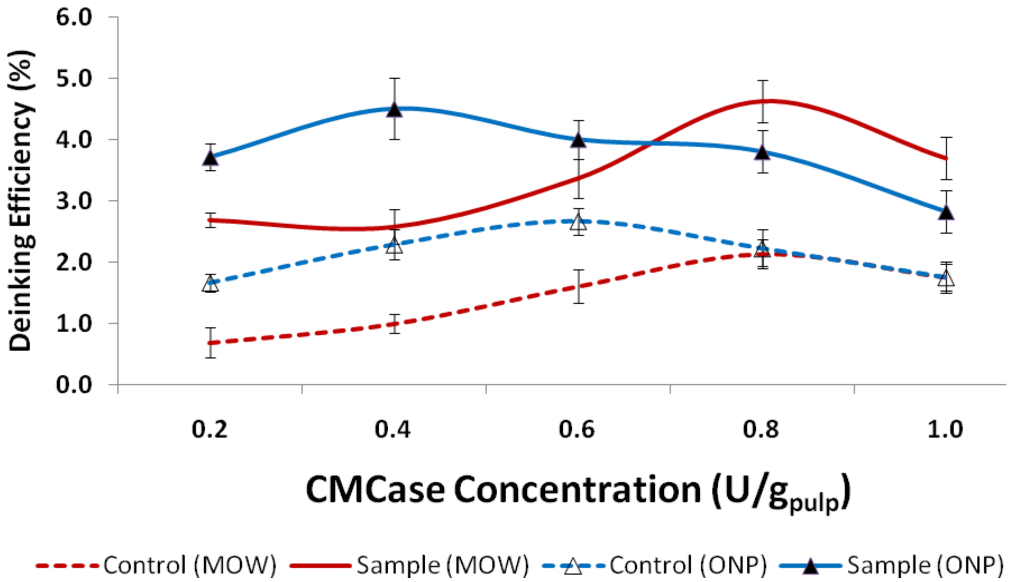
Effect of pH

MOW and ONP share a similar optimum deinking pH of 5.5. Under the optimum pH, the highest deinking efficiency of 2.8% and 3.9% were detected for MOW and ONP, respectively (Fig. 4). Statistical analysis only indicated significant differences ($P<0.05$) in deinking of ONP. In contrast, Lee et al. (2007) obtained the highest deinking efficiency at pH 3.5. The highest deinking efficiency obtained at acidic pH was probably due to the synergistic effect of acidification of the pulp slurry and enzymatic hydrolysis, which in turn led to decrease in the average ink particle sizes (Vieturs et al., 1999). Although a number of researchers reported the use of alkaline deinking processes (Prasad 1993; Franks and Munk 1995; Vyas and Lachke 2003), the present work indicated that low pH also can be efficient; similar findings were reported by Prasad et al. (1992, 1993) and Jeffries et al. (1994).



Effect of enzyme concentration

The enzyme mixture used in this study was mainly cellulase and xylanase. The highest deinking efficiencies of 4.6% and 4.5% were obtained when the enzymes concentrations of 4.0 U (xylanase) and 0.8 U (CMCase) as well as 2.0 U (xyalanse) and 0.4 U (CMCase) per gram air-dried pulp were used in deinking of MOW and ONP, respectively (Fig. 5A and 5B). However, statistical analysis only showed significant difference ($P<0.05$) in deinking of MOW. Some researchers reported that high enzyme loading led to the reduction in brightness as a result of accumulation of enzyme particles on the surfaces of the fibers (Jeffries et al. 1994), which was similar with findings in the present work. Only half of the enzyme concentration was adequate in deinking of ONP to obtain almost the same deinking efficiency of MOW. This is probably due to the fact that impact inks used in newspaper printing are easier to deink even by conventional deinking processes (Pala et al. 2004). Unlike ONP, the difficulty in deinking of MOW was due to the toner fused onto the paper fiber during the printing process (Jeffries et al. 1994, 1995; Viesturs et al. 1999). Meanwhile, previous researchers have used various enzyme concentrations in deinking of MOW and ONP (Prasad 1993; Morkbak and Zimmermann 1998; Viesturs et al. 1999; Vyas and Lachke 2003). The results obtained showed different deinking efficiency, although the enzymes used were generally cellulase and hemi-cellulases or xylanase.



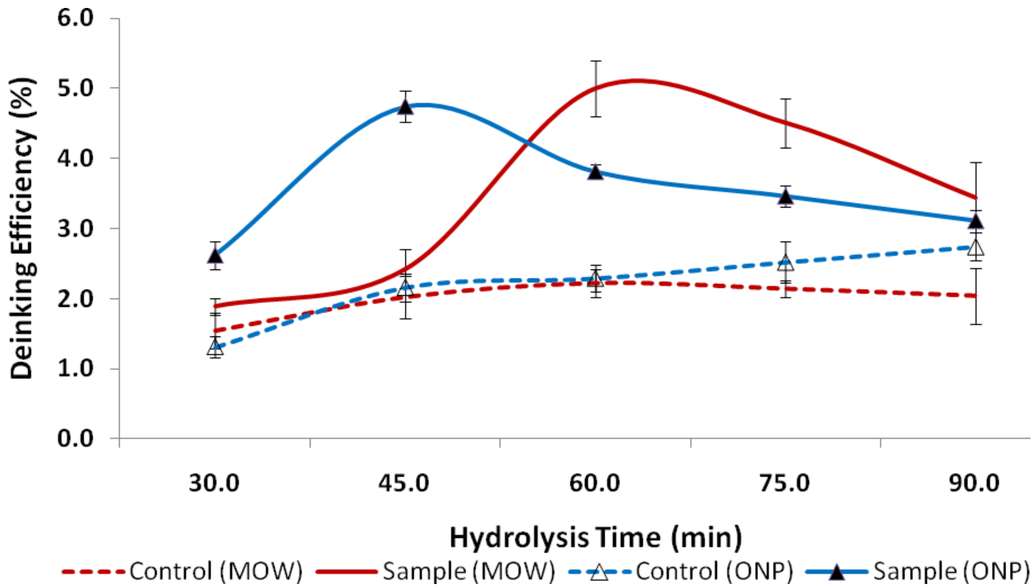
Effect of Incubation Time

The determination of the optimal enzyme hydrolysis time on pulp is very crucial, since excessive hydrolysis time may deteriorate the fiber and thus affect the paper strength and its quality. The highest deinking efficiencies of 4.7% and 5.0% were obtained when hydrolysis of ONP and MOW were carried out for 45 minutes and 60 minutes, respectively (Fig. 6). Nevertheless, prolonged hydrolysis time resulted in a slight apparent decrease in deinking efficiency, but this was not significant ($P>0.05$). This was probably due to enzymatic hydrolysis after 60 minutes incubation was adequate for maximum hydrolysis or due to the reduction in particle sizes, which were too small to be effectively removed by the flotation process (Xia et al. 1996). So far, there is no credible explanation for this reduction in the size of the ink particles (Bajpai and Bajpai 1998).

Effect of the Flotation Process

Effect of flotation pH

Under the optimum flotation pH of 8, deinking efficiencies of 5.6% and 4.9% were obtained for MOW and ONP, respectively (Fig. 7). However, the result obtained exhibited significant differences ($P<0.05$) for deinking of ONP. Previous researchers found out that an acidic deinking flotation process can improve the removal of ink and to be more compatible with the acidic enzymatic treatment of wastepaper (Prasad et al. 1992; Viesturs et al. 1999). Nevertheless, the present work showed optimum enzymatic hydrolysis with an acidic condition (pH 5.5) but optimum flotation process at alkaline pH (pH 8.0). In contrast, Prasad (1993) obtained good deinking efficiency when carrying out enzymatic hydrolysis at pH between 9.0 and 9.5 and a flotation process at pH 9.0.



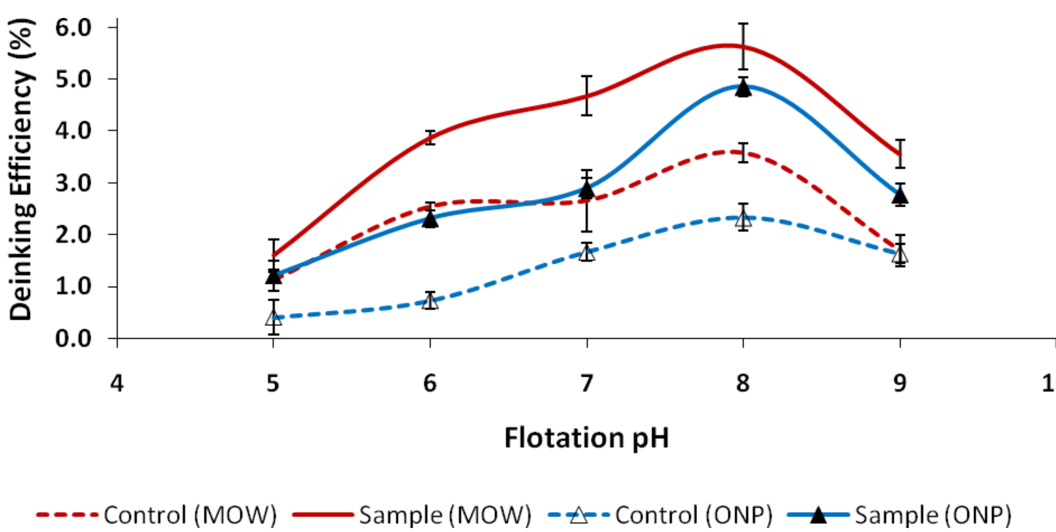
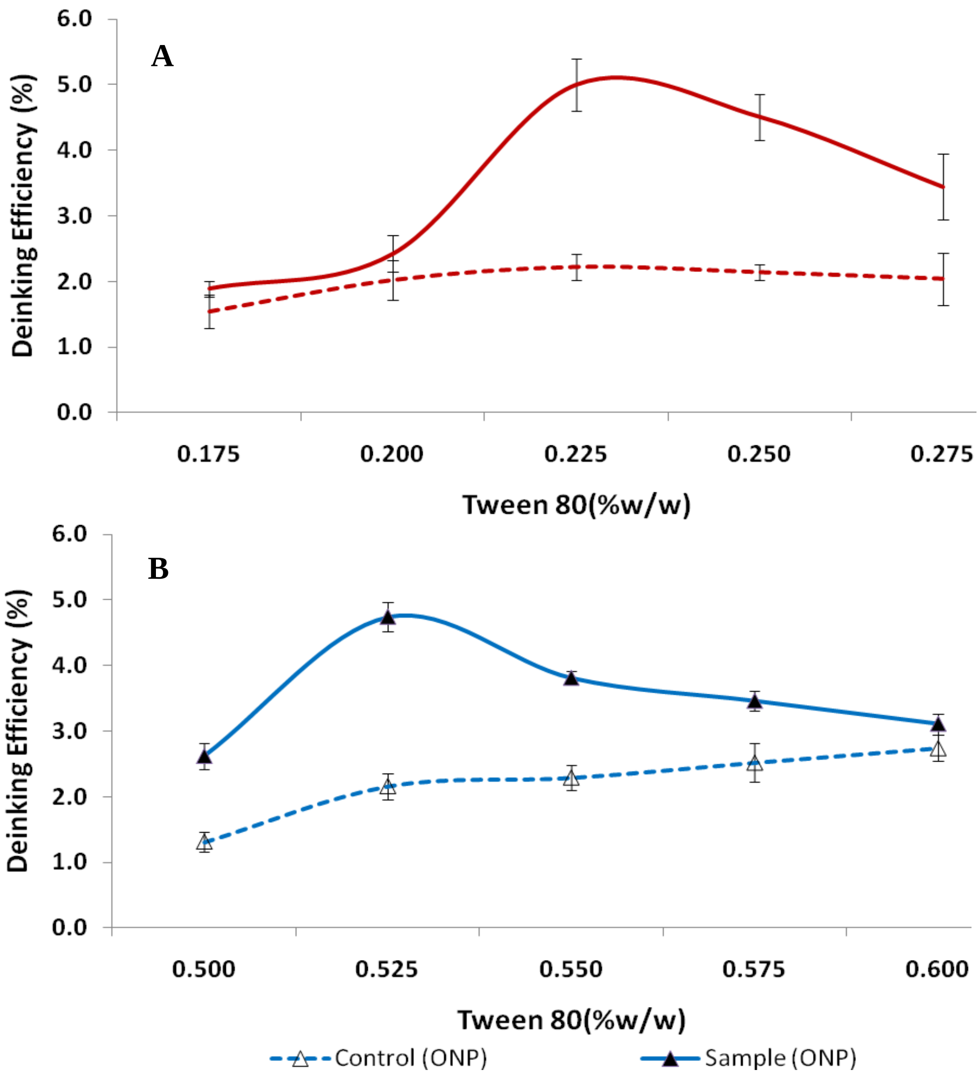


Fig. 7. Effect of flotation pH on ink removal using MOW and ONP.
Note: The flotation process was set at 280L/min of air supplied; room temperature and 0.24% consistency. Arrow bars indicate means with standard error of three replicates

Effect of surfactant concentration

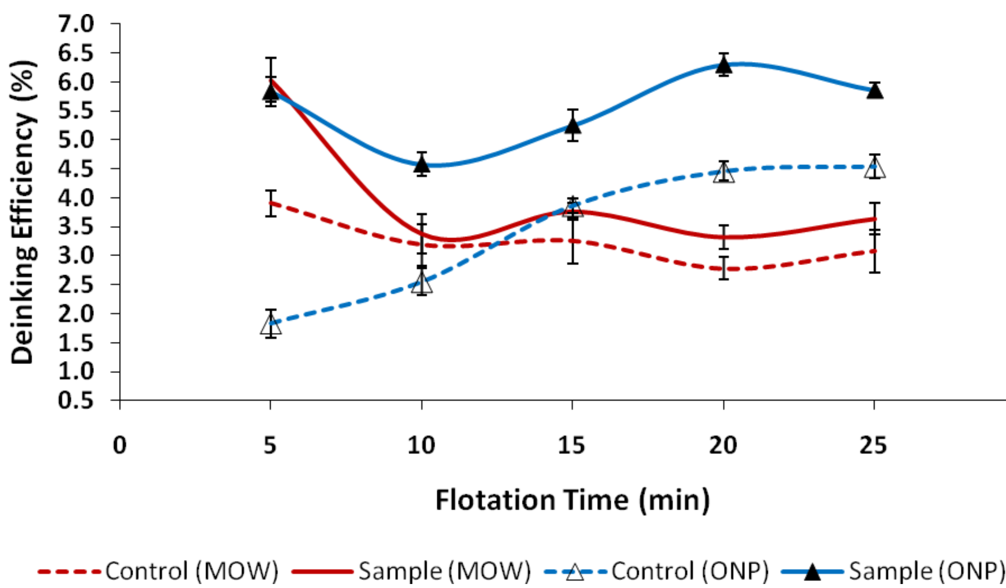
Surfactant is essential in flotation systems because it plays a role as collector of agglomerate toner particles and by altering the hydrophobicity of the agglomerates so that they can be lifted up by the air bubbles as the air flows through the fibers (Viestur et al.

1999). Under the respective optimum Tween 80 concentration of 0.200% and 0.550% (w/w), the highest deinking efficiencies of 5.8% and 6.0% were obtained during deinking of MOW and ONP. Previous researchers used different surfactant concentrations in deinking processes (Jeffries et al. 1994; Gubitz et al. 1998; Viesturs et al. 1999; Vyas and Lachke 2003). These results suggested that the concentration of surfactant required for a deinking process was dependent on the type of waste paper, surfactant and the amount of ink that were present on the waste papers. The concentration of Tween 80 used in deinking of ONP was about 3-fold higher than the concentration used in deinking of MOW. This probably due to the oil-based ink that used in printing ONP has anti-foam properties and neutralizes some of the foam generated by surfactant. Therefore, more surfactant was required in deinking ONP.



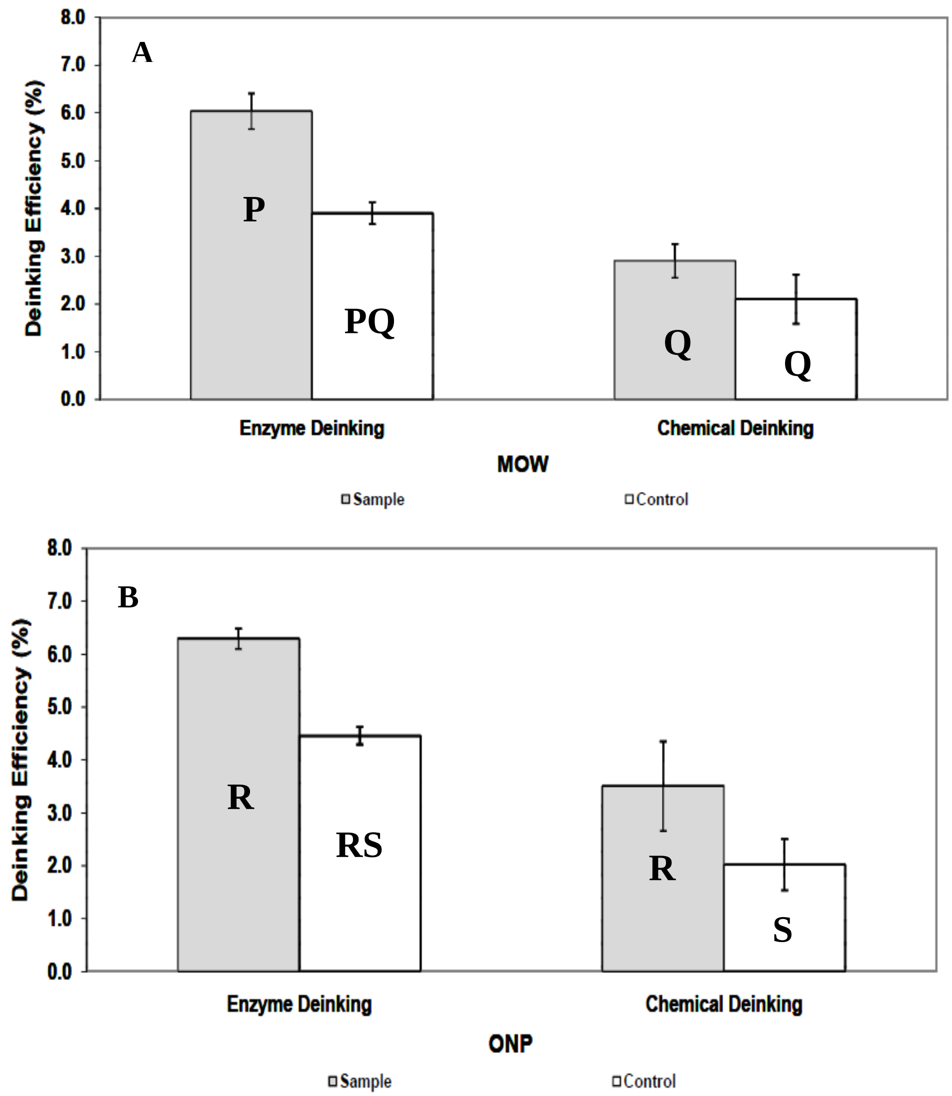
Effect of flotation time

There were no consistent patterns of deinking efficiency observed for deinking process carried out for different flotation times (Fig. 9). Deinking efficiency decreased after 5 minutes of flotation time but increased again with prolong flotation time. This suggests that ink may have re-deposited after 5 minutes of flotation time but became removed when the flotation time was prolonged. Enzymatic deinking of MOW and ONP showed quite similar ink-re-deposition profiles, with evidence of ink removal taking place between 5 to 10 minutes. The highest deinking efficiency of 6.0% and 6.3% were obtained after 5 and 20 minutes of flotation process using MOW and ONP, respectively. However, significant difference ($P<0.05$) in deinking efficiency was obtained by MOW. Under the optimized deinking condition, the highest brightness obtained by MOW and ONP pulp were 83.6% and 41.9%, respectively. In comparison, Morkbak and Zimmermann (1998) reported a brightness of 82.9% after 15 minutes of flotation time.



Comparison with Chemical Deinking Method

Enzymatic deinking is a suitable alternative to a conventional deinking process. This is because the latter requires the use of large quantities of chemicals, which are not environmentally friendly and require costly waste water treatment to meet the environmental regulations (Prasad et al. 1992). The deinking efficiency detected by enzymatic deinking was about 2-fold higher than chemical deinking process using MOW and ONP. The deinking efficiencies of 2.9% and 3.5% were obtained by the chemical deinking process using MOW and ONP, respectively. Statistical analysis showed significant difference ($P<0.05$) in deinking efficiency obtained by enzymatic deinking of MOW compared to chemical deinking process (Fig. 10A and 10B). Ink removal (control sample) in enzymatic deinking was higher compared to the chemical deinking process, which may be due to acidification of the pulp slurry. This is because the pulp hydrolysis in enzymatic deinking was performed under acidic conditions. Meanwhile, in the chemical deinking process the hydrolysis was carried out at an alkaline condition. Alkaline pH may result in accumulation of alkaline-solubilized contaminants and a yellowing effect on the recycled pulp (Morkbak et al. 1999).



It can be concluded that the optimum deinking efficiency obtained by enzymatic deinking process was primarily dependent on the types, sources, and dosage of enzymes used, as well as different type of wastepaper to be deinked. In addition, there is a good potential for enzyme to be used in the deinking process to reduce/replace the chemical used in conventional deinking process. As indicated in this work and in other publications, enzymatic deinking is able to achieve better overall results compared to a conventional chemical deinking process.

CONCLUSIONS

1. Enzymatic deinking processes for MOW and ONP have been developed and optimized.
2. The optimum conditions for the deinking of MOW and ONP were different.
3. The highest brightness obtained from enzymatic deinking of MOW and ONP were 83.6% and 41.9%, respectively.
4. Under respective optimized conditions, the highest enzymatic deinking efficiency obtained using MOW and ONP were 6.0% and 6.3%, respectively.
5. In addition, about 2-fold higher deinking efficiency was achieved for enzymatic deinking compared to the chemical deinking process.
6. The findings obtained from present work suggested that enzymes have high potential to be used in deinking of ONP and MOW. This was found using a newly developed pilot scale deinking system

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