Research Article

Enzymatic Deinking of Xerographic Waste Paper with Non-ionic Surfactant

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Received: 10 October 2019; Revised: 13 December 2019; Accepted: 24 December 2019; Published online: 31 January 2020

Abstract
Cellulase enzyme deinking of xerographic waste paper was investigated in this work. Enzyme action was assisted by using non-ionic surfactant (Triton X-100). Deinking steps were done as followed: converting xerographic waste paper to pulp, mixing pulp with enzyme and surfactant at 50°C for 30 min without pH adjustment, then removing ink from pulp slurry with flotation process. The results showed that cellulase deinking effectively removed ink from pulp slurry. That was indicated by the significantly decrease of ERIC (Effective Residual Ink Concentration) and pulp brightness improvement. Furthermore, the physical properties such as tensile index and tear index were improved with deinking that was influenced by enzyme dosage and surfactant addition.

Keywords: Deinking, Enzyme, Non-ionic surfactant, Waste paper

1 Introduction

Nowadays, Thai people consume over 4.7 million tons of paper yearly [1]. One of major usage is xerographic papers as an information media in both business and industrial sectors. These papers were produced from good quality fiber that is very suitable for paper recycling process. Generally, waste paper is contaminated by ink particles which may cause the low quality of new paper products. Consequently, Researchers paid more and more attention to develop new deinking technologies. Bio de-inking technology has one of newly choices to open up a new way for paper deinking process [2].

Deinking is the most crucial step in which it has the ability to detach ink from the fibers and produce dispersed ink paticles that can remove from subsequent ink removal process. In paper industry, two main deinking processes have been used: Filtration separation process or wash deinking and flotation deinking. Wash deinking uses screen to retain wood fibers and the unwanted ink to pass through it. The advantage of wash deinking is the ability to separate inks from other chemicals because it is a size-dependent method. However, pulp loss is quite high. In contrast, flotation deinking selectively removes hydrophobic particles using surfactant and air bubbles from solution that produces less pulp loss [3].

Loosely bound ink is removed quite easily in comparing with aged and strongly bound hydrophillic inks which can be extracted by combination of various techniques including chemical, physical and enzymatic deinking [4]. Various steps involved in the deinking of pulp are shown in Figure 1.

Please cite this article as: P. Chandranupap and P. Chandranupap, "Enzymatic deinking of xerographic waste paper with non-ionic surfactant," *Applied Science and Engineering Progress*, vol. 13, no. 2, pp. 136–145, Apr.–Jun. 2020.

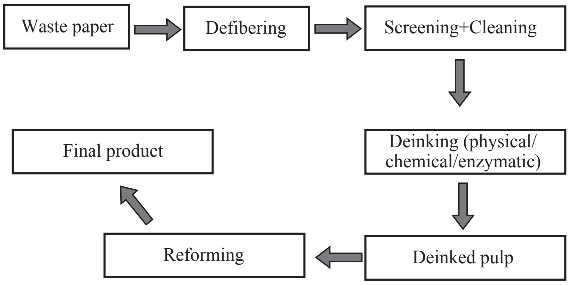


Figure 1: Basic principle of deinking process (adapted from Saxena and Chauhan [5]).

Enzymatic deinking was paid more attention from pulp and paper industries for several years because this method contributed to the avoidance of hazadous chemicals used in deinking of paper. One of enzymes that was used extensively is cellulases (from fungal and bacteria). These enzymes are components of complex species (endo-1,4-β-gluconases, exo-1-4-β-gluconases and 1-4-β-glusidases) which hydrolyse crystalline cellulose for degradation of the polymer into monomers [6]. However, mechanisms of enzymes deinking have not been compleately explained. For example, the propable mechanism for cellulase deinking is the ability to react with cellulose microfibrils. In this proposed mechanism, cellulose fibrils at the fiber surface were peeled off by cellulase and the ink particles dispersed in solution. The peeling mechanism involved in pulp freeness increases after enzymatic treatment of secondary fiber [7]. The schematic diagram of this mechanism was illustrated in Figure 2.

Surfactant plays an important role in deinking. It can reduce surface tension between ink particles and stabilize air bubbles. There are four types of surfactant depending on the hydrophilic electronic charge: non-ionic, anionic, cationic and amphiprotic surfactants. Non-ionic surfactant is likely to provide good ink detachment from fibers during deinking process. The Critical Micelle Concentration (CMC) of surfactant also play an important role in determining how much surfactant required in deinking [8].

This presented work tried to investigate xerographic wastepaper deinking in laboratory scale in order to better understanding the effect of enzyme and surfactant dosages on the ink removal and on the physical properties of the recycle pulp such as tensile index and tear index. Cellulase enzyme and non-ionic surfactant (Triton™ X-100) were used in this experiment. In order to examine the impact of deinking on the environment, Chemical Oxygen Demand (COD) of the deinking effluents were also investigated in this work.

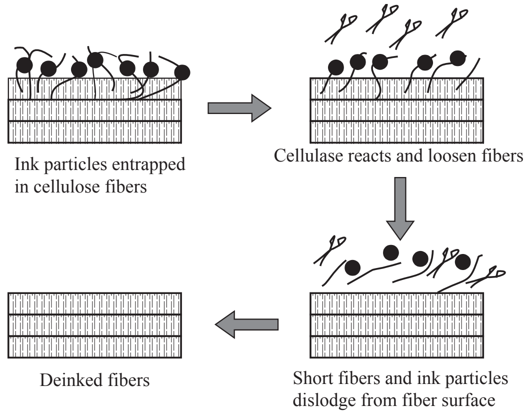


Figure 2: Schematic diagram for cellulase deinking.

2 Materials and Methods

2.1 *Preparation of paper pulp*

The commercial papers (A4-type, 80 g/m^2) were printed with toner ink from xerographic machine on one side of each sheet with 50% coverage. Papers were cut to pieces by paper shredder machine and soaked overnight in water at room temperature. The papers were then disintegrated into pulp slurry at 5% consistency. As a controlled run, small portion of the un-deinked pulp slurry was made to handsheet of 60 g/m^2. Brightness and Effective Residual Ink Concentartion (ERIC) measurements were done as reference values.

2.2 *Experimental design*

Full factorial design was utilized to study the effects that experimental variables can have on a response.

Two deinking variables were identified and investigated by full factorial design. These variables were: 1) amount of enzyme (A), 2) amount of surfactant (B), expressed as percentage of oven-dried pulp (% o.d. pulp). All deinking process combinations were concluded in Table 1.

The values of obtained responses allow the calculation of Analysis of varience (ANOVA) at significant level of 0.05 in order to determine the significant of each variable on pulp properties. The trial version of statistical software, Minitab® v.18. was used in this work.

Table 1: Deinking process variables

Run no	Enzyme (% o.d. pulp)	Surfactent (% o.d. pulp)	Run no	Enzyme (% o.d. pulp)	Surfactent (% o.d. pulp)
1	0	0	11	0.1	0
2	0	0.1	12	0.1	0.1
3	0	0.5	13	0.1	0.5
4	0	0.9	14	0.1	0.9
5	0	1.2	15	0.1	1.2
6	0.05	0	16	0.15	0
7	0.05	0.1	17	0.15	0.1
8	0.05	0.5	18	0.15	0.5
9	0.05	0.9	19	0.15	0.9
10	0.05	1.2	20	0.15	1.2

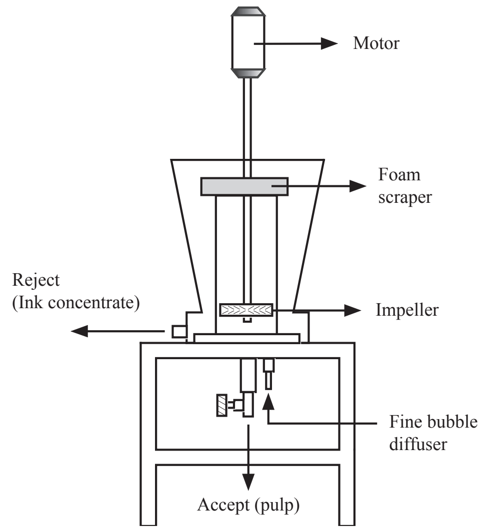


Figure 3: Laboratory-scaled flotation cell.

2.3 *Enzyme treatment*

Cellulase enzyme for deinking process was supplied by Sigma-Aldrich (25 KU, C-1184 SIGMA). This enzyme was kept in powder form at 4°C prior to use. The enzyme solution was prepared by dissolving 1 g solid enzyme in 100 mL of distilled water. Enzyme dosages were varied in the range of 0–0.15% (base on oven-dried (o.d.) pulp). Non-ionic surfactant (Triton X-100) was selected to assist deinking, employing 0–1.2% (o.d. pulp). These values covered the CMC of surfactant (1% wt. on o.d. pulp [9]). Enzyme reaction progressed under continuous slow mixing at 50°C for 30 min. All enzyme treatments were done at neutral pH. After treatment, the pulp slurry was heated at 80°C for 10 min in order to de-activate the enzyme activity. In order to reduce environmental impact, deinking processes was done without adding any chemical agents for pH adjustment.

2.4 *Flotation deinking*

Flotation deinking experiments were done in a 5-l custom-made laboratory flotation cell (Figure 3). The slurries from enzyme treatment were diluted in tap water to 0.5% consistency. The prepared pulp slurries were then passed through flotation cell and the air flotation operation was done at 5 L/min for 10 min at room temperature. Deinked pulps were collected to determined pulp loss. Freeness of deinked pulps were determined by TAPPI standard T 227 om-04.

2.5 *Handsheets preparation and testing*

Paper handsheets from deinked pulps were made according to TAPPI standard T205 sp-95. Deinking efficiency was expressed on the ERIC and brightness of the paper. The paper brightness (% ISO) was determined by TAPPI standard T452 om-98 and the paper ERIC (ppm) was determined by TAPPI standard T567 om-04. Both measurements were done by using brightness and ERIC tester (Color-Touch® PC, Technidyne Corporation). Tensile index of the deinked papers was determined based on the TAPPI standard T494 om-01 (Stograph E-S, Toyoseiki SHO), while tear index was determined based on the internal resistance of the handsheet using the Electronic tearing tester (Model Protear, Thwing-Albert, Thwine-Albert Instrument) as described in TAPPI standard T414 om-98.

2.6 *Chemical oxygen demand measurement*

Waste water from flotation processes were collected for COD measurement by using spectroscopic method that was described elsewhere [10].

3 Results and Discussion

3.1 *Statistical analysis of deinking*

Analysis of varience (ANOVA) at significant level of 0.05 was tested in order to determine the significance of each experimental variables (enzyme and surfactant amounts) on pulp properties in full factorial design. The results are brief summarized in Table 2. It is important to note that *p*-value range from 0 to 1 is a probability that measures the evidence against the null hypothesis. If *p*-vaule is less than or equal to the chosen significant value (0.05), the test suggested that observed variables (pulp properties) are inconsistent with the null hypothesis, so the null hypothesis is rejected and experimental variables had influences on properties of deinked pulp. The R^2 values that obtained from Minitab® calculation indicated that the experimental variables had a strong relation to observed variables. The influences of experimental variables on each pulp property were discussed in many parts of this section.

Table 2: ANOVA results of deinking variables on deinked pulp

Properties of Deinked Pulp	Variables' *p*-value			R^2
	Enzyme (A) (% o.d.)	Surfactant (B) (% o.d.)	Interaction Term (A*B)	
Pulp loss	0.001	<0.001	0.001	0.941
ERIC	<0.001	<0.001	<0.001	0.999
Brightness	<0.001	<0.001	<0.001	0.985
Freeness	<0.001	<0.001	<0.001	0.997
Tensile index	<0.001	<0.001	<0.001	0.941
Tear index	<0.001	<0.001	0.043	0.965

3.2 *Pulp loss*

Within the limit of the study the pulp loss ranged from about 20% to 53% (Figure 4). It can be seen that amount of surfactant has greatly effect on pulp loss. This is due to the ability of surfactant to act as a collector that can adsorbs on pulp. This makes pulp more hydrophobic and resulting in high fiber loss [11] that is clearly seen in the plot of sole surfactant effect on pulp loss in Figure 5(b). Moreover, the increase of surfactant concentration beyond 0.9% dramatically increased pulp loss due to the incapability to hold froth of such a small flotation cell. However, enzyme pretreatment commonly increased hydrophilicity of fiber via hydrolysis that increase fiber-water interaction. This evidence was shown in sole effect of enzyme on Figure 5(a) and interaction effect between surfactant and enzyme in Figure 5(b). This combination resulted

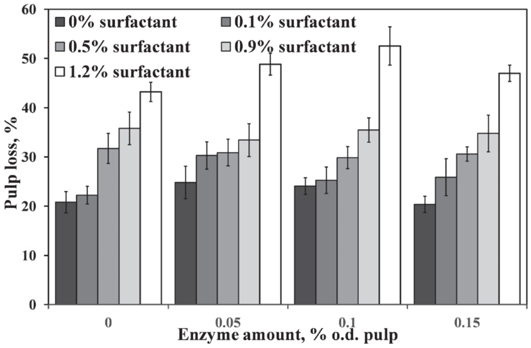
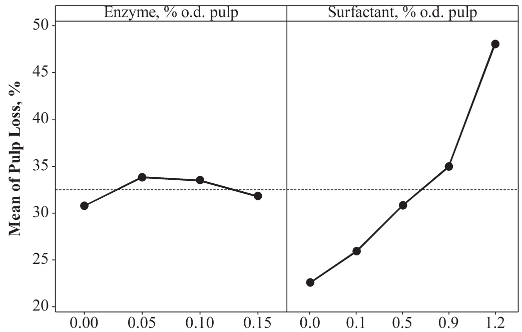
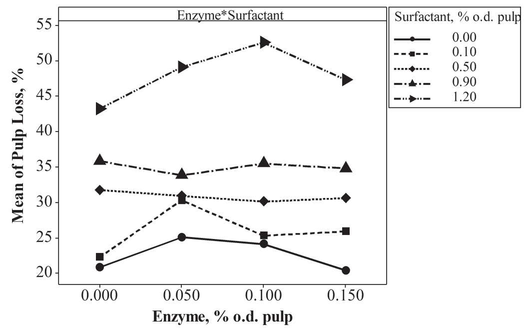


Figure 4: Pulp loss of deinked pulps at various conditions.



(a)



(b)

Figure 5: (a) Main effect plot and (b) interaction effect plot of experimental variables on pulp loss.

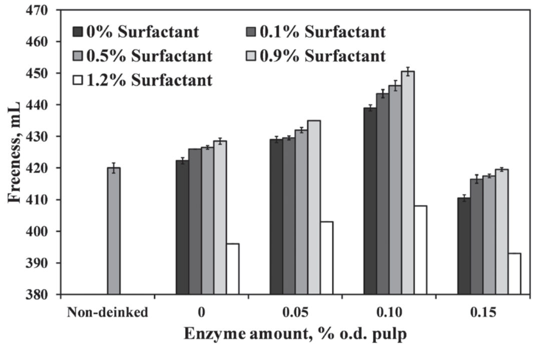


Figure 6: Freeness of deinked pulps at various conditions.

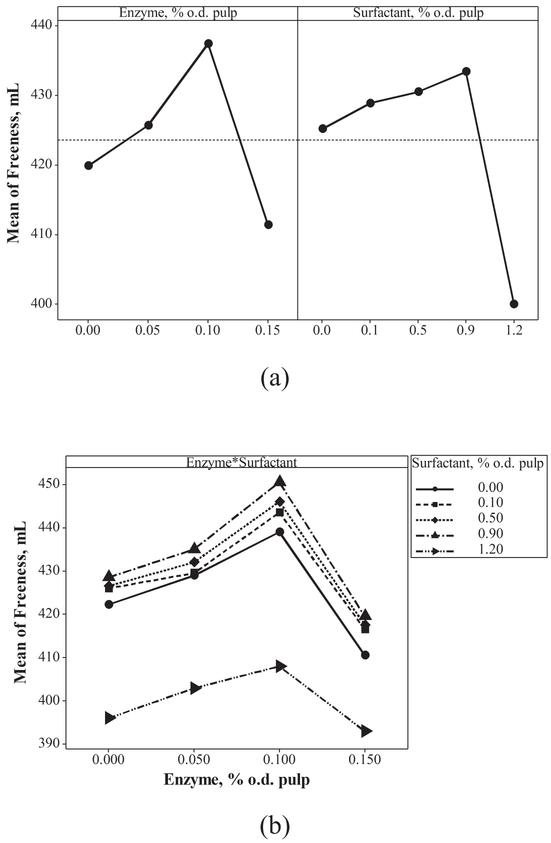


Figure 7: (a) Main effect plot and (b) interaction effect plot of experimental variables on freeness.

in the reduction of pulp loss at high surfactant and enzyme amounts.

3.3 *Freeness*

Freeness is a measure of how quickly water is able to drain from a fiber furnish sample. This property directly related to specific surface area of pulp [12]. The results showed that maximum freeness of the deinked pulp is 450.5 mL at 0.1% enzyme in conjunction with 0.9% surfactant (on o.d. pulp) (Figure 6). This result was clearly seen in main and interaction plot for freeness in Figure 7. Freeness of the pulp increased when the dosage of enzyme and surfactant concentration increased. It can be explained that cellulase eliminated small microfibrils from fiber and this reduced fiber's specific surface area. However, freeness significantly decreased when enzyme concentration exceeded 0.15%. This may be due to excessive degradation of fiber that produced more branches on fiber surface that can entangle each others. This phenomenon increased water retention of fibers and reduced freeness. Moreover, if surfactant percentage exceeds CMC (greater than 1.0%), freeness significantly decreased. This may be due to large amount of suspended ink that might blocked water drainage. This result was the same as increasing of ERIC value when surfactant amount was above CMC.

3.4 *Effective residual ink concentration*

ERIC method evaluates the residual ink by measuring the absorbed light in the infrared range as 950 nm because ink and only ink absorbs light at that wavelength.

The results from ERIC measurement (Figure 8) showed that ERIC greatly decreased whereas dosage of enzyme and surfactant concentration increased. ERIC was reduced as much as 91% (350 ppm of non-deinked pulp to 35 ppm at 0.1% enzyme and 0.9% surfactant of o.d. pulp). This is due to the interaction of cellulase with fibers that caused better detachment of ink particles from fibers. Moreover, the flotation process can improve the deinking efficiency because surfactant molecules can bind with the ink particles and accumulate ink particles to the suitable size for flotation (10–100 µm) [11]. The main effect and interaction effect plot from Minitab analysis [Figure 9(a) and (b)] also revealed the ERIC value at the condition beyond surfactant's CMC. At surfactant amount of 1.2%, ERIC slightly increased due to forming of surfactant's micelle that reduced ability to bind with ink particles.

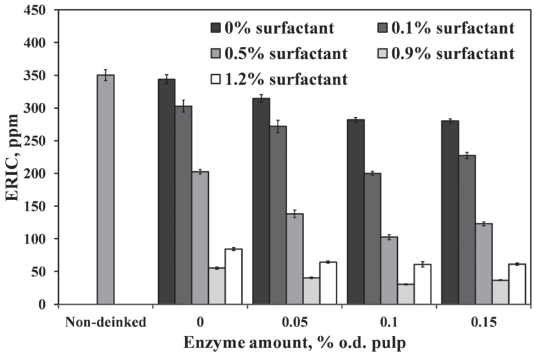


Figure 8: ERIC of deinked pulps at various conditions.

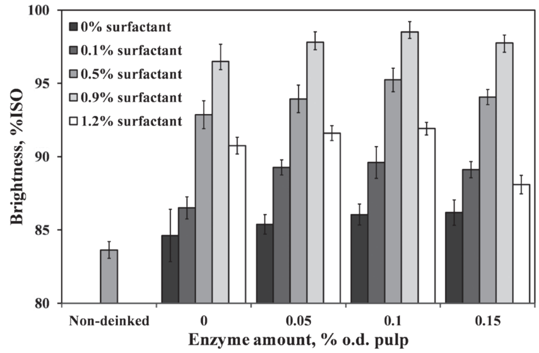
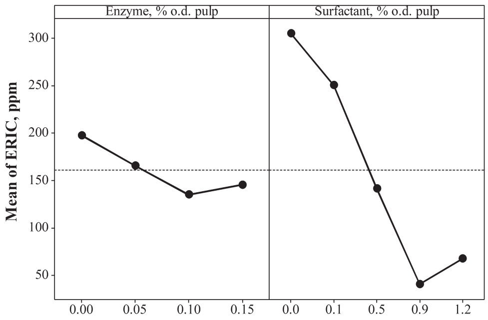
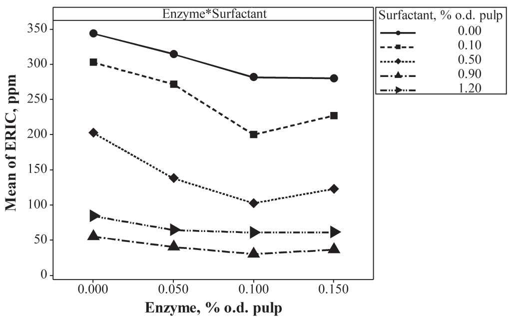


Figure 10: Brightness of deinked pulps at various conditions.

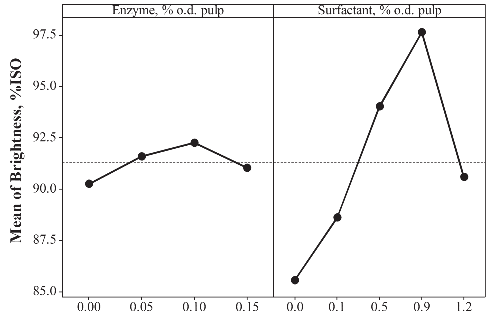


(a)



(b)

Figure 9: (a) Main effect plot and (b) interaction effect plot of experimental variables on ERIC.



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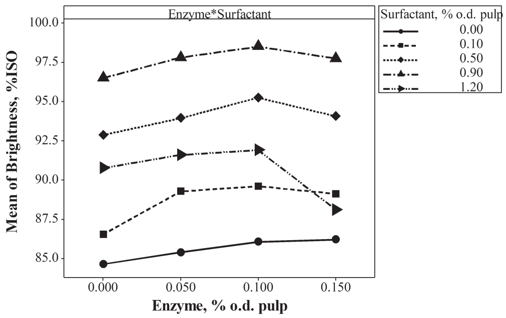

(b)

Figure 11: (a) Main effect plot and (b) interaction effect plot of experimental variables on brightness.

3.5 *ISO brightness*

ISO brightness is the reflectance of blue light at an effective wavelength of 457 nm [13]. This value is commonly investigated along with ERIC in order to determine the effectiveness of deinking process. The results in Figure 10 and effect plots in Figure 11 revealed that brightness increased while amount of cellulase and surfactant increased. The maximum value of brightness was 98.5% ISO at 0.1% enzyme in conjunction with 0.9% surfactant. Similar to ERIC, brightness decreased when surfactant concentration was above CMC. This due to the forming of surfactant micelle that reduced ability to bind ink particles.

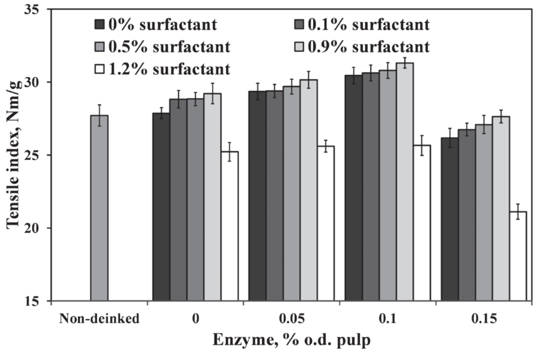

Figure 12: Tensile index of deinked pulps at various conditions.

3.6 *Tensile index*

Tensile index is the value of tensile strength in N/m divide by grammage (g/m^2) of paper. Tensile index usually reflects fiber bonding ability in paper. It was clearly seen that tensile index improved as much as 13% with certain combination of cellulase and surfactant (at 0.1% enzyme and 0.9% surfactant) (Figure 12). This came from the elimination of short fiber by enzyme treatment and left more long fibers that had high bonding ability. Furthermore, the increase of surfactant also eliminated more short fibers and enhanced fiber bonding. However, at cellulase concentration more than 1.0% and surfactant concentration was exceed CMC, tensile index significantly decreased [Figure 13(a) and (b)]. This evidence may caused by the large amount of long fiber's degradation by enzyme overdosage. In addition, the surfactant amount of 1.2% overloaded the ability of flotation cell to operate and led to the loss of fiber as discussed earlier.

3.7 *Tear index*

Tearing strength refers to the ability of the paper to resist the application of tearing forces measured in millinewton (mN). The tear index is calculated as the tearing strength divided by the grammage. Fiber length is a key influence on tear index. The results of tear index at various enzyme and surfactantn amounts in Figure 14. It is clearly seen that tear index increased with the amount of enzyme increased. The deinking improved the tear index of non-deinked paper as high as 15% (at 0.1% enzyme and 0.9% surfactant). This is due to enzyme treatment that eliminate short fibers and left more long fibers. Increasing of surfactant amount also helped to remove short fibers from deinking process. However, Minitab analysis in Figure 15(a) and (b) clearly showed that tear index dropped dramatically when enzyme amounts reached 0.15% of o.d. pulp.

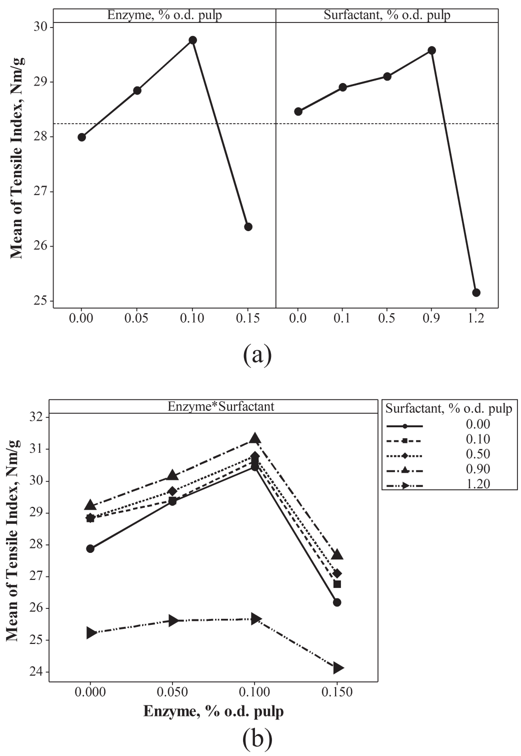

Figure 13: (a) Main effect plot and (b) interaction effect plot of experimental variables on tensile index.

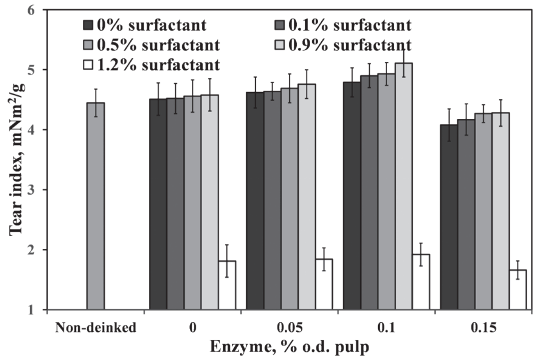

Figure 14: Tear index of deinked pulps at various conditions.

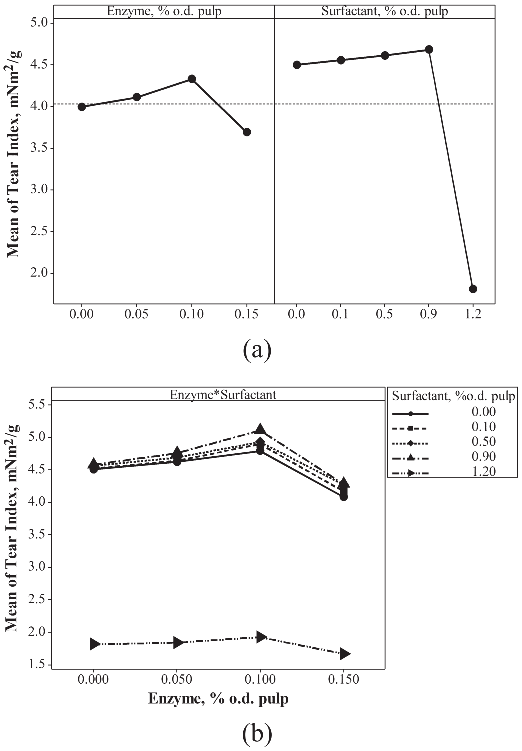

Figure 15: Main effect plot (a) and interaction effect plot (b) of experimental variables on tear index.

Adding surfactant exceeded CMC in this small flotation cell greatly decreased tear index. (from around 4 to the average of 2 Nm^2/g). This indicated not only short fiber loss but also long fiber loss due to surfactant overdosage. Nevertheless, there were a number of reports that have investigated the effect of enzymes on the tear index. However, the effects on tear index have both positive and negative effects [14], [15]. Therefore, different enzyme preparations and fiber compositions are likely responsible for the difference in the tear effect of deinked pulp [16].

For all investigations, it was noticed that at the condition of 0.15% enzyme and 1.20% surfactant provided different results from the rest. This was recognized as enzyme and surfactant overdosage for small lab-scale flotation cell.

3.8 *COD analysis*

Chemical oxygen demand (COD) is the total amount of oxidisable organics (biodegradable and nonbio-

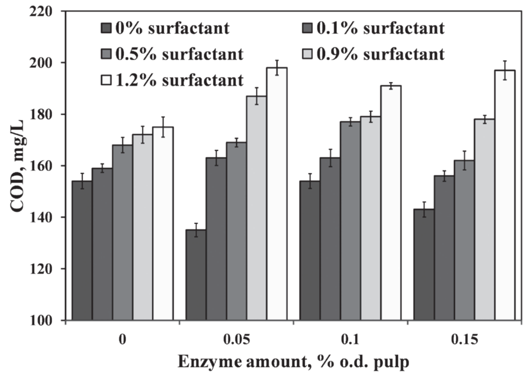

Figure 16: COD of deinked pulps effluents at various conditions.

degradable and both dissolved and particulate), measured by the amount of oxygen in the form of oxidising agent required for the oxidation of organic matters [17]. In Thailand, COD for wastewater that can be released to the environment is under 120 mg/L but not exceed 400 mg/L [18]. Figure 16 showed the COD analysis from various conditions of cellulase deinking. In this experiment, The influence of cellulase was quite unpredictable. This may be due to the complex structure of cellulase that is hard to determine the oxidation reaction . In contrast, amount of surfactant had a clear influence on COD value. This is due to the organic structure of Triton X-100 that can be oxidized in COD analysis. Furthermore, these COD values were still higher than the COD requirement. Water treatment of deinking effluent was still necessary. However, enzymatic deinking still released effluent that has COD value of 20–30% lower than conventional chemical deinking [19].

3.9 *Enzymatic deinking versus chemical deinking*

In order to compare the efficiency of enzymatic deinking with traditional chemical treatment, some pulp properties were compared with chemical deinking that used mixture of naphthalene and amyl acetate (20:80 weight ratio) as a deinking agent. Ink particles from chemical treated pulp samples were then removed with flotation [9] and washing [20] deinking process. All results were compared at the same experimental condition, i.e. 0.5% of pulp consistency, 0.9% of Triton X-100 and 10 min. of surfactant contact time. The results were presented in Table 3.

Table 3: Comparison of pulp properties among enzymatic deinking and chemical deinking

Properties	This Work (Enzyme)	Chemical Treatment	
		Washing [20]	Flotation [9]
Pulp loss, %	35.5	Not report	69.8
Brightness, %ISO	98.50	78.84	84.86
ERIC, ppm	30.35	169.56	29.9
Tensile index, Nm/g	31.31	0.46	0.58
Tear index, mNm^2/g	5.11	6.53	8.1

It is clearly seen that enzymatic deinking had less pulp loss than chemical flotation deinking due to enzyme interaction on fibre surface that increase hydrophilicity that can retain pulp in flotation cell. Washing deinking is quite ineffective in deinking at this condition (high ERIC, low brightness). Tensile index from enzymatic deinking is much higher than chemical deinking. This indicated better fiber bonding in the sample that resulted from enzyme interaction on fiber's surface. This result indicates the possibility to use this enzyme-treated pulp as a reinforcement material in green composite. Nevertheless, tear index is lower than pulp from chemical deinking. This result clearly indicated the shortening of fiber length from enzyme treatment that did not happen on chemical treatment.

4 Conclusions

The experimental results lead to the following conclusions: Both cellulase and Triton X-100 have important roles to effectiveness of deinking process. It can be seen from the decrease of ERIC and the increase of brightness. Physical properties improved with the increase of enzyme and surfactant dosage. However, surfactant dosage beyond CMC significantly reduced several properties of pulps including tensile and tear index due to excessive degradation of cellulose fibers and flotation cell overloading. Enzyme-treated deinked pulp at certain condition has some advantages such as brightness, ERIC, and tensile strength over chemical-treated deinked pulp.

Acknowledgments

The authors are grateful for the support of King Mongkut's University of Technology North Bangkok (University Grant number KMUTNB-GEN-55-23, budget year of 2011). The authors also thank Department of Chemical Engineering, Department of Industrial Chemistry, King Mongkut's University of Technology North Bangkok and Department of Imaging and Printing Technology, Faculty of Science, Chulalongkorn University for supporting research equipment and properties analysis of paper handsheets.

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