

Effects of Sequencing Enzyme Application and Refining on DIP Properties Produced From Mixed Office Waste Paper

Mohammad-Ali Hossein,^a Mohammad Talaeipour,^{b,*} Amir-Hooman Hemmasi,^b Behzad Bazyar,^b and Saeed Mahdavi Feyz-abadi^c

The effect of changing the sequence of refining and enzymatic treatment on the properties of deinked pulp from mixed office waste paper (MOW) was investigated. The sequences included refining before and after enzymatic treatment. Refining was applied for 700 and 1500 revolutions, and the consistency of pulps during enzymatic treatment was 5, 8, and 13%. Enzymatic treatment was applied for 20, 40, and 60 min. After each of the sequences, the deinking stage was the same. When refining was applied after the enzymatic treatment, the freeness of pulp was greater than that of the pulp on which refining was conducted before the enzymatic treatment, at a constant refining speed. Better strength properties were produced when refining was carried out before the enzymatic treatment. Also, the results of testing the optical properties of deinked pulp showed that brightness and ERIC improved when refining was carried out before the enzymatic treatment.

Keywords: Cellulase; Refining; Deinking; ERIC; MOW; Optical properties

Contact Information: a: Department of Wood and Paper Science and Technology, Science and Research Branch, Islamic Azad University, Tehran, Iran; b: Department of Wood and Paper Science and Technology, Science and Research Branch, Islamic Azad University, Tehran, Iran; c: Wood & Paper Science Division, Research Institute of Forests and Rangelands, Iran; * Corresponding author: m.talaeipour@srbiau.ac.ir

INTRODUCTION

Population growth and technological progress, along with an ever-increasing demand for paper on the one hand and significant deforestation and environmental issues on the other hand, necessitate the review and implementation of new and innovative approaches to improve the current processes in the pulp and paper industry.

Refining is a process in which, by applying both mechanical and hydraulic energies, the physical and mechanical properties of pulp are changed. As a result, fibers are influenced by several factors, such as fibrillation, fines production, and fiber shortening as the most pronounced outcomes. External fibrillation involves the separation of fibrils from the fiber surface, which consequently increases the surface area. Internal fibrillation causes the separation of the inner layers of the cell wall, which improves the flexibility of the fibers (Bhardwaj *et al.* 2007; Ny and Messmer 2007; Talaeipour 2008). In addition to the positive effects, the refining process also comes with some undesirable outcomes, including fiber shortening as a result of shear forces and an increase in the amount of fines in the pulp, which causes freeness reduction and difficulties in the dewatering of pulp.

Today a large variety of enzymes are used in different sectors of the pulp and paper industry. Bajpai (2010), mentioned that there have been a wide variety of enzymes in use for debarking, pitch control, bio-pulping, bio-bleaching, refining, deinking, sticky control,

and waste water treatment. However, changes in recycled fiber characteristics have been the main subject of much research in recent years (Dourado *et al.* 1999; Pala *et al.* 2001; Dienes *et al.* 2004; Dienes 2006; Znidarsie-Plazl *et al.* 2009; Mayeli and Talaeipour 2010; Lee *et al.* 2011; Ibarra *et al.* 2012). The combination of enzymes with the refining process is one of the major applications of enzymes. The enzymes bring about an improvement in the dewatering of pulp. Accordingly, the other studies also support that enzymatic treatment increases the freeness, and, when treated pulps were compared with control pulps, the time to reach the specified freeness was reduced in treated pulps by refining (Eriksson *et al.* 1998; Garcia *et al.* 2002; Kim *et al.* 2006; Gil *et al.* 2009; Loosvelt 2009; Bajpai 2010; Maximino *et al.* 2010; Torres *et al.* 2012; Zhang *et al.* 2013; Cui *et al.* 2015). Bajpai (2010) and some other scientists addressed the hydrolysis of fines as the reason for the drainage improvement, believing that fines have a high surface area that induces negative effects on the drainage and strength properties of paper. In the enzymatic treatment, fines are hydrolyzed, which later helps to improve the drainage and strength properties of paper. Moreover, the presence of enzymes results in fiber shortening and fibrillation (Znidarsie-Plazl *et al.* 2009; Zhang *et al.* 2013; Cui *et al.* 2015).

The above researchers mentioned that by applying enzymatic treatment before refining the drainage will be improved, though almost all of the paper strength and other properties will be decreased. In the above mentioned studies, the authors believed that enzymes had an effect on fiber shortening and fiber fibrillation, and it was assumed that by applying the refining process after the enzymatic treatment these effects may become intensified and influence the quality of the final paper. Hence, in the present research, the refining process was conducted both before and after enzymatic treatment, and the corresponding results were compared for these two approaches in terms of drainage and optical and mechanical properties of the deinked pulp.

EXPERIMENTAL

The raw material in this study was prepared by Latif Papermaking Company, a producer of tissue in Iran, out of 70% waste office paper and 30% book paper. Pulp samples were produced by soaking for 20 min, followed by repulping performed under constant conditions: a time of 30 min, consistency of 10%, and temperature of 40 °C.

Produced pulp was washed on 80-mesh and 400-mesh screens and divided into two fractions: a long fiber fraction (the fibers which remained on the 80-mesh screen) and a short fiber fraction (the fibers which passed through the 80-mesh screen and remained on the 400-mesh screen). There was also a mixed fraction of pulp that was not washed on the screens. The long fiber fraction was selected for experiments and treated via two methods: an enzymatic treatment with cellulase, then refining before deinking (ERD method), and refining, then an enzymatic treatment with cellulase before deinking (RED method).

Enzymatic Treatment

Commercial Biotouch[®] C35 was obtained from AB-Enzymes, Darmstadt, Germany. The cellulase enzyme was derived from *Trichoderma reesei*; its main activity is endo-1,4-β-D-glucanase. The enzyme activity and treatment temperature were selected as 30 U/g and 60°C, respectively. The temperature selection was according to the enzyme specifications provided by the supplier. For enzyme activity, since various dosage of cellulase enzyme from 1 U/g to 600 U/g have been used by different scientists (Pala *et al.*

2001; Park *et al.* 2006; Das *et al.* 2013; Efrati *et al.* 2013; Ebner *et al.* 2014; Patil and Dhake 2014), as well as the dosage recommended by the supplier, the authors decided to consider a mean amount of 30 U/g and since this dosage level was identical for all the treatments, it was considered that its effects can be consistently compared. The treatment time were 20, 40, and 60 minutes. The consistencies of treated pulps were 5, 8, and 13%. The treatment pH was adjusted to 5.8, according to the data sheet guidelines (a pH of 5 to 6.5), by 0.25% sulfuric acid.

Refining

Refining was conducted by a PFI mill “Model VI- No 513, Manufactured by Hamar Company, Norway” before and after the enzymatic treatment. Refining conditions were a gap of 0.2 mm, 30 g of pulp (oven dry basis), a consistency of 10%, and speeds of 700 and 1500 revolutions.

Deinking

Deinking was performed on all of the pulps, whether refining was performed before or after the enzymatic treatment. Deinking was performed in a standard laboratory flotation cell, produced by Fan-Azma Industrial Equipment Manufacturing Co, Tehran, Iran, for 10 min at 1% consistency, using 0.5% nonylphenoethoxylate (based on oven-dried weight of pulp) as the surfactant. The process temperature was kept constant at 40 °C. Froth was scraped off manually, and flotation accepts were washed on a 400-mesh screen.

Handsheets

Handsheets were made according to TAPPI standard T 205 (1995) for the evaluation of mechanical strengths, optical properties, and effective residual ink concentration (ERIC) measurement.

Several TAPPI test methods were used for pulp and paper analysis: freeness, TAPPI standard T 227 (1999); tear strength, TAPPI standard T 414 (2004); tensile strength, TAPPI standard T 494 (2001); burst strength, TAPPI standard T 403 (2002); opacity, TAPPI standard T 425 (2006); brightness, TAPPI standard T 452 (2008); and the Kubelka-Munk theory to evaluate the ERIC.

Statistical analysis was used to determine the effect of treatment conditions on the pulp and paper properties using multivariate SPSS analysis. In cases where differences between the treatment averages were observed, Duncan multi-comparison tests were used for ranking the averages.

RESULTS AND DISCUSSION

Freeness and Drainage

The average initial freeness of the pulp was 620 mL before any treatment. The procedures of the ERD and RED methods are presented below.

ERD method

After the enzymatic treatment using the ERD method, the freeness was determined. The effect of consistency in the enzymatic treatment on the freeness of pulp was significant.

Figure 1 depicts the freeness variation at different consistencies. The results shown in this figure are consistent with the results of other studies (Kim *et al.* 2006; Loosvelt

2009; Znidarsie-Plazlet *et al.* 2009; Bajpai 2010; Torres *et al.* 2012; Zhang *et al.* 2013; Cui *et al.* 2015). This Figure shows a similar downward trend at 5% and 8% consistency for different treatment times. The highest freeness of pulp was achieved at 13% consistency. At this consistency, a sharp decline occurred, but, as time increased, the drainage improved.

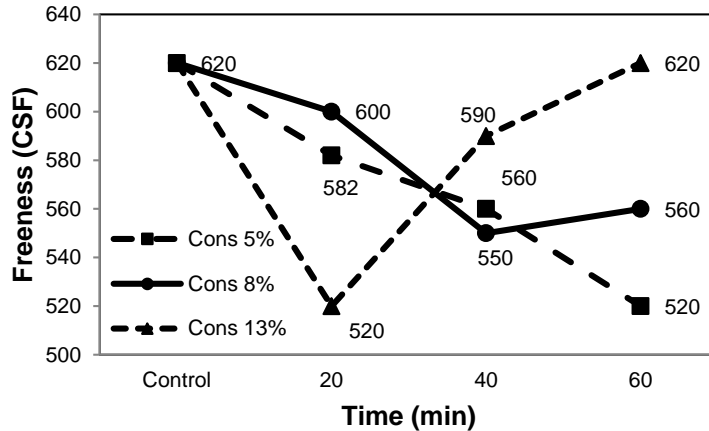


Fig. 1. Freeness variation at different consistencies and times using the ERD method

After the enzymatic treatment, the refining stage was implemented, and the results are shown in Fig. 2.

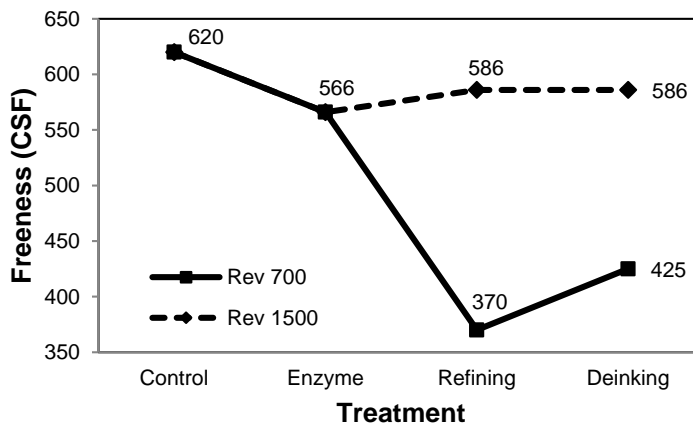


Fig. 2. The total trend of treatments using the ERD method on the freeness of treated pulps

In Fig. 2, a sharp decline was observed at 700 revolutions of refining that may be due to the fiber fibrillation. It seems that the enzyme treatment caused a severe fibrillation of the fiber surface. At 1500 revolutions of refining, an unusual pattern was observed: by refining, the degree of freeness increased. It seemed that the influence of the enzyme weakened the fiber cell wall, leading to fiber shortening (Znidarsie-Plazl *et al.* 2009) at higher revolutions. Thus, more fines were produced, and these passed through the freeness tester mesh (Fig. 3).

Using the ERD method, the deinking stage was implemented after refining, and, at 700 revolutions of refining, the freeness increased slightly. This result could be due to the removal of fiber fines from the pulp at this stage. There was no difference in freeness after the deinking of pulp refined at 1500 revolutions.



Fig. 3. Drained water containing fines and shortened fibers (a) using the ERD method, along with no fines (b) using the RED method

RED method

There was an opposite trend for the RED method: by refining, the freeness decreased (Fig. 4). The next stage in this method was the enzymatic treatment, which also decreased the freeness, possibly due to fiber fibrillation, as mentioned above. The effect of consistency and time on freeness was also significant.

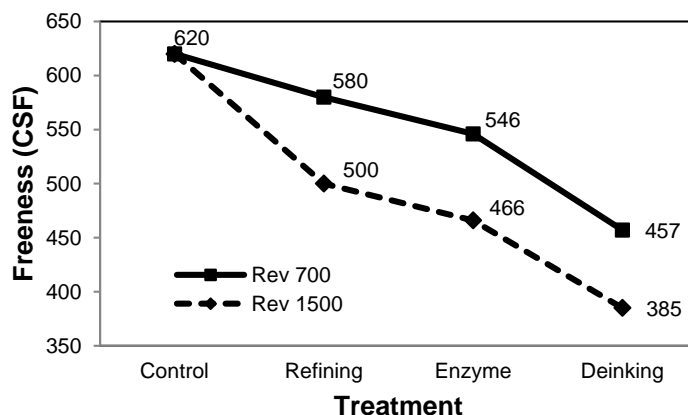


Fig. 4. The total trend of treatments using the RED method on the freeness of treated pulps

According to the above-mentioned description, a decreasing trend was observed using the RED method, but with the ERD method this trend was unusual. After enzymatic treatment and refining, the sum of the freeness reduction was 22.2% and 12% using the ERD and RED methods, respectively. Thus, it can be concluded that better drainage was obtained with the RED method.

Mechanical Properties

For all of the mechanical properties (Table 1), somewhat different trends were observed using the two methods. It was observed that the enzymatic treatment stage had an adverse effect on mechanical properties in both sequences, even after severe mechanical shear applied on fibers by refining in RED method. This can be attributed to the removal of secondary fines generated by refining which contribute effectively on paper strength improvement (Hawes and Doshi 1986; Sirvio and Nurminen 2004).

Table 1. Comparison of Mechanical Strength of Handsheets between the ERD and RED Methods

Mechanical Properties	Control	RED Method						ERD Method					
		R		E		D		E	R		D		
		700	1500	700	1500	700	1500	-	700	1500	700	1500	
Tear (mN.m ² /g)	12.3	11.9	14.2	8.8	8.2	8.2	7.6	7.78	6.8	6.3	6.41	6.2	
Tensile (N.m/g)	28.3	41.9	43.2	38.2	39.9	35.4	37	27.3	30	30.5	28.9	30.4	
Burst (kPa.m ² /g)	1.6	2.6	3.2	1.6	1.8	1.8	1.7	1.3	1.1	1.05	0.99	0.9	

Tear index

Tear index is normally representative of long fibers and a sound cell wall. As long as the fiber cell wall and fiber length are intact, high tear resistance is expected.

Using the ERD method, the application of enzymatic treatment led to decreased tear resistance due to fiber cutting or fiber shortening (Znidarsie-Plazlet *et al.* 2009; Zhang *et al.* 2013). It was expected that the refining stage would worsen the tear strength, as shown in Table 1, by fiber weakening. In the deinking stage, no significant changes were observed in the tear strength. The tear strength reduction after the RE stages was 15% lower than after the ER stages. After deinking with the RED method, the average amount of tear strength was 28.5% and 21.4 % higher than with the ERD method at 700 and 1500 revolutions of refining, respectively.

Tensile index

Tensile strength is an index of fiber bonding. By using a specific amount of enzyme, which varied in different studies (Gil *et al.* 2009; Bajpai 2010; Ibarra *et al.* 2012; Zhang *et al.* 2013), a decrease in tensile strength was observed after enzymatic treatment with both the RED and ERD methods. As is noted above in the mentioned studies, with greater enzyme consumption a significant decrease in tensile index was observed. The trends are shown in Table 1. Using the RED method, tensile strength greatly increased after refining (by about 50% on average) but after enzymatic treatment decreased by about 8.2%. With both methods, the effect of deinking did not have a statistically significant influence on tensile strength. Tensile strength increased by about 27.8% with the RED method but only about 4.7% with the ERD method. Thus, the tensile strength with the RED method was nearly six times higher than with the ERD method.

Burst index

Generally, burst strength can be representative of fiber bonding. It is believed that most enzymes lead to an improvement in drainage but a reduction in strength. This idea can be explained by the fact that during enzymatic treatment, the strength of an individual fiber decreases, so, despite internal and external fibrillation, the mechanical properties of paper decrease (Pomieret *et al.* 1989; Jackson *et al.* 1993; Bajpai 2010; Zhang *et al.* 2013). In this regard, Pala (2009) also claimed that there was no difference between the RED and ERD methods, which is inconsistent with the results obtained in the current study. It is notable that the refining stage in the ERD method caused a burst strength reduction of about

17.6%, but in the RED method it caused an 84% increase in burst index. It seems that applying enzymatic treatment before refining weakened the fiber cell wall, leading to fiber shortening and significantly lowering the strength.

Burst index results revealed that, by using the RED method, this strength was increased by about 10.6% (on average), but, using the ERD method, burst index decreased by about 70% (on average) compared to the control. Thus, it can be concluded that the correct sequence can be very effective on paper properties.

Optical Properties

Some of the optical properties of paper, such as brightness, opacity and ERIC, were determined. In terms of all of these properties, the same trend was observed with both of the methods.

Opacity

Nearly the same downward trend was observed for opacity with both the RED and ERD methods (Fig. 5.). There was no significant difference between not-deinked and deinked pulp in terms of opacity with the ERD and RED methods.

Opacity reduction after enzymatic treatment with both methods could have been due to cell wall degradation and fines generation. Fibers were ready to easily collapse the shape of fibers, which happened in the refining stage and reduced opacity (Cui *et al.* 2015). It can be noted in Fig. 6 that the slope of opacity reduction using the RED method was gentler, while a sharp decline happened with the ERD method, especially after the refining stage. Also, it is apparent that the opacity reduction using the ERD method was nearly double (about 14% in average).

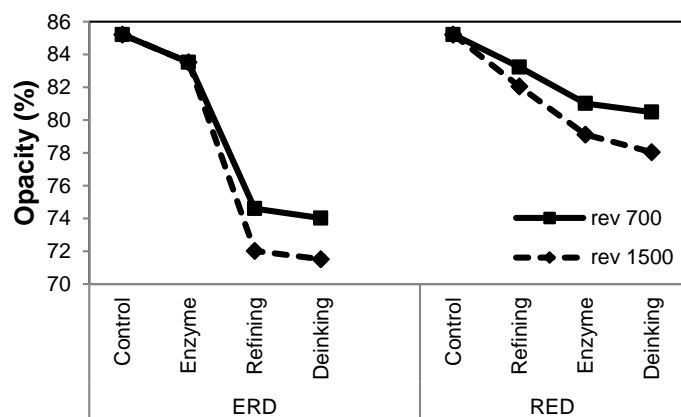


Fig. 5. The effect of the RED and ERD methods on the paper opacity

To discuss the different properties of DIP produced from two methods, optical properties, especially scattering coefficient and opacity, should be considered. As shown in Fig. 5, the higher refining (revolution 1500) and thus higher fines resulted in lower opacity which derives from the nature of the produced fines that are mostly assumed to be fibrillar. As previously indicated (Kangas and Kleen 2004; Seth 2003; Taipale *et al.* 2010; Lee *et al.* 2011), fibrillar fines increase density and bonding between fibers allowing the light to pass through the paper. In the RED method, the amount of fines generated by refining is high, and the enzyme is partly able to hydrolyze them; therefore, opacity is

observed to be higher in the RED method than ERD. On the other hand, the sharp slope in opacity decrease in ERD method after refining might indicate localized degradation of lamella resulting in a significant weakening of fibers (Gurnagul *et al.* 1992), which might be exacerbated by refining not averting the passing light in scattering and opacity measurements.

Opacity and bulk of paper have a direct relationship; the amount of bulk is an indication of opacity and paper stiffness (Chen *et al.* 2013). Therefore, the RED method is expected to produce more bulky paper.

Brightness

As shown in Fig. 6, after enzymatic treatment the brightness of handsheets decreased with both methods. This phenomenon could have been a result of the enzymatic effect on the ink particles' size (Aryaie *et al.* 2012) and ink removal from the fiber surface (Jeffries 1992; Mayeli and Talaeipour 2010).

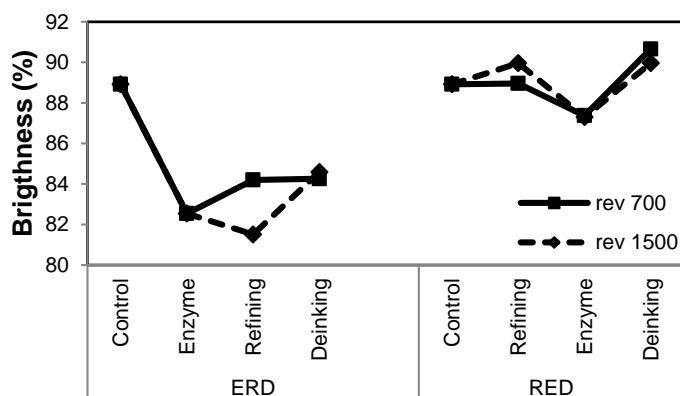


Fig. 6. The effect of the RED and ERD methods on the paper brightness

It is noteworthy that different responses were observed at 700 and 1500 revolutions with the ERD and RED methods. With 700 revolutions of refining, a slight increase was observed in brightness with both methods. It is believed that refining can cause a slight increase in brightness, possibly due to the filler and ash elimination during refining (Talaeipour 2008). However, at 1500 revolutions it is notable that the brightness significantly decreased using the ERD method, especially when the consistency increased, but this led to a brightness increase with the RED method. It seems that more detachment of particles and also more ink removal happened when enzymatic treatment was applied after refining. As shown in Fig. 6, the brightness using the RED method increased by about 1.5% over the control pulp, while decreasing by about 5% using the ERD method.

Effective residual ink concentration (ERIC)

As shown in Fig. 7 and Fig. 8, the trend by which the residual ink concentration changed was almost the reverse of that of brightness. The effect of enzyme treatment and refining was found to be statistically significant relative to ERIC with both methods. This increase in ERIC showed that ink particles had become dispersed after enzymatic treatment and refining (Mayeli 2010). The particle size will be decreased in both ERD and RED methods. As regards the RED method, there are large numbers of free ink particles with small size in this method. Firstly, that can be explained by the refining stage applied, that

as an intense treatment can cause weakening the attachments of fiber surface and ink particles. Secondly, enzymatic treatment and the following hydrolysis in this method can complete the process of detachment. These free particles, consequently, can be easily removed during washing process. On the other hand, the number of small ink adhered on fibers were high in the ERD method. In such case, ink particles on the fibers in the ERD method will be kept in the paper and later give rise to low brightness and high ERIC values.

Figure 8 shows an 83% (951 ppm, on average) increase in ERIC after the ER stages of the ERD method over the control pulp, but, after deinking, the reduction in the ERIC values was just 1% (20 ppm, on average). With the RED method, as seen in Fig. 7, the total increase in ERIC after the RE stages was just 29% (333 ppm, on average), but, after deinking, reduction in the ERIC values was about 5% (92 ppm, on average).

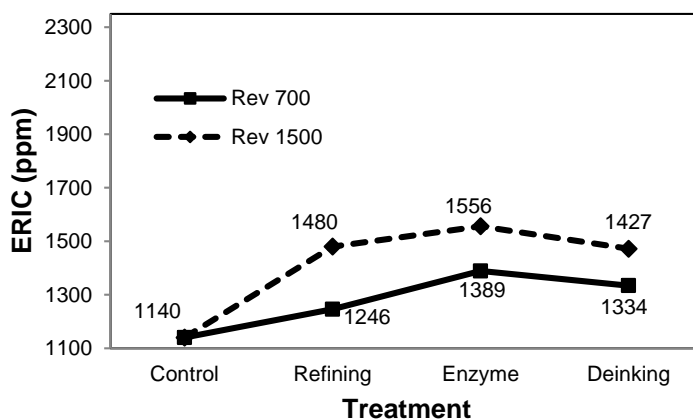


Fig. 7. The total trend of treatments in the RED method on the ERIC of treated pulps

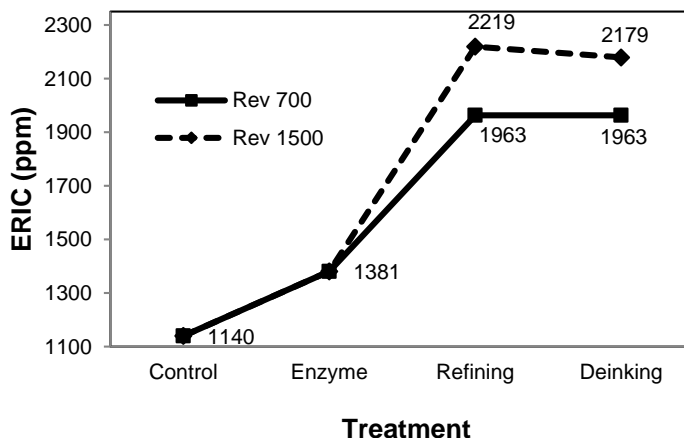


Fig. 8. The total trend of treatments in the ERD method on the ERIC of treated pulps

It seems that the increase in the ERIC using the ERD method represented two undesirable side effects: paper darkening and ink particle size reduction. These two effects led to a decrease in flotation efficiency. Thus, the flotation efficiency of the RED method was determined to be about five times higher than the ERD method. Table 2 shows the comparison between the final paper properties using the RED and ERD methods.

Table 2. Comparison between Final Paper Properties for RED and ERD Methods

Methods	Tear (mNm ² /g)	Tensile (N m/g)	Burst (kPam ² /g)	Opacity (%)	Brightness (%)	ERIC (ppm)
ERD	6.3	29.6	0.94	73	84.4	2071.6
RED	7.9	36.2	1.77	79.2	90.3	1380.8
Competitive Advantages (%)	25.3	22.3	88.3	8.5	7	33.3

The results revealed that the pulp treated with the RED method had better properties. In addition, refining before or after enzymatic treatment significantly affected the mechanical and optical properties of the resulting paper.

CONCLUSIONS

1. Regardless of the sequence of applied treatments (ERD or RED), enzymatic treatment caused a decrease in mechanical properties, optical properties, and pulp freeness, but the ERIC increased. Unlike the enzymatic treatment, it is noteworthy that the order of the refining stage in the treatment sequence (either before or after enzymatic treatment) produced very different effects.
2. When considering enzymatic treatment and refining, subjects including fibrillation, fines generation and hydrolysis, fiber shortening, and surface activation, *etc.*, can be discussed. It was expected that the RED method would generate more fines, firstly due to the primary refining, and then these fines would be removed from the pulp or eliminated by the enzymatic hydrolysis and finally improve the drainage. But the obtained results not only showed drainage improvement, but better strength and optical properties. Such results are of interest because different fines imply intricate effects on paper properties.
3. It seems that applying the refining stage after the enzymatic treatment (ERD method) led to undesirable effects on fibers and ink particles. Refining using the ERD method worsened the mechanical properties, except tensile strength. Brightness and opacity decreased, and ERIC increased significantly, leading to flotation efficiency reduction.
4. Conversely, better results were obtained using the RED method (applying refining before enzymatic treatment). With the RED method, better drainage was obtained compared to the ERD method. It is noteworthy that, after refining, not only all of the mechanical properties increased significantly, but the final strengths also increased in comparison to the ERD method. Different responses were observed according to different refining revolution values, but the brightness increased significantly using the RED method. The opacity decreased using the RED method, but the slope of this reduction was also gentler than with the ERD method. The results for ERIC of treated pulps were noticeable. Although the ERIC increased with both methods (RED and ERD), using the RED method it increased with a gentle slope and was much lower than with the ERD method. Thus, the flotation efficiency of the RED method was determined to be about five times higher than the ERD method.

ACKNOWLEDGMENTS

This paper was extracted from a Ph.D thesis under the supervision of Dr. Mohammad Talaeipour and Dr. Amir Hooman Hemmasi. The Shahid Beheshti University Science and Research Park and the packaging department of the Iranian Standard and Industrial Research Organization are gratefully acknowledged for providing testing facilities.

REFERENCES CITED

- Aryaie Monfared, M. H., Resalati, H., and Ghasemian, A. (2012). "Enzymatic deinking of office waste papers in the comparison with conventional chemical method: Part 1- Appearance and optical properties of pulp," *Wood & Forest Science and Technology* 18(4), 59-76.
- Bhardwaj, N. K., Hoang, V., and Nguyen, K. L. (2007). "Effect of refining on pulp surface charge accessible to polyDADMAC and FTIR characteristic bands of high yield kraft fibers," *Bioresource Technology* 98 (4), 962-966.
- Bajpai, P. K. (2010). "Solving the problems of recycled fiber processing with enzymes," *BioResources* 5(2), 1311-1325.
- Cui, L., Meddeb-Mouelhia, F., Laframboise, F., and Beauregard, M. (2015). "Effect of commercial cellulases and refining on kraft pulp properties: Correlations between treatment impacts and enzymatic activity components," *Carbohydrate Polymers* 115, 193-199. DOI:10.1016/j.carbpol.2014.08.076.
- Das, A., Paul, T., Halder, S. K., Jana, A., Maity, C., Mohapatra, P. K. D., Pati, B. R., and Mondal, K. C. (2013). "Production of cellulolytic enzymes by *Aspergillus fumigates* ABK9 in wheat bran-rice straw mixed substrate and use of cocktail enzymes for deinking of waste office paper pulp," *Bioresource Technology* 128, 290-296.
- Dienes, D., Egyhazi, A., and Reczey, K. (2004). "Treatment of recycled fiber with Trichoderma cellulases," *Industrial Crops and Products* 20(1), 11-21. DOI: 10.1016/j.indcrop.2003.12.009.
- Dienes, D. (2006). "Effect of cellulase enzymes on secondary fiber properties," Ph.D. thesis, Budapest University of Technology and Economics Department of Agricultural Chemical Technology.
- Dourado, F., Mota, M., Pala, H., and Gama, F. M. (1999). "Effect of cellulose adsorption on the surface and interfacial properties of cellulose," *Cellulose* 6(4), 265-282. DOI: 10.1023/A:1009251722598.
- Ebner, G., Vejdovszky, P., Wahlström, R., Suurnäkki, A., Schrems, M., Kosma, P., Rosenau, T., and Potthast, A. (2014). "The effect of 1-ethyl-3-methylimidazolium acetate on the enzymatic degradation of cellulose," *Journal of Molecular Catalysis B: Enzymatic* 99, 121-129.
- Efrati, Z., Talaeipour, M., Khakifirouz, A., and Bazayr, B. (2013). "Impact of cellulase enzyme on strength, morphology and crystallinity of deinked pulp," *Cellulose Chem. Tech.* 47 (7-8), 547-551.
- Eriksson, L. A., Heitmann, J. A., and Venditti, R. (1998). "Freeness improvement of recycled fibers using enzymes with refining," in: *Textbook of Enzyme Applications in Fiber Processing, Chapter 4, pp. 41-54, ACS Books, Wash DC, 1998.* DOI: 10.1021/bk-1998-0687.ch004.

- Garcia, O., Torres, A. L., Colom, J. F., Pastor, F. I. J., Diaz, P., and Vidal, T. (2002). "Effect of cellulase-assisted refining on the properties of dried and never-dried eucalyptus pulp," *Cellulose* 9(2), 115-125. DOI: 10.1023/A:1020191622764.
- Gurnagul, N., Page, D. H., and Paice, M. G. (1992). "The effect of cellulose degradation on the strength of wood pulp fibers," *Nordic Pulp and Paper Research Journal* 3, 152-154. DOI: 10.3183/NPPRJ-1992-07-03-p152-154.
- Gil, N., Gil, C., Amaral, M. E., Costa, A. P., and Duarte, A. P. (2009). "Use of enzymes to improve the refining of a bleached *Eucalyptus globulus* kraft pulp," *Biochemical Engineering Journal* 46(2), 89-95. DOI: 10.1016/j.bej.2009.04.011.
- Hawes, J. M. and Doshi, M. R. (1986). "The contribution of different types of fines to the properties of handsheets made from recycled paper," *The Institute of Paper Chemistry*, Appleton, Wisconsin, 1-8.
- Ibarra, D., Monte, M. C., Blanco, A., Martinez, A. T., and Martinez, M. J. (2012). "Enzymatic deinking of secondary fibers: Cellulases/hemicellulases versus laccase-mediator system," *J. Ind. Microbiol. Biotechnol.* 39(1), 1-9. DOI: 10.1007/s10295-011-0991-y.
- Jeffries, T. W. (1992). "Enzymatic treatments of pulps," in: Rowell, R. M., Schultz, T. P., and Narayan, R. (eds.), *Emerging Technologies for Materials and Chemicals from Biomass*, ACS Symposium Series 476, American Chemical Society, Washington, D.C., pp. 313-329.
- Kim, H. J., Jo, B. M., and Lee, S. H. (2006). "Potential for energy saving in refining of cellulase-treated kraft pulp," *J. Ind. Eng. Chem.* 12(4), 578-583.
- Kangas, H., and Kleen, M. (2004). "Surface chemical and morphological properties of mechanical pulp fines," *Nordic Pulp and Paper Research Journal* 19(2), 191-199.
- Loosvelt, I. (2009). "Modifying the quality of fiber with enzymes," *PaperAge* 20-22.
- Lee, H., Namb, W. S., Sohn, S. D., and Paik, K. H. (2011). "Effect of different types of fines on the properties of recycled chemical pulp," *Journal of Industrial and Engineering Chemistry* 17, 100-104. DOI: 10.1016/j.jiec.2010.12.004.
- Lee, C. K., Ibrahim, D., Ibrahim, C. O., and Wan Rosli, W. (2011). "Enzymatic and chemical deinking of mixed office waste paper and old newspaper: Paper quality and effluent characteristics," *BioResources* 6(4), 3859-3875.
- Mayeli, N., and Talaeipour, M. (2010). "Effect of different HLB value and enzymatic treatment on the properties of old newspaper deinked pulp," *BioResources* 5(4), 2520-2534.
- Maximino, M. G., Taleb, M. C., Adell, A. M., and Formento, J. C. (2011). "Application of hydrolytic enzymes and refining on recycled fibers," *Cellulose Chem. Technol.* 45(5-6), 397-403.
- Ny, C. L., and Messmer, M. (2007). "Potential of refining and dispersing to develop recycled fibre properties," *Pulp and Paper Canada* 108(2), 35-41.
- Pala, H., Lemos, M. A., Mota, M., and Gama, F. M. (2001). "Enzymatic upgrade of old paperboard containers," *Enzyme and Microbial Technology* 29(4), 274-279. DOI: 10.1016/S.
- Pala, H., Mota, M., and Gama, F. M. (2001). "Refining and enzymatic treatment of secondary fibers for paperboard production: cyberflex measurements of fiber flexibility," *COST E20: Wood Fiber Cell Wall Structure Conference Proceedings*.
- Park, S., Venditti, R. A., Abrecht, D. G., Jameel, H., Pawlak, J. J., and Lee, J. M. (2006). "Surface and pore structure modification of cellulose fibers through cellulase

- treatment,” *Journal of Applied Polymer Science* 103, 3833-3839.
DOI: 10.1002/app.25457.
- Patil, M. B., and Dhake, A. B. (2014). “Deinking of news paper pulp by β -glucosidase of *Penicillium purpurogenum*,” *Int. J. Engg. Res. & Sci. & Tech.* 3(2), 276-279.
- Seth, R. S. (2003). “The measurement and significance of fines,” *Pulp and Paper Canada* 104(2), 41-44.
- Sirvio, J., and Nurminen, I. (2004). “Systematic changes in paper properties caused by fines,” *Pulp & Paper Canada*, 105(8), 39-42.
- TAPPI Standard T 205 (1995). “Forming handsheets for physical tests of pulp,” TAPPI Press, Atlanta, GA.
- TAPPI Standard T 227 (1999). “Freeness of pulp (Canadian standard method),” TAPPI Press, Atlanta, GA.
- TAPPI Standard T 494 (2001). “Tensile properties of paper and paperboard (using constant rate of elongation apparatus),” TAPPI Press, Atlanta, GA.
- TAPPI Standard T 403 (2002). “Bursting strength of paper,” TAPPI Press, Atlanta, GA.
- TAPPI Standard T 414 (2004). “Internal tearing resistance of paper (Elmendorf-type method),” TAPPI Press, Atlanta, GA.
- TAPPI Standard T 425 (2006). “Opacity of paper (15/d geometry, illuminant A/2°, 89% reflectance backing and paper backing),” TAPPI Press, Atlanta, GA.
- TAPPI Standard T 452 (2008). “Brightness of pulp, paper, and paperboard (directional reflectance at 457 nm),” TAPPI Press, Atlanta, GA.
- Talaeipour, M. (2008). “The effect of refining on the optical and mechanical properties of chemical deinked pulp,” *Iranian Wood and Paper Research Journal* 24(1), 148-157.
- Taipale, T., Osterberg, M., Nykanen, A., Ruokolainen, J. and Laine, J. (2010). “Effect of microfibrillated cellulose and fines on the drainage of kraft pulp suspension and paper strength,” *Cellulose* 17, 1005-1020. DOI 10.1007/s10570-010-9431-9.
- Torres, C. E., Negro, C., Fuente, E., and Blanco, A. (2012). “Enzymatic approaches in paper industry for pulp refining and biofilm control,” *Appl. Microbiol. Biotechnol.* 96(2), 327-344.
- Znidarsie-Plazl, P., Rutar, V., and Ravnjak, D. (2009). “The effect of enzymatic treatments of pulps on fiber and paper properties,” *Chem. Biochem. Eng.* 23(4), 497-506.
- Zhang, Z. J., Chen, Y. Z., Hu, H. R., and Sang, Y. Z. (2013). “The beatability-aiding effect of *Aspergillus niger* crude cellulase on bleached simao pine kraft pulp and its mechanism of action,” *BioResources* 8(4), 5861-5870.

Article submitted: January 20, 2015; Peer review completed: March 22, 2015; Revised version received and accepted: June 9, 2015; Published: June 16, 2015.

DOI: 10.15376/biores.10.3.4768-4783

APPENDIX

RED Method

Table 3. Untreated Pulp Properties for RED Methods

Pulp	CSF (ml)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
Untreated Pulp	620	88.92	85.21	12.38	28.36	1.60	1140.70

Table 4. The First Stage for RED Method (Refining)

Refining (rev)	CSF (ml)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
700	580	88.96	83.23	11.96	41.98	2.61	1246.97
1500	500	89.96	82.05	14.26	43.23	3.21	1480.94

Table 5. The Second Stage for RED Method (Enzyme Treatment)

Refining (rev)	Enzyme Treatment		CSF (ml)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
	Consistency (%)	Time (min)							
700	5	20	520	86.80	80.55	8.23	38.71	1.60	1361.06
	8	20	518	85.83	82.79	8.24	39.18	1.64	1316.35
	13	20	540	86.10	81.20	8.30	40.23	1.51	1297.54
	5	40	520	87.90	83.09	10.81	38.50	1.88	1283.54
	8	40	570	88.32	82.25	9.07	36.03	1.79	1330.88
	13	40	578	87.23	79.17	9.11	40.50	1.79	1702.12
	5	60	520	88.05	81.51	8.33	39.30	1.67	1387.88
	8	60	580	88.08	77.94	8.83	31.33	1.60	1432.29
1500	13	60	570	87.78	80.66	8.55	40.34	1.70	1397.35
	5	20	460	88.29	78.82	8.31	40.20	2.02	1567.77
	8	20	460	86.96	81.30	7.79	39.97	1.83	1600.67
	13	20	440	87.96	79.25	8.95	42.29	1.77	1631.28
	5	40	460	86.95	78.77	7.78	42.75	1.58	1278.20
	8	40	472	87.02	77.88	8.49	40.50	1.74	1738.11
	13	40	520	86.82	79.66	8.06	36.36	1.79	1634.40
	5	60	490	86.93	77.79	8.20	39.00	2.09	1512.43
8	60	480	86.45	79.05	7.63	40.27	1.73	1530.12	
	13	60	420	88.06	79.50	8.88	38.19	1.72	1518.80

Table 6. The Third Stage for RED Method (Deinking)

Refining (rev)	Enzyme Treatment		CSF (ml)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
	Consistency (%)	Time (min)							
700	5	20	440	89.66	80.54	8.09	31.96	1.70	1337.71
	8	20	488	90.12	80.42	8.70	36.38	1.95	1425.74
	13	20	440	91.54	80.33	7.97	36.86	1.77	1284.90
	5	40	420	90.92	78.85	8.07	35.54	1.99	1359.09
	8	40	460	91.58	80.26	8.94	35.54	2.03	1423.68
	13	40	440	89.98	80.15	7.92	35.80	1.62	1348.99
	5	60	440	90.43	79.61	8.24	34.74	1.68	1258.45
	8	60	460	90.31	83.14	7.55	35.80	1.77	1349.94
	13	60	525	91.44	81.00	8.63	36.67	1.68	1220.57
1500	5	20	440	89.58	78.46	7.79	38.71	1.64	1380.47
	8	20	350	91.08	77.65	7.46	32.92	1.69	1585.60
	13	20	340	89.55	78.47	7.10	36.86	1.62	1352.02
	5	40	430	89.69	78.07	7.78	36.07	1.89	1511.02
	8	40	380	88.47	78.06	7.17	39.25	1.61	1425.36
	13	40	375	90.64	76.52	7.54	35.00	1.66	1526.14
	5	60	440	90.01	78.38	8.62	41.33	2.26	1356.81
	8	60	340	89.55	78.47	7.10	36.86	1.62	1352.02
	13	60	370	91.11	78.34	7.81	36.10	1.73	1357.56

ERD Method

Table 7. Untreated Pulp Properties for RED Method

Pulp	CSF (mL)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
Untreated Pulp	620	88.92	85.21	12.38	28.36	1.60	1140.70

Table 8. The First Stage for ERD Method (Enzyme Treatment)

Enzyme Treatment		CSF (mL)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
Consistency (%)	Time (min)							
5	20	582	81.54	82.91	7.72	32.58	1.30	1529.70
8	20	600	87.40	82.41	7.93	26.76	1.15	1540.53
13	20	520	79.53	81.95	7.92	28.63	1.25	1317.64
5	40	560	80.86	82.96	7.68	26.14	1.41	1424.90
8	40	550	82.62	83.22	7.32	23.17	1.22	1311.83
13	40	590	80.47	84.59	7.60	27.27	1.41	1361.49
5	60	520	82.93	85.41	7.73	28.79	1.39	1405.25
8	60	560	83.65	83.26	8.17	26.98	1.30	1386.57
13	60	620	83.89	85.07	8.04	25.48	1.33	1152.99

Table 9. The Second Stage for ERD Method (Refining)

Enzyme Treatment		Refining (rev)	CSF (mL)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
Consistency (%)	Time (min)								
5	20	700	380	83.80	72.45	7.09	26.54	1.07	2155.60
	20	1500	620	81.01	71.62	6.18	31.25	1.01	2246.24
8	20	700	350	85.88	71.55	7.76	31.83	1.09	1966.65
	20	1500	435	81.40	74.94	6.27	35.58	1.26	2075.54
13	20	700	290	85.10	73.76	7.87	31.59	1.15	1911.59
	20	1500	400	77.93	71.31	6.70	28.08	1.01	2129.00
5	40	700	390	80.96	75.59	6.35	21.90	1.08	1917.71
	40	1500	640	85.56	70.91	6.48	30.92	1.11	2507.36
8	40	700	420	83.23	71.20	7.08	32.07	1.08	1926.27
	40	1500	610	80.75	69.30	6.81	28.05	1.08	2431.27
13	40	700	410	82.88	75.86	6.21	29.52	1.12	2214.60
	40	1500	560	81.70	70.66	6.14	32.05	1.07	2225.09
5	60	700	360	86.39	77.10	6.34	32.42	1.02	1630.53
	60	1500	530	81.82	73.56	6.25	30.39	0.85	2162.18
8	60	700	390	84.93	76.63	6.49	32.83	1.02	1994.98
	60	1500	580	86.71	70.81	5.81	28.09	0.99	2098.78
13	60	700	340	84.68	77.30	6.31	31.56	1.24	1956.57
	60	1500	500	76.75	74.78	6.47	30.70	1.15	2102.01

Table 10. The Third Stage for ERD Method (Deinking)

Enzyme Treatment		Refining (rev)	CSF (mL)	Brightness (%)	Opacity (%)	Tear (mN.m ² /g)	Tensile (Nm/g)	Burst (kPa.m ² /g)	Eric (ppm)
Consistency (%)	Time (min)								
5	20	700	520	81.62	73.15	6.54	31.04	0.84	2155.62
	20	1500	570	82.55	71.57	6.07	31.26	0.56	2158.07
8	20	700	340	84.62	75.04	6.41	23.81	1.13	1966.65
	20	1500	640	87.98	71.41	5.94	20.04	0.58	2089.79
13	20	700	330	83.61	74.46	6.22	31.49	1.03	1911.59
	20	1500	420	83.44	72.74	6.20	32.33	0.96	2094.12
5	40	700	460	84.22	75.26	6.28	31.76	0.96	1917.71
	40	1500	520	83.94	71.92	6.14	32.66	1.08	2018.74
8	40	700	400	84.81	73.84	6.32	22.26	0.94	1926.27
	40	1500	640	84.28	71.03	6.66	30.52	0.86	2256.46
13	40	700	520	84.01	72.82	6.40	30.58	0.96	2214.60
	40	1500	640	86.83	71.38	6.22	28.17	0.97	2282.53
5	60	700	420	84.82	77.38	6.31	29.42	1.05	1630.53
	60	1500	700	88.20	69.66	6.62	30.64	0.69	2500.52
8	60	700	420	86.12	73.93	6.60	30.49	1.06	1994.98
	60	1500	540	80.59	72.37	6.17	34.77	1.25	2042.39
13	60	700	420	84.61	74.81	6.32	29.81	0.94	1956.57
	60	1500	610	83.58	71.51	6.23	33.43	1.11	2171.31