IMPACT OF ACID WASHING AND CHELATION ON $Mg(OH)_2$-BASED HYDROGEN PEROXIDE BLEACHING OF MIXED HARDWOODS CMP AT A HIGH CONSISTENCY

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The removal of transition metal ions is crucial for improving the efficiency of subsequent peroxide bleaching. Acid-washing and chelation have been proposed for such a purpose. However, their influences on the $Mg(OH)_2$-based peroxide bleaching of hardwood pulps at a high consistency have not been well documented in the literature. In this work, we studied the influence of acid-washing using sulfuric acid or chelation using diethylenetriaminepentaacetic (DTPA) on the $Mg(OH)_2$- or NaOH-based hydrogen peroxide bleaching efficiency, effluent properties of bleaching filtrates, and paper properties. The results showed that for $Mg(OH)_2$-based peroxide bleaching, the pulp yield and water retention value of acid-washed pulp were higher than those of the chelated pulp; the chemical oxygen demand (COD) and turbidity of the bleaching filtrates for the acid-washed pulp were lower than those of the chelated pulp. The bleached acid-washed pulp had lower strength properties than bleached chelated pulp did. Additionally, at a high pulp consistency (25%), the $Mg(OH)_2$-based process had a higher bleaching efficiency and superior bleaching effluent properties, but a lower strength properties, in comparison with the NaOH-based process.

Keywords: Acid washing; DTPA chelation; Peroxide bleaching; CMP; Hardwood; $Mg(OH)_2$; NaOH

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INTRODUCTION

Hydrogen peroxide bleaching has been practiced in industry for the bleaching of mechanical pulps or chemical pulps for many years. It is well known that the transition metal contents of pulps significantly affect the performance of a peroxide bleaching stage (Colodette et al. 1989; Prasakis et al. 1996; Lapierre et al. 1997; Ni et al. 1998; Kangas et al. 2002). The decomposition mechanism of peroxide in the presence of metal ions, which impairs the efficiency of peroxide bleaching, has been comprehensively reported in the literature (Colodette et al. 1989; Prasakis et al. 1996; Ni et al. 2000; Kangas et al. 2002; Wekesa and Ni 2003).

Acid washing of pulp is one method for removing the metal ions of pulp prior to the peroxide bleaching (Li et al. 2000; Suess et al. 2001; Babineau et al. 2003; Wójciak 2006). Acid washing has the advantages of low cost and simplicity. It was reported that the effluent of acid washing can be recycled 8 times for the pretreatment of pulp

(Bouchard et al. 1995). However, the selectivity of acid washing in removing metal ions is low. Thus, the restoration of alkaline earth metal ions is necessary before a peroxide bleaching stage (Lapierre et al. 1997).

Chelation has also been applied as a practical method for removing the metal ions prior to peroxide bleaching. There are different proposals for the improving efficiency of peroxide bleaching in the presence of chelating agents. These include making ring structures with metal ions, thus inhibiting their catalytic effect (Prasakis et al. 1996), buffering action (Fairbank et al. 1989), and interruption of free radical processing through the formation of a stable intermediate (Coldoette et al. 1989). In the literature, it was reported that the acid washing can be substituted by diethylenetriaminepentaacetic (DTPA) chelation prior to oxygen delignification (Bouchard et al. 1995). One objective of this study was to compare the performance of acid-washing with that of DTPA chelation prior to $Mg(OH)_2$-based peroxide bleaching.

The substitution of $Mg(OH)_2$ for NaOH as the alkali source in the peroxide bleaching of various wood species has been well documented in the literature (Johnson et al. 2002; Li et al. 2005; He et al. 2006; Wójciak 2006; Wong et al. 2006; Wang et al. 2008; Hou et al. 2010), and the process has been commercialized. The advantages of the $Mg(OH)_2$-based process over the conventional NaOH-based process have been proved. For example, the effluent characteristics, formation of anionic trash, pulp yield, and minimization of deposit formation are improved via the $Mg(OH)_2$-based process in comparison to the NaOH-based process (He et al. 2004, 2005, 2006; Yu and Ni 2006). Another objective of this study was to investigate the performance of $Mg(OH)_2$- and NaOH-based peroxide bleaching at a high consistency for a hardwood chemimechanical pulp (CMP).

In this work, an unbleached CMP was pretreated with an acid washing stage or DTPA chelation. Then, it was subjected to an alkaline hydrogen peroxide bleaching stage at a pulp consistency of 25% using either $Mg(OH)_2$ or NaOH as the alkali source. The bleaching performance, pulp properties, and effluent characteristics were then systematically compared. The results of this research are directly dealt with the application of chelation or acid-washing prior to peroxide bleaching of the CMP sample at a high pulp consistency.

EXPERIMENTAL

Raw Materials

The chemimechanical pulp (CMP) sample via the sulfite process was obtained from a mill located in northern Iran (Mazandaran Wood and Paper Industries Co.). The species used in producing the CMP pulp were hornbeam, beech, and birch at mass fractions of 60%, 20%, and 20%, respectively. The pulp was stored at a consistency of 10 %(wt.) in a cold room prior to use. Diethylenetriaminepentaacetic (DTPA) sodium salt, sulfuric acid, NaOH (1M), and $Mg(OH)_2$ (1 M) were purchased from Merck, Co. Germany, while silicate was from Carlo Erba Co. Italy.

Acid-Washing or Chelation

The acid-washing was performed using sulfuric acid at a pH of 2.5, and 5% consistency at 70 ºC for 30 min. The DTPA chelation stage was conducted using 0.2% DTPA at a 10% consistency, 70 ºC for 30 min. Then, the pulps were washed with deionized distilled water and the filtrates passing a 200-mesh funnel were recycled to collect the fines.

Peroxide Bleaching

The conditions of peroxide bleaching on chelated or acid-washed pulps using $Mg(OH)_2$ or NaOH as the alkali source are listed in Table 1. The bleaching experiment was conducted in plastic bags using a water bath. In the case of NaOH-based peroxide bleaching, water, sodium silicate, sodium hydroxide, and hydrogen peroxide were mixed in a beaker, and the liquor was subsequently added to the pulp. In the case of $Mg(OH)_2$-based peroxide bleaching, DTPA and $Mg(OH)_2$ were added together to the pulp suspension. After thorough mixing, the pulp was placed into the water bath at 70 ºC for 5 min. Subsequently, the required amount of hydrogen peroxide was added to the pulp suspension and the bleaching was conducted under the conditions specified in Table 1. After the bleaching, supernatants were collected via vacuum-filtering of the pulps.

Table 1. Conditions of NaOH-Based or $Mg(OH)_2$-Based Peroxide Bleaching

Alkali, % (wt.) on pulp	DTPA, %(wt.) on pulp	Silicate, % (wt.) on pulp	H_2O_2, %(wt.) on pulp	Temperature, ºC	Time, min	Consistency, % (wt.)
NaOH, 2.1	-	3	3	70	150	25
Mg $(OH)_2$ 1, 1.5, 2	0.1	-	3	70	150	25

Pulp and Effluent Characterizations

The contents of the metal ions, i.e., iron, copper, magnesium, and manganese were measured by using an atomic absorption spectrometer, PU 9400X, PHILIPS, UK according to TAPPI T 266. The water retention of value (WRV) of the bleached pulps was determined according to TAPPI UM 256 at 900g for 30 min using a centrifuge, Z206A, HERMLE, Germany. The chemical oxygen demand (COD) of the bleaching effluent was determined based on the dichromate oxidation method according to the APHA standard method using a PALINTEST 8000 Photometer, PALINTEST, UK. The turbidity of the bleaching effluent was measured using a turbidity meter, IRTB100, EUTECH, Singapore.

Paper Making Properties

The handsheet papers of the bleached pulps were prepared according to TAPPI T255. The brightness, and opacity were measured according to TAPPI T452 using an ELREPHO 2000, Data Color, Switzerland. The tensile, tear, and burst indices were determined according to TAPPI T495, T 414, and T 403, respectively, using tensile, tear, and burst testers (Lorentzen & Wettre, Kista, Sweden). The bulk was determined according to TAPPI T500.

RESULTS AND DISCUSSION

Metal Ion Profiles

The metal contents of the unbleached pulp, the acid-washed, and the chelated pulps are listed in Table 2. As can be seen, the acid-washing or chelation was able to remove 81% or 27% of the magnesium present in the pulp, respectively. In the literature, the acid-washing (at a pH of 3 and 50 ºC for 30 min) or chelating with a DTPA dosage of 0.6% (at 50 ºC for 30 min) reduced 83% or 26% of the Mg ion content of unbleached hardwood kraft pulp (Bouchard et al. 1995). The lower Mg ion reduction via DTPA application was attributable to lower specificity of chelation to the magnesium in the pulp (Bouchard et al. 1995). Magnesium, when present at an optimum amount in the pulp, can promote peroxide bleaching performance (Colodette et al. 1989; Abbot 1991; Prasakis et al.1996). The significant reduction in the magnesium content from the acid-washing can be compensated for by the subsequent addition of $Mg(OH)_2$ in the peroxide bleaching (Bouchard et al. 1995). Additionally, the acid-washing decreased the Mn ion content present in the pulp by 18%, while the chelation decreased it by 56% (Table 2). The decomposition of H_2O_2 in the presence of metals ions, especially Mn ion, has been well documented in the literature (Prasakis et al. 1996; Abbot 1991, Wekesa and Ni 2003; Ni et al. 2000). Based on these results, one can conclude that the acid-washing was not as effective as chelation for managing the Mg and Mn ion profiles of the unbleached pulp prior to peroxide bleaching. Also, both treated pulps had similar amounts of Fe and Cu, which is consistent with the results reported in the literature (Bouchard et al. 1995).

Table 2. Metal Contents of Pulps after Acid-washing or DTPA Chelation Treatment

Metal	Fe, ppm	Mg, ppm	Cu, ppm	Mn, ppm
Untreated pulp	7.5	395	1.5	147
Acid-washed pulp	4.1	75	0.45	120
Chelated pulp	3.6	285	0.4	65

Peroxide Bleaching

The properties of various bleached pulps are listed in Table 3. As expected, by increasing the dosage of $Mg(OH)_2$, the bleaching yield was reduced, regardless of the pretreatment conditions. It was reported that by increasing the dosage of $Mg(OH)_2$ in the peroxide bleaching of chelated TMP pulp, the yield was reduced (He et al. 2004). Evidently, the bleaching yield was 2% higher for the acid-washed pulp than for the chelated pulp at the same $Mg(OH)_2$ dosage applied. Furthermore, the WRV of the bleached acid-washed pulp was relatively higher than that of the bleached chelated pulp. It is also noted in Table 3 that by increasing the dosage of $Mg(OH)_2$ during the peroxide bleaching, the WRV decreased, regardless of the bleaching pretreatment. It has been reported that the adsorption of Mg ions on the fiber via ion exchange during $Mg(OH)_2$-based peroxide bleaching might decrease the swelling ability of fibers (He et al. 2006). Also, it can be found that the final pH after bleaching was higher as a result of the increase in the dosage of $Mg(OH)_2$ applied.

It is further noted in Table 4 that the bleaching yield was about 4% lower if NaOH was used instead of $Mg(OH)_2$ in the peroxide bleaching. In the literature, by using 2% of NaOH or $Mg(OH)_2$ in the peroxide bleaching of TMP at 11% consistency, a yield of

96.08% or 97.36% was obtained, respectively (He et al. 2004). The WRV of NaOH pretreated pulp was much higher than that of $Mg(OH)_2$-based bleached pulp.

Table 3. Pulp Properties Obtained after Various Conditions of Peroxide Bleaching

$Mg(OH)_2$ or NaOH %	Yield, %		WRV, g/g		Final pH		Reference
	Acid washed	Chelated	Acid washed	Chelated	Acid washed	Chelated	
1%, $Mg(OH)_2$	97.4	95.3	2.4	2.2	8	8.1	PS[1]
1.5%, $Mg(OH)_2$	96.2	94.2	2.3	2.1	8.2	8.3	PS
2%, $Mg(OH)_2$	96.3	94.5	2.2	2.1	8.4	8.8	PS
2.1%, NaOH	-	91.2	-	2.61		9.2	PS
2%, NaOH	-	-	-	2.7	-	7.71	He et al. 2006
2%, $Mg(OH)_2$	-	-	-	2.8	-	7.61	He et al. 2006
2%, $Mg(OH)_2$	-	97.36	-	-	-	7.93	He et al. 2004
2%, NaOH	-	96.08	-	-	-	8.13	He et al. 2004

[1]:present study

Bleaching Effluent Characterization

The turbidity and COD of the peroxide bleaching effluent are listed in Table 4. As can be seen, the turbidity for the acid-washed pulp was about 4-5 units lower than that of the chelated pulp. Additionally, the COD for the acid-washed pulp was 3-4 kg/ton lower than that for the chelated pulp. These results imply that more organic compounds were dissolved during the peroxide bleaching of chelated pulp than that of acid-washed pulp. These results are consistent with the pulp yield results (Table 3) in that a lower pulp yield was obtained for the chelated pulp than for the acid-washed pulp under the same bleaching conditions.

It is also notable in Table 4 that the turbidity and COD of the bleaching effluent from the NaOH-based process was significantly higher than those from the $Mg(OH)_2$-based peroxide process. These results can be attributed to the dissolution of more organic materials from pulp during the peroxide bleaching when NaOH is used as the alkali source (He et al. 2005). The effluent characteristics of the peroxide bleaching of hardwood TMP (at 10% consistency, 70 ºC for 5 h) are also included in Table 4. It is evident that the COD and turbidity of the NaOH-based peroxide bleaching was significantly higher than those of the $Mg(OH)_2$-based peroxide bleaching. In the literature, the higher COD and turbidity of NaOH were attributed to the higher alkalinity of NaOH, which hydrolyzed the long-chain lignocellulosic materials (or anionic trashes) of the pulps (He et al. 2005; Zeinaly et al. 2009).

Table 4. Effluent Characteristics of Acid-Washed and Chelated Pulps

$Mg(OH)_2$ or NaOH, %	Turbidity, NTU		COD, kg/ton		References
	Acid-washed	Chelated	Acid-washed	Chelated	
1%, $Mg(OH)_2$	6.8	11.4	5.6	9.6	PS[1]
1.5%, $Mg(OH)_2$	7.0	11.6	5.7	9.7	PS
2%, $Mg(OH)_2$	7.3	12.0	6.1	9.6	PS
2.1%, NaOH	-	26.4	-	19.1	PS
2%, $Mg(OH)_2$	-	10.7	-	37.7	He et al. 2004
2%, NaOH	-	103	-	51.9	He et al. 2004

[1]:present study

Paper Properties

The properties of papers made of bleached pulps from either acid-washed or chelated pulp, are listed in Table 5. The tensile, burst, and tear indices of the chelated pulp were significantly higher than those of the acid-washed pulp. The higher strength of the chelated pulp is attributed to the dissolution of more hydrophobic organics (higher yield loss) during peroxide bleaching. However, the opacity and bulk of the chelated pulp were slightly lower than those of the acid-washed pulp, which are in agreement with the higher strength properties. Also, the optimum dosage of $Mg(OH)_2$ was 1.5 % for obtaining the maximum strength for the bleached chelated pulp. In the literature, it was reported that the optimum dosage of $Mg(OH)_2$ for obtaining the maximum strength depended on the wood species and other bleaching conditions (He et al. 2004, 2005). Furthermore, the brightness of the chelated pulp was approximately 5 % ISO higher than that of the acid-washed pulp under the conditions studied. In the literature, the brightness of the chelated hardwood kraft pulp was 2.5 %ISO higher than the acid-washed pulp when applying 5% peroxide charge at a pH of 3, 90 ºC for 3 h (Bouchard et al. 1995).

Included in Table 5 are the strength results of peroxide bleaching when NaOH was used as the alkali source. It is evident that the tensile, burst, and tear indices of the bleached pulp from the NaOH-based process were substantially higher, while the bulk and opacity were lower than those from the $Mg(OH)_2$-based process. In one study, 2% of NaOH or $Mg(OH)_2$ was applied in the 4% peroxide bleaching of TMP at 10% consistency at 70 ºC for 5 h (He et al. 2006). In another study, a hardwood CMP pulp was first chelated under the conditions of 3% pulp consistency, 0.3% DTPA (o.d. on pulp), a pH of 6, 70 ºC for 30 min, and then bleached via 3% H_2O_2 dosage at 10% consistency, 0.1% DTPA dosage, at 70 ºC for 2 h (Zeinaly et al. 2009). The strength properties of these two studies are also listed in Table 5. Evidently, the strength properties of the papers were higher for the bleached pulp from the NaOH-based peroxide process than those from the $Mg(OH)_2$-based process.

Table 5. Properties of Papers Made of Bleached Acid-washed or Bleached Chelated Pulp

$Mg(OH)_2$, %	Tensile index, Nm/g		Burst index, $kPam^2/g$		Tear index, Nm^2/kg		Ref.
	Acid washed	Che-lated	Acid washed	Che-lated	Acid washed	Che-lated	
1%, $Mg(OH)_2$	11.2	15.9	0.47	0.52	1.41	1.52	PS[1]
1.5%, $Mg(OH)_2$	11.0	18.3	0.43	0.54	1.40	1.74	PS
2%, $Mg(OH)_2$	10.3	16.0	0.41	0.50	1.71	1.72	PS
2.1%, NaOH	-	24.1	-	0.68	-	2.01	PS
2.25%, NaOH	-	36.5	-	2.24	-	6.7	Zeinaly et al. 2009
1.8%, $Mg(OH)_2$	-	35.8	-	2.22	-	6.72	Zeinaly et al. 2009

$Mg(OH)_2$, %	Bulk, cm^3/g		Opacity, %		Brightness, %ISO		Ref.
	Acid washed	Che-lated	Acid washed	Che-lated	Acid washed	Che-lated	
1%, $Mg(OH)_2$	3.4	3.3	87.7	87	63.4	69.4	PS[1]
1.5%, $Mg(OH)_2$	3.0	3.0	86.8	86.3	65.2	70.8	PS
2%, $Mg(OH)_2$	3.2	3.4	87.3	86.9	65.0	69.7	PS
2.1%, NaOH	-	2.1	-	80.0	-	70.1	PS
2.25%, NaOH	-	2.39	-	-	-	74.2	Zeinaly et al. 2009
1.8%, $Mg(OH)_2$	-	2.4	-	-	-	75.9	Zeinaly et al. 2009

[1]: present study

Acid-Washing versus Chelation

The results in Table 2 showed that the chelation was more effective than acid-washing for removing the harmful transition metal ions from the pulp; therefore, the peroxide oxidation/bleaching would be expected to be more effective for the DTPA chelated pulp than for the acid-washed pulp. As a result, the bleaching yield was lower for the pulp pretreated with DTPA chelation (Table 3), and the bleaching effluent for the DTPA chelated pulp had higher COD and turbidity (Table 4). The post-treatment of bleaching effluent may be a bottleneck for some mechanical pulp mills, which affects their production capacity. Therefore, if acid-washing is used as the pretreatment step prior to peroxide bleaching, a lower load is induced to the bleaching effluent, thus a higher production capacity would be obtained in the mills. Additionally, the higher bleaching yield of acid-washed pulp will also contribute to the increase in the production capacity of the mill (Table 3). However, the optical and strength properties of pulp may suffer to some extent (Table 5), which should be considered.

CONCLUSIONS

1. The results showed that the magnesium removal from unbleached pulp was greater in an acid-washing stage than in a chelation stage, while the manganese removal from the pulp was more in a DTPA chelation stage.
2. For the $Mg(OH)_2$-based peroxide bleaching, a higher yield and WRV were obtained for the acid-washed pulp than for the chelated pulp; however, the COD and turbidity of the bleaching filtrates for the acid-washed pulp were lower than those for the chelated pulp; additionally, the bleached acid-washed pulp had lower strength properties than bleached chelated pulp.
3. A higher yield was obtained for the $Mg(OH)_2$-based peroxide bleaching than for the NaOH-based peroxide bleaching at a brightness target of 70 %ISO. Correspondingly, the turbidity and COD of the filtrates of the $Mg(OH)_2$-based peroxide bleaching were lower than those of the NaOH-based peroxide bleaching, whereas the strength properties of the NaOH-based bleached pulp were higher than those of the $Mg(OH)_2$-based bleached pulp.

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