

The Use of Mg(OH)₂ in the Final Peroxide Bleaching Stage of Wheat Straw Pulp

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Magnesium-based alkali is an attractive alkaline source for the peroxide bleaching of high-yield pulp. However, little information is available on Mg(OH)₂ application in the final peroxide bleaching stage of wheat straw pulp. The use of Mg(OH)₂ was demonstrated as a partial replacement for NaOH in the peroxide bleaching of a chelated oxygen-delignified wheat straw pulp. The yield, viscosity, and strength properties of bleached pulp significantly increased with increasing replacement ratio of Mg(OH)₂, while the chemical oxygen demand load (COD) of filtrate was decreased. For similar brightness of bleached pulp at a 24% replacement ratio of Mg(OH)₂, the tensile and tear indices were higher, by 2.1 Nm.g⁻¹ and 1.75 mN*m².g⁻¹, respectively, than that of control pulp bleached with NaOH as the sole alkaline source. When the MgSO₄ was eliminated and the dosage of Na₂SiO₃ was decreased in the bleaching process, the tear and burst indices of the bleached pulp were also enhanced, with the brightness maintained. Scanning electron microscopy (SEM) showed that more swelling occurred in the fibers of bleached pulp from the Mg(OH)₂-based bleaching process. Fiber analysis indicated that peroxide bleaching with Mg(OH)₂ increased the proportion of fiber lengths between 0.20 to 1.20 mm and 1.20 to 7.60 mm.

Keywords: Mg(OH)₂; Wheat straw pulp; Peroxide bleaching; Strength properties

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INTRODUCTION

Paper is a fundamental part of modern society, and its use is expected to continue to increase. Due to environmental damage and protection of forestlands, papermakers, especially in countries with a shortage of forest resources, are searching for alternative fiber sources, such as bagasse, bamboo, and wheat straw. Agricultural residuals such as wheat straw continue to be a main alternative fiber source based on their high growth rate and large production in Asia, Europe, and America. Meanwhile, studies and practices have indicated that wheat straw is a valuable raw material for manufacturing paper (Zhang *et al.* 2011; Roncero *et al.* 2003). Countries with a shortage of pulpwood, for example China, India, Mexico, and Egypt, are increasingly using wheat straw in commercial pulping applications. In North America, though there are abundant forest resources, using straw for papermaking is also proposed to be a profitable disposal method, because burning straw is harmful to the environment. Moreover, plowing the straw back into the ground is labor- and cost-intensive.

Pulp bleaching with hydrogen peroxide is typically carried out under alkaline conditions. The dissociation of hydrogen peroxide in alkaline media can be expressed as

follows: $\text{H}_2\text{O}_2 + \text{HO}^- \rightarrow \text{HOO}^- + \text{H}_2\text{O}$. The hydrogen peroxide anion (HOO^-) is mainly responsible for the reduction in concentration of chromophores. The alkali source used in hydrogen peroxide bleaching is conventionally sodium hydroxide (NaOH). However, it has been indicated that the use of NaOH as a bleaching auxiliary has undesired side effects in production practice. NaOH can cause the dissolution of carbohydrates into the bleach effluent and lead to increased COD and BOD load to the secondary treatment system. Also, the high alkalinity of the process leads to the formation of a significant amount of anionic trash, which is carried onto the wet-end of the paper machine. This can have negative consequences on papermaking operations, such as increased polymer/additive costs, reduced drainage, and decreased product quality (Ni 2005). In recognition of the drawbacks associated with the NaOH-based process, peroxide bleaching using magnesium hydroxide ($\text{Mg}(\text{OH})_2$) has received a lot of recent attention. In fact, studies performed by many researchers on various wood species have shown very interesting results (Zeinaly *et al.* 2009; He *et al.* 2008; Li *et al.* 2004). $\text{Mg}(\text{OH})_2$ in a peroxide bleaching process brings many benefits for pulp and paper mills, including a lower chemical oxygen demand, less formation of anionic trash, lower oxalate scaling, and higher bulk of the bleached pulp. However, regarding the brightness and physical strength properties of bleached chemi-mechanical pulp, inconsistent results have been reported in the literature on the effect of replacing NaOH with $\text{Mg}(\text{OH})_2$. Nyström *et al.* (1993) showed that using $\text{Mg}(\text{OH})_2$ instead of NaOH as the alkaline source in the peroxide bleaching of stoneground spruce wood caused a decrease in tensile strength of the bleached pulp and an increase in brightness (Nyström *et al.* 1993). Zhang *et al.* observed that with maple chemi-thermomechanical pulp (CTMP), the $\text{Mg}(\text{OH})_2$ -based peroxide bleaching process gave a lower brightness and tensile strength of bleached pulp compared with that of the conventional NaOH-based process (Zhang *et al.* 2004). However, Li *et al.* reported that no significant change in the brightness or strength properties of the bleached (spruce/a small amount of fir) thermal mechanical pulp (TMP) occurred when replacing NaOH with $\text{Mg}(\text{OH})_2$ during the peroxide bleaching process (Li *et al.* 2005).

Recent research by the authors has indicated that $\text{Mg}(\text{OH})_2$ significantly improves the bleaching efficiency and selectivity of wheat straw soda-AQ pulp in a single peroxide bleaching stage (Liu *et al.* 2011); peroxide bleaching was performed in the last stage of a TCF bleaching sequence to increase the brightness and brightness stability of the ultimate pulp. Because the effect of $\text{Mg}(\text{OH})_2$ on a single peroxide bleaching stage of wheat straw soda-AQ pulp was found to be positive, the present paper further investigates the use of $\text{Mg}(\text{OH})_2$ in the final peroxide bleaching stage of a chelated oxygen-delignified (OQ) wheat straw soda-AQ pulp.

Compared to unbleached soda-AQ pulp of wheat straw, the contents of residual lignin and transition metals are lower in a chelated oxygen-delignified pulp. In addition, the reactivity of residual lignin decreased because the lignin undergoes condensation reactions under oxygen delignification conditions. It can be deduced that the residual lignin and transition metals have impacts on peroxide bleaching with $\text{Mg}(\text{OH})_2$ for a chelated-oxygen delignified (OQ) wheat straw pulp. There are two aspects of this problem to be addressed. The first question involves how partial substitution of NaOH with $\text{Mg}(\text{OH})_2$ may affect the characteristics of bleached pulp and filtrate. The second problem relates to the effect of $\text{Mg}(\text{OH})_2$ on the fiber morphology and fiber surface of bleached pulp. Therefore, the results will be discussed in terms of brightness, viscosity,

yield, strength properties, and fiber morphology parameters. Scanning electron microscopy (SEM) was used to observe the surface morphology of bleached pulp fibers.

EXPERIMENTAL

Materials

An oxygen-delignified wheat straw soda-AQ pulp was collected from the lab of the forest products business unit of Alberta Innovates Technology Futures, Alberta, Canada. The pulp had a kappa number of 4.76, a viscosity of $969 \text{ mL}\cdot\text{g}^{-1}$, and a brightness of 51.0% ISO. A 50% (W/W) H_2O_2 solution, 50% (W/W) NaOH solution, diethylenetriamine pentaacetic acid sodium salt (DTPA), and $\text{MgSO}_4\cdot 7\text{H}_2\text{O}$ were analytical reagents, and $\text{Mg}(\text{OH})_2$ powder was of USP/FCC grade. All of them were supplied by Fischer Scientific. The 40% (W/W) Na_2SiO_3 solution was supplied by EMD Chemicals, Inc. All chemical charges used in pulping and bleaching experiments were expressed as the percentage on a pulp oven dry (o.d.) basis.

Bleaching Methods

Chelation stage (Q): a chelating process with DTPA is used to decrease the metal ion content to maximize the peroxide consumption for the bleaching reaction. The metal chelation procedure prior to peroxide bleaching was performed for 60 min at 70°C and 3.0% consistency in a beaker with a stirring rate of 450 rpm. The DTPA charge was 0.3%. The initial pH was adjusted to 6.0 to 6.5 with diluted H_2SO_4 . After the chelation stage, the pulp was washed thoroughly with distilled water. The chelated oxygen-delignified pulp had a kappa number of 3.9, a viscosity of $965 \text{ mL}\cdot\text{g}^{-1}$, a brightness of 55.2% ISO, and an ash content of 0.62%.

Peroxide bleaching stage (P): the bleach liquors were prepared by adding, in order, the required amounts of sodium silicate, sodium hydroxide, diethylenetriamine pentaacetic acid sodium salt (DTPA), magnesium sulfate, and hydrogen peroxide solution to the beaker containing distilled water. Then, the mixed solution was added to the pulp in a polyethylene bag. When $\text{Mg}(\text{OH})_2$ was needed, it was added directly into the pulp before the bleach liquor. Good mixing was achieved with hand kneading for two minutes. The polyethylene bag was sealed and placed in a water bath for the desired retention time. The conditions of peroxide bleaching for the chelated oxygen-delignified (OQ) wheat straw pulp are listed in Table 1. The $\text{Mg}(\text{OH})_2$ replacement ratio was defined as the weight equivalent of $\text{Mg}(\text{OH})_2$ that replaced NaOH, on a total alkaline charge of 3.00% NaOH, and is reported here as percent replacement. After the required retention time, a liquid sample was obtained to determine the residual H_2O_2 and COD. The pulp slurry was washed thoroughly with distilled water.

Pulp Analyses

The brightness pads were prepared according to TAPPI T218 and were measured with a Technibrite Micro TB-1C. The viscosity was performed based on ASTM D1795-96. The lignin content was measured with the kappa number method according to TAPPI T236. The pulp freeness was determined according to PAPTAC C.1. A Kajaani 300 was used to measure the fiber morphology. The laboratory handsheets with unbleached and bleached pulps were prepared according to TAPPI T255. The tensile index, tear index, and burst index were measured according to GB/T 453-2002, GB/T455-2002, and DCP-

NPY 1200, respectively. The residual H_2O_2 and COD of the filtrate were determined according to PAPTAC J.16 and PAPTAC H.3, respectively.

Surface Analysis by SEM

Samples of laboratory sheets were made from the pulps for analysis. SEM images were obtained using a SF-3700 microscope (Hitachi Co. Ltd.), operated in secondary electron mode at a beam current of 100 mA and an accelerating voltage of 10 kV. Samples were coated with a gold film before scanning.

Table 1. Operating Conditions of Peroxide Bleaching for a Chelated Oxygen-Delignified (OQ) Wheat Straw Pulp

Mg(OH) ₂ replacement ratio (%)	Temperature (°C)	Time (min)	Consistency (%)	DTPA (%)	Silicate (%)	MgSO ₄ (%)	H ₂ O ₂ (%)	Initial pH
0(control)	85	240	10	0.20	3.00	0.50	4.00	10.74
24	85	240	10	0.20	3.00	0.50	4.00	10.67
48	85	240	10	0.20	3.00	0.50	4.00	10.33
24	85	240	10	0.20	2.00	0.50	4.00	10.62
24	85	240	10	0.20	2.00	0.00	4.00	10.65

RESULTS AND DISCUSSION

Brightness and Residual H_2O_2

Figure 1 compares the pulp brightness and the content of residual hydrogen peroxide in the filtrate for various magnesium hydroxide replacement ratios in the peroxide bleaching stage. The data show that a brightness of 83.3%ISO was achieved for bleached pulp at a 24% Mg(OH)₂ replacement ratio, which is comparable to that of the control (83.5%ISO). However, the brightness of bleached pulp at a 48% Mg(OH)₂ replacement ratio was 2.8% ISO lower than that of the control. The result was inconsistent with previous research on wheat straw soda-AQ pulp, which revealed a significant brightness enhancement with increasing Mg(OH)₂ replacement ratio from 24% to 73% (Liu *et al.* 2011).

Compared with wheat straw raw stock, the reactivity of residual lignin in a chelated oxygen-delignification pulp was low. Further oxidation of the chromophores in residual lignin required much higher activation energy of H_2O_2 . On the other hand, a change in pH value led to the formation of HOO^- , which may be responsible for the elimination of the chromophores during pulp bleaching. Due to the lower solubility and alkalinity of Mg(OH)₂, the initial pH value (10.33) at a 48% Mg(OH)₂ replacement ratio was lower than that of the control (pH=10.74), which limited the generation of HOO^- and reduced the activation energy of H_2O_2 . Therefore, the brightness of bleached pulp at a 48% Mg(OH)₂ replacement ratio was lower than that of the control.

Meanwhile, Fig. 1 also indicates that for a similar brightness of bleached pulp at 0% and 24% replacement ratios of NaOH with Mg(OH)₂, the residual H_2O_2 at a 24% Mg(OH)₂ replacement ratio was comparable to that of the control. When the brightness of bleached pulp decreased from 83.5% ISO to 80.7% ISO, the residual H_2O_2 was increased from 3.40 g/L to 3.83 g/L.

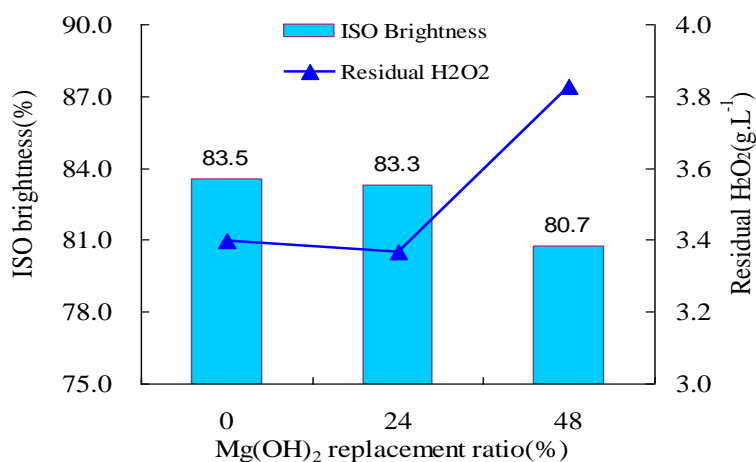


Fig. 1. Effect of Mg(OH)₂ replacement ratio on the pulp brightness and residual H₂O₂ content in the filtrate

Viscosity and Yield

Mg(OH)₂ had a positive effect on the viscosity and yield of wheat straw OQ pulp, as shown in Fig. 2. The viscosity and yield of bleached pulp increased consistently with increasing Mg(OH)₂ replacement ratio. At a 48% Mg(OH)₂ replacement ratio, when the brightness of bleached pulp was 2.8% ISO lower than that of the control, the viscosity and yield of bleached pulp were higher by 112 mL.g⁻¹ and 2.9%, respectively. Compared with the bleached pulp from the Mg(OH)₂-based bleaching process, the yield loss of the control mainly came from the degradation of carbohydrates because the kappa number of wheat straw OQ pulp was low (just 3.9), equal to 0.5% lignin content. A sounder explanation was that the decomposition of H₂O₂ in the higher pH of NaOH-based peroxide bleaching stage produced hydroxyl radicals (OH·), which then oxidized the polysaccharides, and the oxidized polysaccharides underwent chain cleavage by beta-elimination and subsequent degradation reactions. Hence, partial substitution of Mg(OH)₂ for NaOH promoted carbohydrate protection and the yield of bleached pulp.

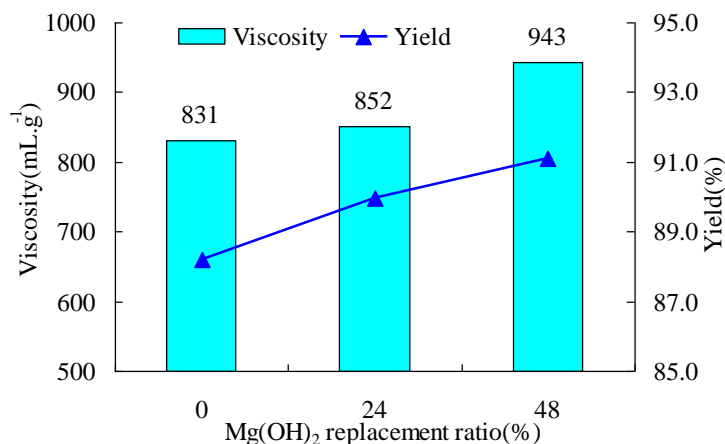


Fig. 2. Effect of Mg(OH)₂ replacement ratio on the pulp viscosity and yield

COD

As indicated by Fig. 3, the COD load of bleaching filtrates at 24% and 48% $\text{Mg}(\text{OH})_2$ replacement ratios were lower by 22% to 31% than that of the control. The milder alkalinity of the $\text{Mg}(\text{OH})_2$ system was believed to be the cause of the lower COD formation. The high COD value of filtrate from the control was attributed to the strong alkalinity of NaOH, with a higher pH than hydrolyzed carbohydrates, which caused the dissolution of carbohydrates, particularly the hemi-celluloses, into the bleach effluent and was responsible for the increasing COD load in the secondary treatment system. Consequently, the costs associated with effluent treatment would be significantly decreased as a result of the substantial reduction of COD.

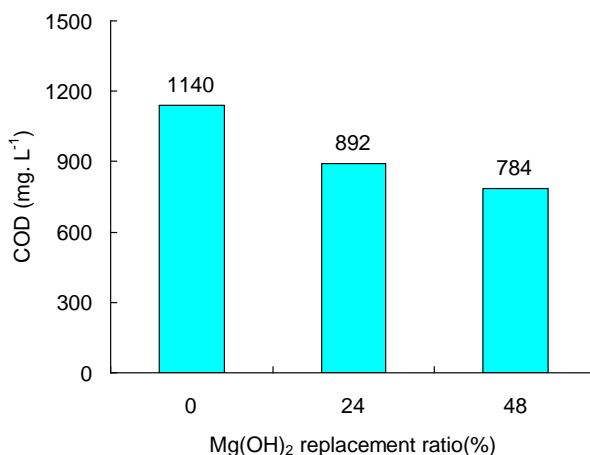


Fig. 3. Effect of $\text{Mg}(\text{OH})_2$ replacement ratio on the COD of the filtrate

Pulp Strength Properties

A comparison of the strength properties of the two bleached pulps is shown in Table 2. For a similar brightness of bleached pulp at a 24% $\text{Mg}(\text{OH})_2$ replacement ratio, the tensile index and tear index were higher than that of the control by $2.1 \text{ Nm}\cdot\text{g}^{-1}$ and $1.75 \text{ mN}\cdot\text{m}^2\cdot\text{g}^{-1}$, respectively. However, other researchers reported that the strength properties of wood bleached mechanical pulp remained unchanged or decreased slightly in the $\text{Mg}(\text{OH})_2$ -based peroxide bleaching process, compared with that from the conventional NaOH-based bleaching process (Zhang *et al.* 2004; He *et al.* 2006). The reasons for this difference are that the peroxide bleaching of wood mechanical pulp was a procedure of strength enhancement due to the lignin dissolution under alkalinity conditions. The milder alkalinity of $\text{Mg}(\text{OH})_2$ gave rise to much lower dissolved lignin, leading to decreased fiber swelling and fiber-fiber bonding, thus limiting the strength enhancement of bleached pulp from the $\text{Mg}(\text{OH})_2$ -based bleaching process (He *et al.* 2006). However, for a chelated oxygen-delignified wheat straw pulp, bleaching reduced the strength because of the degradation of carbohydrates (Hedjazi *et al.* 2009). The milder alkalinity of $\text{Mg}(\text{OH})_2$ reduced the degradation of carbohydrates, especially the hemicelluloses, as indicated by the increased yield of bleached pulp and the COD reduction of the filtrate from the $\text{Mg}(\text{OH})_2$ -based bleaching process. The high hemicellulose content of chemical pulp has a beneficial effect on interfiber bonding, resulting in dense paper sheets with high strength properties, particularly for tensile strength (Kordsachia and Patt 1988).

Table 2. Effect of Mg(OH)₂ Replacement Ratio on the Strength Properties of Bleached Pulp

Mg(OH) ₂ replacement ratio (%)	Freeness (mL)	Tensile index (Nm.g ⁻¹)	Tear index (mN*m ² .g ⁻¹)	Burst index (Kpa*m ² .g ⁻¹)
OQ pulp	366	74.1	4.54	4.75
0(control)	478	40.0	5.77	2.48
24	432	42.1	7.52	2.58
48	421	43.9	7.85	2.80

Fiber Morphology

The differences in fiber morphologies are important factors influencing the physical properties of pulp (Guo *et al.* 2009). The fiber morphology of bleached pulp was measured to illustrate the increase in the tensile and tear indexes. As shown in Table 3, the average fiber dimensions, *i.e.*, weighted fiber length and fiber width of the control resembled those from the Mg(OH)₂-based bleaching process. However, the distribution proportion of fiber length between 0.20 and 1.20 mm increased with Mg(OH)₂ replacement ratio. The proportion of fiber lengths between 1.20 and 7.60 mm decreased with Mg(OH)₂ replacement ratio. Hence, the fiber proportion between 0.20 to 1.20 mm and 1.20 to 7.60 mm increased with the Mg(OH)₂ replacement ratio. The increase was significant enough to cause some differences in the properties of paper derived from these pulps. When the average length of the fibers is similar, improved physical properties of bleached pulp are obtained with increasing medium-length fibers.

Pulp fines also play an important role in the development of paper strength properties, and their functions in improving bonding and related mechanisms have been recognized and explained extensively in the literature (Kang and Paulapuro 2006; Lin *et al.* 2007). Table 3 also makes it clear that the fines of bleached pulp increased with the Mg(OH)₂ replacement ratio. The curl index of fibers became larger, while the kink index of fibers became smaller. All of these factors may promote the strength properties of handsheets made from bleached pulp with Mg(OH)₂.

Table 3. Effect of Mg(OH)₂ Replacement Ratio on the Fiber Morphology of Bleached Pulp

Mg(OH) ₂ Replacement Ratio (%)	0	24	48
Weight length L_w (mm)	1.07	1.07	1.05
Weight width W_w (um)	15.77	15.95	15.68
Curl index (%)	16.92	17.02	17.30
Kink index (1.m ⁻¹)	3479.3	2317.1	2297.8
Fines (%)	29.82	34.63	38.74
Fiber length (0.20 to 0.50 mm)	11.1	13.5	14.7
Fiber length (0.50 to 1.20 mm)	29.6	33.5	34.6
Fiber length (1.20 to 3.20 mm)	54.1	49.9	48.0
Fiber length (3.20 to 7.60 mm)	3.8	1.3	0.7
Fiber length (0.20 to 1.20mm) / Fiber length (1.20 to 7.60 mm)	0.70	0.92	1.01

Scanning Electron Microscopy

In Figure 4, the surface morphology of pulp fibers from the oxygen-delignified pulp without peroxide bleaching and the peroxide bleached pulps both with NaOH-based and Mg(OH)₂-based processes is illustrated. As for the initial pulp without peroxide

bleaching in photograph (a), the surface morphology looks smooth and has less fibrillation. The fibers of bleached pulp from the NaOH-based process in photograph (b) are rougher and more heterogeneous, with small filaments on the surface. This means that the fibers were damaged during the peroxide bleaching process. The same phenomena are seen in photograph (c), which shows the pulp bleached at a 48% $\text{Mg}(\text{OH})_2$ replacement ratio. However, the cross-section contraction of the fiber surface was more noticeable in photograph (c), which means that more swelling occurred in the fiber structure, resulting in higher bonding strength properties of bleached pulp. The milder alkalinity of $\text{Mg}(\text{OH})_2$ promotes the protection of hemicelluloses. The bleached pulp fibers from the $\text{Mg}(\text{OH})_2$ -based process were more swollen than those from the control.

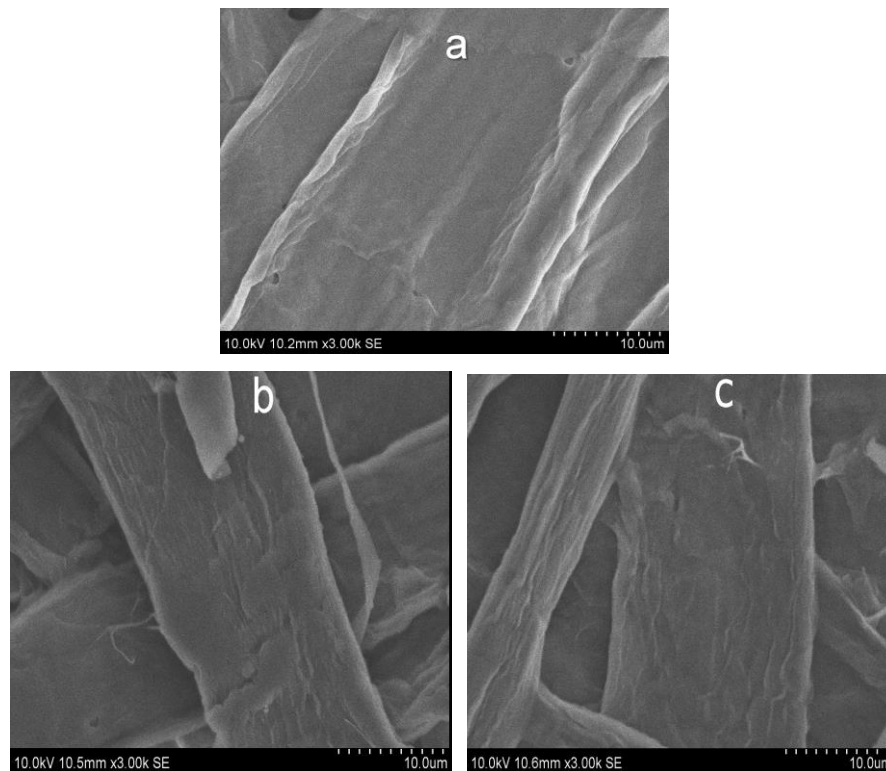


Fig. 4. SEM photographs of wheat straw pulp: (a) chelated oxygen-delignified pulp; (b) pulp bleached with NaOH as the sole alkaline source; (c) pulp bleached at a 48% $\text{Mg}(\text{OH})_2$ replacement ratio

Na_2SiO_3 and MgSO_4

In the peroxide bleaching stage, MgSO_4 was added as a carbohydrate protector, and Na_2SiO_3 provided alkalinity and pH buffering. It is known that $\text{Mg}(\text{OH})_2$ provides pH buffering, and $\text{Mg}(\text{OH})_2$ has both a functional anion (OH^-) and cation (Mg^{2+}) to provide alkalinity and cellulose protection. Because $\text{Mg}(\text{OH})_2$ can supply an abundance of Mg^{2+} ions in addition to OH^- ions, the dosage of Na_2SiO_3 was decreased and MgSO_4 was eliminated from the 24% $\text{Mg}(\text{OH})_2$ substitution bleaching stage. As shown in Table 4, compared with the control, when the dosage of Na_2SiO_3 was decreased from 3.00% to 2.00% and MgSO_4 was eliminated from the bleaching stage, a comparable brightness of bleached pulp was achieved, but with a higher tear index and burst index. Compared with sample 1# indicated in Table 4, the brightness of bleached pulp changed little, the

viscosity decreased from 852 to 751 mL/g, and the tear index decreased from 7.52 to 6.45 mN*m²/g, but the burst index increased from 2.80 to 3.09 kPa*m²/g.

Table 4. Effect of Mg(OH)₂ with Various Doses of Na₂SiO₃ and MgSO₄

Samples	Mg(OH) ₂ replacement ratio(%)	Silicate (%)	MgSO ₄ (%)	ISO Brightness (%)	Viscosity (mL.g ⁻¹)	Tear Index (mN*m ² /g)	Burst Index (kPa*m ² /g)
control	0	3.00	0.50	83.5	831	5.44	2.48
1#	24	3.00	0.50	83.3	852	7.52	2.58
2#	24	2.00	0.50	82.5	786	6.94	2.80
3#	24	2.00	0.00	82.6	751	6.45	3.09

CONCLUSIONS

1. For a chelated oxygen-delignification wheat straw pulp, a brightness of 83.3% ISO was achieved at a 24% Mg(OH)₂ replacement ratio, which was similar to pulp bleached with a 100% NaOH charge (83.5% ISO). The yield, viscosity, and strength properties of bleached pulp significantly increased with the Mg(OH)₂ replacement ratio. The COD load of bleaching filtrates from 24 to 48% Mg(OH)₂ replacement ratios was lower by 22 to 31% than that from bleaching using NaOH as the sole alkaline source. The lower solubility and alkalinity of Mg(OH)₂ was partly responsible for this favorable effect on the final peroxide bleaching of wheat straw chelated oxygen-delignification pulp.
2. Scanning electron microscopy (SEM) showed that more swelling occurred in the fibers of bleached pulp from the Mg(OH)₂-based bleaching process. Fiber analysis indicated that Mg(OH)₂ increased the proportion of fiber lengths between 0.20 to 1.20 mm and 1.20 to 7.60 mm. These were proposed to be responsible for the favorable effect of Mg(OH)₂ on the strength properties of bleached pulp.
3. When the dosage of Na₂SiO₃ decreased from 3.00% to 2.00% and MgSO₄ was eliminated from the 24% substitution of NaOH with Mg(OH)₂, the tear index and burst index of bleached pulp were enhanced, respectively, by 1.01 mN*m²/g and 0.61 kPa*m²/g compared to that of pulp bleached with a 100% NaOH alkaline source.

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