

HYDROGEN PEROXIDE BLEACHING OF TMP PULPS USING $Mg(OH)_2$

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ABSTRACT

A brief review of the chemistry of peroxide bleaching with $Mg(OH)_2$ is given, and contrasted to conventional peroxide bleaching using sodium silicate and sodium hydroxide as the base. The results of laboratory experiments are also presented comparing conventional and $Mg(OH)_2$ bleaching of TMP pulp. When both the caustic and silicate were replaced with an optimal level of $Mg(OH)_2$, the pulp brightness equaled or exceeded the control bleaching at the same peroxide charge. At equivalent brightness, the residual hydrogen peroxide in the $Mg(OH)_2$ /peroxide bleaching stage was higher than for the control caustic/silicate/peroxide bleaching and suggests the possibility of recycling of peroxide for greater efficiency. The $Mg(OH)_2$ /peroxide bleaching stage also had lower COD and lower conductivity in the filtrate.

INTRODUCTION

Mechanical pulps are produced from wood using mechanical means only. There are four mechanical pulping processes in use commercially; stone groundwood (SGW), pressurized stone groundwood (PSGW), refiner mechanical (RMP) pulping, and thermomechanical (TMP) pulping. The stone groundwood process, and its variant, the pressurized stone groundwood process, use wood bolts, while the refiner mechanical and thermomechanical pulping processes use chips. In terms of productive capacity, the stone groundwood process is the leading process. However, it is rapidly being replaced by the thermomechanical pulping process, because there are distinct economies that arise from using chips rather than wood bolts, and because the resultant thermomechanical pulp is inherently stronger. In mechanical pulping, no active chemical other than water is used to facilitate fiber liberation. Virtually all of the chemical constituents in the wood are retained in the pulp. Mechanical pulps are bleached using two predominant bleaching agents, sodium dithionite in reductive bleaching, and hydrogen peroxide in oxidative bleaching.

Potentially, $Mg(OH)_2$ could find a place in the mechanical pulping industry. Peroxide bleaching was chosen as the subject of this work because it is performed under alkaline conditions, and is thus compatible with the alkaline nature of magnesium hydroxide $Mg(OH)_2$.

OBJECTIVES AND SCOPE

The objective of this study is to examine the use of $Mg(OH)_2$ as a replacement for NaOH and/or sodium silicate as an alkali source in peroxide bleaching of mechanical pulp. $Mg(OH)_2$ is a weaker base than sodium hydroxide, but may be cost competitive especially if it can be shown to have multiple benefits. The work of Vincent et al. (1997) has shown that magnesium oxide (MgO) can be used economically in bleaching chemi-mechanical pulp produced using the cold soda process. It needs to be shown that $Mg(OH)_2$ has the same potential.

Hydrogen peroxide bleaching experiments were performed on hardwood TMP pulps from two northeastern mills using standard mill conditions, and then magnesium hydroxide was substituted for silicate and caustic in the bleaching process.

RELATED LITERATURE

A search of technical literature on the use of magnesium compounds in the bleaching of high yield or thermo-mechanical pulp (TMP) revealed that limited research has been conducted and published in this field.

Soteland (1988) at the Norwegian Pulp and Paper Institute studied the replacement of caustic soda and sodium silicate with either MgO or CaO in the bleaching of groundwood and chemi-thermo-mechanical pulp (CTMP). The brightness gain was about one point lower with MgO than with either CaO or the sodium compounds. However, the particle size of the MgO was quite large, which no doubt contributed to this result. The lower brightness gain could be offset in the case of CTMP if bleaching was carried out at high consistency (30%). Soteland makes the conclusion that "Use of divalent bases in peroxide bleaching increases the yield and reduces pollution." However, no data or proof is offered within the paper.

Maughan and associates (1992) at Australia Newsprint Mills studied the use of MgO as an alkali source in refiner bleaching of pine TMP pulp, with a 1% peroxide dosage. One of their objectives was to determine if lower dissolved solids in the effluent would result from the substitution of MgO for NaOH. They found that a reduction of 37% in dissolved solids could be obtained by substituting 0.5% MgO for 0.8% NaOH. Their results also indicated the importance of retaining the pulp consistency at 25% for 30 minutes to obtain full brightness gain.

A second paper involving the same mill (Griffiths and Abbot, 1993) evaluated several local sources of MgO in comparison with technical grade MgO. The local magnesite ore does not have high purity (94 to 96% MgO and significant silica). The conclusion of this study was that MgO could not match NaOH as an alkali source.

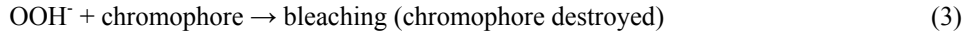
Vincent, Rizzon, and Zooeff (1997) carried out work on a pulp process they call "CCS pulp", which is essentially CTMP. In their study, they replaced caustic soda and sodium silicate with MgO and DTPA. Their work stressed the need for a two stage DTPA treatment - a pre-treatment directly to the pulp to tie up transition metals from the wood fiber, and a second dosage with the peroxide bleaching liquor to contain any metals entering with the MgO or Mg(OH)₂ source. One interesting finding was the deterioration in brightness gain when silicate was combined with MgO. Mill trials were conducted, and confirmed the lab results that the substitution of MgO for NaOH reduced the peroxide requirement by 1% on pulp to obtain target brightness (from 3.1% on pulp to 2.1%). An assessment of cationic content was also made. The test determined the amount of retention aid chemical required to neutralize the bleached pulp. Using MgO/DTPA bleaching, the average retention chemical requirement was reduced by 50% in comparison to NaOH/ silicate bleaching.

Nystrom, Pykalainen, and Lehto (1993) examined the peroxide bleaching of SGW spruce with alternate alkalis that would not require silicate for stabilization, including CaO, Ca(OH)₂, MgO, Mg(OH)₂, and NaHCO₂. In lab work, they found that at the same chemical charge, Mg(OH)₂ could give better results than NaOH, even without silicate. Silicate could improve results with Mg(OH)₂. Alkalis other than caustic gave lower handsheet tensile index values, which they attribute to the probable reduction in crosslinking between fibers, which correlated somewhat with the increased freeness of the alternative alkali pulps. All alternative alkalis resulted in lower COD values. They attribute the high COD values of NaOH to its being a strong alkali, with a high pH, which hydrolyzes the long-chain molecules in the pulp to smaller ones. An advantage of the Mg alkalis was their low solubility, resulting in a lower pH. Pilot scale trials were also carried out, with very similar results to the lab work. Nystrom et al. (1993) concluded that Mg(OH)₂ was a good alternative to NaOH, giving good brightness results, and a substantial COD reduction.

Suess and associates (2001) examined the use of alternative sources of alkali in the peroxide bleaching of mechanical pulps, including sodium carbonate, magnesium oxide, and magnesium hydroxide. Sodium carbonate could replace sodium hydroxide over a narrow range, resulting in similar brightness levels and a 20% reduction in COD. Magnesium hydroxide could replace both sodium hydroxide and silicate, with similar brightness results. At very high peroxide charges, Mg(OH)₂ gave somewhat poorer brightness results than caustic, and required slightly higher charges of peroxide to achieve equivalent brightness. A small amount of silicate was found to improve the Mg(OH)₂ results, but Mg(OH)₂ was much less sensitive to silicate than NaOH. The researchers found that the final pH must be above 7 or the reaction stops. Mg(OH)₂ as the alkali source resulted in higher peroxide residual, indicating fewer side reactions than with caustic. However, this residual could not be used to increase brightness response by either increasing reaction time or temperature. COD generation was reduced by more than 30% with the use of Mg(OH)₂. Mg(OH)₂ was found to be an attractive alternative to NaOH, since conventional conditions could be used, the COD was substantially decreased, a smaller charge of Mg(OH)₂ was needed than with NaOH for the same brightness, and the silicate charge could be reduced.

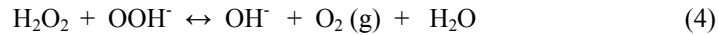
REACTIONS DURING $Mg(OH)_2$ /PEROXIDE BLEACHING

Magnesium hydroxide reacts with hydrogen peroxide to produce the perhydroxyl ion (OOH^-) needed for the bleaching reaction as follows:



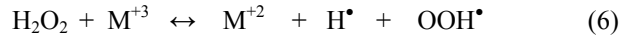
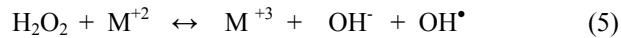
$Mg(OH)_2$ dissociates to produce hydroxyl ions (OH^-). The hydroxyl ion reacts with the peroxide to produce perhydroxyl ion. The perhydroxyl ion in turn reacts with the colored chromophore groups, destroying the group, and therefore bleaching the pulp. $Mg(OH)_2$ is attractive as an alkali source in peroxide bleaching because it is a solid and would be released slowly into the system, thus acting as a buffer to prevent rapid changes in pH.

Other competing reactions can take place which would consume peroxide without providing bleaching effects. Peroxide can be decomposed by the perhydroxyl ion with the following reaction:

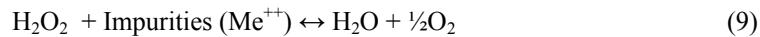


This can happen when reaction (1) proceeds to the right so rapidly that an excess of hydroxyl ions is present. For $Mg(OH)_2$ at optimal levels, this reaction may proceed more slowly than with caustic, since the solubility of $Mg(OH)_2$ is low, therefore limiting the OH^- available. This should result in reaction (2) being reduced, and enhancing reaction (3), the bleaching reaction. Increasing the reaction temperature would also drive these reactions.

Peroxide can also be decomposed by excess impurities, in the form of multivalent metal ions, as in the following reactions from Presley and Hill (1996):

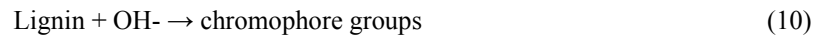


or more simply as:



Again, the peroxide would be consumed without bleaching taking place.

Pulp can also darken due to alkali darkening reactions, simply described as:



where phenolic lignin units may be oxidized by alkali to light absorbing structures. Allison and Graham (1990) found that peroxide bleached pulp could show alkali darkening at the final bleaching pH of ~9 if all of the active peroxide was consumed. The peroxide potentially can create and destroy the precursors to alkali-generated chromophores, and also eliminate them once they are formed.

Figure 1 shows a simplified representation of some of the various competing reactions taking place during bleaching. Increasing the peroxide charge will result in increasing brightness, to a maximum value. Increasing alkali at a peroxide level will also result in increased brightness up to a maximum, after which the brightness will decrease. This may be due to the excess alkali driving reaction (1) to the right, increasing hydroxyl ion, which in turn would drive reaction (2), producing perhydroxyl ion too rapidly to be used. Therefore, reaction (3) would proceed, decomposing the peroxide before it was used in bleaching. Increasing metals content would generally

result in lower brightness, since reactions (5) to (8) would proceed to decompose peroxide without providing bleaching effects.

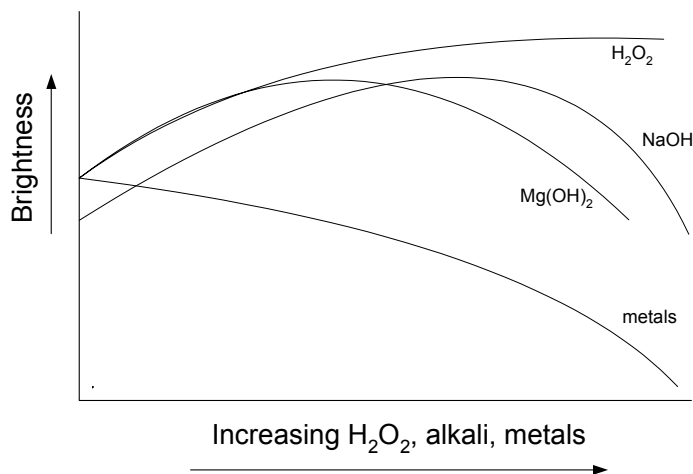


Figure 1. Competing reactions during peroxide bleaching.

PROCEDURES

Softwood TMP sampled after refining was obtained from two northeast mills, Pulp A and Pulp B. In both cases, the species used in producing the TMP were predominantly eastern spruce and balsam fir. The brightness of the brownstock was 62.0 % ISO for the Pulp A, and 63.5% for Pulp B pulp. The magnesium hydroxide used was supplied by Martin Marietta Magnesia Specialties Inc. at 3 μ m particle size, 9 ppm Fe content, and was in slurry form.

The conditions used in bleaching followed mill conditions as closely as practicable for the control case. Bleaching experiments were performed using a chelation stage followed by a hydrogen peroxide stage. The pulp was then washed and tested. General bleaching conditions are found in Table 1.

Table 1. Bleaching Conditions.

	Mill Condition (Control) Experiments	Mg(OH) ₂ Experiments
Chelation Stage (10% consistency, 70°C, 30 min)		
% DTPA	0.3	0.2
Pulp squeezed to ~25% consistency		
Hydrogen Peroxide Stage (10% consistency, 70°C, 2 hrs Pulp A, 5 hrs Pulp B)		
% sodium silicate	3	--
% NaOH	1.5	--
% DTPA	--	0.1
% H ₂ O ₂	2	Varied
% Mg(OH) ₂	--	Varied
Pulp stirred with 10l total of water, squeezed to ~25%		

* all chemical charges as % on dry pulp

RESULTS

Preliminary work involved Pulp A. A series of runs was done where the Mg(OH)₂ was kept at 0.5%, and the NaOH charge was varied, but no silicate was added. Increasing the caustic with Mg(OH)₂ in the system worsened the results. With just Mg(OH)₂ in the system, the brightness approached that of the control system (Figure 2). Other

experiments, not shown here, have shown that adding silicate to the $Mg(OH)_2$ system can improve the brightness results.

The peroxide residuals for these experiments, shown in Figure 3, show the residual for the $Mg(OH)_2$ /peroxide system to be much higher than in the control $NaOH$ /silicate/peroxide system. As seen in Figure 4, the initial pH of the $Mg(OH)_2$ /peroxide system is lower than the control system or the $NaOH/Mg(OH)_2$ system. In the $Mg(OH)_2$ system, the initial pH is below the final pH, while in the control and $NaOH/Mg(OH)_2$ systems the pH drops during the retention time. The $Mg(OH)_2$ system final pH is somewhat lower than the final pH of the control or $NaOH/Mg(OH)_2$ system. With the $Mg(OH)_2$ /peroxide system, bleaching takes place at a lower pH than with the conventional system, and the pH changes less during bleaching.

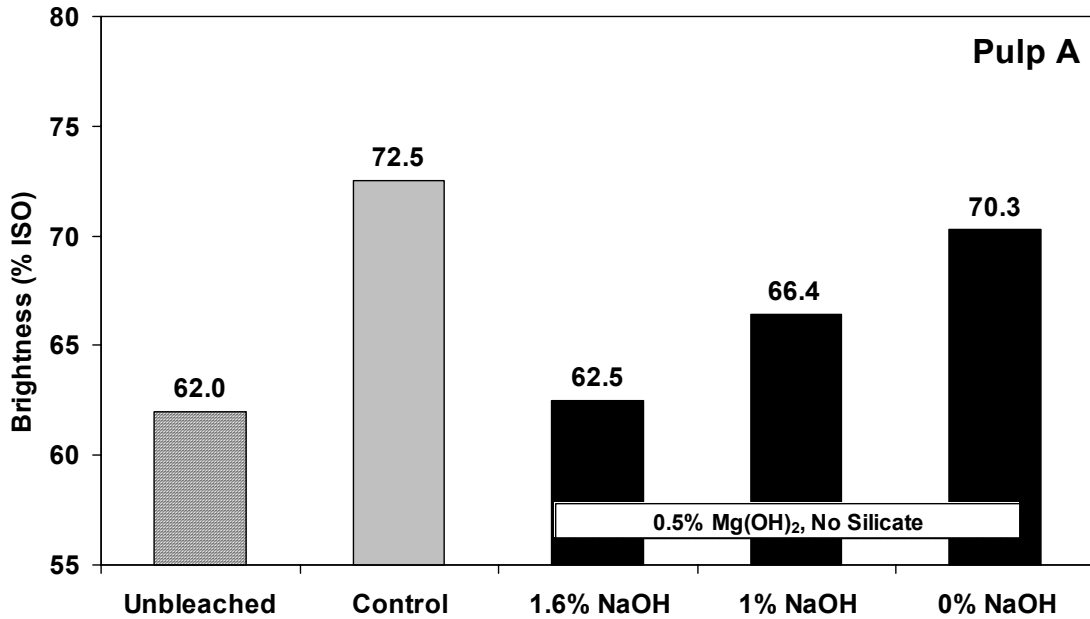


Figure 2. Pulp A brightness results.

Control - Q: 0.3% DTPA; P: 2% peroxide, 3% silicate, 1.6% NaOH

$Mg(OH)_2$ - Q: 0.2% DTPA; P: 0.1% DTPA, 0.5% $Mg(OH)_2$, 2% peroxide, various NaOH.

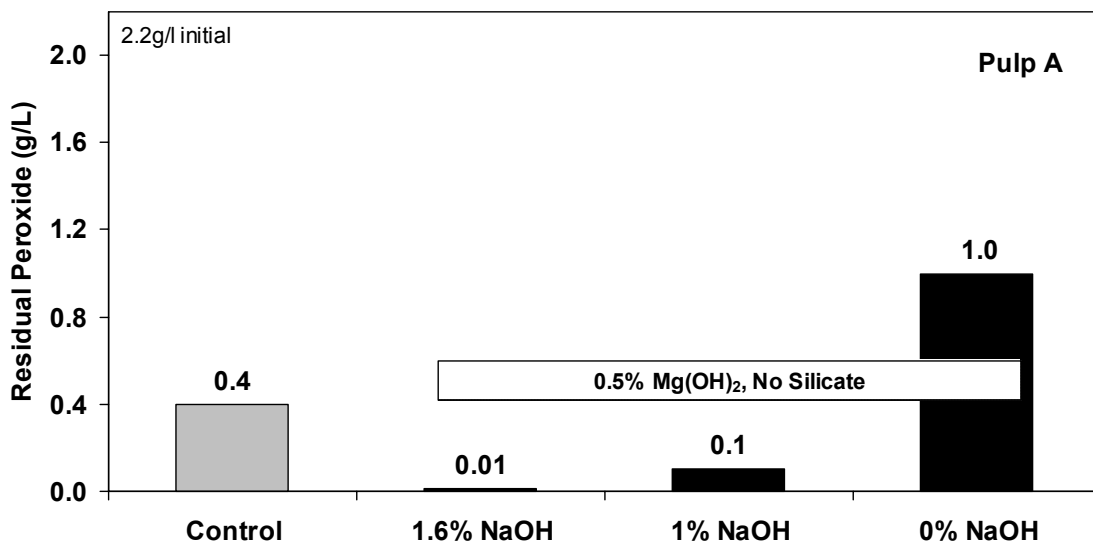


Figure 3. Pulp A residual peroxide results. (see Figure 2 caption for conditions)

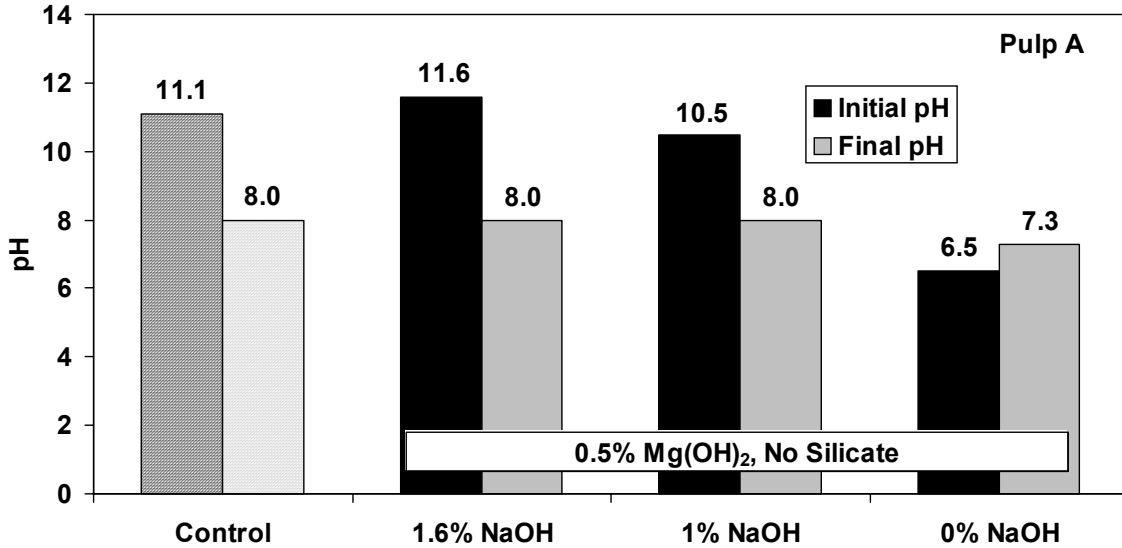


Figure 4. Pulp A initial and final pH. (see Figure 2 caption for conditions)

After these preliminary experiments, an $Mg(OH)_2$ response curve was done on Pulp A, using only $Mg(OH)_2$ and peroxide in the bleach liquor. As seen in Figure 5, an optimum brightness value was obtained at 0.5% charge of $Mg(OH)_2$, with the brightness decreasing with additional $Mg(OH)_2$. For this pulp, this optimum brightness was 70% ISO, which was lower than the conventional bleaching system. Brightness of both the control and the $Mg(OH)_2$ bleaching could be increased by increasing peroxide charge.

The peroxide residual, shown in Figure 6, shows a steady decrease in residual with increasing $Mg(OH)_2$ charge. The residual at the optimal brightness level with the $Mg(OH)_2$ was much higher than for the control. The initial pH is much lower for the $Mg(OH)_2$ bleaching than for the conventional bleaching (Figure 7). It increases with increasing $Mg(OH)_2$ charge. The final pH also increases with increasing $Mg(OH)_2$ charge, and at higher $Mg(OH)_2$ charges, approaches the conventional bleaching final pH. However, when the $Mg(OH)_2$ final pH value meets the conventional final pH value, the $Mg(OH)_2$ level is higher than optimal for brightness.

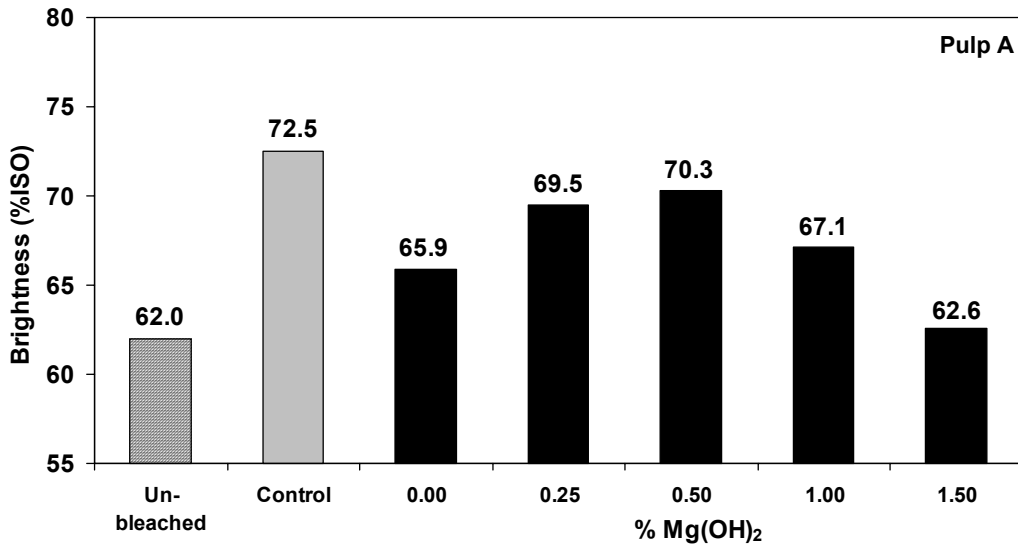


Figure 5. Pulp A brightness results, $Mg(OH)_2$ response curve.
 Control - Q: 0.3% DTPA; P: 2% peroxide, 3% silicate, 1.6% NaOH
 $Mg(OH)_2$ - Q: 0.2% DTPA; P: 0.1% DTPA, varying $Mg(OH)_2$, 2% peroxide.

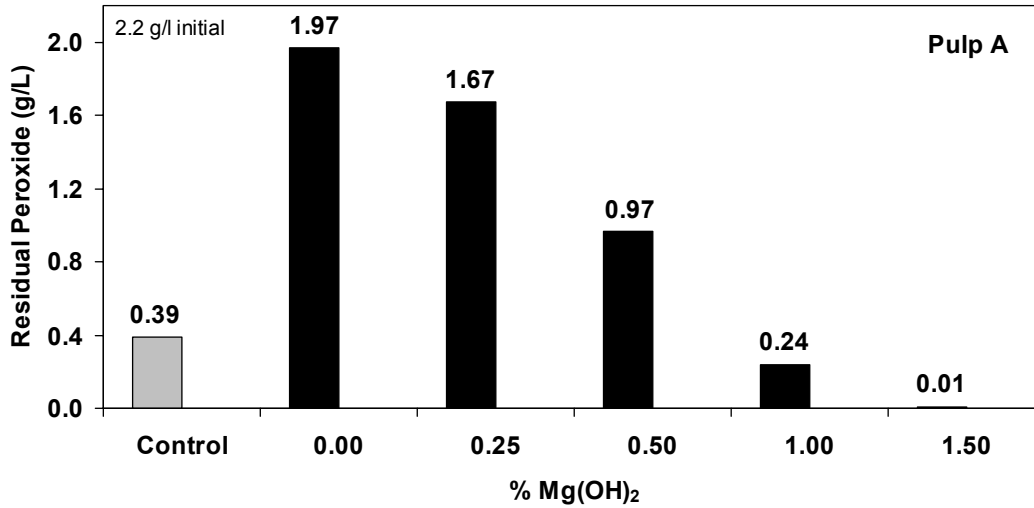


Figure 6. Pulp A residuals, 2% peroxide, varying Mg(OH)₂ levels. (see Figure 5 caption for conditions)

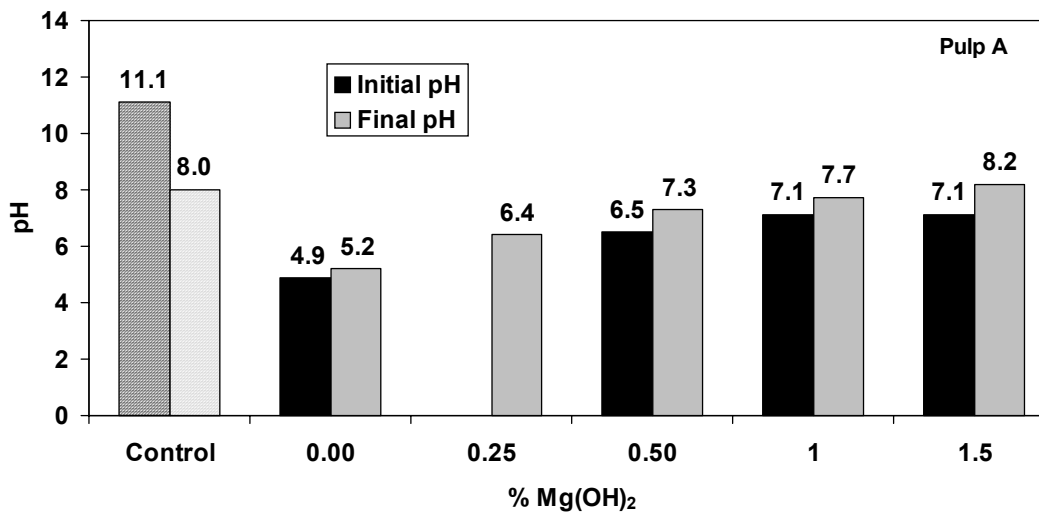


Figure 7. Pulp A pH, varying Mg(OH)₂ levels. (see Figure 5 caption for conditions)

Pulp B was used to produce an Mg(OH)₂ response curve. The curve (Figure 8) showed the optimal brightness value for this pulp to occur at 1% Mg(OH)₂ charge. At this Mg(OH)₂ level, the brightness exceeded the control bleaching brightness. As with Pulp A, there was again much more residual at the optimal brightness for the Mg(OH)₂ system than for the conventional bleaching, and the residual decreased with increasing Mg(OH)₂ levels (Figure 9). Figure 10 holds the initial and final pH data, which show the same trends as Pulp A data.

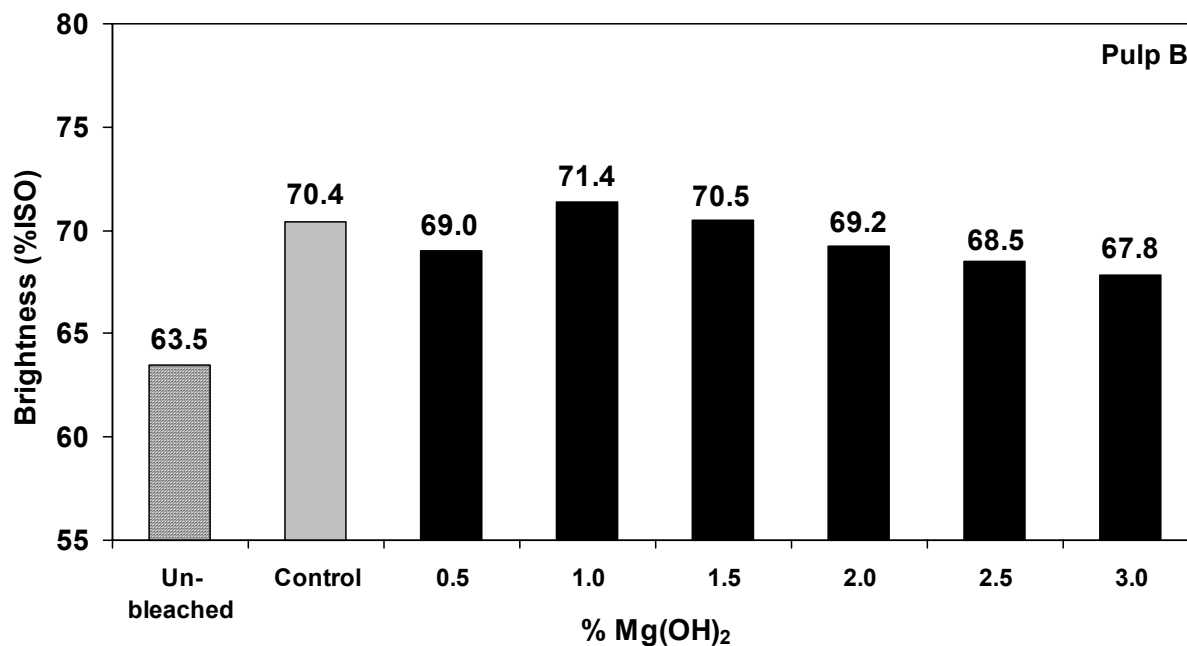


Figure 8. Pulp B Mg(OH)₂ Response Curve - Brightness.
 Control - Q: 0.3% DTPA; P: 2% peroxide, 3% silicate, 1.6% NaOH
 Mg(OH)₂ - Q: 0.2% DTPA; P: 0.1% DTPA, varying Mg(OH)₂, 2% peroxide

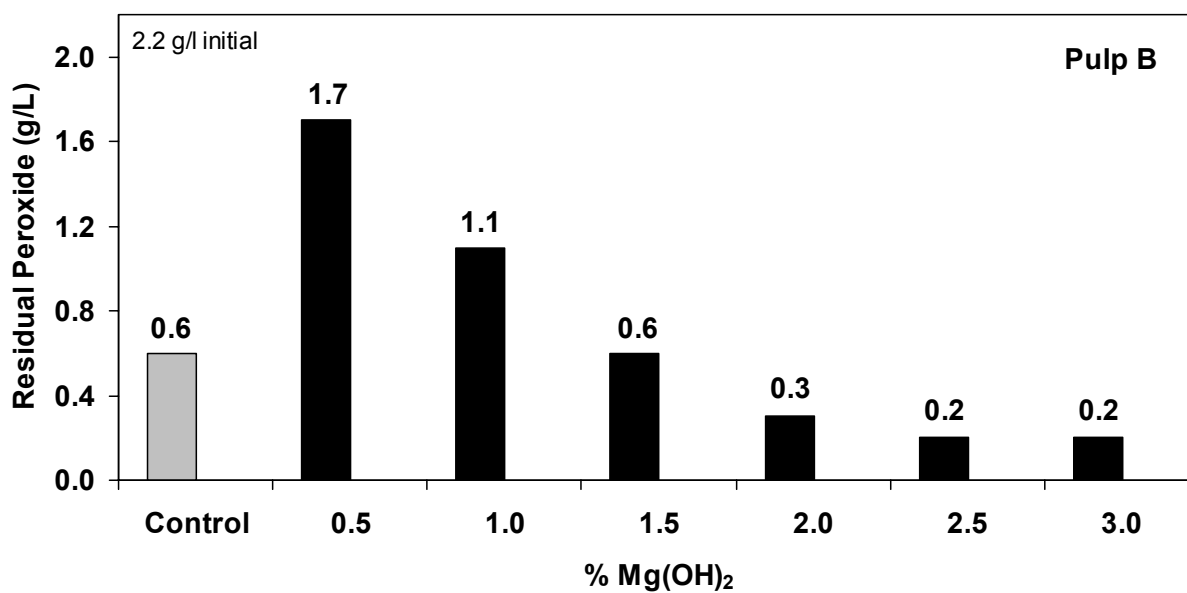


Figure 9. Pulp B Mg(OH)₂ Response Curve – peroxide residual. (see Figure 8 caption for conditions)

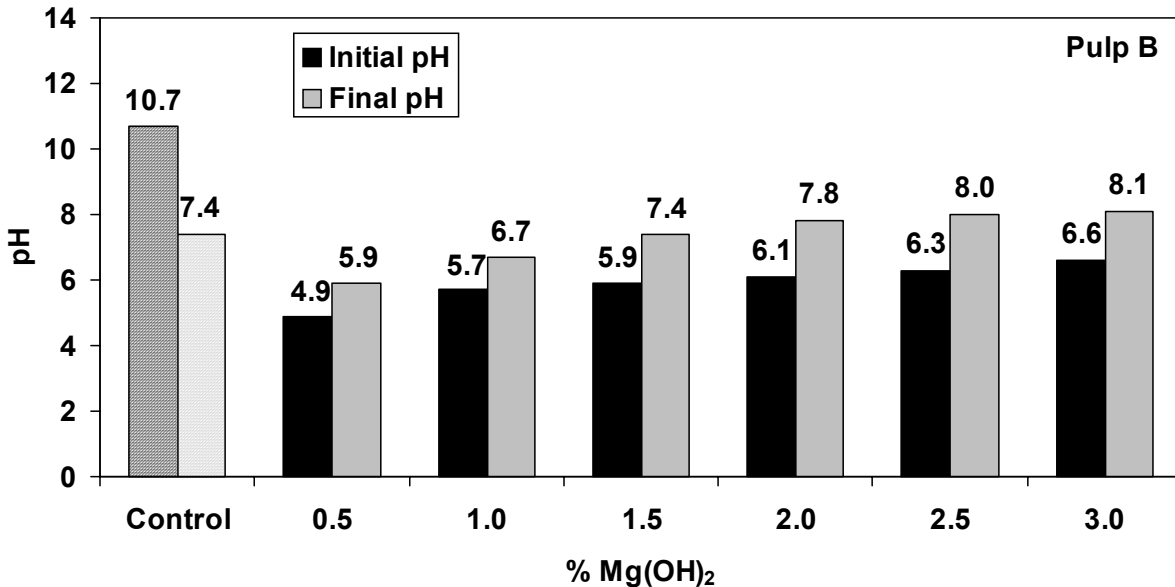


Figure 10. Pulp B Mg(OH)₂ Response Curve – initial and final pH. (see Figure 8 caption for conditions)

The brightness gain (Table 2) for Pulp A control was 10.5 points, and for Pulp B 6.9 points. The Mg(OH)₂ charges for optimal brightness were 0.5% for Pulp A and 1% for Pulp B. At these charges, the brightness gain for Pulp A was 8.3 points, and for Pulp B, 7.9 points. Pulp A and Pulp B had similar peroxide residuals, 1 and 1.1g/L, respectively, at optimal charges. However, at a given Mg(OH)₂ charge, Pulp B had more peroxide residual than Pulp A. The Pulp B control peroxide residual was also higher than for Pulp A. Both of these pulps have been tested more than once, with pulp samples collected up to a year apart, and each have shown similar results.

Table 3 shows the metals analyses of the unchelated, unbleached pulps. The largest differences between these pulps were the Al and the Mn contents. Pulp B had a very high Al level, since alum is used in the mill process before the sampling point for this pulp. Pulp A had nearly twice the Mn content of Pulp B.

None of the bleaching sequences were optimized for chelation. In these experiments, metals content was not measured directly after chelation, so the effectiveness of the chelation stage is not known. Optimizing chelation on both pulps could affect results of both the control and Mg(OH)₂/peroxide bleaching. Later, we will look at metals for handsheets from Pulp B, which show fairly low levels of Mn and Fe for both control and Mg(OH)₂ bleaching, which would indicate sufficient chelation.

From the bleaching results, there are differences in the responses of the pulps, both to control and Mg(OH)₂ bleaching, but data we have concerning these pulps do not explain the differences.

Table 2. Points brightness gain, and peroxide residual, for Pulps A and B.

	Control	0.5%Mg(OH) ₂	1% Mg(OH) ₂	1.5% Mg(OH) ₂
Points Brightness Gain				
Pulp A	10.5	8.3*	5.1	0.6
Pulp B	6.9	5.5	7.9*	7.0
Peroxide residual, g/L				
Pulp A	0.4	1.0*	0.24	0.01
Pulp B	0.6	1.7	1.1*	0.6

*at optimal brightness

Table 3. Metals analysis of unchelated, unbleached Pulps A and B

	Fe ppm	Mn ppm	Mg ppm	Al ppm
Pulp A	17	180	126	21
Pulp B	33	103	94	1510

The COD values for Pulp B, shown in Figure 11, are substantially less for the $Mg(OH)_2$ system, even at higher $Mg(OH)_2$ levels. At the 1% $Mg(OH)_2$ charge, which gave the optimal brightness (71.4%), the COD is 61% of the control value (70.4% brightness). This decrease in COD indicates a preservation of pulp yield, and a lowering of effluent clean-up requirements.

The conductivity values are also much less for the $Mg(OH)_2$ system (Figure 12), and remain so at higher levels of $Mg(OH)_2$. At the 1% $Mg(OH)_2$ charge, the conductivity is 52% of the control value. Lowering conductivity should lower ionic trash to the paper machines, and reduce the need for retention chemicals.

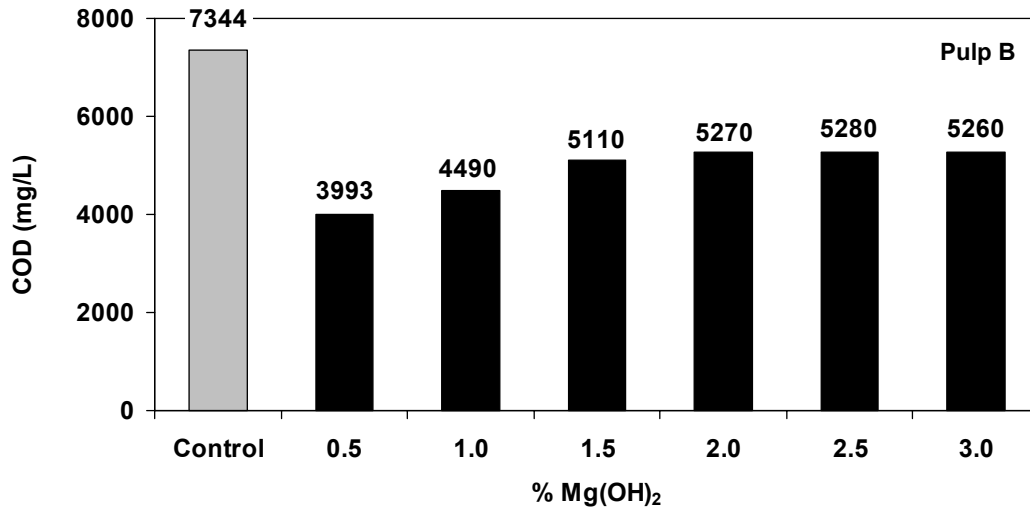


Figure 11. Pulp B COD values for $Mg(OH)_2$ Response Curve experiments. (see Figure 8 caption for conditions)

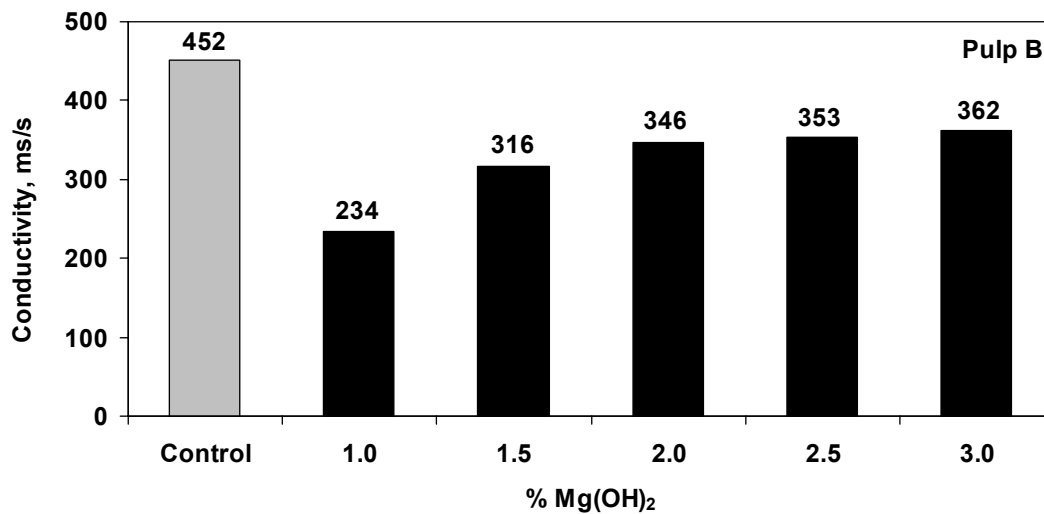


Figure 12. Pulp B conductivity for $Mg(OH)_2$ Response Curve experiments. (see Figure 8 caption for conditions)

As seen in this work, and in all other work we have done with $Mg(OH)_2$, the residual peroxide level is very high as compared to $NaOH$ bleaching. We have tried various strategies to utilize this peroxide, such as split addition of the $Mg(OH)_2$ and changing bleaching conditions, but have not succeeded in lowering the peroxide residual. However, there is the potential to recycle the peroxide in the post bleaching filtrate back into the bleaching liquor. Bleaching runs have been done, with the bleach liquor made up of a combination of fresh peroxide, and peroxide contained in filtrates from previously bleached pulps. Figure 13 shows brightness results, and Figure 14 shows residual results. The brightness and residual results were the same for experiments with the same total charge, regardless of what proportion of the peroxide was fresh or from filtrate. The response to additional peroxide was fairly modest here, at least in part because the $Mg(OH)_2$ was under the optimal dosage.

The filtrate can be reused a number of times. Figure 15 and Figure 16 show results of work done by bleaching pulp, then using the filtrate as partial charge of peroxide for another experiment on fresh pulp, then using filtrate from this pulp in the next bleaching, etc. The brightness and residual results for all three bleach runs were the same, showing that the peroxide residual is still fully active.

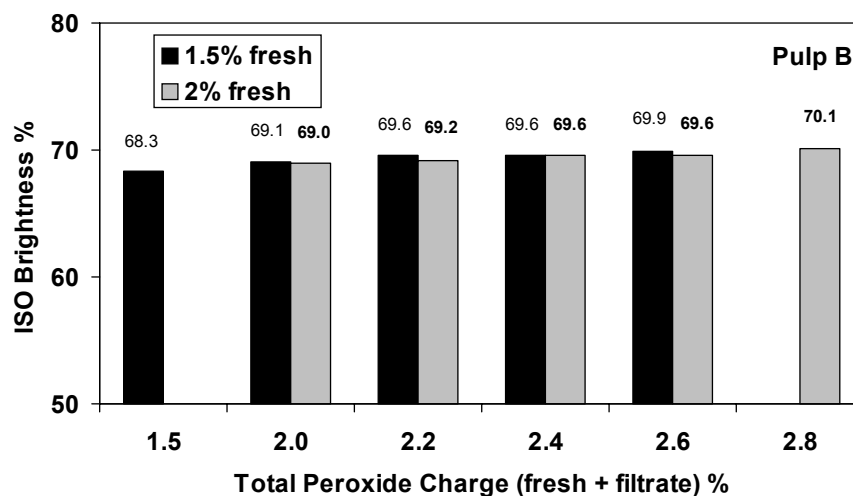


Figure 13. Potential for recycling filtrate, brightness results.

$Mg(OH)_2$ – Q: 0.2% DTPA; P: 0.1% DTPA, 0.5% $Mg(OH)_2$, 1.5 or 2% fresh + filtrate peroxide.

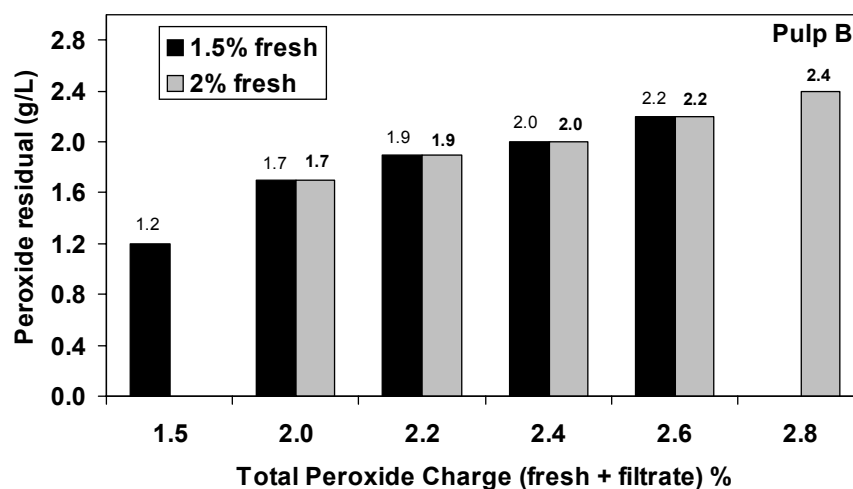


Figure 14. Potential for recycling filtrate, peroxide residual results.

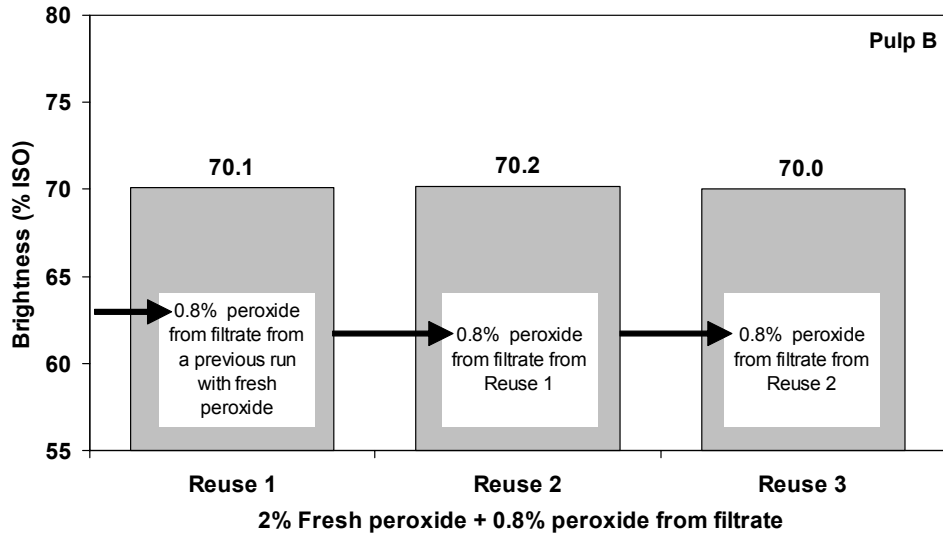


Figure 15. Multiple reuse of residual, brightness.

Mg(OH)₂ – Q: 0.2% DTPA; P: 0.1% DTPA, 0.5% Mg(OH)₂, 2% fresh + 0.8% filtrate peroxide.

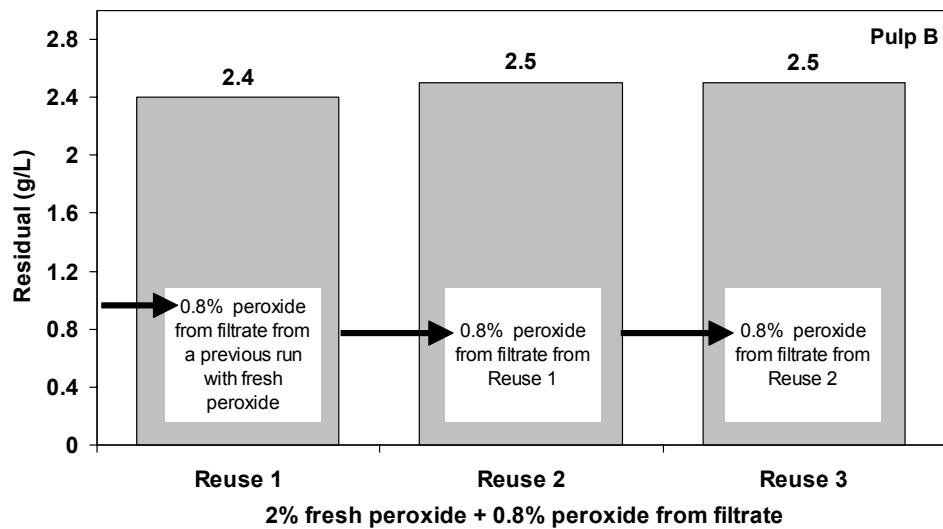


Figure 16. Multiple reuse of residual, peroxide residual.

Figure 17 shows the physical properties of handsheets made from pulp peroxide bleached conventionally, and at two levels of Mg(OH)₂. Pulp strength comparisons were done at similar brightness levels. Freenesses were not measured. Values do not vary greatly from the control, with the Mg(OH)₂ bleached values within 10% of the control values.

Handsheets made from bleached pulp were analyzed for metals content, shown in Table 4. The control obviously had a higher Na content, and the Mg(OH)₂ handsheets a higher Mg content, but otherwise values are comparable.

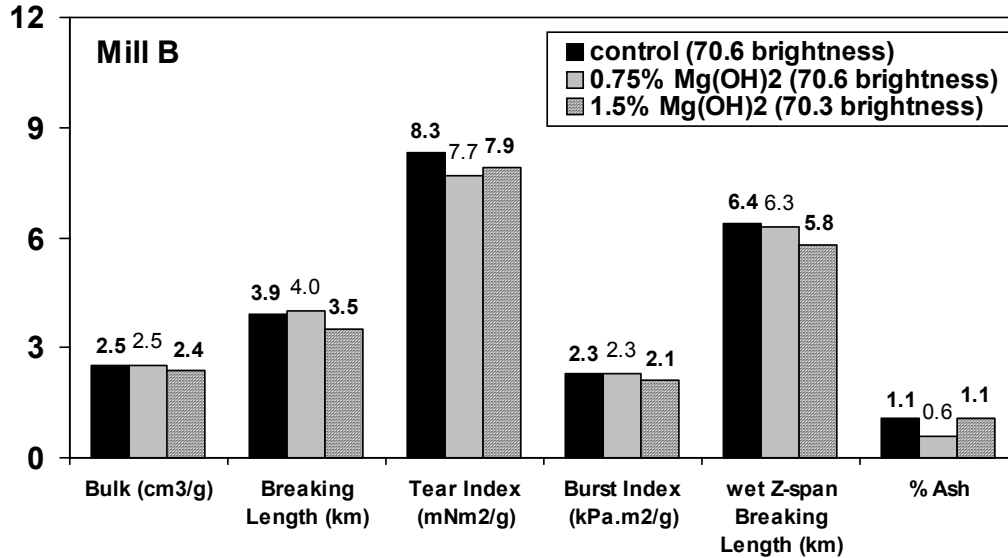


Figure 17. Physical properties of handsheets.

Table 4. Metals analysis of Pulp B handsheets, after bleaching.

Sample	ppm Na	ppm Mg	ppm Fe	ppm Mn
Control	401	17	7	0.5
0.75% Mg(OH) ₂	63	720	9	2.1
1.5% Mg(OH) ₂	32	976	8	1.6

MILL TRIALS

Mill trials have been conducted that successfully replaced caustic soda with magnesium hydroxide on a partial substitution basis and on a complete replacement basis in peroxide bleaching of mechanical pulp. Several multi-day to multi-week trials at one TMP mill allowed for evaluation of magnesium hydroxide at various dosages and its performance on a wider range of pulp grades. Variable speed peristaltic pumps, combined with mass flow meters, were used to control the magnesium hydroxide addition rate to the process. The magnesium hydroxide slurry entered the process at the refiners prior to the bleaching tower. Based on the trial results, complete replacement of caustic soda with magnesium hydroxide, elimination of sodium silicate, and a reduction in chelant usage, while maintaining pulp brightness, was achieved. Additional benefits observed during the mill trial included a reduction in mill scale and improved operating costs. Further optimization and quantifying reductions in chemical oxygen demand and anionic trash will be evaluated in future extended mill trials.

A shorter trial was conducted at a CTMP mill using magnesium hydroxide as an additive replacement for magnesium sulfate to reduce sulfate scale. After bleaching in a two-stage peroxide system, equivalent final pulp brightness was achieved without any negative results. An extended trial will be necessary to confirm and quantify expected process benefits.

CONCLUSIONS

Mg(OH)₂ can be used to replace NaOH and silicate in peroxide bleaching of TMP, reaching target brightness levels, and either reaching or exceeding the brightness of silicate/NaOH bleached pulp at the same peroxide charge. Optimal levels of Mg(OH)₂ will differ with pulp sample and peroxide charge.

At the optimal brightness level of $\text{Mg}(\text{OH})_2$, bleaching with 2% peroxide, the residual peroxide is considerable greater than for the control bleaching, indicating that the $\text{Mg}(\text{OH})_2$ system has fewer peroxide decomposition reactions. By releasing alkali more slowly due to its low solubility, $\text{Mg}(\text{OH})_2$ avoids some of the perhydroxyl ion decomposition reactions.

This level of peroxide residual also should make the recycling of the residual possible, resulting in decreased peroxide use. The peroxide residual has been shown to work as effectively as fresh peroxide.

At this $\text{Mg}(\text{OH})_2$ level, the COD is ~40% lower, the conductivity is ~50% lower, and the strength values are similar.

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