

Refiner Bleaching With Magnesium Hydroxide (Mg(OH)₂) And Hydrogen Peroxide

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ABSTRACT

Conventional bleaching of mechanical pulp with magnesium hydroxide and hydrogen peroxide has shown significant economic benefits for pulp mills over bleaching with caustic soda and sodium silicate. Magnesium hydroxide (Mg(OH)₂) bleaching generates lower effluent BOD/COD, higher pulp yield, lower anionic trash in the paper machines, reduced oxalate scaling and in some cases, higher bulk.

NORPAC has taken this bleaching process one step further through their patented refiner bleaching technology, which provides an enhanced and more efficient peroxide bleaching process. The excellent mixing, high temperature and high consistency of the refining environment solubilize the magnesium hydroxide and provide an excellent bleach reactor with the addition of hydrogen peroxide. By adding magnesium hydroxide into the refining system, refiner energy demand is reduced by 100-200 KW-hr/odmt and pulp strength is improved by 5-10%. This paper describes NORPAC's development of refiner bleaching with magnesium hydroxide and hydrogen peroxide, University of Maine lab refiner studies that support NORPAC's work and the additional benefits this technology provides to the mill.

INTRODUCTION

North Pacific Paper Corporation (NORPAC), a newsprint mill in Longview, WA began investigating alternative alkalis for the bleaching of mechanical pulp in 1999. The initial emphasis to consider a different alkali source was driven by a severe calcium oxalate deposition problem when the mill converted to neutral papermaking. Later the supply and cost variability of NaOH added emphasis to pursue an alternate alkali source. After several lab and pilot plant studies over the next year, Mg(OH)₂ was chosen as the most viable alternative alkali from a cost, supply and operating simplicity standpoint. The development of refiner bleaching technology, the mill's trial experiences, further lab research and a practical discussion as to why benefits were achieved will be described in this paper.

Process Description

The NORPAC TMP mill has a patented inter-stage, alkaline peroxide bleaching process converted to Mg(OH)₂ in 2004. The two TMP mills contain a total of nine two-stage double disc refiners, with a peak production capacity of over 1,500 ODMT/day. Brightness ranges between 52 to 72 ISO depending on the grade. The three newsprint machines average over 2,000 ODMT/day of newsprint and ground wood specialty communication papers.

The simplified process flow is depicted in Figure 1. After washing, chips are fed to pressurized digesters for pre-steaming at 45 psig for 2½ minutes. The chips are then treated with DTPA before being fed to the eye of the pressurized refiner along with dilution water to aid in feeding. The bleach chemicals, Mg(OH)₂ and hydrogen peroxide, are also added into the refiner eye. Pulp exits the pressurized refiner via a blow line into the steam recovery system. After steam recovery, the pulp is fed by a screw conveyor to a high consistency bleaching tower with 45 minutes of retention time. The pulp is then re-diluted down to 5% consistency and then thickened to 30% consistency with screw presses just prior to atmospheric secondary refining.

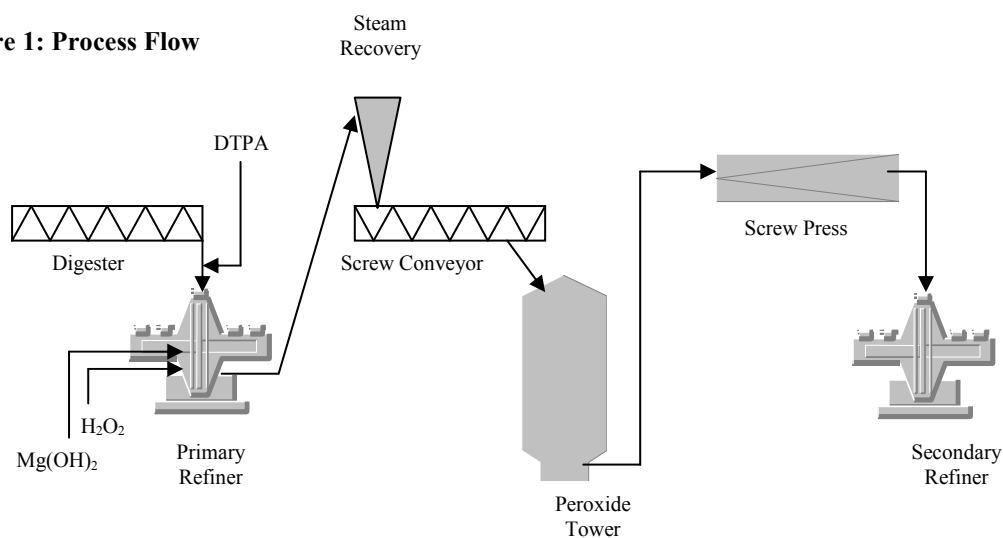
The NORPAC mill implemented a patented process for bleaching thermo-mechanical pulp at high temperature and high consistency. Hydrogen peroxide at 40% concentration is pumped directly to the primary refiners. Diethylenetriamine pentaacetic acid sodium salt (DTPA) at a 5% solution concentration is added to the chip feed to

the primary refiners. An aqueous, synthetic grade 61% $Mg(OH)_2$ slurry provided by Martin Marietta Magnesia Specialties is added directly to the primary refiners. Specifications are shown in Table I.

Table I: $Mg(OH)_2$ Specifications

Dry Solids Basis	Typical	Specification
$Mg(OH)_2$ purity, %	98.8	98.6 min
Fe, %	0.07	0.10 max
Mn, ppm	100	120 max
Cu, ppm	1	10 max
Median Particle Size, microns	3	---
% passing 325 mesh	99.9	99.0 min

Figure 1: Process Flow



MILL TRIALS

NORPAC conducted several lab and pilot plant trials prior to permanently converting the mill over to $Mg(OH)_2$. The lab and pilot plant data indicated that peroxide bleaching with $Mg(OH)_2$ could provide significant environmental and operational benefits to the mill such as:

- 1) Reduction of BOD and COD by 20% - 30% along with a projected yield improvement of 2% - 3%.
- 2) Energy reduction of 10% due to adding the alkali into the refiner.
- 3) Bleach cost reduction.
- 4) Potential to eliminate calcium oxalate deposition.

Encouraged by the lab and pilot plant studies, mill trials were run from early 2001 thru 2003. The initial short term trials were performed using tote bins and peristaltic tube pumps to deliver the $Mg(OH)_2$ to the refiners. The duration of the trials ranged from a few days to six weeks. The short term trials confirmed that 100% of the NaOH and Na_2O/SiO_2 could be replaced with $Mg(OH)_2$ while maintaining the same bleaching response. The mill also found that overdosing $Mg(OH)_2$ created no significant operating issues either in bleaching or refining as opposed to NaOH where overdosing would revert the pulp brightness and impact the stability of the refiner operation. Also it was concluded that less alkali dosage was needed with $Mg(OH)_2$ than NaOH to get an equivalent brightness response.

Although the short term trials were promising, a longer term trial (six months) evaluation was needed to examine all of the impacts to the mill operation. A temporary $Mg(OH)_2$ storage tank was installed (provided by the $Mg(OH)_2$ supplier) in February of 2003 to allow for tanker truck deliveries. NORPAC has two TMP mills. The long term trial

using $Mg(OH)_2$ as the alkali was done on #2 TMP, leaving #1 TMP on $NaOH/Na_2SiO_3$ bleaching for comparative purposes.

Key findings from the long term trial:

- There was no reduction in pulp strength when comparing #1 TMP vs. #2 TMP at the same CSF levels.
- Refining energy was reduced by 100 to 150 kW-hrs/MT.
- Calcium oxalate deposits were eliminated, allowing the scale inhibitor to be removed.
- Retention aid chemistry on the paper machines was changed due to the reduction in anionic trash from TMP.
- Peak brightness capability was maintained with $Mg(OH)_2$ as the alkali.
- There was a reduction in BOD/COD on #2 TMP effluent that directly correlated with the conversion to $Mg(OH)_2$.
- A 1% overall TMP yield improvement resulted even though only half the TMP production was converted to $Mg(OH)_2$.
- Bleach cost savings alone could justify capital expenditure of a permanent $Mg(OH)_2$ storage and delivery system with a payback of less than six months.

The mill approved a permanent $Mg(OH)_2$ system in June of 2003. Start up of the #2 TMP system was in December of 2003 with the #1 TMP system starting up the following month (January 2004). The system has been running successfully with 100% uptime to date. A modification in May of 2004 that introduced hydrogen peroxide directly into the refiner rather than in the downstream blow line improved bleaching efficiency by over 10% due to improved mixing and increased bleaching consistency. U.S. patents on the bleaching process were issued in June 2004 and April 2005. A Canadian patent on the process was issued in January 2005.

RESULTS AND DISCUSSION

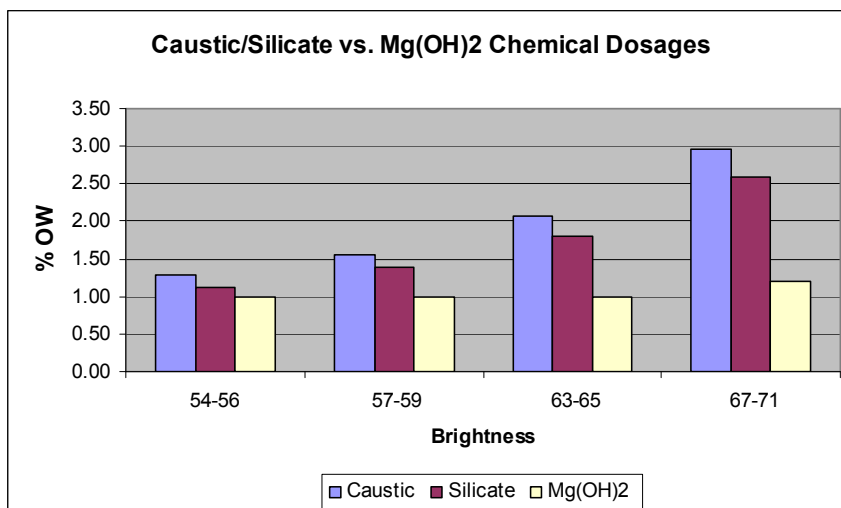
Bleaching Cost

The reduction in bleach costs using $Mg(OH)_2$ as the alkali source is achieved for two fundamental reasons:

- 1) Peroxide stabilization and the pH buffering provided when $Mg(OH)_2$ is the alkali source allow sodium silicate to be removed without impacting bleach efficiency. This reaction is described by Nyström et. al. [1].
- 2) The number of acidic groups generated during bleaching determines the demand for $Mg(OH)_2$; in most cases, as long as the pH is above 7.0, the amount of $Mg(OH)_2$ is sufficient.

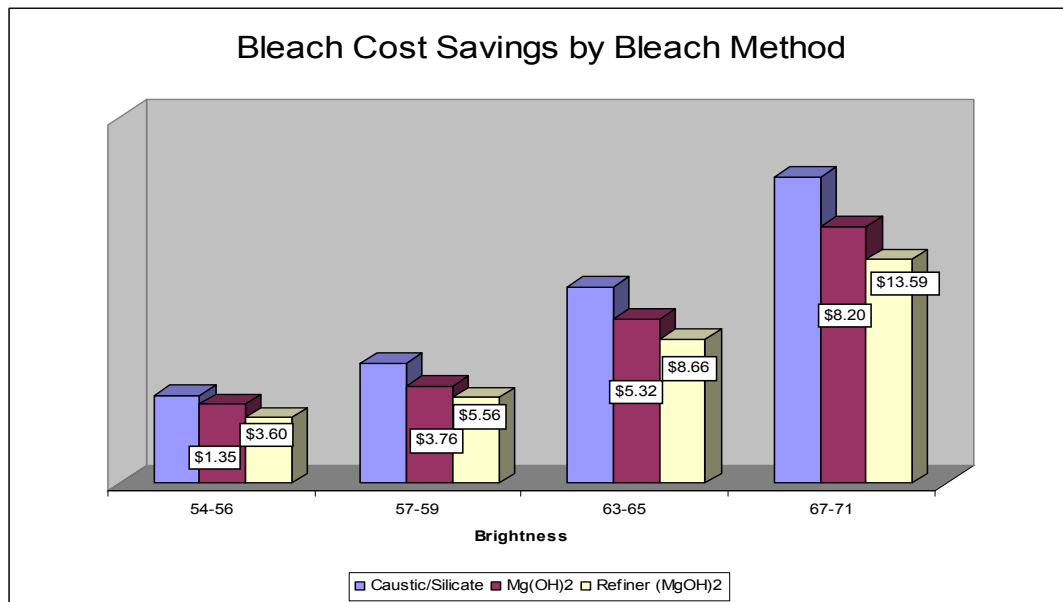
Consequently, there is a savings potential, and the alkalinity demand for caustic soda is higher than for $Mg(OH)_2$. This is reported by Seuss et. al. [2] and is confirmed with mill results. As the brightness is increased, the caustic/silicate must be increased along with peroxide. Magnesium hydroxide, however, remains constant at 1% OW (on wood) with increasing peroxide dosages and is only raised to 1.2% OW to reach peak brightness capability; this is demonstrated in Figure 2.

Figure 2:



The bleach cost savings on a per ton basis is displayed in Figure 3. The savings are by brightness target and use the caustic/silicate bleaching as the base case for comparison. The Mg(OH)₂ case shows the savings with Mg(OH)₂ added to the refiner and hydrogen peroxide added downstream in the blow line. The refiner Mg(OH)₂ case displays the savings the mill experienced when Mg(OH)₂ and hydrogen peroxide are both added in the refiner. The additional savings are due to improved bleach response due to improved mixing of chips and bleaching chemicals and a higher consistency, which result in a lower peroxide dosage to achieve the same final brightness target.

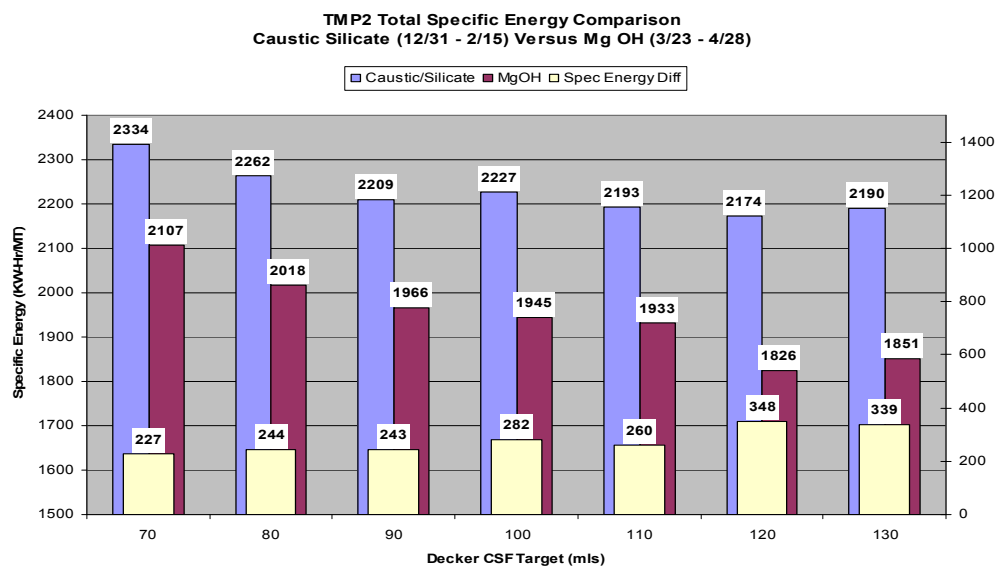
Figure 3:



Refining Energy

The fiber swelling and loosening of the lignin bonds when an alkali source is added during or prior to refining has a significant impact on refiner specific energy consumption. It is well documented by Kappel [3]. Figure 4 shows the energy reduction by freeness target when Mg(OH)₂ was applied directly into the refiner versus the mill's previous process configuration where caustic and silicate were added downstream of the primary refiner in the blow line.

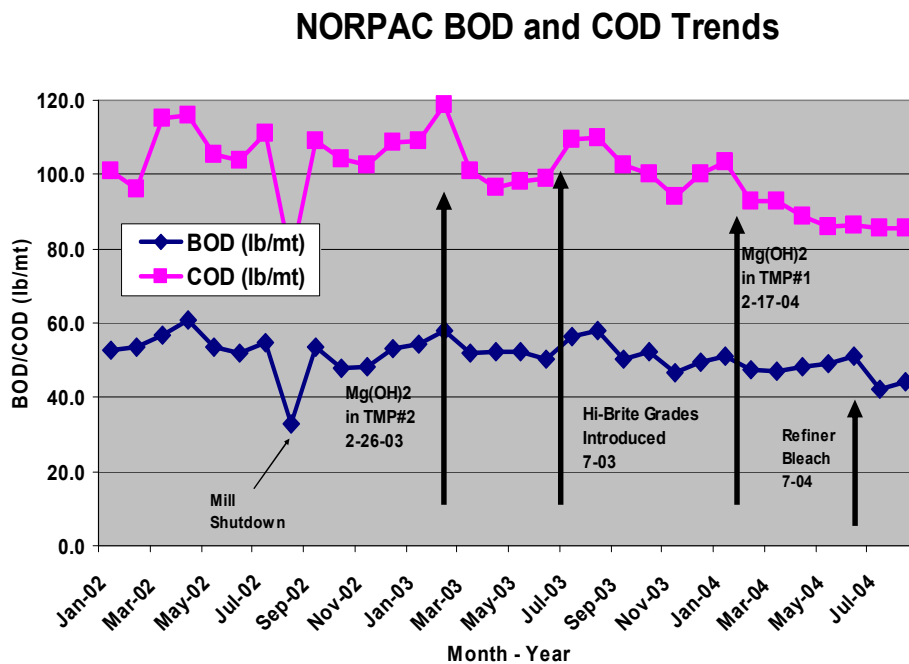
Figure 4:



BOD & COD

Typically in mechanical pulping, COD/BOD is generated from the defiberization and bleaching processes. In the case of peroxide bleaching with caustic soda, as the brightness target increases, the caustic application is increased resulting in higher COD/BOD loads. When using $Mg(OH)_2$ as the alkali source, the relationship between alkalinity and the bleach reaction changes (as seen in figure 2). The lower alkalinity needed to achieve the bleach response with $Mg(OH)_2$ helps reduce COD/BOD loading. The low solubility and alkalinity provided by the divalent base $Mg(OH)_2$ is another factor that lowers COD/BOD loading. The reduction in COD/BOD loading using $Mg(OH)_2$ versus caustic soda is well documented in literature [2], [4], [5] and generally states a reduction of between 20 to 40%. Mill data has confirmed a significant reduction in BOD/COD with the conversion to $Mg(OH)_2$ as seen in figure 5. From the mill data, it is obvious that there were step changes when #2 TMP was converted to $Mg(OH)_2$ and again a year later when #1 TMP was converted to $Mg(OH)_2$.

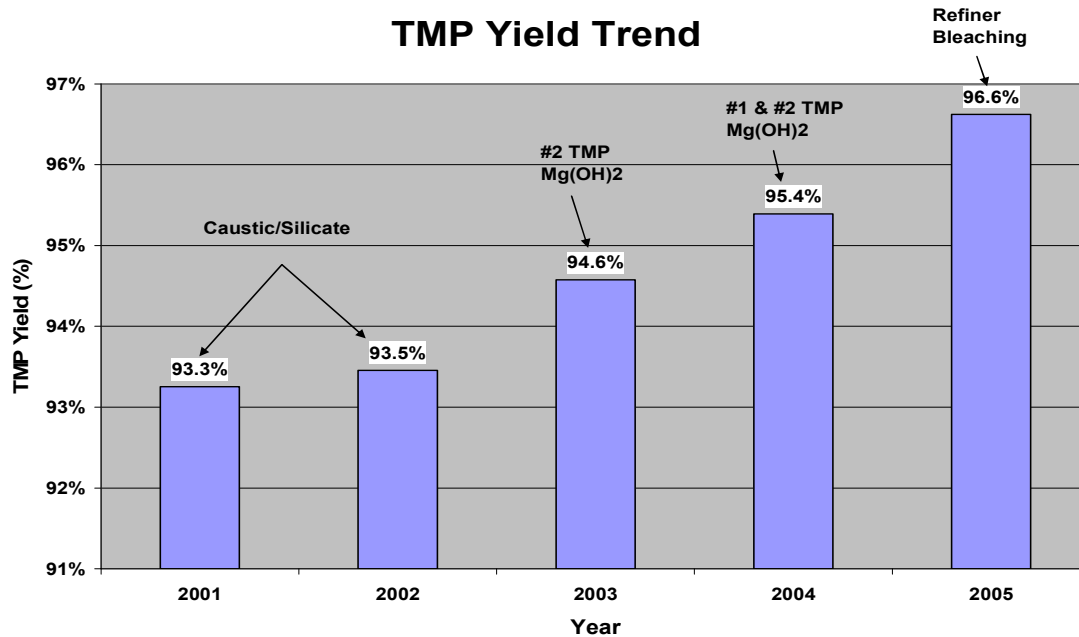
Figure 5:



Yield

Peroxide bleaching of mechanical pulps can reduce yield from 2% to 5% depending on bleach conditions and wood species. Using $Mg(OH)_2$ causes less dissolution of the pulp than caustic soda, resulting in not only lower COD/BOD but increased pulp yield. A typical TMP yield for the mill with caustic soda as the alkali source for bleaching was 93.5 %, shown in Figure 6 for the years 2001 and 2002. When #2 TMP was converted to $Mg(OH)_2$ in 2003, the TMP yield increased by 1%. With the conversion of the other TMP to $Mg(OH)_2$ in 2004, the yield increase was 2% over the caustic/silicate base case. In 2005 when peroxide was added to the refiner bleaching, efficiency improved by over 10%, and another point 1% yield improvement was achieved.

Figure 6:



Calcium Oxalate

The initial emphasis for the mill to investigate an alternative alkali was due to excessive calcium oxalate deposits when the mill converted to neutral paper making. Peroxide bleaching is the major source of oxalate generation in the mill and necessitated the need for a costly scale inhibitor program to prevent calcium oxalate scale from depositing in pipes and on machine surfaces. The formation of oxalate from the $Mg(OH)_2$ peroxide bleaching was investigated by L. Yu, M. Rae and Y. Ni [6]. They concluded that the $Mg(OH)_2$ bleached pulp generated the same amount of oxalate as the caustic soda bleached pulp. The difference is that the $Mg(OH)_2$ bleached pulp forms oxalate in a soluble state, as opposed to the caustic soda bleached pulp where a large portion of the oxalate generated is in the precipitate form. Thus the $Mg(OH)_2$ bleached pulp significantly reduces or eliminates oxalate related scaling issues. The impact of $Mg(OH)_2$ bleaching on calcium oxalate scale was clearly observed in the TMP mill. Bleaching with caustic soda, the mill used a scale inhibitor and still had scale deposits that had to be removed physically with pressure washing. Once the mill was fully converted to $Mg(OH)_2$ bleaching, the scaling issues were reduced to the point where the scale inhibitor was removed and clean up of piping and equipment due to oxalate scale deposits was eliminated.

Ease of Operation

Previous attempts to refiner bleach have met with varying success. There have been two major drawbacks with silicate/caustic soda based bleaching: 1) silicate scaling causing the refiner plates to fill in; 2) caustic soda can affect the viscosity of the pulp resulting in unstable refiner loading. The mill has had no issues with plate filling or impacts to refiner stability since the conversion to $Mg(OH)_2$. The $Mg(OH)_2$ refiner bleaching process couples refining with bleaching, thus fully taking advantage of the alkaline environment to improve fiber development and decrease specific energy.

Bleach variability has been greatly reduced with $Mg(OH)_2$. The caustic/silicate system needs to be continuously monitored to ensure the caustic application is not too low, creating high peroxide residuals and low bleach efficiency, or too high, where peroxide residual is totally consumed and brightness reversion occurs. With the $Mg(OH)_2$ system, peroxide is dosed to achieve a given brightness target, and due to the buffering nature of $Mg(OH)_2$, it can be applied at a 1% OW or lower dosage regardless of the brightness target. If overdosing of $Mg(OH)_2$ occurs, there is virtually no impact on bleach response. Using the refiner as the bleach mixer allows the bleaching chemicals to be applied neat, thus eliminating the need for chemical make down systems.

FURTHER LAB STUDIES

Since conventional refiner systems do not incorporate an interstage bleach tower to allow additional time for the peroxide reaction to occur, laboratory work was performed to determine if an increase in brightness could still be achieved if refiner bleaching is followed by a dilution step (e.g. latency chest).

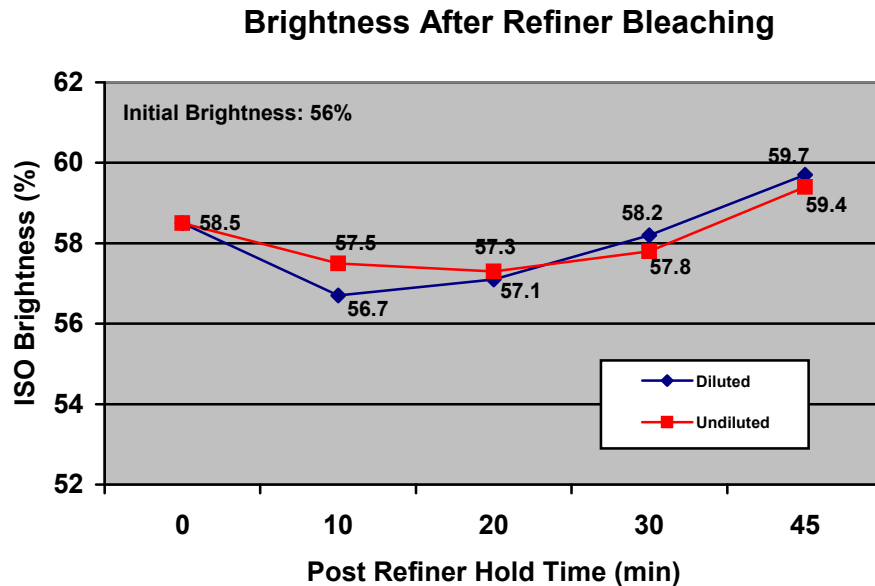
University of Maine conducted lab refiner studies using $Mg(OH)_2$ and peroxide to bleach TMP in a refiner system. The pulp sample used in the study was obtained from another TMP mill from the eastern U.S. Softwood TMP that was mill refined through primary and secondary refiner systems, bleached with hydrosulfite, and screened, was evaluated in the lab. The objective of the study was to determine the feasibility of refiner bleaching with $Mg(OH)_2$ and peroxide in the mill's downstream reject refiner system.

The pulp sample as received measured 360 mL CSF and 28% consistency. Pulp was mixed in a Hobart mixer with bleach liquor consisting of 2% peroxide, 0.75% $Mg(OH)_2$ (see Table I for specifications) and 0.2% DTPA in 500 mL of water. The pulp mixture was then processed through a 12-inch Sprout-Bauer mechanical refiner to a target freeness of 140-150 mL CSF at the refiner discharge. White water from the mill was used as dilution water during refining which resulted in 12% - 15% pulp consistency at the refiner discharge.

During the run, pulp samples were removed from the receiving tank of the refiner, bagged, sealed and placed in a water bath at 80°C. Since the mill's reject refiner is followed by a reject hold tank that dilutes the pulp down to 7% consistency and has a retention time of 45 minutes, the post-refiner bleached pulp was also diluted with white water to 7% - 10% consistency and held in the water bath at various retention times up to 45 minutes. As a comparison, undiluted, post-refiner bleached pulp (12% - 15% consistency) was also held in the water bath at the same time intervals.

Figure 7 compares the brightness of the undiluted and diluted refiner bleached pulps. The brightness of the pulp increased immediately after refiner bleaching, with no hold time, from 56% ISO unbleached to 58.5% ISO. At the 10, 20 and 30 minute intervals however, brightness decreased slightly. At the end of 45 minutes, brightness recovered for both samples as a 3+ point brightness gain was achieved after refiner bleaching. The brightness curves for the diluted and undiluted samples were similar indicating that a low consistency step after refiner bleaching did not greatly impact brightness development. This lab study demonstrated that mills with a conventional refiner system and a latency chest after the refining system can still benefit from refiner bleaching with $Mg(OH)_2$ and peroxide.

Figure 7:



CONCLUSION

The Mg(OH)₂ refiner bleaching system has provided significant environmental and process benefits to NORPAC. The mill has been operating with this bleach method for over two years and it has proven to be safe, reliable and simple to operate. The benefits achieved include: 1) reduced bleach costs, 2) lower refiner specific energy, 3) decrease in BOD/COD, 4) increased yield, and 5) elimination of calcium oxalate scaling issues. The technology is adaptable to various refiner configurations and easy to trial due to the simplicity of the chemistry and application method. NORPAC has obtained patents [7,8,9] for refiner bleaching with Mg(OH)₂ and has licensed the technology to Martin Marietta Magnesia Specialties.

References

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4. Dionne, P.Y., Seccombe, R., Vromen, M.R., and Crowe, R., "The Use of Soda Ash and Magnesium Oxide As Alkali Sources for the Hydrogen Peroxide Bleaching of Mechanical Pulp", *International Mechanical Pulping Proceedings*, pp. 403-408 (1993).
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Refiner Bleaching With Magnesium Hydroxide and Hydrogen Peroxide



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Overview

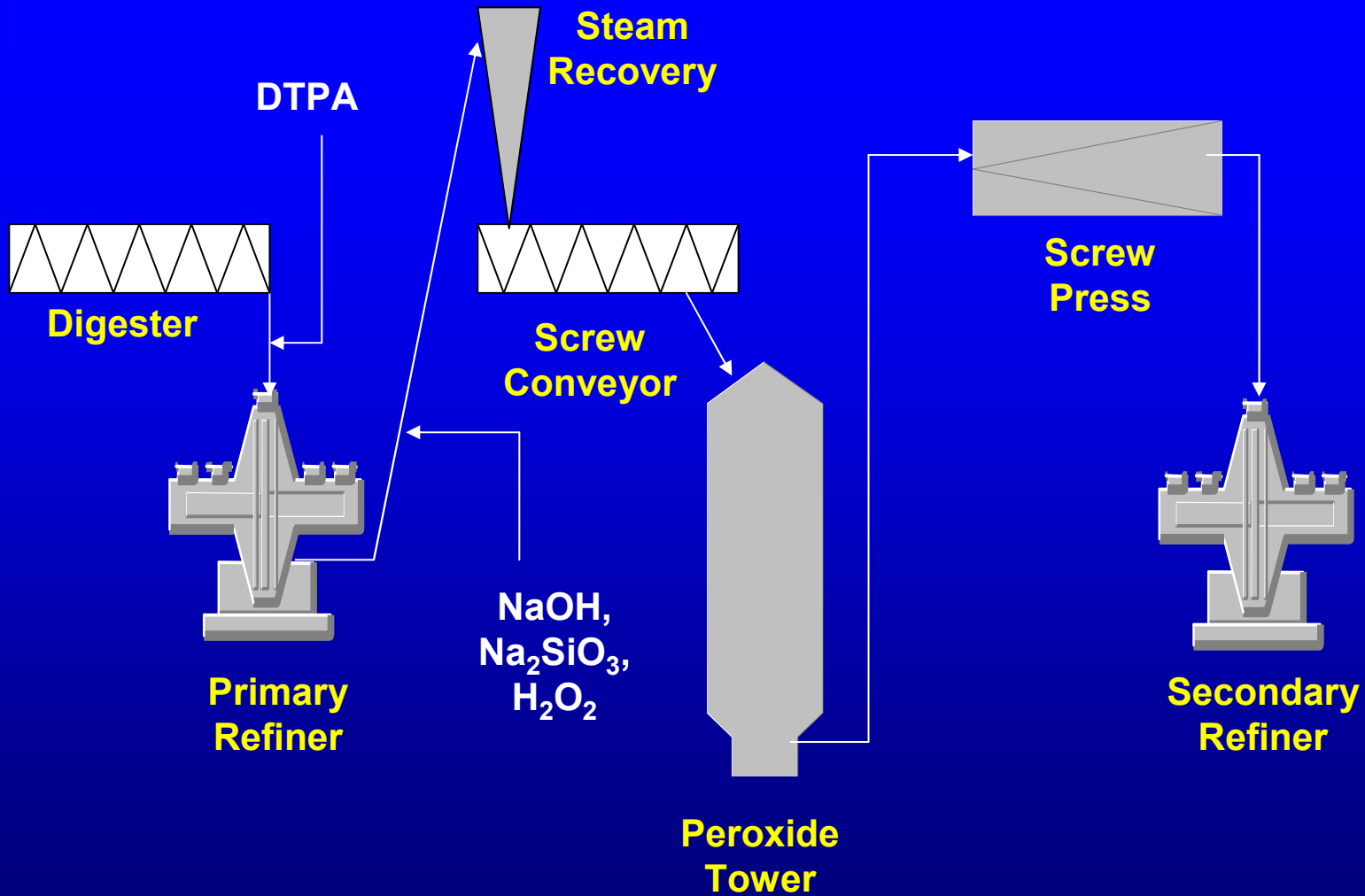
- **Mill Background**
- **Why Refiner Bleach?**
- **Mill Trials**
- **Results and Discussions**
- **Further Lab Trials**
- **Conclusions**

NORPAC

- 2000 TPD Newsprint and GWD Specialty Communication Papers
- Located in Longview, Washington USA
- 1400 TPD TMP Using 9 Two-Stage Refiner Lines
- TMP Brightness Range: 50% – 70% ISO



Original Process Flow



Original Drivers for Alternative Alkali Consideration

- **Conversion to Neutral Papermaking Created Calcium Oxalate Scale Issues**
- **Increasing Caustic Prices/Supply Shortages**
- **High Brightness Development Caused Refiner Runnability Issues and Drainage Issues**
- **Cost Reduction**

Why Refiner Bleaching?

- **Alkalinity Addition to the Refiner Lowers Energy Demand and Increases Strength**
- **Depending on Process Configuration Refiner Bleaching Offers a Low Capital Bleach System**
- **High Brightness Can Be Achieved**
- **Mg(OH)₂ Eliminates the Negatives Often Seen with Refiner Bleaching, i.e. Scaling, Refiner Runnability**
- **Easy to Trial**

Mill Trials

- Initial Short Term Trials Were Conducted Using Skid-Mounted Tote Systems
- Peristaltic Pumps Injected $\text{Mg}(\text{OH})_2$ to the Refiners
- Results Were Positive



Magnesium Hydroxide Slurry Properties

Aqueous Suspension

Dry Mg(OH)₂ Solids

Viscosity

Typical

60%

200 cps

Dry Solids Basis

Mg(OH)₂

Fe, %

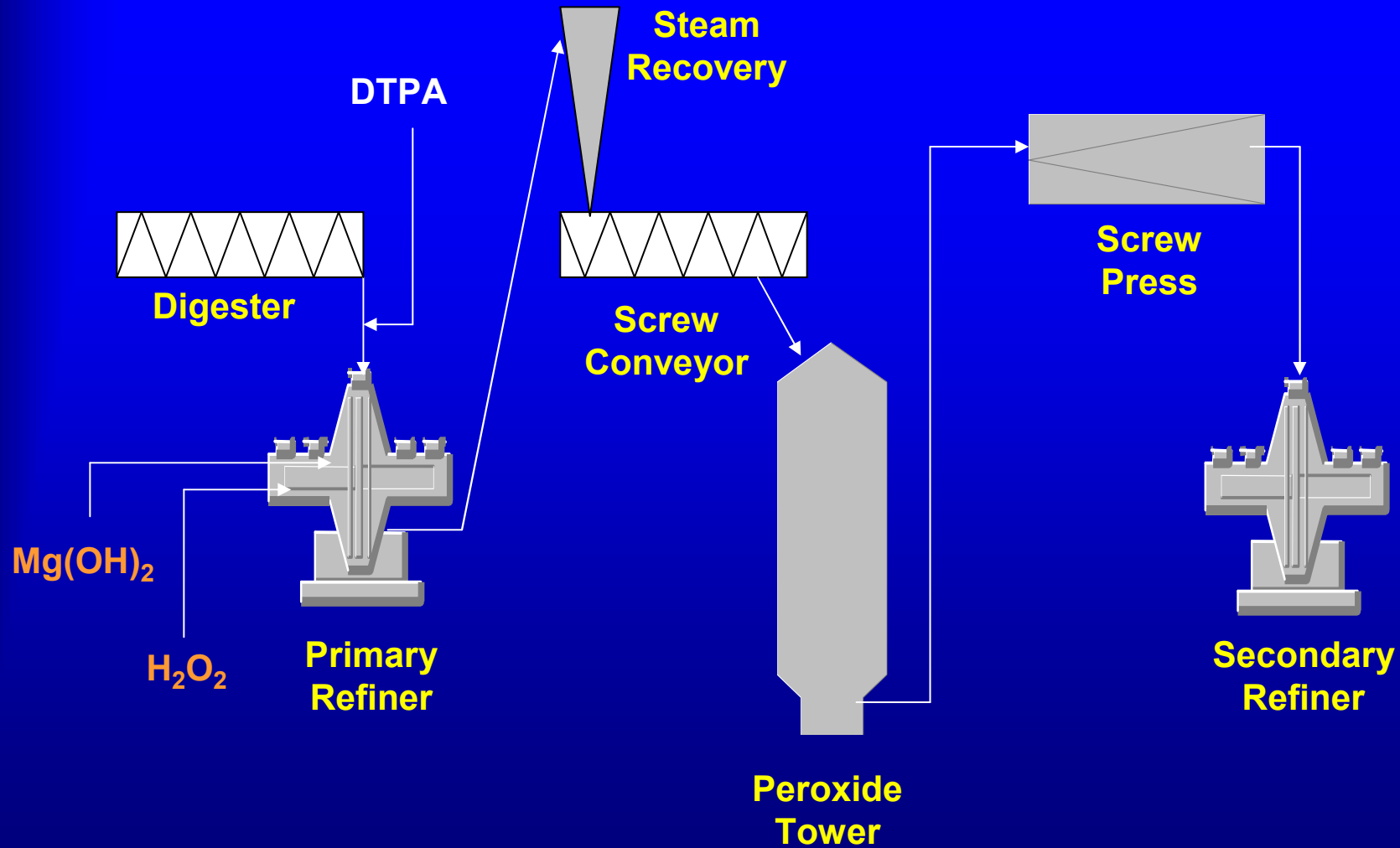
Mn, %

98.8%

0.07%

0.01%

Modified Process Flow



Mill Trials

- Longer Term Trial (6 months) Employed Bulk Storage Tanks for #2 TMP Mill
- Trial Objectives:
 - Confirm Short Term Trial Results
 - Evaluate All Impacts of $\text{Mg}(\text{OH})_2$ Refiner Bleaching to Mill Operation



Key Findings from Trial

- **No Reduction in Pulp Strength**
- **Reduction in Refining Energy by 100-200 kW-hr/MT**
- **Calcium Oxalate Scaling Was Eliminated**
- **Reduction in Anionic Trash**

Key Findings from Trial

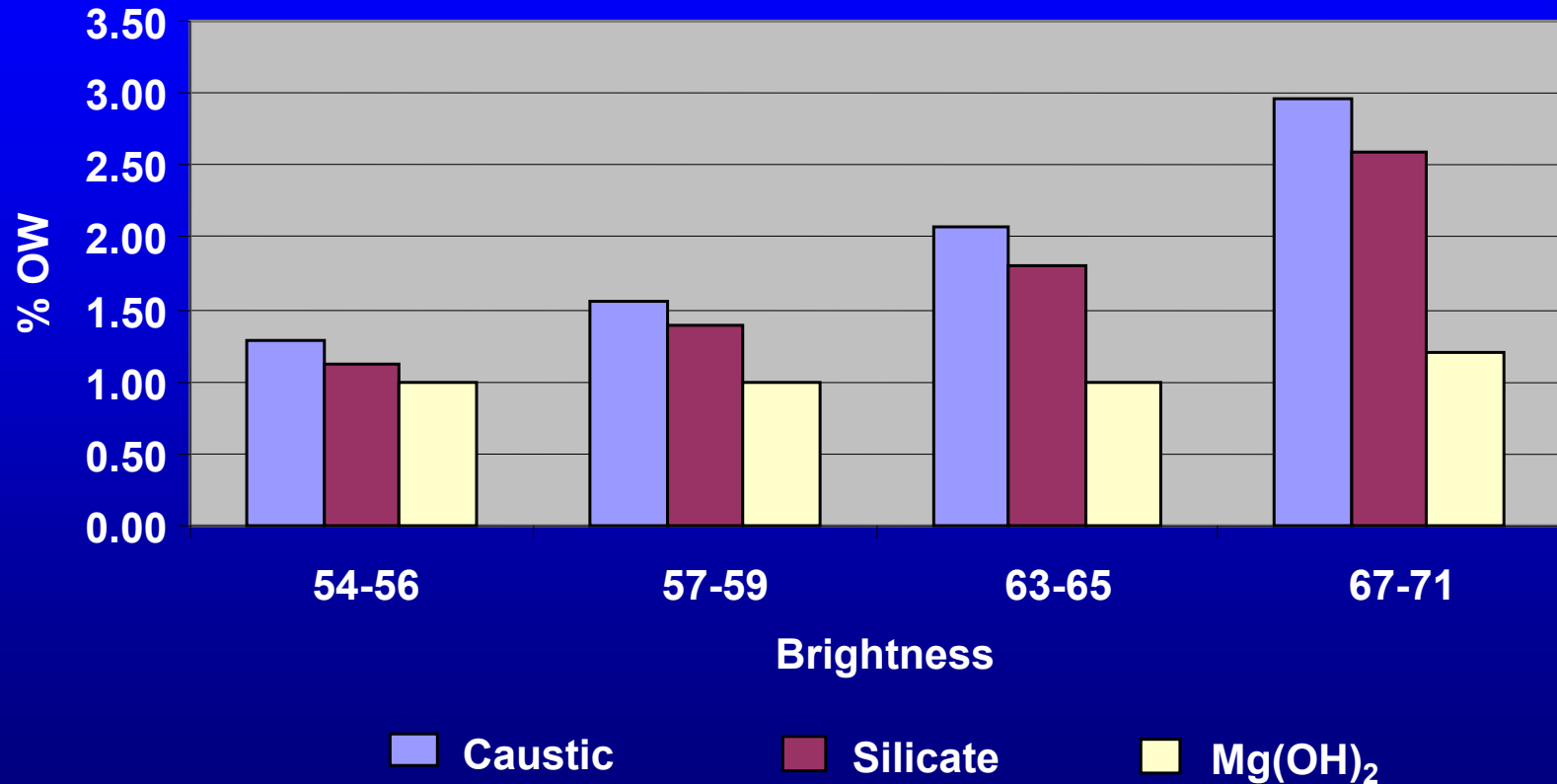
- **Brightness Was Maintained**
- **Reduction in BOD/COD**
- **Increase in TMP Yield by 1%**
- **Confirmed Bleach Cost Savings**

Results – Bleach Costs

- Bleach Costs Are Reduced Since $\text{Mg}(\text{OH})_2$ Provides pH Buffering; Allows Na_2SiO_3 to be Eliminated
- Unlike $\text{NaOH}/\text{Na}_2\text{SiO}_3$, the $\text{Mg}(\text{OH})_2$ Dosage Requires Only a Small Increase to Achieve Higher Brightness
- Peroxide Bleaching is More Efficient With Refiner Bleaching

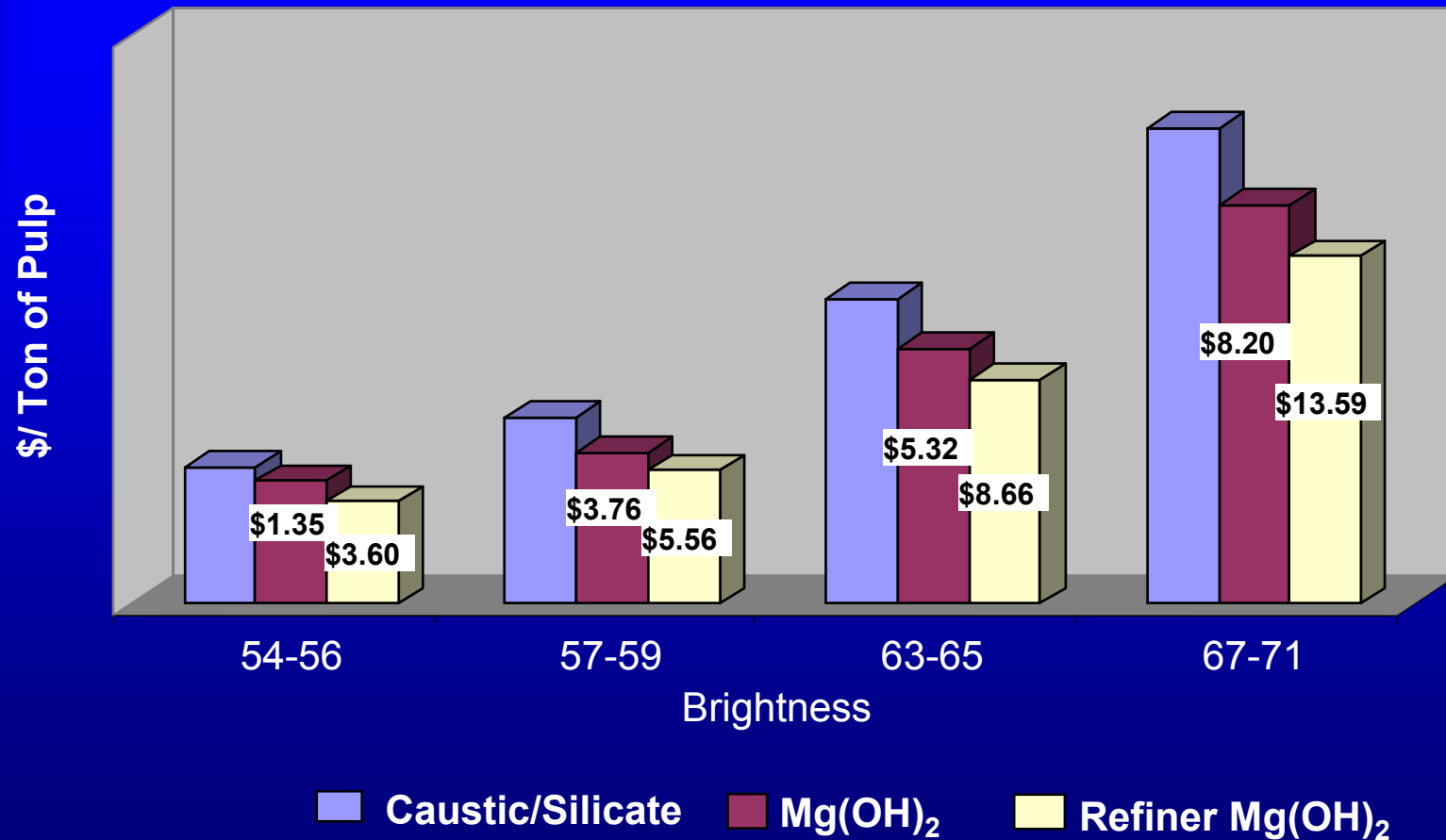
Results – Bleach Costs

Caustic/Silicate vs. $Mg(OH)_2$ Chemical Dosages



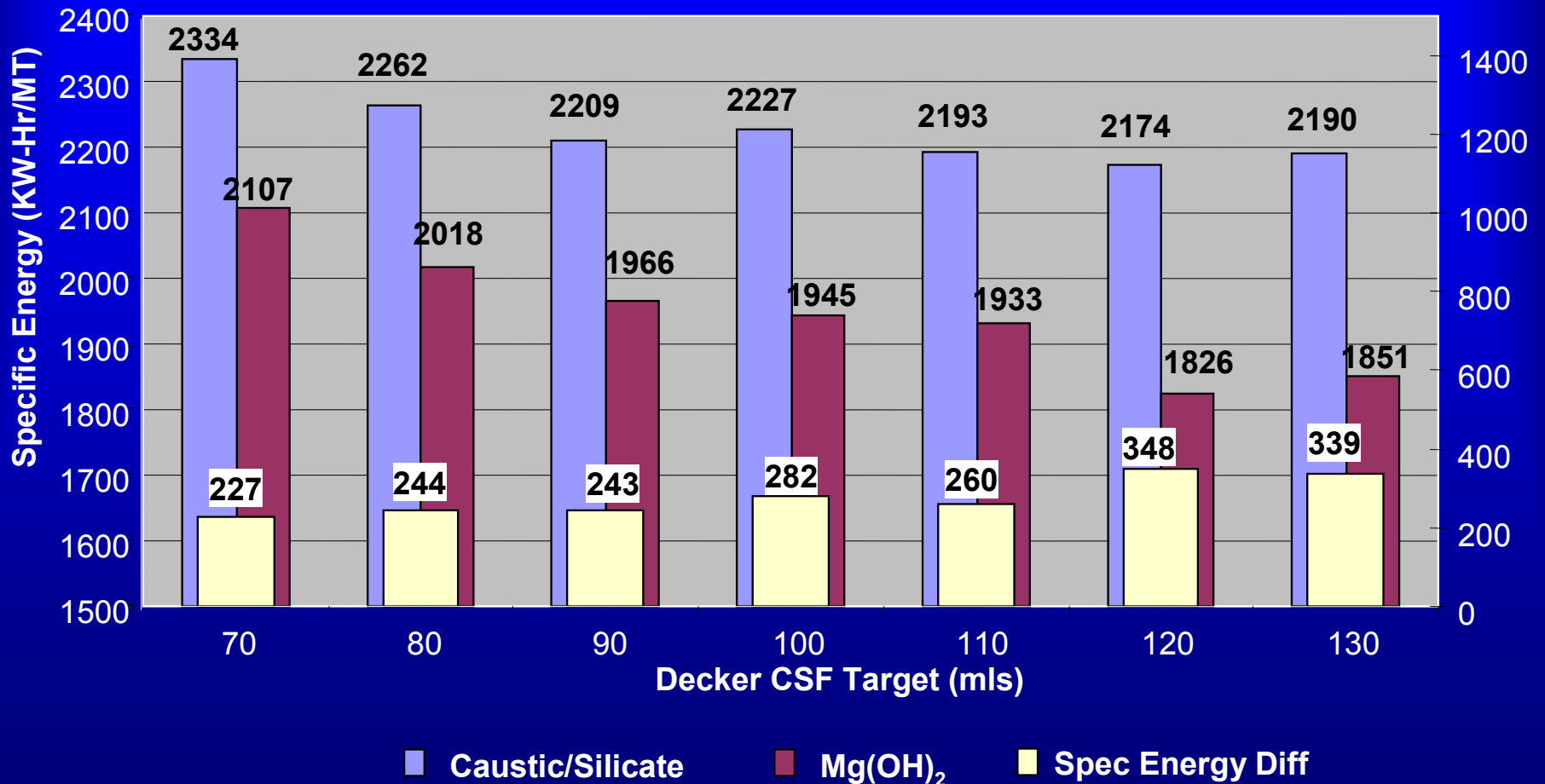
Results – Bleach Costs

Net Bleach Cost Savings by Bleach Method

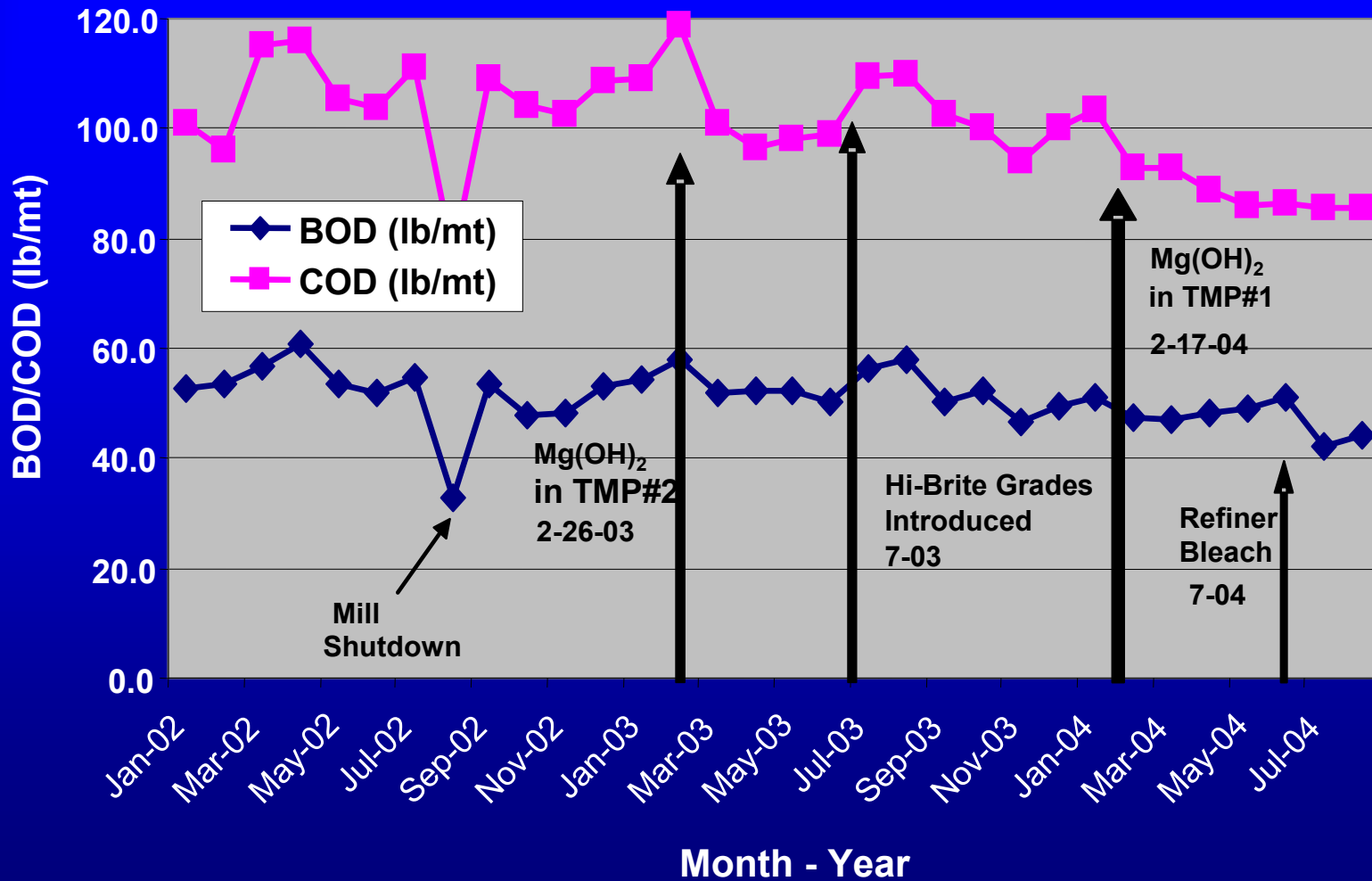


Results – Refiner Energy

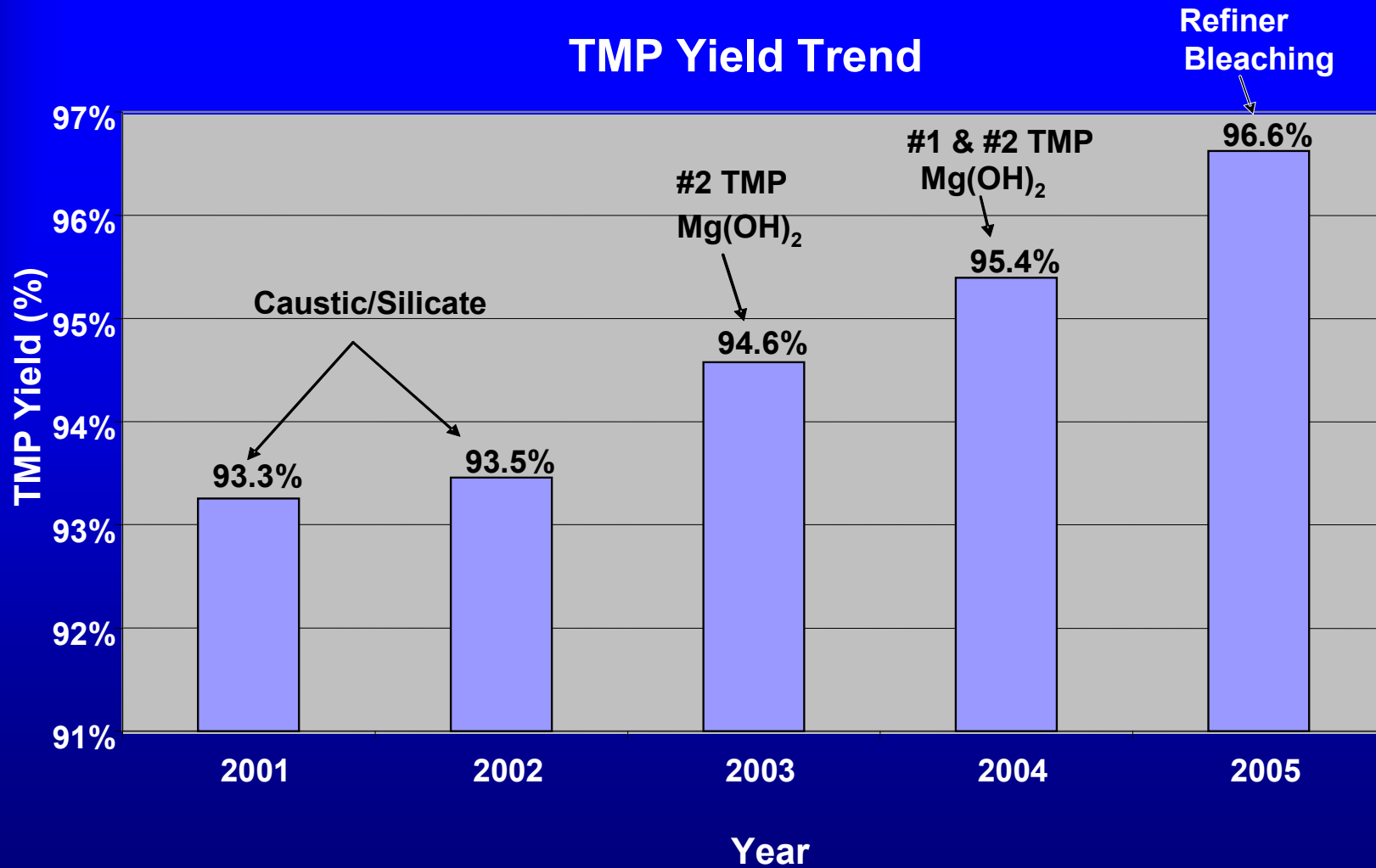
TMP2 Total Specific Energy Comparison
Caustic Silicate (12/31 - 2/15) Versus Mg(OH)₂ (3/23 - 4/28)



Results – BOD/COD



Results – Pulp Yield



Results – Calcium Oxalate

- **Oxalate Scaling is Eliminated Since $\text{Mg}(\text{OH})_2$ Forms Soluble Oxalate Salts**
- **Scale Inhibitors and Scale Removal Operations Are Eliminated**

Results – Ease of Operation

- **No Runnability Issues**
- **Bleaching Variability Has Been Reduced Due to $\text{Mg}(\text{OH})_2$ Buffering Ability**
- **No Chemical Make Down Systems Are Necessary**

Mill Benefits Achieved

Benefit	Estimated Savings
Refining Energy Reduction	100-200 KW-Hr/ODMT
Bleach Chemical Cost Reduction: 1% Mg(OH) ₂ Replaces	2% Caustic 2% Silicate
Improved Peroxide Bleaching Efficiency Due to Improved Mixing and Increased Consistency	15%
BOD and COD Reductions	20% to 30%
Yield Improvement	2% to 3%
Strength Improvement Due to Alkali in Refining System vs. Post Refiner Bleaching	5% to 10%
Oxalate Scale Elimination	Lower Chemical, Cleaning Costs
Retention Aid Reductions on Paper Machines	Depends on PM Chemistry

Additional Benefits Achieved

- **Simplified Bleach System and Controls**
- **Lowers Retention Time Requirements**
- **Safer Chemical Handling**

Further Lab Studies

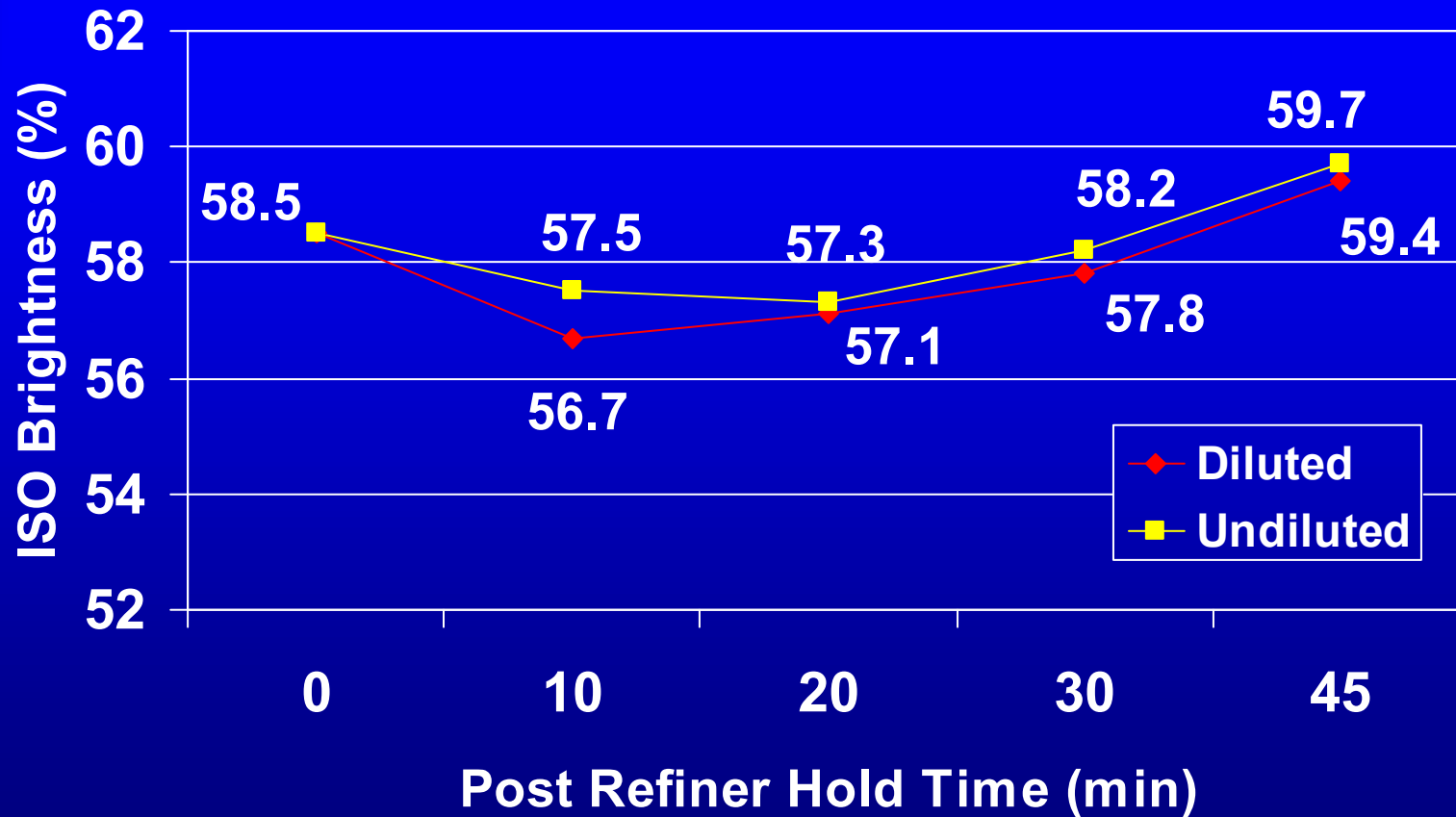
- **Feasibility of Refiner Bleaching in Conventional Refiner Systems (i.e. No Interstage Bleaching)**
- **Determine Brightness Gain After Refiner Bleaching and Dilution Step (e.g. Latency Chest)**
- **Evaluated Softwood TMP from Eastern U.S. in Lab Refiner**

Further Lab Studies

- **Refiner Bleaching Conditions**
 - 15% Pulp Consistency
 - 2% H₂O₂, 0.75% Mg(OH)₂ and 0.2% DTPA
 - Target 140-150 mL CSF
- **Dilution Step**
 - 7% Pulp Consistency
 - 80°C
 - 45 Minutes Retention Time

Brightness After Lab Refiner Bleaching

Initial Brightness: 56% ISO



Conclusions

- **Refiner Bleaching With $\text{Mg}(\text{OH})_2$ and H_2O_2 Offers Significant Environmental, Process and Economic Benefits**
- **Adaptable to Conventional Refiner Systems**

Acknowledgements

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Questions?