# Enhanced Bleachability of Chemi-Thermomechanical Pulp by Hydrogen Peroxide Bleaching in Ethanol-Water Media

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Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) bleaching in ethanol-water media was used to improve the bleaching efficiency of the chemi-thermomechanical pulp (CTMP). The brightness of CTMP increased and the yellowness decreased when the ethanol ratio was increased to 50%. However, when more than 50% ethanol was added, the brightness decreased, and the vellowness increased. In general,  $H_2O_2$  bleaching in ethanol-water media, except for 100% ethanol, improved the optical properties of CTMP compared to conventional H<sub>2</sub>O<sub>2</sub> bleaching. Moreover, the chemical composition of CTMP did not alter significantly. The best bleaching condition for CTMP was observed in 50% ethanol at a bleaching temperature and time of 80 °C and 120 min, respectively. The brightness of CTMP under this condition increased by approximately 22% compared with conventional H<sub>2</sub>O<sub>2</sub> bleached CTMP. Principal component analysis (PCA) revealed that the optical properties of bleached CTMP were affected by the structural changes of the aromatic ring and carbonyl group in lignin and carbonyl group in hemicellulose. Consequently, the specific optimal ratio of ethanol that could improve the optical properties of CTMP was determined. The results can serve as a reference for fabricating improved CTMP for its practical application in high-grade papermaking.

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#### INTRODUCTION

Chemi-thermomechanical pulp (CTMP) is a high-yield pulp produced using thermal and mechanical energy. Usually, a refiner is used to manufacture CTMP by fibrillating and cutting the fibers. Although CTMP is less expensive than chemical pulp owing to its manufacturing method and properties, its applications are limited. Recently, it has been used in the manufacturing of magazines, low-grade printing papers, newsprint, and corrugated board. However, the use of CTMP for high-grade papermaking is associated with certain disadvantages such as low strength, a large number of shives, and low brightness of the final product. Improving the bleachability of CTMP is an effective way to diversify its use. CTMP bleaching is typically conducted using hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), bisulfite, and dithionite. Compared to bisulfite and dithionite, which contain sulfur, causing troubles in wastewater treatment, CTMP can be bleached more effectively with  $H_2O_2$ . However, because the process requires more than 2 h to achieve the desired quality, there have been numerous attempts to improve the efficiency of  $H_2O_2$  bleaching of CTMP.

Organic solvents have been used to improve the pulping process. In the early stages, organic solvents were used to separate lignocellulose into its components to study them (Holmberg and Runius 1925; Hunter et al. 1939; Sarkanen and Schuerch 1957; Fullerton 1983; Sousa et al. 1986) owing to their lower surface tension than water, which enhances the penetration of chemical solutions. Moreover, organic solvent-based pulping has been primarily used to mitigate the disadvantages of kraft pulping, such as high capital equipment costs, bleaching efficiency, and environmental emission issues (Pye and Lora 1991; Lora and Pye 1992). Certainly, it is difficult to use organic solvents in mills. However, technology is advancing to make it possible, and Alcell has been successfully commercialized as a representative organic solvent-based process. Unlike the extensive research on organic solvent-based pulping, very few studies have been conducted on bleaching using organic solvents. Initially, organic solvent-based bleaching was used to extract holocellulose from wood. However, this process was closer to pulping than to bleaching (VanBeckum and Ritter 1937; Leopold 1961). Schmidt isolated the carbohydrate portion from extractive-free wood using chlorine dioxide (ClO<sub>2</sub>) in a 30% pyridine solution; however, it took approximately a month to produce holocellulose pulp (VanBeckum and Ritter 1937). Furthermore, organic solvents have been used to increase the selectivity of ozone during bleaching, and some organic solvents have been shown to improve delignification without decreasing pulp viscosity (Solinas and Murphy 1996; Van Lierop et al. 1996; Zhang 1994). However, Kolar and Lindgren reported a 10-fold decrease in the reaction rate of ClO<sub>2</sub> and styrene with an increase in the butanol ratio in the medium (Kolar and Lindgren 1982). In addition, according to Brogdon et al., ClO<sub>2</sub> bleaching in ethanol-water media slightly improves the delignification and bleaching efficiency (Brogdon 1997).

H<sub>2</sub>O<sub>2</sub> bleaching in ethanol-water media has not been extensively studied. Generally, ethanol has a lower wettability for lignocellulose than for water. Nevertheless, the lower surface tension of ethanol than water enables increasing the permeability of bleaching chemicals by adjusting the ratio of ethanol. In addition, although the use of ethanol may cause an increase in cost, it is possible to use a recovery system such as flash evaporation or distillation (Kleinert 1971; Peter *et al.* 1984). In this study, ethanol was used for H<sub>2</sub>O<sub>2</sub> bleaching of CTMP to increase the bleaching efficiency of CTMP and expand its field of use. Non-destructive analysis was conducted to analyze the factors affecting the optical properties of CTMP.

#### EXPERIMENTAL

#### Materials

CTMP was obtained from Jeonjupaper (Jeonju, Korea) and manufactured using Korean redpine. Hydrogen peroxide, sodium hydroxide, and sodium silicate were purchased from DAEJUNG Chemicals (Siheung, Korea), and ethylenediaminetetraacetic acid (EDTA) was purchased from Sigma-Aldrich (St. Louis, MO, USA).

#### Hydrogen Peroxide Bleaching

H<sub>2</sub>O<sub>2</sub> bleaching was conducted to evaluate the bleachability of CTMP based on the ethanol ratio, bleaching time, and temperature. The bleaching liquor consisted of 12% H<sub>2</sub>O<sub>2</sub>, 8% NaOH, 2% Na<sub>2</sub>SiO<sub>3</sub>, and 0.5% EDTA (% on dry pulp) by mass. The bleaching conditions are listed in Table 1. The bleaching liquor was premixed and placed in a plastic bag with CTMP slurry. Subsequently, the plastic bag was immersed in a water bath at a convenient temperature (Table 1) for bleaching. The CTMP slurry was mixed every 10 min, and all samples were washed with 500 mL of hot water after bleaching.

Pulp Consistency (%)	Temperature (°C)	Time (min)	Water:EtOH Ratio (%)	
10	70	60	100:0 70:30	
		90		
		120	50:50	
	80	60	30.70	
		90	0.100	
		120	0.100	

Table	1.	Bleaching	Conditions	of	CTMP
			•••••••	•••	• • • • • • •

#### Chemical Composition of Pulp

The pulp samples were analyzed to study the effect of H<sub>2</sub>O<sub>2</sub> bleaching in ethanolwater media on the chemical composition of the pulp under different bleaching conditions. The extractives of the pulp were measured using the alcohol-benzene extraction method according to NREL/TP-510-42619. The acid-insoluble lignin content of the pulp was measured after the extraction step according to NREL 510-42618, and the acid-soluble lignin content of filtrate was determined using a UV-vis spectrophotometer at 205 nm. An HPLC (Agilent 1100, Agilent Technologies, Santa Clara, CA, USA)/ RID (refractive index detector, Agilent 1200) instrument equipped with an Aminex HPX-87H column was used to analyze the sugar content of the pulp samples. All analyses were performed in triplicate.

# **Optical Properties of Pulp**

The optical properties of the pulp were measured using a spectrophotometer (Elrepho, Lorentzen & Wettre, Kista, Stockholm, Sweden) to evaluate the bleaching efficiency according to ISO 2470-1.

# ATR-IR and Principal Component Analysis

Infrared (IR) spectra of the pulps were obtained using attenuated total reflection infrared spectroscopy (ATR-IR, Bruker Optics, Karlsruhe, Germany). A total of 32 scans per sample were performed from 4000 to 400 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup>. The IR analysis was repeated 10 times per sample, and IR spectra were used for principal component analysis (PCA). Before performing PCA, IR spectra were preprocessed into the second derivative according to the 7<sup>th</sup> equation based on the Savitzky-Golay algorithm (Savitzky and Golay 1964). PCA was performed on the IR spectra over the range 1800 to 800 cm<sup>-1</sup> using Unscrambler software (v. 9.8, CAMO software Inc., Woodbridge, USA) to better understand the chemical differences among samples.

### **RESULTS AND DISCUSSION**

#### **Analysis of Chemical Composition**

Figures 1 through 4 show the chemical composition of CTMP with varying ethanol ratio and bleaching temperature during H<sub>2</sub>O<sub>2</sub> bleaching. The ethanol ratio did not significantly affect the lignin content of CTMP, while most of the extract was removed under similar conditions. In addition, the lignin content of the bleached CTMP increased slightly compared with that of the original CTMP; however, the increase in the ratio was relative to the removal of the extract. Figures 3 and 4 show the sugar composition of CTMP under different bleaching conditions.



Fig. 1. Lignin contents of BCTMP with varying ethanol ratio





Fig. 2. Extracts of BCTMP with varying ethanol ratio





When a small amount of xylan was removed by adding alkali during H<sub>2</sub>O<sub>2</sub> bleaching, the glucan content increased slightly; however, the bleaching temperature did not significantly affect the sugar composition of CTMP. In H<sub>2</sub>O<sub>2</sub> bleaching, HOO<sup>-</sup> ions destroy the chromophore structure of lignin and induce an increase in brightness; however, unlike chlorine dioxide or chlorine-based bleaching, lignin was not removed. In addition, ethanol decomposes into ethoxide and hydrogen ions in NaOH solution easily, and it is known that ethoxide ions are strongly nucleophilic (Parsons *et al.* 2011). Nucleophilic ethoxide ions can neutralize the hydroxide ions generated in NaOH. As a result, neutralization can suppress the hydrolysis and peeling reaction of carbohydrates during bleaching, minimizing the loss of glucan, making it suitable for bleaching high-yield pulps, such as CTMP.

#### **Optical Properties of Bleached CTMP**

The optical properties of pulp are the most useful indicators for evaluating its bleaching efficiency. In this study, the brightness and yellowness were measured to evaluate the optical properties of bleached CTMP with varying ethanol ratio.

Figure 5 shows the brightness of CTMP as a function of ethanol ratio during  $H_2O_2$  bleaching at 70 °C. As the ethanol ratio increased to 50%, the brightness of the CTMP gradually increased, and it was greater than that of the conventional  $H_2O_2$  bleached pulp with 0% ethanol. However, when the ethanol ratio exceeded 50%, the brightness of the bleached CTMP decreased. In addition, as shown in Fig. 6, the bleached CTMP with 50% ethanol-water media had the lowest yellowness, and it was the same even when bleaching at 80 °C (Fig. 8).



Fig. 5. Brightness of BCTMP with varying ethanol ratio (70  $^{\circ}$ C)

Fig. 6. Yellowness of BCTMP with varying ethanol ratio (70  $^{\circ}$ C)

As shown in Fig. 7, when CTMP was bleached for 120 min at 80 °C in 50% ethanolwater medium, the brightness of CTMP was 68.5%, and the brightness of CTMP prepared by conventional H<sub>2</sub>O<sub>2</sub> bleaching was 46.51%. It was confirmed that 50% ethanol-water media increased the brightness of CTMP by approximately 22%. The surface tension of ethanol decreases as the concentration increases, and the surface tension of 50% ethanol at 25 °C was approximately 25 mN/m (Sharma *et al.* 2019), which was significantly lower than the surface tension of water, 71.78 mN/m (Gianino 2006), indicating a positive effect on the penetration of chemical solutions. In addition, the ethanol-water solution showed highest viscosity with a value of 50% (Khattab *et al.* 2012). According to Darcy's law, the permeability of a solution is proportional to its viscosity. In this study, the bleaching efficiency was estimated to increase as the permeability of the chemical solution improved under 50% ethanol ratio condition, where the viscosity of ethanol was the highest. However, when the ethanol ratio exceeded 50%, the bleaching efficiency decreased because of the decrease in the penetration rate of the chemical. Therefore, it is expected that adequate ethanol can reduce bleaching time compared to conventional H<sub>2</sub>O<sub>2</sub> bleaching.





Fig. 8. Yellowness of BCTMP with varying ethanol

**Fig. 7.** Brightness of BCTMP with varying ethanol ratio (80 °C)

#### **IR and Principal Components Analysis**

 $H_2O_2$  bleaching did not change the chemical composition of CTMP; however, a change in the optical properties was observed. Therefore, IR analysis and PCA were performed to identify the major factors influencing the optical properties of CTMP.

ratio (70 °C)

Figure 9 shows the typical IR spectra of the pulp under different bleaching conditions; however, it was difficult to detect the differences in the IR spectra obtained under different bleaching conditions. The IR region of 1800 to 800 cm<sup>-1</sup> contains most of the main components (cellulose, hemicellulose, and lignin) in the spectroscopic analysis of pulp. It is important to identify this region of the IR spectrum to observe the lignin in pulp bleaching studies. To observe the change in the main component of CTMP by  $H_2O_2$  bleaching in more detail, the region of 1800 to 800 cm<sup>-1</sup> of the IR spectra was extracted. A representative peak for each bleaching condition was selected from the extracted area and a PCA score plot was prepared. When creating a PCA score plot, the direction of dispersion was divided into four categories using representative components, and the points with the highest dispersion among the dispersion directions, divided from PC1 to PC4, were set as PC1. In addition, the dispersion direction with the best classification accuracy for each bleaching condition was adopted.



Fig. 9. IR spectra of BCTMP under different bleaching conditions

Figure 10 shows the PCA plot and principal components by PCA of the BCTMP with respect to ethanol ratio at 80 °C. The clusters of each sample were classified as PC1 and PC4. Therefore, the components of PC1 and PC4 were traced back through the second differential spectra. In Fig. 10(a), score plots of pulp bleached in 50% ethanol, which had the highest brightness, were clustered in the upper-right corner of the graph, and score plots of pulp bleached in 0 and 100% ethanol with relatively low brightness were clustered on the left. Furthermore, when the ethanol ratio was 30 to 70%, the clusters of the score plots for each condition were classified based on the components of PC4. The peaks of components that had the greatest effect on classification by PC1 were 1508 (aromatic skeletal vibrations), 1462 (C-H bending in CH<sub>2</sub> and CH<sub>3</sub>), and 1266 cm<sup>-1</sup> (C-O stretching) corresponding to the peaks of lignin (Fig. 10(b)). Gellerstedt, and Agnemo (1980) studied the alpha-carbinol structure and reported that the reaction with H<sub>2</sub>O<sub>2</sub> proceeds via the formation of a quinone methide intermediate by a Dakin-like reaction. When quinone methide reacts rapidly with superoxide ions, hydroperoxide is formed and the hydroperoxide derivative further forms a para-quinone-type structure. In the absence of reactive species at the alpha position, nucleophilic attack by hydroperoxide is directed to the ortho position, resulting in the removal of the methyl group to form an ortho-quinone type derivative, and the ortho- and para-quinone rings can be opened by hydroperoxide anion attack (Ma et al. 2015; More et al. 2021). Therefore, it is considered that the structural change of the aromatic ring in lignin had a major effect on the brightness of bleached CTMP, which could be easily analyzed through PCA without chemical analysis. In contrast, the main peaks affecting the classification of PC4 were 1592 (COO<sup>-</sup> stretching),

1105 (C2-OH stretching), and 1018 (C6-OH stretching dominant) cm<sup>-1</sup>, corresponding to the peaks of carbohydrates (mainly hemicellulose and cellulose, Fig. 10(b)). NaOH addition during H<sub>2</sub>O<sub>2</sub> bleaching has been reported to elute hemicellulose (Suess 2010), and virtually eliminate the carbonyl groups of hemicellulose, which is one of the representative chromophores (Perrin *et al.* 2014). In this study, it was assumed that the removal of carbonyl groups in hemicellulose by an alkali shows an additional effect on the brightness of the CTMP.



Fig. 10. PCA score plots (a) and main peaks of each principal component (b) of BCTMP with varying ethanol ratio (80 °C, 120 min)

Figure 11 shows the PCA plot and principal components by PCA of the bleached CTMP with respect to the bleaching temperature. In Fig. 11(a), the score plots of the pulp before and after bleaching were classified as PC1, and those of BCTMP by bleaching temperature were classified as PC3. Therefore, the components of PC1 and PC3 were traced back through the second differential spectrum. As shown in Fig. 11(b), the peaks of the most influential components for classification by PC1 were 1654 (C=O stretching of coniferyl/sinapyl aldehyde), 1508 (aromatic skeletal vibration), and 1020 (C-O stretching) cm<sup>-1</sup> corresponding to lignin.



**Fig. 11.** PCA score plots (a) and main peaks of each principal component (b) of BCTMP with varying bleaching temperatures (50% ethanol, 120 min)

The phenolic cinnamaldehyde structure of the coniferyl aldehyde type was easily cleaved by alkaline H<sub>2</sub>O<sub>2</sub> to produce the corresponding aromatic aldehyde, such as vanillin, whereas, the alpha-carbonyl structure of the aromatic aldehyde was cleaved by the nucleophilic attack of H<sub>2</sub>O<sub>2</sub> (Gellerstedt and Agnemo 1980; Ma *et al.* 2015). Conversely, clusters of score plots with varying bleaching temperatures were classified as PC3, caused by the COO<sup>-</sup> stretching, the C2-OH stretching of hemicellulose at 1592 and 1105 cm<sup>-1</sup>, and the C-H bending in CH<sub>2</sub> and CH<sub>3</sub> of lignin at 1462 cm<sup>-1</sup>. As a result, a change in the brightness of bleached CTMP with temperature was observed owing to the change in the carbonyl structure of lignin and hemicellulose.

Figure 12 shows the score plots with varying bleaching time. The classification accuracy was low compared to other conditions, and both PC1 and PC4 components were found to have an effect. The score plots of CTMP bleached for 60 and 90 min showed a tendency to be classified as PC1, and the components that had the greatest influence on the classification of PC1 were the aromatic skeletal vibrations (1508 cm<sup>-1</sup>), the C-O stretching (1266 cm<sup>-1</sup>), and the C-H of aromatic in-plane bending (1158 cm<sup>-1</sup>), all of which mean lignin. CTMP bleached for 120 min was classified with other samples as PC4, which were also analyzed by the COO- stretching, O-H in-plane bending, and the C3-OH stretching of hemicellulose and/or cellulose at 1592, 1318, and 1055 cm<sup>-1</sup>. Therefore, it was considered that the structural change in lignin mainly occurs in the initial stage of H<sub>2</sub>O<sub>2</sub> bleaching of CTMP, and thereafter the change in hemicellulose occurs predominantly.



Fig. 12. PCA score plots and main peaks of each principal component of BCTMP with varying bleaching time (50% ethanol, 80 °C)

# CONCLUSIONS

- 1. In this study, the bleaching efficiency of the chemi-thermomechanical pulp (CTMP) was improved using ethanol-water media for H<sub>2</sub>O<sub>2</sub> bleaching. No significant effect was observed on the overall chemical composition of CTMP.
- 2. The brightness of CTMP increased with the increasing ethanol ratio and subsequently decreased when the ethanol ratio exceeded 50%. In addition, CTMP bleached for 120 min at 80 °C in 50% ethanol showed the highest brightness.

- 3. Principal component analysis revealed that the structural change of the aromatic ring and carbonyl structure in lignin had a dominant influence on the change in the brightness of CTMP; moreover, the structural change of the carbonyl group of hemicellulose was observed to induce additional brightness improvement.
- 4. Therefore, H<sub>2</sub>O<sub>2</sub> bleaching in ethanol-water media of CTMP improved the optical properties without affecting the chemical composition. It was found that a specific ratio of ethanol in H<sub>2</sub>O<sub>2</sub> bleaching could increase the added value of CTMP.

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