

EFFECTS OF DIFFERENT ACTIVATION PROCESSES ON H₂O₂/TAED BLEACHING OF *Populus nigra* CHEMI-THERMO MECHANICAL PULP

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Tetra acetyl ethylene diamine (TAED) was used as an activator in H₂O₂ bleaching to improve bleaching efficiency. The present work was aimed at confirming different activations for various H₂O₂/TAED bleaching processes, including the addition of acetic anhydride and the step-addition of sodium hydroxide. The results showed that an acetic anhydride dosage of 1%, an acetic anhydride treatment time of 10 min, and an addition time of 45 min were the optimal treatment conditions. The optimum processes of NaOH step-addition treatment in H₂O₂ bleaching and in H₂O₂/TAED bleaching also were confirmed, respectively. The o-quinone contents of H₂O₂ bleached lignin and H₂O₂/TAED bleached lignin were determined. The results indicated that H₂O₂/TAED bleached lignin has a lower o-quinone content than H₂O₂ bleached lignin, which is one of the reasons for the H₂O₂/TAED bleaching process having better bleaching efficiency than H₂O₂ bleaching.

Keywords: *Populus nigra*; H₂O₂; TAED; Acetic anhydride; o-quinone

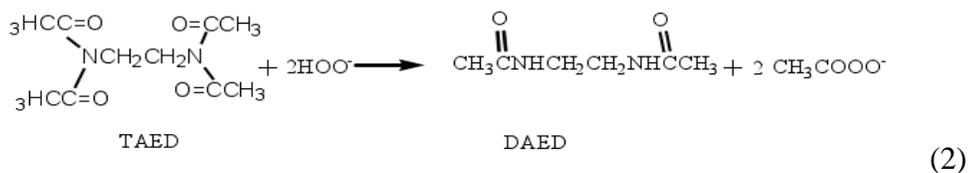
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INTRODUCTION

Hydrogen peroxide has been widely used as an environmentally responsible bleaching agent for high-yield pulp bleaching processes. The mechanism of hydrogen peroxide bleaching in obtaining high-yield pulp is mainly due to the damage of chromophoric groups in the lignin, rather than delignification (Qin *et al.* 1999). Hydrogen peroxide is easily decomposed due to its instability and due to the catalytic effects from metal ions present in pulps during the bleaching process, which leads to an increase in running cost. To improve the bleaching efficiency, many methods, such as a chelation pretreatment, addition of a bleaching activator, and a step-addition of NaOH to activate the residual hydrogen peroxide, were employed in this work.

Conventional peroxide bleaching is typically conducted under conditions of high temperature and pH, which causes significant fiber strength loss (Andrews and Singh 1979). This is especially problematic for fibers having poor strength properties (Erdumlu and Ozipek 2008; Wang *et al.* 2009). Using bleaching activators, such as TAED, the bleaching process can be conducted at a lower temperature and for a reduced time relative to conventional peroxide bleaching, thereby leading to reduced fiber damage.

The reaction mechanism of H_2O_2 and TAED is shown in Formulas 1 and 2: the HOO^- produced in the H_2O_2 bleaching system reacts with TAED to produce CH_3COOO^- . The CH_3COOO^- , which makes the delignification reaction selectively occur at relative lower temperature and in shorter time, has better oxidative ability than that of HOO^- (Zhao *et al.* 2003).



Per-acids, such as peroxy-monosulphuric acid and peracetic acid or a mixture of the two, have been proposed as a replacement of chlorine compounds in delignification and bleaching of pulp, though per-acids have not been used commercially (Siddhartha *et al.* 2010). Per-acid bleaching processes not only can reduce the bleaching pollutant load but also bleach the fibers at low temperatures. The peracetic acid bleaching process also has some disadvantages, such as poor stability, intense irritating smell, and the potential danger of explosion. The addition of acetic anhydride to activate the hydrogen peroxide in bleaching processes can, however, allow for the mentioned disadvantages to be avoided. The reaction mechanism of H_2O_2 and acetic anhydride is as follows: the H_2O_2 reacts with acetic anhydride to produce CH_3COOOH and CH_3COOH as indicated in Formula 3. The CH_3COOO^- has better oxidative ability than that of HOO^- .



Sodium hydroxide is usually completely added at the beginning of bleaching in conventional hydrogen peroxide bleaching processes. This addition style of NaOH has been described as being unreasonable according to the mechanism of hydrogen peroxide bleaching (Wang 2004). The $\text{H}_2\text{O}_2/\text{TAED}$ bleaching system also has a similar problem. The step-addition of sodium hydroxide can make the OH^- concentration distribution reasonable and activate the residual H_2O_2 , resulting in an increased bleaching efficiency.

The use of TAED as an activator in H_2O_2 bleaching has been successfully used in cotton fabric bleaching (Hashem *et al.* 2003). The application of this process in pulp bleaching also has been studied, due to its outstanding advantages (Zhao 2010; Sain *et al.* 2009). This work presents different activation processes of $\text{H}_2\text{O}_2/\text{TAED}$ bleaching for *Populus nigra* CTMP, such as the addition of acetic anhydride and the step-addition of NaOH, to enhance the bleaching efficiency further.

EXPERIMENTAL

Materials

Populus nigra (five years) CTMP pulp was obtained from a company in the east of China (Shan Dong Hua Tai Group). The pulp brightness was 47.2 %ISO. All chemicals used for bleaching were purchased from Beijing Chemical Reagent Company.

Chelation Pretreatment

The chelating process was carried out before bleaching to reduce the metal ion content. Chelation of the pulp was conducted using a 10% pulp consistency, a pH of 5.0, and 70°C for 30 min, with 0.5% EDTA, 3% Na₂SiO₃, and 0.05 % MgSO₄ (the contents of all added chemicals are reported based on oven-dry pulp).

Bleaching Process

The bleach liquors were added in order as follows: deionized water, magnesium sulfate, sodium silicate, sodium hydroxide, TAED, and hydrogen peroxide. Then the polyethylene bag was sealed and placed into a water bath at the design temperature. When bleaching was complete, the spent liquor was filtered through a 200-mesh screen for collecting the fines. The filtered spent liquors were collected for residual peroxide determination and pH testing. The bleached pulps were used to make handsheets and the brightness, PC No. (yellowing value), absorption coefficient, and scattering coefficient of the handsheets were tested according to methods in the literature (Shi and He 2003). The bleaching efficiency was obtained by the following formula:

$$\text{Bleaching efficiency} = \text{Brightness gain}(\%) / \text{H}_2\text{O}_2 \text{ consumption}(\%) \quad (4)$$

Evaluation of the Pulp Optical Properties

For evaluation of the optical properties, the bleached pulps were prepared in a laboratory sheet former. The optical properties were tested according to the standard methods listed in the literature (Shi and He 2003). The ISO brightness of the pulp handsheets was measured using a YQ-Z-48B tester.

Content Determination of o-quinone

The dioxane lignins of the raw material, H₂O₂ bleached pulp, and H₂O₂/TAED bleached pulp were prepared according to the literature (Zhao 2010). Ultraviolet absorption values of the different lignins were obtained with a UNICO-UV3802 UV-Vis spectrophotometer. The detection wavelength was at 550 nm.

Reduction with sodium borohydride

Solutions of lignin samples (200 mg each) in methyl alcohol (8 mL) and 0.1 N NaOH (4 mL) were treated separately with NaBH₄ (40 mg) and H₂O (8 mL). The solutions were protected with N₂ and reacted in the dark for 24 h. The pH values of the solutions were adjusted to 4 with 1N HCl, and the lignins were centrifugally separated. The dried lignins were refined with a dioxane-ether refining procedure.

The 15 mg lignin samples (un-reduced by NaBH₄) were dissolved into 3.5 mL dioxane-methanol (4:6v/v) solution, respectively. Then, 1 mL Na₂SO₃ (c = 10 mM, in

dimethyl sulfoxide:H₂O = 1:1 solution) and 0.5 mL FeCl₃·6H₂O (c = 10 mM, in dimethyl sulfoxide solution) solutions were added. Finally, 1 mL of pyridine was added to the above solution slowly. The absorbance of the solution at 550 nm was determined. The reference solution included all the compounds above except FeCl₃.

Then 15 mg of each of the three lignin samples (reduced with NaBH₄ solution), respectively, were weighed and treated following the above procedure. Tests of the absorbance of the solution were obtained at 550 nm. Calculations were then carried out as follows,

$$\text{o-quinone content} = (A_1 - A_2) * 100 / (C \cdot a \cdot L) \quad (5)$$

where A_1 is the lignin absorbance after NaBH₄ reduction, A_2 is the lignin absorbance before NaBH₄ reduction, C is the lignin molar concentration (mol/L), a is the molar absorption coefficient of catechol ($1050 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$), and L is the thickness of the colorimetric dish (cm).

RESULTS AND DISCUSSION

Effects of Acetic Anhydride on Hydrogen Peroxide Bleaching

Organic peroxides are compounds containing the peroxide functional group (ROOR'). Such compounds tend to decompose easily into free radicals of the form RO·; RO· then reacts with the hydrogen group of the matrix and cellulose fibers.

The mechanism of acetic anhydride activation involves the production of peracetic acid from the reaction between acetic anhydride and hydrogen peroxide, which benefits the bleaching effect in the anaphase bleaching. The present work studied the effects of acetic anhydride dosage, addition time, and reaction time on bleaching effects. The main factors were as follows:

Acetic Anhydride Dosage

The acetic anhydride dosages of 0, 1%, 3%, and 5% were used when bleaching reaction time was at 45 min, and the bleaching results achieved are presented in Fig. 1.

As shown in Fig. 1, the brightness gain increased at the beginning and then decreased as the dosage of acetic anhydride increased. This phenomenon suggested that the addition of acetic anhydride activated the hydrogen peroxide in a specific dosage range. On the other hand, the excessive acetic anhydride addition may have led to an excessively high concentration of peroxy-acid, which led to the undesired side reaction, producing a worse brightness gain. The results in Fig. 1 show that the brightness gain decreased as the dosage increased past 1%. According to the analysis above, the suitable dosage of acetic anhydride was judged to be 1%.

Table 1 shows the effects of acetic anhydride on other indexes of the bleaching process. The residual H₂O₂, bleaching efficiency, and PC No. decreased as the acetic anhydride dosage increased continuously. Comparably, the bleaching efficiency with 1% acetic anhydride was relatively high, and the absorption coefficient changed in accordance with the change in brightness.

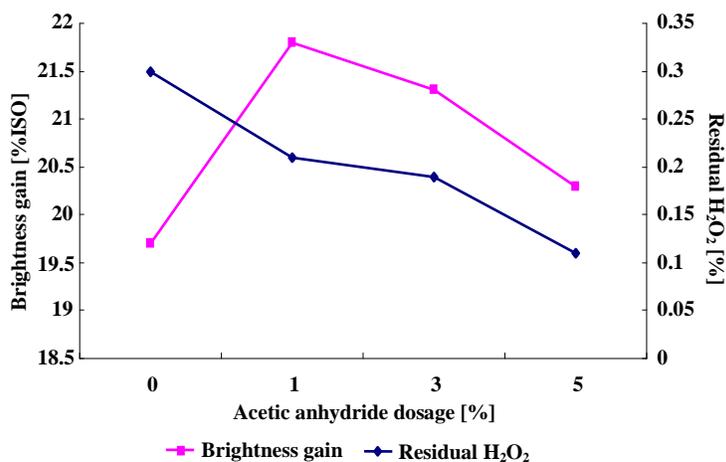


Fig. 1. Effects of acetic anhydride usage on the bleaching results.

Other reaction conditions: acetic anhydride added at the time of 45 min, treatment time of 10 min, H₂O₂ dosage of 4%, and NaOH dosage of 2%, T:P=0.3.

Table 1. Usage of Acetic Anhydride, Effect on the Bleaching

Dosage of Acetic Anhydride/%	Residual H ₂ O ₂ /%	PC No.	Adsorption Coefficient/M ² ·kg ⁻¹	Scattering Coefficient /M ² ·kg ⁻¹	Bleaching Efficiency
0	0.30	2.7	2.42	33.37	2.81
1	0.21	2.3	2.47	33.19	2.76
3	0.18	2.2	2.36	31.42	2.63
5	0.11	2.0	2.34	31.35	2.28

Acetic Anhydride Treatment Time

To confirm the optimum acetic anhydride treatment time, 5 min, 10 min, and 15 min reaction times were employed in bleaching. The bleaching results are shown in Fig. 2. As shown, the brightness gain and residual hydrogen peroxide were affected by the duration of bleaching. The brightness gain of the handsheets achieved the highest value when the treatment time was 10 min, which indicated that the suitable reaction time improved the lignin activation efficiency and increased the bleaching efficiency of the H₂O₂/TAED bleaching system. Meanwhile, the residual hydrogen peroxide also was at a relatively high level when the treatment time was 10 min. From the analysis, it can be found that the acetic anhydride activation time of 10 min was optimal.

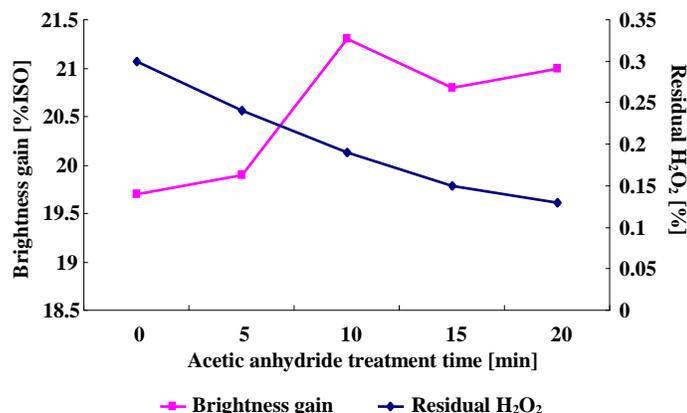


Fig. 2. The treatment time of acetic anhydride effect on the brightness. Other reaction conditions: acetic anhydride added at the time of 45 min, acetic anhydride dosage of 1%, H₂O₂ dosage of 4%, and NaOH dosage of 2%, T:P = 0.3.

Addition Time of Acetic Anhydride

The addition time of acetic anhydride was also an important factor for the H₂O₂/TAED bleaching system.

Results shown in Figure 3 demonstrate that the brightness gains of the handsheets were a response to the addition time of acetic anhydride. Early in the bleaching period, the concentration of peracetic acid was enough to launch the bleaching reaction. The addition of acetic anhydride at this time, such as at 30 min, resulted in only a slight increase in brightness gain.

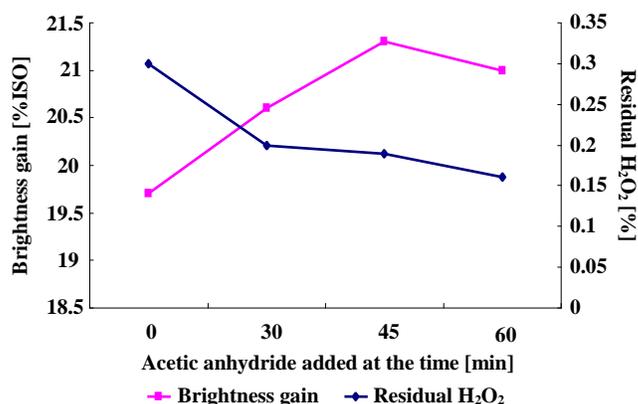


Fig. 3. Addition time of acetic anhydride effect on the brightness. Other reaction conditions: acetic anhydride treatment time of 10 min, acetic anhydride dosage of 1%, H₂O₂ dosage of 4% and NaOH dosage of 2%, T:P = 0.3.

As the bleaching process continued, the concentration of peracetic acid was substantially consumed. At this time the further addition of acetic anhydride activates the residual hydrogen peroxide to produce more peracetic acid, which benefits the bleaching reaction.

Table 2. Effects of Acetic Anhydride Adding Time on the Bleaching Results

Addition Time/min	Residual H ₂ O ₂ /%	PC No.	Adsorption Coefficient /M ² ·kg ⁻¹	Scattering Coefficient /M ² ·kg ⁻¹	Bleaching Efficiency
30	0.18	2.3	2.44	33.78	2.58
45	0.17	2.2	2.36	33.42	2.59
60	0.16	1.9	2.20	31.57	2.50

*Other reaction conditions: acetic anhydride treatment time 10 min, acetic anhydride dosage of 1%, H₂O₂ dosage of 4%, and NaOH dosage of 2%, T:P = 0.3.

It can be found from Fig. 3 that the greatest brightness gain was obtained when acetic anhydride was added over a period of 45 min. If the addition time was prolonged to 60 min, then the brightness gain was smaller than that of addition at 45 min. From the analysis above, the optimal addition time of acetic anhydride was therefore 45 min.

The data in Table 2 reflects the effects of addition time of acetic anhydride on bleaching efficiency and other optical indexes. It can be concluded that the brightness stability increased, *i.e.* the PC No. decreased, as the addition time was prolonged from 30 min to 60 min. On the other hand, the bleaching efficiency of hydrogen peroxide was best at the time of 45 min. Taking the bleaching efficiency and optical indexes of the handsheets into consideration, the addition of acetic anhydride at the time of 45 min was suitable.

Effects of Step-Addition Alkali on Peroxide Bleaching

The bleaching reaction rate has been found to decrease due to lower pH at the later stage in conventional hydrogen peroxide bleaching (Pan 2001; Lachenal 1990; Wang 2004), which leads to undesired decomposition of hydrogen peroxide and causes the bleaching efficiency to decrease.

Sodium hydroxide is usually completely added at the beginning of conventional hydrogen peroxide bleaching processes. This addition style was judged to be unreasonable according to the bleaching mechanism of hydrogen peroxide. The H₂O₂/TAED bleaching system also has a similar problem. The step-addition of the sodium hydroxide process was carried out in these two bleaching processes to avoid a sharply decreasing concentration of OH⁻.

Dosage of the Second Added Portion of NaOH

The dosage of sodium hydroxide had a great influence on the hydrogen peroxide and hydrogen peroxide activated by TAED bleaching processes. Early in the bleaching period, the pH was high and there was enough OH⁻ to react with H₂O₂, so the addition of alkali was not necessary. The pH decreased as the bleaching reaction continued, leading

to a decrease in HOO^- production, which reduced the oxidation ability of the lignin and caused the bleaching stagnation.

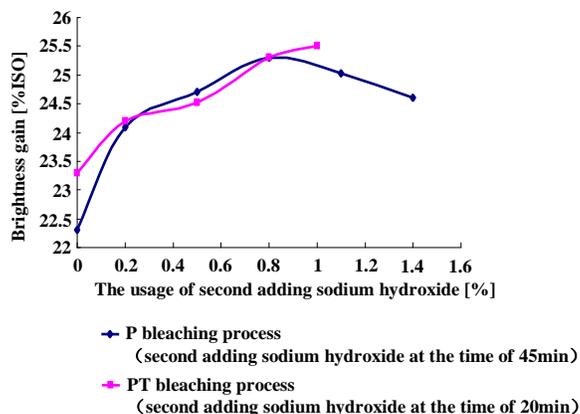


Fig. 4. The usage of second adding NaOH effects on brightness. Other reaction conditions: H_2O_2 dosage of 4% and original NaOH dosage of 2%, T:P = 0.3; total reaction time was 75 min.

Figure 4 illustrates the effects of step-addition sodium hydroxide on hydrogen peroxide bleaching at the time of 45 min and on $\text{H}_2\text{O}_2/\text{TAED}$ bleaching at the time of 20 min. The plot shows that the brightness gain improved as the second-addition of sodium hydroxide dosage increased, though the best addition dosage was different for the two bleaching systems. In the hydrogen peroxide process, the highest brightness gain was obtained with 0.8% sodium hydroxide addition, and the excessive addition caused the brightness gain to decrease. For hydrogen peroxide activated by TAED bleaching, the most favorable dosage of second addition of NaOH was 1%. This may have been caused by the different bleaching mechanisms of the two bleaching processes. The consumption of OH^- in the $\text{H}_2\text{O}_2/\text{TAED}$ bleaching process was larger than in the H_2O_2 bleaching process, and resulted in the need for more sodium hydroxide addition.

From Table 3, it is seen that the brightness improved and the residual H_2O_2 decreased as the NaOH dosage was increased, which indicates that the NaOH addition accelerated the decomposition of H_2O_2 and increased the bleaching efficiency.

For the $\text{H}_2\text{O}_2/\text{TAED}$ bleaching process, the step-addition of NaOH slowed down the production rate of peracetate ion at the beginning and made the reaction controllable. In this way, the step-addition of NaOH decreased the chance for possible side reactions. TAED reacts most favorably in pulp bleaching when the pH is close to 10.5. In the presence of TAED, the perhydroxyl ion reacts with the TAED to form the peracetate ion. Peroxide primarily works in alkaline conditions when the perhydroxyl ion, HO_2^- , is formed (Hsieh *et al.* 2006). Late in the bleaching period the decrease of the pH leads to the reduction of peracetate ion. The second addition of NaOH accelerates peracetate ion production and increased the bleaching efficiency. The bleaching efficiency was 2.51, and the PC No. was 1.9, which were relatively good values in the experimental range, when the NaOH dosage was 1%.

Table 3. The Usage of Second Adding Sodium Hydroxide Effects on Bleaching Results (PT)

Second-Addition Dosage of NaOH /%	PC No.	Adsorption Coefficient /M ² ·kg ⁻¹	Scattering Coefficient /M ² ·kg ⁻¹	Bleaching Efficiency
0	2.5	2.35	32.98	2.24
0.20	2.4	2.24	31.12	2.41
0.50	1.7	2.18	37.17	2.48
0.80	1.6	2.40	29.09	2.54
1.00	1.9	2.35	29.53	2.51
1.20	2.2	2.08	30.46	2.47

Time for the Second Addition of NaOH

The best time of implementation of the second addition of NaOH had great influence on brightness and bleaching efficiency. Addition of NaOH too early would lead to the side reaction and decrease the bleaching rate. The acceleration effect of the addition of NaOH only occurs at the specific time according to the bleaching mechanism of the two bleaching processes. Step-addition of NaOH can make the pH change reasonable during bleaching, which keeps the main bleaching reaction smooth and inhibits the undesired decomposition of hydrogen peroxide.

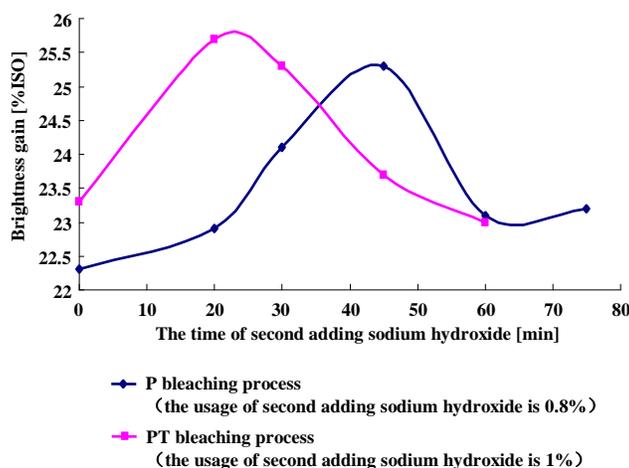


Fig. 5. The time of second adding sodium hydroxide effects on the brightness. Other reaction conditions: H₂O₂ dosage of 4% and original NaOH dosage of 2%, T:P = 0.3; total reaction time was 75 min.

Figure 5 shows that the brightness of the handsheets increased with the second-addition of NaOH in both the H₂O₂ and H₂O₂/TAED bleaching processes. As the bleaching process was going on, the pH, the production rate of HOO⁻, and the reaction activity of lignin all decreased, which led to stagnation of the bleaching process. At the

beginning of the bleaching process, the concentration of OH^- was high and could easily promote H_2O_2 and OH^- to react as shown in Formula 1 to produce OOH^- . Late in the bleaching process, the OH^- consumption increased and the bleaching reaction rate decreased. The addition of NaOH at this time would increase the pH, accelerate the H_2O_2 decomposition, and enhance the bleaching rate further.

As the addition time increased, the consumption of H_2O_2 decreased while the brightness of the pulp increased; this combination of effects indicates that the addition of NaOH can decrease the undesired decomposition of H_2O_2 and improve the bleaching efficiency. The brightness of the pulp decreased with the addition of NaOH as the bleaching time prolonged to a specific point. This is due to the consumption of H_2O_2 and undesired reactions increasing dramatically near the bleaching stagnation; the addition of NaOH was mainly used to neutralize the acids produced by the side reactions, not to accelerate the bleaching reaction.

Table 4. Effects of the Time of Second Addition of Sodium Hydroxide Portion on the H_2O_2 /TAED Bleaching Results

Second Addition Time /min	PC No.	Adsorption Coefficient / $\text{M}^2 \cdot \text{kg}^{-1}$	Scattering Coefficient / $\text{M}^2 \cdot \text{kg}^{-1}$	Bleaching Efficiency
0	1.5	2.55	32.98	2.24
20	1.6	1.94	33.07	2.51
30	1.8	2.40	33.35	2.41
45	1.7	2.45	37.17	2.34
60	2.0	2.51	33.68	2.32
75	2.1	2.59	33.42	2.33

It was found that brightness was greatest when the second-addition time of NaOH was 45 min for the H_2O_2 bleaching process and 20 min for the H_2O_2 /TAED bleaching process, which indicated that there was a different bleaching mechanism governing each of the two processes. The bleaching reaction was basically complete at the time of 40 min for the H_2O_2 /TAED bleaching process, and much less than that for the H_2O_2 process. Table 4 illustrates other bleaching index changes with the second-addition time for the H_2O_2 /TAED bleaching process. The bleaching efficiency was 2.51, the highest value, when the second-addition time was 20 min. On the other hand, the adsorption coefficient and PC No. were also suitable at the time of 20 min.

O-quinone Content of H_2O_2 Bleaching vs. H_2O_2 /TAED Bleaching

Quinone compounds are the main chromophoric groups in lignin (Lin and Kringstad 1970). The o-quinone and parquinones, as the main chromophoric groups of lignin, were produced in lignin formation and decomposition processes. Most parquinones were soluble in term of units, while o-quinone was residual in the lignin macromolecule. A study on high-yield pulp of Masson pine indicated that o-quinone is the main color resource of lignin (Li 1991).

The o-quinone structure was reduced by NaBH_4 into a phenol structure and formed a stable chelate compound having a characteristic absorption at 550 nm. The o-quinone content was tested according to the above mechanism. The catechol structure in lignins can chelate with Fe^{3+} to form catechol- Fe^{3+} chelate compound, which has a characteristic absorption at 550 nm.

Table 5 illustrates the o-quinone contents of different lignins. The data indicates that H_2O_2 bleaching and $\text{H}_2\text{O}_2/\text{TAED}$ bleaching can decrease the o-quinone content, which increases the brightness. This is because the HOO^- that is present in H_2O_2 bleaching liquid can oxidize the carbonyl groups of lignin macromolecule side chains and decolorize compounds having the quinone structure (Reeves and Pearl 1965). Comparatively, $\text{H}_2\text{O}_2/\text{TAED}$ bleaching decreased the o-quinone content much more than the H_2O_2 bleaching process; the content decreased from 0.0818 % to 0.0065 %. This is because the peracetic acid produced in the $\text{H}_2\text{O}_2/\text{TAED}$ bleaching process had better bleaching selectivity and oxidation ability than those of H_2O_2 bleaching alone. The nucleophilic reaction of CH_3COOO^- and lignin macro molecule becomes the main reaction as more CH_3COOO^- is produced in the $\text{H}_2\text{O}_2/\text{TAED}$ bleaching process. Quinone structure compounds were formed into organic acids by the Baeyer-Villiger reaction. In addition, the α -carbonyl groups were oxidized into ring-oxides and decomposed (Chen *et al.* 2000).

This result was in accordance with that of FTIR analysis for the lignin treated by the two bleaching processes (Zhao 2010). The results also explained the reason for achieving higher brightness and a shorter bleaching time with the $\text{H}_2\text{O}_2/\text{TAED}$ process.

Table 5. O-quinone Contents of Bleached *Populus nigra* Lignins

Lignin Style	A1	A2	Absorption Differential	O-quinones Contents % (Molar Content)
HO	0.2239	0.1802	0.0437	0.0818
HP	0.2401	0.1937	0.0464	0.0761
HPT	0.2213	0.2175	0.0038	0.0065

* Note: HO:raw material lignin, HP: H_2O_2 bleached lignin, HPT: $\text{H}_2\text{O}_2/\text{TAED}$ bleached lignin

CONCLUSIONS

The goal of the present work was to confirm different activation methods for the $\text{H}_2\text{O}_2/\text{TAED}$ bleaching process, including the addition of acetic anhydride and the step-addition of sodium hydroxide. The results show that an acetic anhydride dosage of 1%, an acetic anhydride treatment time of 10 min, and an addition time of 45 min were the optimal treatment conditions. The optimum processes of NaOH step-addition treatment in H_2O_2 bleaching and in $\text{H}_2\text{O}_2/\text{TAED}$ bleaching also were confirmed. The o-quinone contents of H_2O_2 bleached lignin and $\text{H}_2\text{O}_2/\text{TAED}$ bleached lignin also were determined. The results indicated that $\text{H}_2\text{O}_2/\text{TAED}$ bleached lignin has a lower o-quinone content than H_2O_2 bleached lignin, which was one of the reasons why the $\text{H}_2\text{O}_2/\text{TAED}$ bleaching process had better bleaching efficiency than H_2O_2 bleaching.

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