Bleaching of Bamboo (*Phyllostachys bambusoides*) Kraft-AQ Pulp with Sodium Perborate Tetrahydrate (SPBTH) after Oxygen Delignification

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The most prominent environmental problems facing the paper industry are those due to bleaching processes that use chlorine compounds. In this study, totally chlorine free (TCF) bleaching sequences were applied to *Phyllostachys bambusoides* bamboo unbleached kraft anthraquinone (AQ) pulp, using different conditions with Oxygen (O) delignification and Sodium Perborate Tetrahydrate (SPBTH) stages. The effects of oxygen pressure, SPBTH ratio, and bleaching time were studied to maximize the brightness gain at the lowest viscosity loss. Unbleached kraft-AQ bamboo pulp was applied to first stage oxygen delignification for bleaching with under 5 bar, 3% NaOH, and 12% concentration conditions. Following the chelated bleaching, Sodium Perborate Tetrahydrate (SPBTH) bleaching was carried out as the final stage. The optimum bamboo kraft pulp bleaching conditions were SPBTH level: 4%, $MgSO_4$: 0.5%, Na_2SiO_3: 3%, bleaching time: 80 min., reaction temperature: 70°C, and concentration: 12%. An overall increase in the physical properties of paper was evident up to an SPBTH level of 4%. When the SPBTH level and bleaching time increased, the kappa number, viscosity, opacity, and yellowness were decreased, but the brightness was increased. Oxygen delignification with chelatation and SPBTH as a bleaching sequence was shown to be a promising alternative to produce high-quality pulp from bamboo for cleaner paper.

Keywords: Bamboo; Boron compounds; Environment-friendly bleaching; Oxygen delignification;

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INTRODUCTION

Bamboo is widely used as a non-wood fibrous raw material for the production of paper and paperboard in Asia (Atchison 1998). Because of its long fibers (often comparable with softwood), bamboo chemical pulps are used as reinforcing fibers for blending with hardwood and non-wood pulps in many paper products. Bamboo responds well to kraft pulping (Misra 1981).

Many kraft pulp mills use oxygen delignification before bleaching to reduce the amount of chlorinated organic compounds in the bleach plant effluent. Organic chlorinated compounds are the main source of pollution from kraft pulp mills that produce bleached pulp. Even the best current waste liquor treatment facilities only partially remove these compounds. The oxygen delignification stage can remove only half of the lignin present in the pulp before serious pulp strength losses occur (Springer and McSweeny 1993). The delignification response in the oxygen stage is dependent on many factors: reaction conditions, chemical charge, and the history of the pulp (cooking method, brown stock washing, *etc*.). It has also been experimentally shown that the temperature and the alkali charge have the most important influence on delignification, and that the effects of time and oxygen pressure are minor (Gierer and *et al.* 1977; Li *et al.* 1996).

Due to environmental pressure, increasing the use of hydrogen peroxide as a total or partial substitute for chlorine based bleaching agents with elemental chlorine free (ECF) and total chlorine free (TCF) bleaching has been demonstrated (Tutuş *et al.* 2002). The most important problem in TCF bleaching is cellulose degradation. A possible solution to this problem is to introduce non-degrading bleaching agents in the sequence. In addition, it has been demonstrated in some studies that selectivity in oxygen delignification is increased by certain chemical pretreatments of the unbleached pulp. Activated bleaching with sodium perborate is used in the textile industry. Sodium perborate has been found to have promising effects in relation to bleach pulps. It forms peroxy compounds, which have a higher redox potential than that of hydrogen peroxide. Sodium perborate is a good brightening agent when it is utilized under optimum conditions (Lopez *et al.* 2002). Sodium perborate tetrahydrate is formed by a mixture of sodium hydroxide and borax by adding hydrogen peroxide. The molecular formula of the sodium perborate tetrahydrate anion has two peroxide bridges such as $[(HO)_2BOO]_2^{2-}$ (Köroğlu and *et al.* 2003). Sodium perborates are an inexpensive, reliable, and easily stored oxidant (Mckillop and *et al.* 1988). In comparison to hydrogen peroxide, the efficiency of the sodium perborate tetrahydrates can be increased at low temperatures by using TEAD type activators (Bayça *et al.* 2004). Also, hydrogen peroxide bleaching is 16% more costly than sodium perborate bleaching (Leduc *et al.* 1996).

The aim of this study was to investigate the possibilities of producing effective bleachable bamboo pulp without the loss of strength properties, in addition to bleaching experiments with non-wood raw materials, and examining the balance between oxygen delignification and sodium perborate tetrahydrate (SPBTH).

EXPERIMENTAL

Raw Material

The bamboo material used in the study was *Phyllostachys bambusoides* from Rize/Pazar in Turkey. The chemical composition of this material is shown in Table 1.

Table 1. Some Characteristic Properties of Bamboo (*P. bambusoides*)

Component	Composition of bamboo dry solids (%)
Holocellulose	70.5
Cellulose	50.1
Alfa cellulose	43.3
Lignin	24.5
Silica	1.03
Hot Water	6.47
1% NaOH	25.1
Alcohol-Benzene	3.94

Methods of Analysis of Raw Materials and Pulp and Paper Sheets

The bamboo samples were 3 to 5 cm in diameter, and the nodes and rotten part was removed from the body. For pulping, the bamboo chips were screened (SCAN-CM 40:94), air-dried, and stored at a dry solids content of 92%.

Analyses of raw material and pulp of bamboo were made according to TAPPI methods with the exception of holocellulose content determined by Wise's sodium chlorite method (Wise and Murphy 1946) and cellulose content according to Kurscher and Hoffner's nitric acid method (Rowell 1984). Using the respective TAPPI methods, alpha cellulose content was determined by T 203 cm-99, lignin content was determined by T 222 om-02 (acid insoluble lignin), silica content was determined by T 245 cm-98, water solubility of wood was determined by T 207 cm-99, alcohol-benzene extraction was determined by T 204 cm-97, and 1% sodium hydroxide solubility of wood was determined by T 212 om-98.

Determination of kappa numbers of pulp samples was carried out following a standard procedure described in T 236 om-99. According to this standard, the kappa number is the volume (in milliliters) of 0.1 N potassium permanganate solution consumed by 1 g of moisture-free pulp under the conditions specified in this method. The results are corrected to 50% consumption of the permanganate added. The determination of kappa was carried out three times for each pulp.

The pulp obtained was used to determine viscosity in accordance with Scan-C 15:62, according to this standard cellulose depolymerisation by measuring the 0.5 M cupri-ethylenediamine CED solution. The following formula was used to convert the SCAN pulp viscosity to DP of cellulose. The determination of viscosity was carried out in three times for each pulp.

$$DP = (0.75*[\eta scan])^{1.105} \quad (1)$$

Handsheets of 60 g/m^2 were formed, and their properties were evaluated in accordance with the TAPPI methods. The handsheets were conditioned in accordance with T 402 sp-98, the burst index of handsheets was measured by method T 403 om-97, the tensile index of handsheets was measured by method T 404 cm-92, and the tear index of handsheets was measured by method T 414 om-98.

Brightness, yellowness, and opacity were calculated to the ISO standard; and finally the amount of bleaching yield was calculated to the dry solids content of the pulps.

Kraft-AQ Pulping

The pulp from the bamboo chips was produced with the kraft-AQ method. The cooking conditions and pulp properties are shown in Table 2. Bamboo was chopped to 30 x 20 x 4 mm dimensions manually. Pulping was performed in an electrically heated rotary laboratory digester 15 L in volume and monitored with a digital temperature control system.

At the end of the pulping, the pressure was discharged to atmospheric pressure. The resulting pulps were washed and screened on a Noram type pulp screen with a 0.15 mm slotted plate.

Table 2. Cooking Conditions and Properties of the Bamboo (*P. bambusoides*)

EA, on o.d. bamboo (as NaOH) (%)	18
Sulfidity (%)	25
AQ ratio (%)	0.1
Liquor-to-bamboo ratio (l/kg)	4/1
Cooking Temperature (ºC)	165
Time (min.)	90
Viscosity (mL/g)	1164
Kappa no	20.03
Screened Yield (%)	45.8
Reject (%)	0.25
Total Yield (%)	46.05

Bleaching Method

Oxygen delignification was chosen for the first stage of bleaching. An oxygen delignification optimization study was performed at different oxygen pressures (Table 3). Next, the bleached pulps were beaten to 50 ±3ºSR (Schopper-Riegler) freeness with a PFI mill.

Table 3. Oxygen Delignification Conditions

NaOH, on o.d. pulp (%)	3
$MgSO_4$, on o.d. pulp (%)	0.5
Concentration (%)	12
O_2-pressure (bar)	3, 5, 7
Temperature (ºC)	100
Treatment time (min)	60

The economic, physical, and optical properties, in terms of determining the most appropriate conditions, were identified as the first stage of oxygen bleaching. Before the SPBTH bleaching stage, the pulp was pretreated with 0.3% EDTA for 60 min at 80°C to eliminate undesirable heavy-metal ions (Chelating Stage). The pulp concentration was 12% and the pH ranged from 5 to 7 in this stage. Then, the SPBTH bleaching stage was applied as the last stage. In this bleaching stage, the amount of SPBTH and applied time were set as independent variables (Table 4).

Table 4. Sodium Perborate Tetrahydrate (SPBTH) Bleaching Conditions

SPBTH Level (%)	2, 4, 6
Treatment time (min)	60, 70, 80
$MgSO_4$ (%)	0.5
Temperature (ºC)	70
Concentration (%)	12

Statistical Analysis

A one-way ANOVA test was used to examine if a statistically significant difference occurred on a brightness level, tensile index, burst index, and tear index of the paper pulps when the bleaching time and SPBTH level were changed. Among the post-hoc tests, the Duncan test was used in order to determine the inter-group differences.

RESULTS AND DISCUSSION

Effect of Oxygen Pressure

The physical, optical, and chemical results of the bamboo pulp, to which oxygen delignification was applied, are reported in Table 5 and Fig. 1.

Table 5. Chemical, Optical, and Physical Properties of Oxygen Delignification on Kraft-AQ Bamboo Pulps (50±5 SR°).

O_2 Pres.	Kappa Num.	Pulp viscosity (cm^3/g^{-1})	ISO Bright (%)	Yellow. (E313) (%)	ISO Opac. (%)	Tensile Index ($Nm.g^{-1}$)	Tear Index ($mN.m^2.g^{-1}$)	Burst İndex ($kPa.m^2.g^{-1}$)
Con.	20.03	1164	26.91	45.76	97.33	39.04	7.93	2.41
3 bar	7.08	889	43.59	37.91	88.38	50.72	9.32	3.59
5 bar	6.71	882	53.29	27.41	83.38	46.34	8.85	3.17
7 bar	6.25	750	53.82	27.27	83.90	41.13	8.53	2.86

It can be seen that the oxygen pressure had a significant influence on the kappa number, viscosity, and brightness (Fig. 1). For example, while the brightness was 26.91% with the control sample, the same value was found to be 53.82% when using an oxygen pressure of 7 bars. The bamboo pulp brightness reached its maximum value at an oxygen pressure of 7 bars.

Table 5 and Fig. 2 illustrate the effect of different oxygen pressures on some of the physical properties of the kraft-AQ bamboo handsheets. An increase in the oxygen pressure constant to 3 bars resulted in minor increases in tensile, burst, and tear index values. It can be deduced that this situation is a result of an increase in the rate of lignin removal having a positive impact on the paper's physical properties (Kırcı 2002). However, continued addition of oxygen pressure in the bleaching shows a reversion in the increase of tensile, burst, and tear index value, as illustrated in Fig. 2. The maximum physical properties were obtained with an oxygen pressure charge of 3 bars.

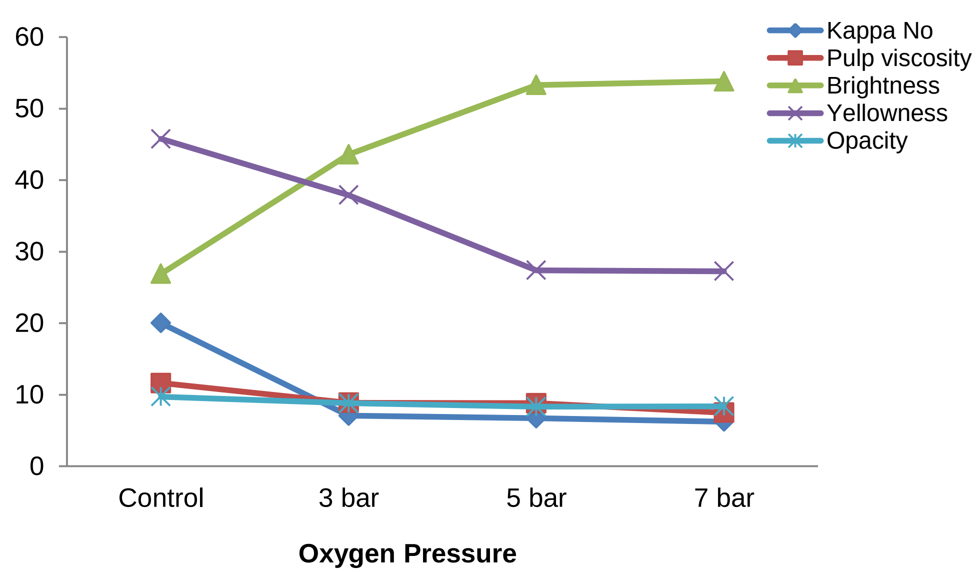

Fig. 1. Effect of oxygen pressure on the chemical and optical properties of bamboo kraft-AQ pulps

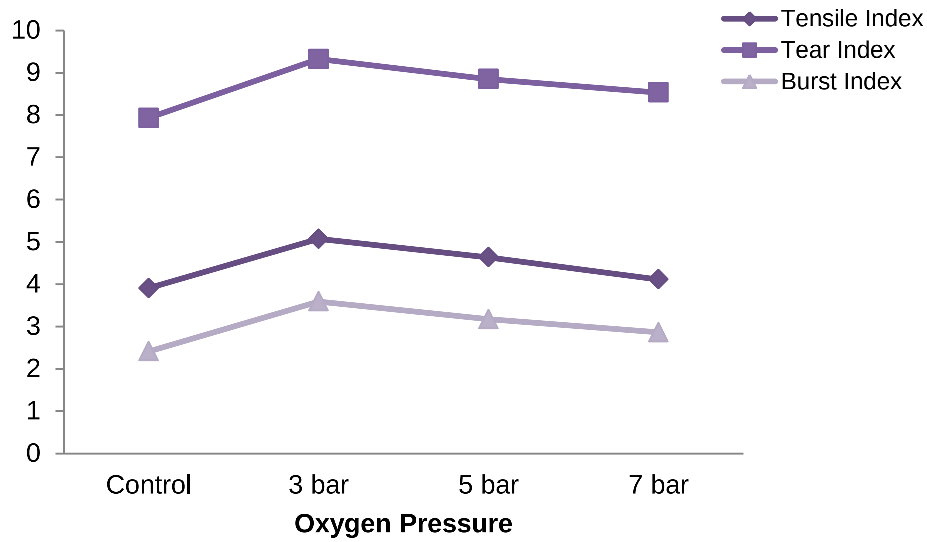

Fig. 2. Effect of oxygen pressure on the physical properties of bamboo kraft-AQ pulps

The values of chemical, optical, and physical changes were in agreement with the existing literature (Mittal *et al.* 1994; Joutsimo 2002; Vu *et al.* 2004; Thomas *et al.* 2007; Dutt *et al.* 2009). These results indicate that the most appropriate oxygen pressure was 5 bars. The reason for this was that the physical properties of the bleached samples showed a very moderate decrease compared to the control sample. Moreover, the kappa number is the desired value for the other bleaching stages without a severe decline in the viscosity. In this study, chelation and SPBTH bleaching was applied after the oxygen delignification reached 5 bar.

Effect of Sodium Perborate Tetrahydrate Level and Bleaching Time

According to Table 6, when all of the other conditions were kept constant, the brightness value increased, but the bleaching yield, kappa number, yellowness, and pulp viscosity decreased with an increasing SPBTH ratio and bleaching time. For example, while the bleaching yield was 98.63% with a 2% SPBTH ratio, the same value was found to be 97.85% with a 4% SPBTH ratio. This result is compatible with the literature (Kırcı et al. 2004).

Table 6. Chemical, Optical, and Physical Properties of SPBTH Bleaching on Kraft-AQ Bamboo Pulps (50±5 SR°)

SPBTH Rate	Time (Min)	Kappa No	Bleach Yield (%)	Pulp vis. (cm^3/g^{-1})	ISO Bright (%)	Yellow (E313) (%)	ISO Opac (%)	Tensile İndex (Nm.g^{-1})	Tear Index (mN.m^2.g^{-1})	Burst Index (kPa.m^2.g^{-1})
2 %	60	4.96	98.63	836.40	60.02	21.62	83.34	42.44	9.39	2.95
	70	4.80	98.57	827.17	60.75	21.17	82.39	53.45	9.47	4.00
	80	4.55	98.06	822.59	61.11	20.99	81.83	54.12	9.59	4.19
4 %	60	4.40	97.85	831.19	61.38	20.04	81.29	51.51	10.78	3.83
	70	4.38	97.29	817.55	64.16	19.85	80.77	53.96	10.81	4.12
	80	4.35	97.20	800.51	64.70	19.45	79.64	57.83	10.99	4.51
6 %	60	4.04	96.23	810.27	65.30	17.79	79.42	51.01	7.99	3.75
	70	3.72	96.21	796.44	65.71	17.63	79.35	51.50	8.08	4.05
	80	2.26	96.06	749.90	65.80	17.47	79.31	52.95	8.17	4.10

Similarly, by using SPBTH for the bleaching of bamboo pulp, the brightness of the pulp was increased from 53.29% to 63.80% ISO. The pulp brightness reached its maximum value at a bleaching time of 80 minutes and a 6% SPBTH level (Fig. 3). Increasing the bleaching time had a positive effect on the brightness level of pulp. For instance, at a 60 min bleaching time the ISO brightness was 60.02%, whereas at 70 and 80 min bleaching times, the ISO brightness was found to be 60.75% and 61.11%, respectively, while the SPBTH level remained constant at 2%. The yellowness of the bleached pulps decreased (27.4 to 17.47%) with an increased percentage of the SPBTH level and bleaching time. Increasing the bleaching time to 60, 70, and 80 min, while keeping other bleaching conditions constant, slightly decreased the pulp opacity. At the same, increasing the SPBTH level to 2%, 4%, and 6% slightly decreased the pulp opacity while the other bleaching conditions were kept constant. In these trials, the reagents $MgSO_4$, Na_2SIO_3, temperature, and concentration were constant at levels of 0.5%, 3%, 70°C, and 12%, respectively. Thus, one can conclude that kraft-AQ bamboo pulp can be bleached at 65.80% ISO brightness with a third stage of bleaching using SPBTH as a chemical agent. Also, the role of SPBTH addition level with respect to optical properties is greater than that of the bleaching time.

SPBTH charges of 2 to 6% and bleaching times of 60 to 80 min applied at the oxygen stage resulted in pulps with lower kappa number and higher viscosity.

Sodium perborate can be prepared from sodium peroxide and boric acid (Mathews 1911). Hence, the bleaching mechanism of sodium perborate is similar to bleaching mechanism of hydrogen peroxide. Hydroperoxide anion (HOO-) is an active species and is responsible for the bleaching action of hydrogen peroxide under alkaline conditions. Otherwise, hydroperoxyl and hydroxyl radicals generated by decomposition of hydrogen peroxide are responsible for delignification. Hydroxyl radicals are capable of attacking practically all types of organic structures, including those containing hydroxyl and ether linkages (Parthasaraty *et al.* 1990). Similar reactions occur with sodium perborate, but its alkalinity is higher than that of hydrogen peroxide; in this respect sodium perborate has significant effects on delignification at same active oxygen charge (Pesman *et al.* 2011).

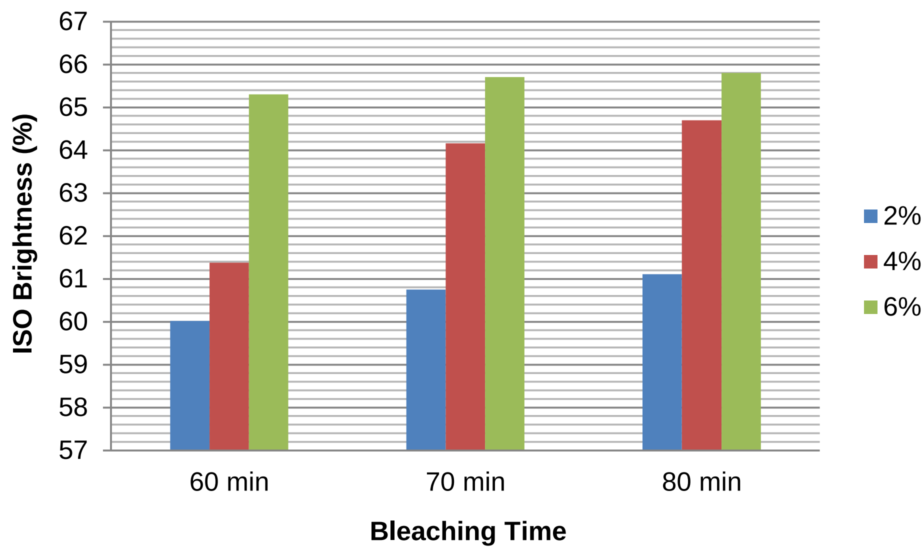

Fig. 3. Effect of SPBTH level and various bleaching times on ISO brightness for kraft-AQ bamboo pulp

A one-way ANOVA test was used to determine changes in the brightness level when the bleaching time was increased to 60, 70, and 80 min at SPBTH levels of 2, 4, and 6%, and it was observed that they formed different groups ($p < 0.05$). According to the results of the Duncan Test, which was made to determine those differences, each bleaching time was included in a different group at a concentration of 2%, while only the bleaching time of 60 min formed a different group at the concentrations of 4% and 6%. Similarly, the bleaching concentration was increased to 2%, 4%, and 6%, and a one-way ANOVA test was used to examine the change in the brightness level at the bleaching times of 60, 70, and 80 min; as a result of the evaluation, the formation of different groups was found ($p < 0.05$). Based on the results of the Duncan test to determine those differences, each bleaching concentration formed a statistically distinct group at bleaching times of 60, 70, and 80 min.

Based on the literature, when hydrogen peroxide is used with sodium perborate monohydrate for physical pulp, the brightness value is increased, but the yellowness value is decreased. While the unbleached pulp brightness value was reported as 50.7 (ISO brightness), the yellowness value was 30.9%; the brightness value increased by adding H_2O_2 at a rate of 2% and SPBTH at a rate of 2% (Celik *et al.* 2008).

The effect on the bleaching time and the SPBTH level on the physical properties of the hand sheets is illustrated in Table 6.

The effect of SPBTH level and bleaching time on the tensile index of bleached bamboo handsheets is reported in Fig. 4. An increase in the SPBTH charge to above 2% showed a positive effect on tensile index. However, tensile index was decreased with selection of the 6% SPBTH level. The maximum tensile index was obtained with a perborate charge selected as 4% and a bleaching time of 80 min. Table 6 and Fig. 4 show that bleaching time was the variable having the main positive effect on the tensile index for SPBTH bleaching.

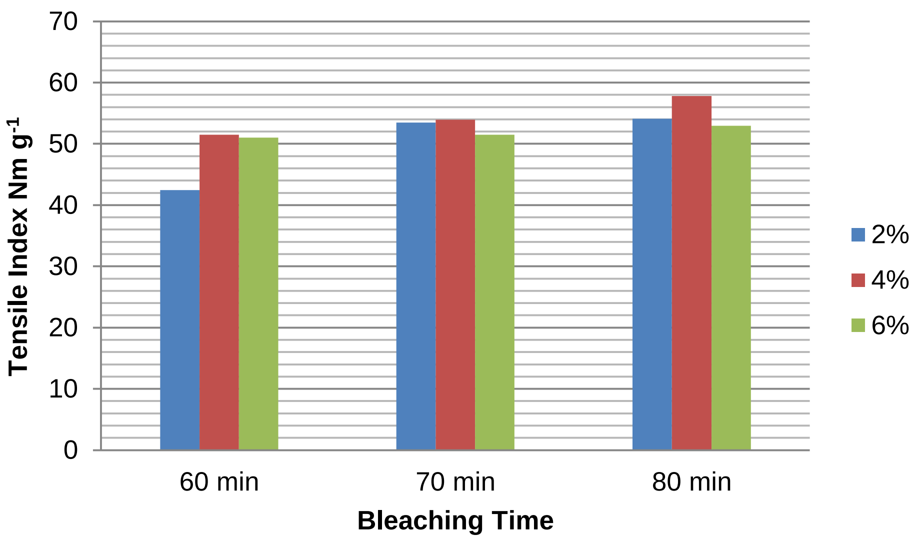

Fig. 4 Effect of SPBTH level and various bleaching times on tensile index for kraft-AQ bamboo pulp

A one-way ANOVA test was used to determine the change in the tensile index at concentrations of 2%, 4%, and 6% when the bleaching time was increased to 60, 70, and 80 min. The formation of different groups was found, except in the case of the concentration of 6% ($p < 0.05$). According to the results of the Duncan test, each bleaching time was included in a different group at a concentration of 4%, while only a bleaching time of 60 min formed a different group at a concentration of 2%. Similarly, a one-way ANOVA test was used to determine the change in the tensile index at bleaching times of 60, 70, and 80 min when the concentration was increased to 2%, 4%, and 6%, and the formation of different groups was found, excluding the bleaching time of 70 min ($p < 0.05$). The results of the Duncan test showed that a bleaching concentration of 2% at a bleaching time of 60 min formed a different group, while a bleaching concentration of 4% at a bleaching time of 80 min formed another different group.

Burst index results for the bleached bamboo handsheets with SPBTH are presented in Fig 5. The bleaching process significantly increased burst index. Moreover, the unbleached pulp showed a rather lower burst index compared to all the bleached pulps. It is notable that this result was obtained despite moderate decrease in viscosity by the bleaching process as tensile index. Figure 5 shows that burst index was generally increased along with increasing of the SPBTH level and bleaching time.

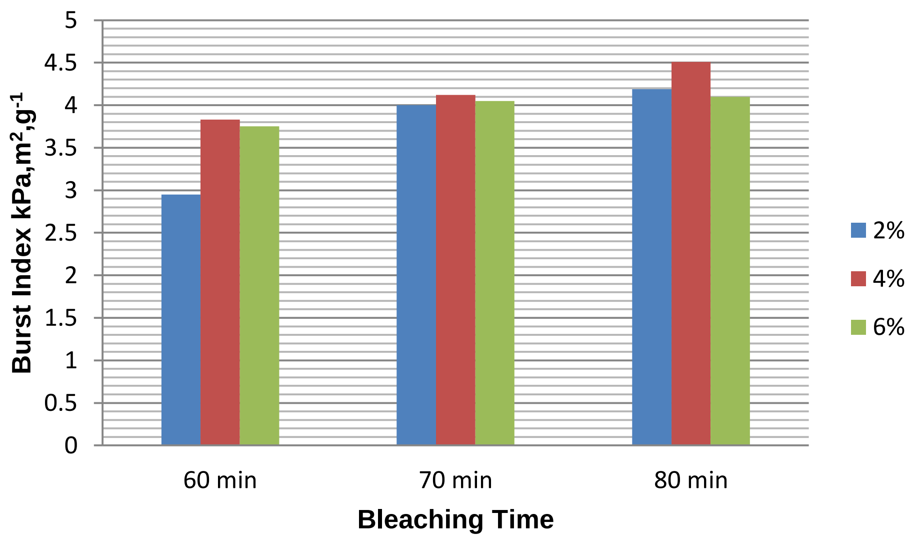

Fig. 5. Effect of the SPBTH level and various bleaching times on the burst index for kraft-AQ bamboo pulp

A one-way ANOVA test was used to determine the change in the burst index at concentrations of 2%, 4%, and 6% when the bleaching time was increased to 60, 70, and 80 min, and the formation of different groups was found ($p < 0.05$). According to the results of the Duncan test, which was done to determine those differences, each bleaching time formed a different group at a concentration of 4%, while only a bleaching time of 60 min was included in a different group at a concentration of 2%; a bleaching time of 70 min at a concentration of 6% formed a different group relative to both 60 min and 80 min. Similarly, a one-way ANOVA test was used to determine the change in the burst index at bleaching time of 60, 70, and 80 min when the concentration was increased to 2%, 4%, and 6%, and the formation of different groups was found, excluding the bleaching time of 70 min ($p < 0.05$). According to the results of the Duncan test, a bleaching concentration of 2% at a bleaching time of 60 min formed a different group, while a bleaching concentration of 2% at a bleaching time of 80 min formed another different group relative to the bleaching concentrations of 4% and 6%.

Tear index depends mostly on fiber length, but tensile index and burst index depend on relative bonded area of handsheet surfaces (Ahmed *et al.* 1998). Figure 6 shows tear index values of bleached bamboo pulps at various SPBTH levels and reaction times. According to this, the tear index increased and then decreased with increasing SPBTH level. Bleaching time had a positive effect on tear index (similarly to burst and tensile index) according to Fig 6. Tear index typically would be expected to show an inverse correlation with both tensile and burst index (Caufield *et al.* 1988). However, in the present study tear index showed a correlation with both tensile strength and burst index.

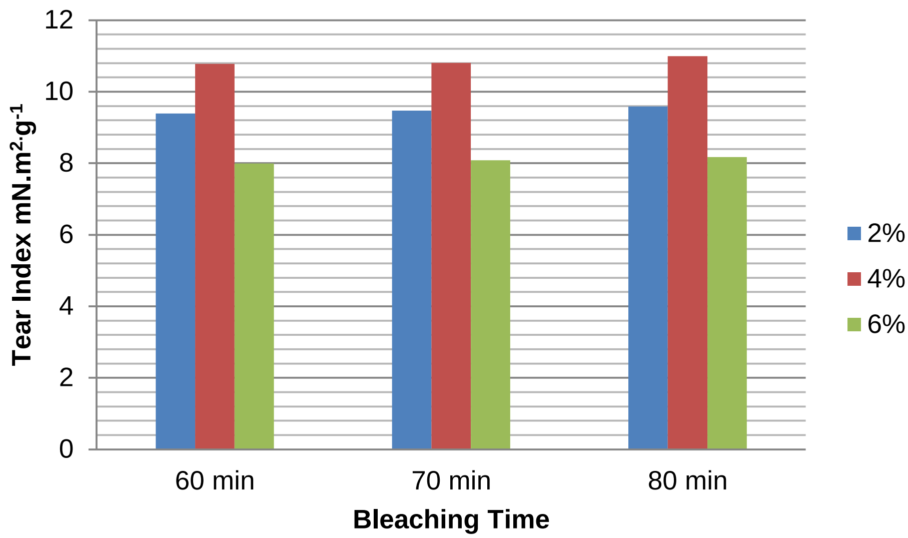

Fig. 6. Effect of the SPBTH level and various bleaching times on the tear index for kraft-AQ bamboo pulp

A one-way ANOVA test was used to determine the change in the tear index at concentrations of 2%, 4%, and 6% when the bleaching time was increased to 60, 70, and 80 min, and the formation of different groups was not found ($p > 0.05$). Similarly, a one-way ANOVA test was used to determine the change in the tear index at a bleaching time of 60, 70, and 80 min when the bleaching concentration was increased to 2%, 4%, and 6%; the formation of different groups was found ($p < 0.05$). According to the results of the Duncan test, which was done to determine those differences, a bleaching concentration of 2% at a bleaching time of 60 min formed a different group relative to a bleaching concentrations of 6% and 4%, while each bleaching concentration formed a different group at the bleaching times of 70 and 80 min.

Figures 4, 5 and 6 indicated that tensile, burst, and tear index first increased and then decreased with increasing SPBTH level, while bleaching time had a positive effect on physical properties.

According to Tutus *et al.* (2009), the oxygen bleaching process was determined as the first stage, and this provided the best result in the view of physical and optical properties. Then, in relation to the second stage, the oxygen and sodium perborate monohydrate bleaching experiments were carried out to determine the optimum bleaching condition for cereal straw. The results of that study revealed that the tear, burst, and tensile index increased while increasing the sodium perborate monohydrate (SPBMH) rate. Moreover, the SPBMH rate was kept constant, and the tear, burst, and tensile indexes increased with the increasing bleaching time (Tutus *et al.* 2009).

CONCLUSIONS

A third stage of oxygen, chelation with sodium perborate tetrahydrate bleaching was carried out under laboratory conditions on bamboo pulp, which was obtained using the kraft-AQ method. The most important findings from different bleaching experiments were:

1. Oxygen pressure affected chemical, physical, and optical properties on bleaching the pulp. A high oxygen pressure (7 bar) resulted in a lower viscosity and kappa, but a higher brightness.
2. As a consequence of three-stage bleaching, pulps with a relatively low kappa number (20.03 to 2.26) were produced, and the viscosity of the pulp was decreased from 1164 cm^3/g to 749.9 cm^3/g with oxygen bleaching done in the presence of SPBTH.
3. As expected, pulp strength properties improved with the usage of SPBTH in oxygen delignification bleaching. It can be deduced that the increase of the lignin removal had a positive impact on the paper's physical properties. As can be seen from the results for Kappa number, SPBTH can play an important role in delignification during oxygen bleaching.
4. The statistical analyses indicated that a 4% SPBTH level and a bleaching time of 80 minutes offer optimum results with respect to the optical and physical characteristics.

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