

Effect of Treating Birch Kraft Pulp with Peracetic Acid before O₂-Delignification on the Properties of Pulp and Consumption of ClO₂ in D₀ED₁ Bleaching

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The influence of pre-treating Kappa number of 18.0 industrial birch kraft pulp with peracetic acid (Paa) before oxygen delignification (OD) was studied relative to the pulps' level of delignification, yield, brightness, intrinsic viscosity (IV), FS-number, tear index (TI), and the consumption of chlorine dioxide (ClO₂) in its subsequent Elemental Chlorine Free (ECF) bleaching. The study showed that such pretreatment is a way to extend the delignification of such pulp quite selectively in OD based on values of IV, FS-number, and TI, and to reduce significantly the consumption of ClO₂ required to bleach this pulp to a brightness of 87%. This reduction depended on the amount of Paa used for pulp pretreatment. For example, when 0.33% and 0.66% as active oxygen (A.O.) of Paa were used, ClO₂ consumption to bleach the pulp to 87% was lowered by 15.4% and 42.3%, respectively. As high as a 61.5% reduction in ClO₂ consumption in its bleaching could be obtained using 0.66% as A.O. on pulp and change of time of OD. This result allows for a significant decrease in the chlorine passing to the filtrates from washing the pulp after the D₀ and E stages to the pulp mill wastewater treatment plant.

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INTRODUCTION

The biggest facilities now manufacturing fibrous papermaking pulps are kraft pulp mills. These mills mostly use the chlorine dioxide (ClO₂) bleaching method to whiten their pulps (Dence 1996). In this process, filtrates containing chlorine compounds harmful to the environment and recovery boiler are produced in D (chlorine dioxide) and E (alkaline extraction) stages. Therefore, they must be disposed of to the pulp mill wastewater treatment plant, which also causes losses of a certain amount of organic substances as fuel for the boiler and generates costs for their processing in the plant. Although these mills fulfill the current environmental legislation requirements in terms of chlorine effluent discharge from them, the regulations may change in the future, and the pulp industry is therefore still seeking new pulp bleaching methods to minimize the amount of filtrates containing chlorine from washing and directing as much as possible of these filtrates to combustion into the recovery boiler (Tripathi *et al.* 2020). This can be achieved by deeper delignification of wood in kraft pulping (Colodette *et al.* 2007), removing a greater amount of lignin (Saldivia 2002) and hexenuronic acids (Andrew *et al.* 2009) from pulp before

bleaching or replacing the D stages in the bleaching with Z (ozone) (Métais *et al.* 2011), P (hydrogen peroxide), OP (pressurized hydrogen peroxide) (Ragnar and Dahllöf 2002), and Paa (peroxy acetic acid) stages.

As for the removal of a greater amount of lignin from the pulp before bleaching, this can be done by optimizing the parameters of oxygen delignification (OD) process or activation of the residual lignin. The activation can take the form of pulp pretreatment using different peroxyacids such as performic acid (Hauque *et al.* 2019); peracetic acid (Paa) (Poukka *et al.* 1999; Turner *et al.* 2004; Chong *et al.* 2015; Zeinaly *et al.* 2019; Sharma *et al.* 2020); persulfuric acid (Allison and McGrouter 1995; Jafari *et al.* 2014; Rizaluddin *et al.* 2015); perphosphoric acid (Springer 1997); (Springer 1997; Rizaluddin *et al.* 2015; Haque *et al.* 2019; Zeinaly *et al.* 2019); salts of these acids (Springer and McSweeney 1993; Allison and McGrouter 1997; Zeinaly *et al.* 2019; Bourchard *et al.* 2000; Miri *et al.* 2015) or more “exotic” substances as polyoxometalates (Bujanovic *et al.* 2011).

This work is utilitarian and presents mainly the effect of pretreatment of industrial birch kraft pulp intended for bleaching (regular grade) with peracetic acid before the OD process on reduction of the consumption of ClO₂ in ECF bleaching (bleaching sequence D₀ED₁). Such a result turned out to be possible due to the extension of the delignification of industrial this pulp in the OD process. Additionally, the influence of deep delignification of this pulp on its strength properties [(FS-number and tear index (TI)] or properties having a significant impact on strength properties [intrinsic viscosity (IV)] was also examined. This work was conducted due to the lack of such a study in the literature on the subject that would determine the effect of peracetic acid pretreatment of industrial birch kraft pulp on chlorine dioxide consumption in its ECF bleaching, in particular in the aspects of the scope of possible obtaining extending of delignification of the pulp thanks to this type of pretreatment and the dependence of chlorine dioxide consumption for complete bleaching of this pulp on the obtained extending of delignification.

It should be mentioned that the work is a continuation of the topic, because this type of research was previously performed on regular grade pine kraft pulp (Danielewicz 2023). For this reason, this work has the status of brief communication.

EXPERIMENTAL

Pulps Used in Bleaching Experiments

Room-dried birch industrial, regular grade papermaking pulp (*i.e.*, intended for bleaching) KN 18.0 birch (*Betula pendula*) kraft pulp was used in this study. It was obtained from a Polish kraft pulp mill.

Applied Bleaching Chemicals

The following chemicals were used in this work: oxygen; distilled 37.5 to 40.5% peracetic acid (Sigma-Aldrich company); EDTA (POCH company); MgSO₄ (POCH company, 2% solution in distilled water); NaOH (POCH company); and ClO₂ (laboratory made from NaClO₂, 6.3 g/L).

Chelation (Q stage)

A sample of defibered and squeezed pulp was placed in a polyethylene bag. Heated distilled water (in the amount necessary to ensure the assumed consistency of a 10% fibrous slurry), sulfuric acid solution (to obtain an initial pH of 4.0 for the pulp slurry), and 0.25%

EDTA based on the oven-dried (o.d.) pulp were added to a beaker. After thorough mixing, the solution was poured into a polyethylene bag containing the pulp sample, from which excess water had been squeezed out. The content was mixed by kneading and heated in a water bath for 60 min at 70 °C. After the preset time, a sample of pulp was poured into a Büchner funnel lined with filter textile, squeezed, washed with two aliquots of 1000 mL distilled water, squeezed again, and stored in polyethylene bags.

Pulp Treatment with Peracetic Acid (Paa)

A sample of pulp was placed in a polyethylene bag. Heated distilled water (in the amount necessary to ensure the assumed consistency of a 10% fibrous slurry) and 0.33% (S1 pulp sample) or 0.66% (S2 and S3 pulp sample) Paa as active oxygen (A.O.) based on the o.d. pulp were added to a beaker. After thorough mixing of the ingredients, the solution was poured into the polyethylene bag containing pulp sample squeezed out of excess water. The content was mixed by kneading and heated in water bath for 30 min at 50 °C and then for 90 min at 70 °C. After treatment, every sample of pulp was washed and stored as described in the case of the Q stage. The A.O. concentration in a solution of Paa was determined by the titration method described by Amini and Webster (1995).

Oxygen Delignification (OD)

A portion of pulp was placed in a polyethylene bag, and 0.5% magnesium sulphate (based on the o.d. pulp) dissolved in 300 mL distilled water was added to it, followed by 1% NaOH (based on the o.d. pulp) along with the remaining amount of water necessary to obtain a pulp suspension consistency of 8 wt%. The content was mixed by kneading and then transferred to the autoclave of a Jayme rotary digester. The autoclave was closed, filled with oxygen to 8 MPa, and the rotating mechanism of the digester was switched on. The autoclave of the digester was heated to 100 °C within 30 min, and heating was then continued for 60 min in the case of samples S0, S1, and S2 and for 120 min in the case of sample S3. After the preset time, the digester was stopped, the autoclave was degassed and emptied, and every sample of pulp was washed and stored as described in the case of the Q stage.

Chlorine Dioxide Bleaching (D₀ and D₁ Stages)

Bleaching of the O₂-delignified pulp samples was performed according to the D₀E D₁ sequence, where D₀ is the first stage of bleaching, in which the residual lignin contained in the pulp is subjected to the action of ClO₂, as a result of which it becomes easily removed in the subsequent alkaline extraction process (E), while D₁ is the final stage of bleaching, in which the pulp is again treated with chlorine dioxide to decolorize the remaining colored lignin moieties. In this work, the total amount of chlorine dioxide used in the bleaching experiments of for S0 sample was 0.4 to 2.6%, while for S1-S3: 0.4 to 2.2% based on oven-dried (o.d.) pulp. In the D₀ and D₁ stages, 65% and 35% of these amounts were used, respectively.

The procedure for bleaching was as follows: a sample of the pulp was placed in a polyethylene bag. Distilled water and calculated amounts of chlorine dioxide solution containing an appropriate amount of ClO₂ to achieve a consistency of 10% of the fibrous suspension were then measured into the beaker. After mixing, the solution was poured into the polyethylene bag containing the pulp sample. The content was mixed by kneading and heated in a water bath for 60 min at 65 °C (D₀ stage) or 90 min at 75 °C (D₁ stage). After the assumed reaction time, the pulp samples were washed and stored as described in the

case of Q stage. The concentration of chlorine dioxide in the solution was determined by the titration method described by Modrzejewski *et al.* (1985).

Alkaline Extraction (E)

A sample of the pulp was placed in a polyethylene bag. The appropriate amount of sodium hydroxide solution containing 1.5% NaOH based on the o.d. pulp was measured into a beaker with distilled water in the amount necessary to achieve the consistency of a 10% fibrous slurry. After mixing, the solution was poured into the polyethylene bag to the pulp, which was mixed by kneading and then heated in a water bath for 90 min at 75 °C. After the preset time, every pulp sample was washed and stored as described in the case of Q stage.

Determination of Pulps' Properties

Standard methods were used to measure the kappa number [PN-70/P50095 (1985)], the brightness of the pulp [ISO 2470 (1999) in SpectroColor 01 (Spectrocolor company) apparatus, and IV [ISO 5351-11 (1981)]. The Pulmac fibre strength number (FS – number) was measured using handsheets of basis weight ~60 g/m² and using paper strips cut out of these handsheets of dimensions 100 mm × 25 mm, which were then torn off in a Troubleshooter Tester TS-100 (Pulmac Corp.) after their wetting with water according to the owner's manual for this apparatus (Cowan 1995; Pulmac 2001). The ~60 g/m² and ~75 g/m² handsheets (used in the measurement of TI of S0-S3 pulp samples) were prepared in standard Rapid-Köthen apparatus according to the PN-EN ISO 5269 (2001). The beating of the S0-S3 pulp samples needed for preparation of 75 g/m² handsheets was performed in a laboratory Jokro mill according to the PN-EN 25264-3 (1999) standard. The beating time was selected to obtain the breaking length of the pulp samples of 9000-10200 m. TI of pulp samples was determined according to the PN-EN ISO 1974 (2012) standard in the ProTear tester (Thwing-Albert, USA).

Elaboration of Results

The oxygen delignification (OD) experiments of birch kraft pulp were conducted twice. As mentioned, characterization of the pulp after OD involved determining the KN and brightness, and this was also performed twice. Therefore, the pulp yields, the KN, and brightness tests are the arithmetic averages of four measurements. The bleaching experiments were carried out only once because of (i) the large number of experiments (total number of bleachings - 26) enabling reliable curves to be drawn for the pulp brightness increase as a function of the total amount of ClO₂ dosed in bleaching, and (ii) the three-stage nature of each experiment (total number of bleaching stages performed - 78). The determination of the FS-number and TI of pulps was performed using 18 and 12 samples of paper cut out from best-formed handsheets. The dispersion of the results of all determinations in this study was presented in the form of deviation of the obtained results from their mean value.

RESULTS AND DISCUSSION

Table 1 shows the effect of the Paa pre-treatment of regular grade KN 18.0 birch kraft pulp on its KN after OD, as well as the yield and ISO brightness.

Table 1. Results of OD of Birch Kraft Pulp

Sample Symbol	Sequences and Amount of chemicals on Oven-dried Pulp	KN	KN Reduction ¹ (%)	Brightness (%)	Pulp Yield (%)	IV, cm ³ /g	FS-number, psi/15 mm	TI, mN·m ² /g
S0	Q, OD (1% NaOH, 100 °C, 60 min)	12.7 ± 0.3	-	44.3 ± 0.2	97.1 ± 0.3	895 ± 10	40.2 ± 1.0	8.1 ± 1.1
S1	Q, Paa (0.33% A.O.), OD (1% NaOH, 100 °C, 60 min)	8.7 ± 0.3	31.5	49.8 ± 0.3	96.6 ± 0.2	902 ± 12	39.5 ± 6.5	8.5 ± 0.6
S2	Q, Paa (0.66% A.O.), OD (1% NaOH, 100 °C, 60 min)	6.3 ± 0.1	50.4	53.4 ± 0.1	96.3 ± 0.1	905 ± 21	36.9 ± 4.1	8.9 ± 0.9
S3	Q, Paa (0.66% A.O.), OD (2% NaOH, 100 °C, 120 min)	4.9 ± 0.2	61.4	56.4 ± 0.2	95.8 ± 0.3	848 ± 26	41.3 ± 2.0	9.9 ± 0.3

¹ Relative to control sample (S0)

² at breaking length 9210-10180 m

As shown in Table 1, the pretreatment of the pulp with distilled Paa in amount of 0.33% and 0.66% of A.O. based on oven-dried (o.d.) pulp before its OD enabled its delignification to a KN of 8.7 and 6.3, respectively, *i.e.*, a 31.5% and 50.4% reduction in the content of substances oxidized by 0.1 n potassium permanganate (KMnO₄) in KN test, respectively, when compared with sample S0. Increasing the amount of NaOH dose in OD from 1% to 2% based on o.d. pulp and extending the OD process from 1 h up to 2 h for sample S3 allowed its further delignification to a KN of 4.9 with a 61.4% reduction in the content of substances oxidized by KMnO₄ compared with sample S0. As already mentioned in the previous work, similar large reductions in the content of substances oxidized with KMnO₄ in KN test after treatment with peroxygen compounds were also presented by other authors (Haque *et al.* 2019; Zeinaly *et al.* 2019), and this may be a result of a fairly wide spectrum of lignin (Delagoutte *et al.* 1999; Jääskeläinen and Poppius-Levlin 1999; Chong *et al.* 2015) and certain unsaponified residues of carbohydrates origin that Paa is able to chemically modify (Tavast *et al.* 2011; Estaves *et al.* 2020).

In addition to the ability to delignify kraft pulps intended for bleaching, Paa's ability to brighten them is also valuable (Tavast *et al.* 2011; Kowalska and Ramos 2014). As shown in Table 1, the brightness of Paa-O pre-bleached S1, S2, and S3 birch pulp samples was increased 5.5%, 9.1%, and 12.1% compared to the S0 sample, respectively, but this was closely related to the decrease in the KN of these samples in the OD process. Apart from this, the 12.1% increase in brightness of this pulp for sample S3 over S0 is noteworthy.

Table 1 also contains the yields of pulp samples S0 to S3 after delignification according to the Q-O and Q-Paa-O sequences. This data indicates that the pulp yield was reduced by 0.5%, 0.8%, and 1.3% as the delignification of the pulp was increased by 31.5%, 50.4%, and 61.4% in the S1, S2, and S3 experiments, respectively, compared with S0.

The influence of the modification of the initial delignification of birch pulp by applying its peracetic acid pretreatment on their strength properties was assessed by determining their IV, the FS-number (Wet Zero-Span Tensile Strength W-ZSTS), and the TI of the pulps before their bleaching. Bleaching with chlorine dioxide is known to selectively affect lignin, and therefore the differences in the fibre strength of the pulp samples that can be noted before bleaching should be retained after this process, especially if a smaller amount of chlorine dioxide was used to completely bleach them after their peracetic acid treatment compared to the reference sample. As for the influence of the peracetic acid treatment on the IV of samples S1-S3, the data in Table 1 indicate that the value of this index remained at the level of the S0 sample, *i.e.*, approx. 900 cm³/g. A decrease in the IV can be observed in sample S3, which is related to the extension of the oxygen delignification process time in the case of this sample. To verify these results, as it was mentioned the strength of the fibers of samples S0-S3 (FS-number index) and TI of sheets of these samples were additionally determined (TI determined at their breaking strength of 9210-10180 m) (Table 1). These results indicate comparable strength of fibers of samples S0, S1, and S3, and even a positive effect of the application of pulp treatment with peracetic acid on this feature of fibers (FS-number of S3 sample) and tear resistance (TI of S0 and S1-S3 samples). It seems that may be partly due to the increase in cellulose content in more delignified samples S1-S3 than S0.

It is self-evident that a decrease in the KN of pulp before bleaching obtained by treating the pulps with Paa before the OD will influence the consumption of bleaching chemicals in its bleaching. To determine the effect that an extension of the delignification of birch kraft pulp in the Q-Paa-O sequence has on the amount of ClO₂ required for full bleaching of this pulp, oxygen delignified pulp samples S0 to S3 were bleached according to the DoED₁ sequence with an increasing amount of ClO₂. The relationship between the amount of ClO₂ dosed in the bleaching process and pulp brightness is shown in Fig. 1.

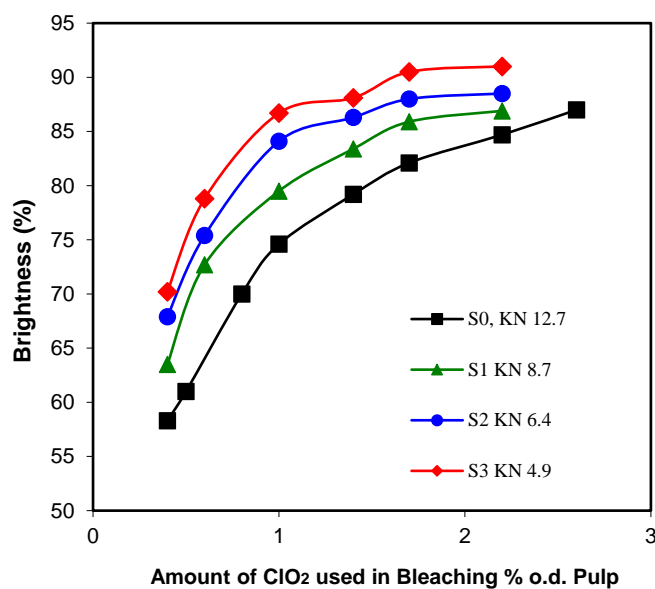


Fig. 1. The dependence of brightness of regular grade birch kraft pulp with different initial KN after OD on the amount ClO₂ used in bleaching

As shown in Fig. 1, the pretreatment of birch kraft pulp samples with Paa before OD enabled a notable reduction in the amount of ClO_2 required to bleach the birch kraft pulp to 87% ISO-brightness. The reduction is shown in Fig. 2.

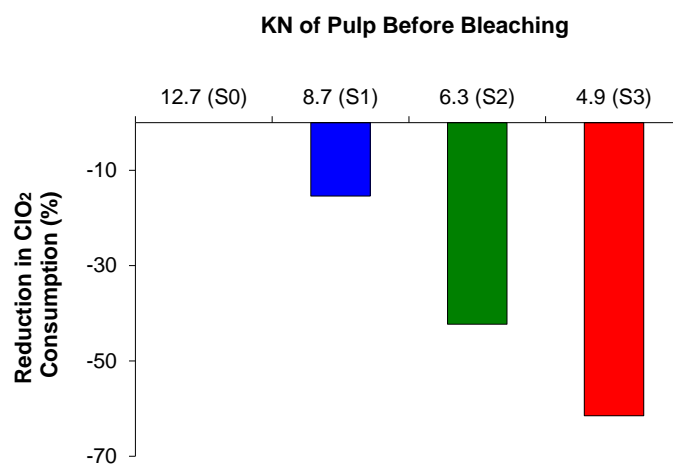


Fig. 2. Reduction of the amount of ClO_2 required for bleaching of regular grade birch pulp to 87% ISO brightness, due to reduction of the content susceptible to oxidation substances in birch pulp before the bleaching process

Figure 2 shows that the pretreatment of birch kraft pulp samples with Paa before OD in the amount of 0.33% and 0.66% A.O. based on oven-dried (o.d.) pulp during 60 min reduced the amount of ClO_2 required to bleach birch pulp to brightness of 87% ISO by 15.4% and 42.3%, respectively. For the S3 pulp sample, which had a KN of 4.9 units before bleaching, a 61.5% relative reduction in ClO_2 consumption was achieved to 87% brightness. As stated earlier (Danielewicz 2023), this large scope of the reduction, apart from the lower content of lignin and the higher brightness of sample S3 compared to S2, can also result from a significant increase in the bleaching susceptibility of sample S3 after its lignin content dropped below the limit level of this increase (Costa and Colodette 2007; Barros *et al.* 2010; Costa *et al.* 2022).

Other issues that should be addressed include the content of chlorine compounds in filtrates from birch pulp bleaching and the method of rational management of filtrate from washing the pulp after the Paa stage.

As for the first issue it is obvious that the removal of more lignin in the OD by introducing the Paa stage before this process allows for a significant reduction in the amount of chlorine used in bleaching and at the same time in the filtrate from the pulp washing after the D_0 stage and the E stage (if we assume that not all of the filtrate from the E stage is used for washing the pulp after the D_0 stage).

In solving the second problem, the results of the work of Rivera *et al.* (2021) could be used. These authors provided few possibilities for reducing the consumption of fresh wash water in such a mill washing department in which bleaching according to $D_0E_1D_1E_2D_2$ is used. For example, they present the possibility of using the acid filtrate from the D_1 stage as part of the liquid used to dilute the pulp suspension before the D_0 stage and wash the pulp after this stage, or even the E_1 stage. It is therefore possible to assume that the filtrate from the Paa stage could be used similarly, especially since this filtrate does not contain chlorine. It's also important to keep in mind that, because the peracetic acid may not have completely reacted, the filtrate from the Paa stage may still have some

residual capacity for delignification. As a result, using this filtrate to wash pulp after the D₀ or D₀E stages may result in an additional effect of delignification or activation of lignin.

CONCLUSIONS

1. Pre-treatment of regular grade birch kraft pulp with peracetic acid (Paa) before an oxygen delignification (OD) stage, as in the case of regular grade pine kraft pulp, was found to be an effective way to extend the delignification of the pulp in this process with a moderate decrease in pulp yield.
2. The extension of delignification of birch kraft pulp in an OD process from Kappa number (KN) 12.7 to 8.7 and 6.3, due to its pretreatment with Paa made it possible to obtain a 15.4% and 42.3% relative reduction of chlorine dioxide consumption in its elemental chlorine free (ECF) bleaching to ISO brightness of 87%, respectively.
3. A decrease in the KN of the regular birch kraft pulp to 4.9 units was due to its pre-treatment with Paa and extension of the time of the OD process caused high, *i.e.*, 61.5% relative reduction in chlorine dioxide consumption for its bleaching.
4. By increasing the amount of lignin removed in the OD stage from the birch kraft pulp thanks to the pretreatment with Paa before ECF bleaching, the amount of chlorine that passes to the filtrates from washing the pulp after the D₀ and E stages sent to the wastewater treatment plant was significantly reduced, and the amount of organic substances in the filtrates after the OD stage directed to a recovery boiler was increased. On the basis of the results presented by other authors, it can be supposed that the filtrate from the Paa stage can be used beneficially in the bleaching department of a kraft pulp mill for reduction of consumption of fresh water for washing and maybe even obtaining additional delignification/activation of lignin of pulp subjected to bleaching.

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