The Effect of Treating Pine Regular Kraft Pulp with Peracetic Acid before O_2-Delignification on the Consumption of ClO_2 in D_0ED_1 Bleaching

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Effects of pre-treating pine kraft pulp kappa number 31.5 with peracetic acid (Paa) (0.33% and 0.66% as active oxygen based on oven-dried pulp) before O_2-delignification were studied relative to the consumption of chlorine dioxide in a subsequent Elemental Chlorine Free (ECF) bleaching process. The study showed that such pre-treatment is an effective way to extend the delignification of the pine pulp and to reduce the consumption of chlorine dioxide required to bleach the pulp to a brightness of 87%. The reduction in chlorine dioxide required to bleach the pine pulp depended on the amount of Paa used for pre-treatment. Furthermore, the research showed that decreasing the kappa number of the pine pulp to approximately 7.5 units (with Paa pre-treatment and lengthening of O_2-delignification process) caused an over-proportional reduction (by approximately 62.5%) in chlorine dioxide consumption to bleach the pulp to 87% brightness.

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INTRODUCTION

The low brightness of kraft pulps (25 to 35%) is one of their basic negative features (Norrstrom 1969; Mróz and Surewicz 1976). Because of this, bleaching them to approximately 88 to 90% requires several stages (Dence 1996). In the late 80s, there was a significant breakthrough in the bleaching of kraft pulps as a result of replacing chlorine with chlorine dioxide, reducing the harmful substances (*e.g.* dioxins) that were formed in bleaching processes using the former chemical (Kachi *et al.* 1980; Voss *et al*. 1980; Carrier 1987). Bleaching kraft pulps with chlorine dioxide, called ECF (elemental chlorine free) technology, is one of the best available bleaching technologies, partly due to the negligible toxicity of the chlorine compounds formed in this process (Adams 1995; McDonough 1996). The significantly lower toxicity of these compounds compared to those formed in bleaching processes using elemental chlorine results from a difference in the reaction mechanism of these two bleaching agents with the residual lignin remaining in the pulp. Unlike chlorine, which can simultaneously chlorinate and oxidize lignin, ClO_2 primarily oxidizes it (Solomon 1996).

In modern bleached kraft pulp mills using ECF bleaching technology, 50 to 90% of the total aqueous discharge originates from the bleaching plant. Although these mills

fulfill the current environmental legislation requirements in terms of effluent discharge, the regulations may change in the future, and the pulp industry is therefore seeking new pulp bleaching methods to minimize the environmental impact. Consequently, one of the main research trends in kraft pulp bleaching over many years has been to reduce the amount of chlorine compounds discharged from bleaching processes to the sewage treatment plants of kraft pulp mills (Karim 2011; Tripathi *et al.* 2020; Starrsjö *et al.* 2021; Oliveira *et al*. 2022). This can be achieved by bleaching pulps with lower levels of lignin and hexenuronic acid residues than conventional, as well as optimizing the parameters of this process. A lower lignin content can be obtained in kraft pulps by deeper delignification of wood in the kraft pulping (Andtbacka and Tibbling 1994; Colodette *et al.* 2007; Segura *et al*. 2016) and oxygen delignification processes. In the latter process, the amount of lignin that can be removed from pulps is limited by the observed decrease in delignification selectivity after the removal of 40 to 50% of its content. This is because, in addition to the decrease in the residual lignin content of the pulp, the amount of lignin moieties having a degradation activation energy equal or even greater than the degradation process of polysaccharides increases (Karhunen *et al.* 1994; Akim *et al*. 2001). To delignify kraft pulps of low residual lignin content more selectively in bleaching, it has been proposed to use two-stage oxygen delignification of the pulps (Saldivia 2002; Johnson *et al*. 2009) or to pre-treat them with substances that activate the residual lignin moieties of reduced susceptibility for delignification (Fossum and Marklund 1988), 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); and salts of these acids (Springer and McSweeny 1993; Allison and McGrouter 1997; Zeinaly *et al*. 2019; Bourchard *et al*. 2000).

Paa can be used in several stages of bleaching kraft pulp. This work investigated the effect of the pre-treatment of regular grade (*i.e.*, intended for bleaching) pine kraft paper pulp with a kappa number of 31.5 before its subsequent oxygen delignification, mainly in terms of the amount of lignin removed in the latter process and the demand for chlorine dioxide to bleach this pulp to ISO 87% brightness.

EXPERIMENTAL

Pulps

Dry pine industrial-grade, regular (*i.e.*, intended for bleaching), kappa number 31.5 kraft pulp (*Pinus sylvestris* wood) was used in this research. It was obtained from a Polish kraft pulp mill.

Applied Delignification and Bleaching Chemicals

Oxygen; distilled 37.5-40.5% peracetic acid (Sigma-Aldrich company); EDTA (POCH company); $MgSO_4$ (POCH company, 2% solution in distilled water); NaOH (POCH company); and ClO_2 (laboratory made from $NaClO_2$, 6,3 g/L) were used in this work.

Chelation (Q stage)

After fiberization (to separate fibre bundles) at a consistency of 2%, the pulp was thickened to about 30% using cheesecloth. 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 thoroughly mixing with the pulp, the mixture was poured into a polyethylene bag. The content was further 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 a polyethylene bag at 4 °C for further processing.

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% (S2 pulp sample) or 0.66% (S3 and S4 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 (O)

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 3% 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 S1, S2, and S3 (process symbol - O_{60}) and for 120 min in the case of sample S4 (O_{120}). 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 (D_0 and D_1 Stages)

Bleaching of the O_2-delignified pulp samples was performed according to the D_0ED_1 sequence, where D_0 is the first stage of bleaching, in which the residual lignin contained in the pulp is subjected to the action of ClO_2, as a result of which it becomes easily removed in the subsequent alkaline extraction process (E), while D_1 is the final stage of bleaching, in which the pulp is again treated with chlorine dioxide to remove the remaining colored lignin moieties. In this work, the total amount of chlorine dioxide used in the bleaching experiments was 0.4-3.2% based on the o.d. pulp for S1 sample; 0.2-2.6% for S2, 0.2-2.6% for S3, and 0.1-1.8% for S4. In the D_0 and D_1 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 ClO_2 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 (D_0 stage) or 90 min at 75 °C (D_1 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)] and the brightness of the pulp [ISO 2470 (1999) in SpectroColor apparatus].

Elaboration of Results

The oxygen delignification experiments of pine kraft pulp were conducted twice. As mentioned, characterization of the pulp after O_2-delignification involved determining the kappa number and brightness, and this was also performed twice. Therefore, the pulp yields, the kappa number and brightness tests are the arithmetic averages of two measurements. The bleaching experiments were carried out once due to: (i) the large number of experiments (total number of bleachings 28) enabling reliable curves to be drawn for the pulp brightness increase as a function of the total amount of ClO_2 dosed in bleaching, and (ii) the three-stage nature of each experiment (total number of stages 84).

RESULTS AND DISCUSSION

Table 1 shows the effect of the Paa pre-treatment of regular pine kraft pulp on its kappa number after oxygen delignification, as well as the yield and ISO brightness.

Table 1. Results of O_2-delignification of Pine Kraft Pulp

Sample Symbol	Sequences and Amount of Peracetic Acid (Paa) on Oven-dried (o.d.) Pulp	Kappa Number	Kappa number reduction[1] (%)	Pulp yield (%)	Brightness (%)
S1	Q, O	13.9 ± 0.6	-	95.9 ± 0.3	37.1 ± 0.9
S2	Q, Paa (0.33% A.O.), O_{60}	11.1 ± 0.8	20.1	95.5 ± 0.2	40.5 ± 0.6
S3	Q, Paa (0.66% A.O.), O_{60}	9.3 ± 0.7	33.1	94.8 ± 0.2	43.3 ± 0.9
S4	Q, Paa (0.66% A.O.), O_{120}	7.5 ± 0.5	46.0	94.5 ± 0.2	46.3 ± 0.4

[1] Relative to control sample (S1)

As can be seen in Table 1, the pre-treatment of 31.5 kappa pine kraft pulp with distilled Paa (0.33% and 0.66% of A.O. based on the o.d. pulp) before its oxygen delignification enabled its delignification to a kappa number of 11.1 and 9.3, respectively, *i.e*., a 20.1% and 33.1% reduction in the content of substances oxidized by $KMnO_4$,

respectively, compared with sample S1. Extending the oxygen delignification process from 1 h up to 2 h in the case of pulp sample S4 allowed its further delignification to a kappa number of 7.5, *i.e*., a 46% reduction in the content of substances oxidized by $KMnO_4$ compared with sample S1. These percentage reductions in the kappa number of pulps samples correspond with the results seen in other studies. For example, Zeinaly *et al.* (2019) pre-treated bagasse pulp with Paa [(obtained from a reaction of tetraacetylethylenediamine (TAED) with hydrogen peroxide)] and achieved an approx. 53% reduction of its kappa number, while Haque *et al.* (2019) treated acacia pulp with performic acid, obtaining a 16 to 35% reduction of the index.

The obtained pulp activation using Paa results from the different chemistry of action of this compound with residual lignin in comparison to oxygen in the alkaline environment because it reacts not only with the phenolic rings of the lignin monomeric units but also with the non-phenolic residues of these units (Delagoutte *et al*. 1999; Chong *et al*. 2015). Moreover, it is reported that the treatment of kraft pulps with Paa effectively reduces the content of certain lignin-carbohydrate complexes (Jääskeläinen and Poppius-Levlin 1999) and hexenuronic acids contained in these pulps (Tavast *et al.* 2011; Estaves *et al.* 2020).

Paa also has the ability to brighten pulp (Tavast *et al*. 2011; Kowalska and Ramos 2014). This can also contribute to a reduction in the amount of bleaching agent required to fully bleach an unbleached pulp. An example of this is the better susceptibility of regular sulfite pulp to bleaching (brightness after pulping 51 to 58%) than kraft pulps (brightness of 25 to 30%). Fletcher *et al*. (1997) reported that sulfite pulp with an initial kappa number of 20 can be completely bleached using a Totally Chlorine Free bleaching sequence, while kraft pulp with an initial kappa number of 20 must be oxygen delignified to a kappa number ~ 10 before such bleaching to achieve comparable brightness. As can be seen in Table 1, the brightness of PaaO pre-bleached S2, S3, and S4 pulp samples was increased by 3.4, 6.2, and 9.2% compared to the S1 sample, respectively, but this was closely related to the decrease in the kappa number of these samples in the oxygen delignification process. For this reason, the ability of Paa to brighten unbleached pine pulp seems to be moderate, although the 9.2% increase in brightness of this pulp for sample S4 over S1 is noteworthy.

Table 1 also contains the yields of pulp samples S1-S4 after delignification according to the QO and QPaaO sequences. These data indicate that the pulp yield was reduced by 0.4, 1.1, and 1.4% as the delignification of the pulp was increased by 20.1, 33.1, and 46.0% in the S2, S3, and S4 experiments, respectively, compared with S1. Although the number of these experiments is small, the pulp yield reduction can be considered normal or even relatively low because part of the loss is residual lignin extracted from the pulp. It is possible that treatment of the pulp with Paa before being subjected to oxygen delignification lowers the activation energy of the residual lignin moieties (activating them), which redirects the harmful action of HO^- anions and active form of oxygen (*e.g.*, HO• radicals) from carbohydrates to the lignin, resulting in them being less susceptible to dissolution in O_2-delignification.

A decrease in the kappa number of pulps before bleaching influences the consumption of bleaching chemicals (Durbeuil *et al*. 2000). To determine the effect that an extension of the delignification of pine kraft pulp in the QPaaO sequence has on the amount of chlorine dioxide required for full bleaching of this pulp, oxygen delignified pulp samples S1-S4 were bleached according to the D_0ED_1 sequence with an increasing amount of chlorine dioxide. The relationship between the pulp brightness and the amount of chlorine dioxide dosed in the bleaching process is shown in Fig. 1.

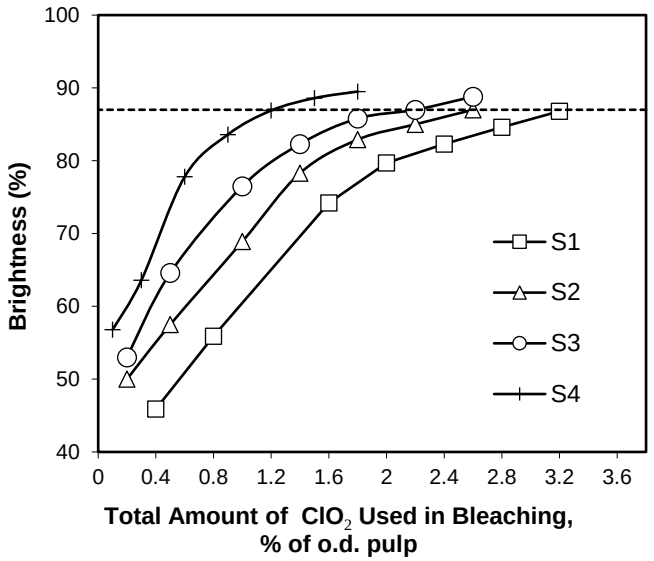

Fig. 1. Interrelation of the ISO brightness of samples of pine kraft pulp delignified with oxygen, with and without the use of peracetic acid pre-treatment, with the total amount of chlorine dioxide used in bleaching

As can be seen in Fig. 1, the pre-treatment of pine kraft pulp samples with Paa before O_2-delignification enabled a notable reduction in the amount of chlorine dioxide required to bleach the pine kraft pulp to 87% ISO-brightness. The reduction is shown in Fig. 2.

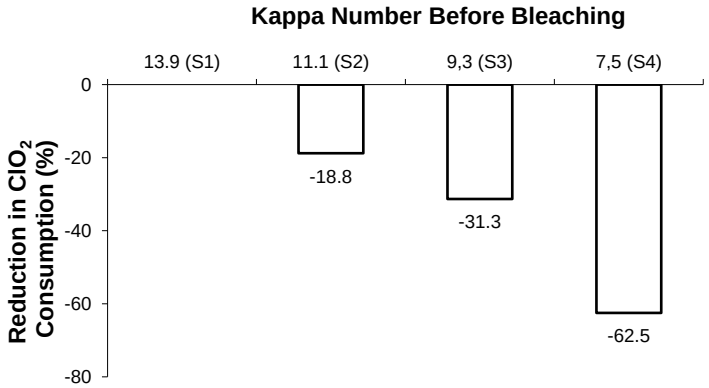

Fig. 2. Reduction of the total amount of chlorine dioxide required to bleach pine kraft pulp samples S1-S4 to 87% ISO brightness (% in relation to sample S1)

Figure 2 shows that the pre-treatment of pine kraft pulp samples with Paa before oxygen delignification in the amount of 0.33% and 0.66% A.O. based on the o.d. pulp reduced the amount of chlorine dioxide required to bleach pine pulp to brightness 87% ISO by 18.8% and 31.0%, respectively. As for the S4 pulp sample, which had a kappa number of 7.5 units before bleaching, an over-proportional (62.5% rel.) reduction in chlorine dioxide consumption was achieved to bleach it to 87% brightness. The observed reduction in consumption of chlorine dioxide resulted not only from the lower kappa number of samples S2 to S4, but also from the higher brightness of these samples before bleaching.

Martino *et al*. (2013) also observed a distinct difference in the bleachability of two eucalyptus kraft pulp samples that differed in their initial brightness, despite the difference in initial brightness being only 4.2%.

These reasons clearly explain the differences in the reduced amount of chlorine dioxide required to brighten the pine pulp to 87% brightness in the case of pulp samples S2 and S3. For these samples, the kappa number factor (KNF), calculated by dividing the amount of chlorine dioxide required to bleach the pulp samples to 87% brightness by their kappa number before bleaching, was almost the same, *i.e.*, 0.23 to 0.24. On the other hand, for pulp sample S4, the KNF was lower, *i.e.*, 0.16.

These results may be due to better bleachability of pulp at higher levels of delignification and brightness. It is known that a pulp containing little residual lignin could in principle be further bleached with a lower number of bleaching stages than pulp with a higher content of this component (Barros *et al.* 2010). This effect may, in turn, be due to the better bleachability of lignin alone of such pulp or/and increase in the content of the non-lignin substances in this pulp that require less chlorine dioxide for their oxidation (Costa and Colodette 2007; Costa *et al.* 2022).

CONCLUSIONS

1. Pre-treatment of pine kraft pulp with peracetic acid before oxygen delignification 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 such pulp in oxygen delignification process from kappa number 13.9 to 11.1 and 9.3, owing to its pre-treatment with peracetic acid in the amount of 0.33% and 0.66% as A.O. based on the o.d. pulp, made it possible to obtain a 18.8 and 31% rel. reduction of chlorine dioxide consumption in its elemental chlorine free (ECF) bleaching to ISO brightness of 87%, respectively.
3. A decrease in the kappa number of the regular pine kraft pulp to 7.5 units owing to its pre-treatment with peracetic acid and the lengthening of the O_2-delignification time to two hours caused an over-proportional (approx. 62% rel.) reduction in chlorine dioxide consumption for bleaching.

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