Chemical pulping

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The effects of high alkali impregnation and oxygen delignification of softwood kraft pulps on the yield and mechanical properties

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Abstract: This study investigated whether the yield improvement after high alkali impregnation (HAI) is maintained after oxygen delignification and whether the potential of oxygen delignification to increase the mechanical properties is affected by high alkali impregnation. The yield improvement achieved by high alkali impregnation (1%) was preserved after oxygen delignification, particularly of glucomannan. The total fiber charge and swelling increased after oxygen delignification regardless of the type of impregnation in the cooking step. The tensile index improvement obtained by oxygen delignification was retained if this was preceded by high alkali impregnation. The stiffness index was higher and elongation slightly lower after HAI impregnation than after a standard (REF) impregnation. Fibers obtained through high alkali impregnation seem to be slightly less deformed and slightly wider than fibers obtained after a standard impregnation.

Keywords: curl index; fiber charge; glucomannans; kraft cooking; tensile index.

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Graphical abstract

Introduction

In chemical pulp mills, kraft cooking is the most frequently used process to remove lignin, with a yield loss of about 50%; but a significant part of the polysaccharides are also lost (Brännvall 2017), due to peeling reactions, where a sugar unit at the reducing end of the carbohydrate chain is removed. Glucomannans are strongly affected by these reactions, already in the initial phase of the cooking, leading to dissolution and severe losses (Sjöström 1993, Paananen et al. 2010), and it is essential to improve the delignification while preventing unnecessary polysaccharide losses.

Impregnation can have a significant impact on the final yield, and by improving the diffusion of chemicals inside the wood chips, the delignification can be improved and carbohydrate degradation reduced (Malkov et al. 2003, Tavast and Brännvall 2017, Brännvall 2018). The chemical transport in the impregnation phase is accomplished by liquor flow into the air-filled voids and by ionic movement in solution. Increasing the initial alkali concentration in the impregnation will increase the gradient concentration and will therefore promote the diffusion rate of chemicals into the chips, giving a faster removal of acetyl groups and a better ion mobility (Määtt-
An increase in alkali concentration can lead to a higher rate of peeling reactions, but also a higher rate of stopping reactions. While the rate of the peeling reaction tends to level off when the alkali concentration is higher than 0.1 mol/l, the rate of the stopping reactions does not level off until 1.5 mol/l, favoring the stopping reactions over the peeling reactions (Paananen et al. 2010, Brännvall and Bäckström 2016). Brännvall and Bäckström (2016) have shown that a uniform distribution of the cooking chemicals contributes to a more efficient cook with less rejects and an increase in the final yield of almost 2%-units.

Oxygen delignification is a well-established process and has recently proved to be an efficient way to improve the tensile index when the fiber charges are sufficiently increased (Esteves et al. 2020, Esteves et al. 2021b). Oxygen can remove a large amount of residual lignin and introduce carboxylic acid groups by oxidation of lignin and carbohydrates leading to a significant increase in swelling, conformability and fibrillation (Laine et al. 1997, Zhang et al. 2005, Zhao et al. 2016, Esteves et al. 2020).

High alkali impregnation has previously been shown to improve the yield of kraft cooking (Brännvall and Bäckström 2016, Brännvall 2018), but it is yet unknown whether this yield increment remains after subsequent oxygen delignification.

To clarify this, the present study has included a comparison between a delignification performed with only kraft cooking to a kappa number of ca. 25 and a combined delignification performed at first with kraft cooking to a kappa number of ca. 50 followed by oxygen delignification to a kappa number of ca. 25. In addition, the study has included a similar comparison where the kraft cook had been carried out with a high alkali impregnation (HAI), in order to see whether oxygen delignification has the same potential to increase the mechanical properties after cooking performed with HAI as it has after REF cooking.

**Materials and methods**

**Material**

A mixture of softwood chips from BillerudKorsnäs Skärblacka Mill (70 % Spruce (Picea abies) and 30 % Pine (Pinus sylvestris)) was used in this study. With a dry solid content between 91–93 % the chips prior to cooking were placed in autoclaves and soaked in water applying a pressure of 5 bar by nitrogen gas overnight. After draining the water, there remained between 1.6–1.9 mL/g water inside the chips, considering the wood fiber saturation point of 30 to 40 % and a void fraction of ca. 73 %, this means that the lumen is most likely filled with water. It is assumed, therefore, that the chemical transport occurs mainly by diffusion and not penetration.

The white liquor was prepared from stock solutions of sodium hydroxide (NaOH) and hydrogen sulphide (Na2S) to obtain the desired effective alkali charge and sulfidity in the kraft cooking.

**Methods**

The chips were subjected to kraft cooking with either standard impregnation (REF) or high alkali impregnation (HAI). For pulps to be subsequently delignified by oxygen, the cooks were stopped at a kappa number of ca. 50 while pulps delignification by only kraft cooking were continued to a kappa number of ca 27. The cooks which were stopped at a high kappa number were further subjected to oxygen delignification to a kappa number of ca 25. The procedure is schematically illustrated in Figure 1. Pulps are denominated KX_OY, where X is the kappa number of the cooked pulp and Y is the kappa number of the oxygen-bleached pulp. REF stands for “standard impregnation” and HAI for “high alkali impregnation”.

**Kraft cooking with standard impregnation (REF)**

The kraft cooks were performed with batches of 2 kg o. d. chips in a digester with recirculation of cooking liquor, with controlled temperature and a forced liquor flow. The effective alkali (EA) charge was 22 % and the sulfidity of 30 % at and a liquor/wood ratio of 4.51/kg.

During the impregnation, the temperature was raised from 20 °C to 100 °C at a rate of 5 °C/min. After 30 min at 100 °C, the temperature was raised to 160 °C at 3 °C/min. The cooks were stopped at different H factors (cooking times) to achieve the desired kappa numbers. After the cooking, the steam flow was stopped, and the spent liquor was drained off and collected for analysis – Table S1.

The delignified chips were washed with deionized water for 10 h in self-emptying metal cylinders and subsequently defibrated and screened in a NAF water jet defibrator (Nordiska Armaturfabriken). The shives were collected, dried at 105 °C, and weighed. The pulp was collected and centrifuged to increase the solid content. For homogenization of the centrifuged pulp, it was passed through a channel with a rotating shaft with horizontal bars that ripped the pulp into smaller dimensions.
Kraft cooking with high alkali impregnation (HAI)

The HAI trials were performed using the same equipment, using an EA charge of 31% and a sulfidity of 52%. The high sulfidity in the impregnation was chosen so that the hydrosulfide ion concentration in the cooking stage would be similar to the hydrosulfide concentration after REF impregnation. The impregnation was done with a temperature increase from 20 °C to 120 °C at a rate of increase of 5 °C/min. After the impregnation, a black liquor sample was collected and the residual alkali was measured to ensure that the EA concentration was ca. 0.9 M – Table S1. Approximately 4 l of free black liquor was removed from the digester, and approximately 4 l, corresponding to the entrapped liquor, remained in the digester. The alkali concentration in the cooking stage was adjusted to 0.5 M by dilution with deionized water and the temperature was raised to 160 °C at a rate of 3 °C/min.

As in the standard kraft cooking, the trials were stopped at different H-factors.

Oxygen delignification

Oxygen delignification was performed with batches of 60 g of oven-dried (o. d.) pulp, placed in polyethylene bags with 3.2% of NaOH, 0.5% of MgSO₄ and water at a consistency of 12%. The bags were closed by heat-welding, kneaded initially by hand and then in a vibrational shaker. The pulp was then removed from the bags and transferred to a pressurized steel autoclave coated with Teflon with a volume of (2.5 dm³). The autoclaves were closed, pressurized with oxygen (7 bars), and then placed in an electrically heated glycol bath at 100 °C, with rotation at a slight inclination. The conditions are given in Figure 1. After the oxygen delignification, the pulps were washed and filtered with distilled water.

Refining and paper sheet making

The pulp samples were refined in a PFI-mill according to ISO 5264-2 and handsheets were prepared according to ISO 5269-1 with deionized water, to a grammage of 60 g/m². The sheets were characterized by grammage (ISO 536), structural thickness (SCAN-P 88:01) and mechanical properties, such as tensile index, stiffness index and elongation (ISO 1924-3).

Pulp analysis

The residual alkali and the HS⁻ concentration were determined in duplicate according to SCAN-N 33:94 and SCAN-N 31:94 respectively.

The kappa number was determined according to the ISO 302:2004 standard. This method assumes that the potassium permanganate consumed is directly related to the lignin content present in the pulp because lignin is oxidized faster than carbohydrates.

The carbohydrate composition was determined according to SCAN-CM 71:09. The xylan, glucomannan and cellulose contents were calculated from the monosaccharides (Table S2) according to (Janson 1974), using a uronic acid content of 4.25 and a glucose:mannose ratio of 4.17. The water retention value (WRV) was determined according to SCAN-C 62:00 and the Schopper-Riegler number was
Table 1: Yield and rejects after kraft cooking and oxygen delignification for REF and HAI cases.

| Wood  | REF-K50 | HAI-K51 | REF-K26 | HAI-K28 | REF-K50_025 | HAI-K51_025 |
|-------|---------|---------|---------|---------|-------------|-------------|
| Total yield, % on wood | 100 | 49.4 | 50.5 | 46.5 | 47.3 | 47.7 | 48.5 |
| Screened yield, % on wood | – | 45.7 | 47.0 | 46.4 | 47.1 | 44.2 | 45.1 |
| Rejects, % on wood | – | 3.7 | 3.5 | 0.1 | 0.3 | – | – |

determined according to ISO 5267-1. All the tests were performed in duplicate.

The total fiber charge was determined by conductometric titration according to the method described by Katz et al. (1984).

The fiber morphology was studied with the L&W Fiber Tester, where fiber parameters such as curl index, fiber width, number of kinks and length were determined (Table S3). The curl index was calculated according to Page et al. (1985).

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\text{Curl Index} (\%) = \frac{1}{\text{shape factor}} - 1. \tag{1}
\]

Results and discussion

Pulping

Kraft and oxygen delignified pulps after conventional kraft cooking (REF) and after kraft cooking using high alkali impregnation (HAI) were compared with regard to yield, chemical composition and fiber charge.

Yield after kraft cooking and oxygen delignification

The total and screened yields of the kraft cooks performed with two types of impregnation – REF and HAI – are shown in Table 1. After HAI, the kraft cooks clearly showed a higher total yield than the kraft cooks performed with standard impregnation (REF), in agreement with previous studies (Brännvall and Bäckström 2016, Brännvall 2018). The amount of rejects was, however, similar regardless of whether the cook was performed with a standard or a high alkali impregnation, which disagrees with the results of previous studies where a decrease of ca. 1 % was seen when higher alkali was used in the impregnation (Brännvall 2018). In the study by Brännvall (2018), the cooking was however performed in autoclaves (with lower sulfidity) and the kappa number was in the range of 60 to 30, the greatest impact on the reject being from kappa number 45 to 60. In the present study, the cooking was performed in a digester with forced circulation (with higher sulfidity) and the starting kappa number was 50. Despite the same reject level, the screened yield was higher in the HAI case.

The yield increment for HAI (1 %-unit at a given kappa number) remained after oxygen delignification. The yield in the oxygen stage was 96.6 % for REF pulps and 96.0 % for HAI.

Chemical composition

Cooking after high alkali impregnation resulted in pulps with higher glucomannan content, as shown in Table 2. The high initial hydroxide ion concentration (1.7 mol/l) increases the rate of the stopping reaction whereas the rate of the peeling reactions remains the same for both REF and HAI, leading to a greater glucomannan preservation, due to the stabilization effect of carbohydrates (Paananen et al. 2010, Jafari et al. 2014, Brännvall and Bäckström 2016, Brännvall 2018). Less xylan remained in the pulps after HAI cooking, in agreement with Brännvall (2018). The decrease in xylan content was expected since xylan dissolves more readily at a higher alkali concentration (Jansson and Brännvall 2011). The total hemicellulose content was similar for REF and HAI pulps, the difference being in the xylan and glucomannan content for each trial; HAI pulp had a higher glucomannan and a lower xylan content than REF.

Oxygen delignification of HAI-cooked pulp from kappa number 51 to 26 seems to be beneficial for the preser-
Table 3: Total fiber charge for REF and HAI pulps after kraft cooking and after subsequent oxygen delignification.

|                  | REF-K50 | HAI-K51 | REF-K26 | HAI-K28 | REF-K50_O25 | HAI-K51_O26 |
|------------------|---------|---------|---------|---------|-------------|-------------|
| Total fiber charges (meq/kg) |   124   |   115   |    74   |    81   |    143      |    136      |

Figure 2: Total fiber charge as a function of kappa number on pulps produced under REF (black squares), HAI (orange squares) and oxygen delignification (open squares) conditions.

In the HAI-trials there was a slightly higher preservation of cellulose than for the REF-trials, in accordance with previous studies Brännvall and Bäckström (2016), Brännvall (2018).

Fiber charges

The total amounts of charged groups are presented in Table 3. Kraft delignification from kappa number 50 to 25 reduced the amount of charged groups in agreement with previous studies (Jafari et al. 2014, Esteves et al. 2021a). Oxygen delignification from kappa number 50 to 25, on the other hand, increased the amount of charged groups, as also reported previously (McDonough 1989, Laine 1997, Yang et al. 2003, Zhang et al. 2005, Esteves et al. 2021a).

As shown in Figure 2, the total amount of charged groups at a given kappa number was similar, whether they were produced according to REF or HAI. The carboxylic groups in unbleached pulp are found mainly in xylan (Laine et al. 1996) consisting of methylglucuronic acid groups (MeGlcA) originally present in xylan, or hexenuronic acid groups (HexA) formed from MeGlcA (Sjöström 1989, Laine 1997). In oxygen delignified pulps, carboxylic groups have been introduced to lignin by oxidation of free phenolic groups into muconic acid structures in lignin (Snowman et al. 1999, Yang et al. 2003, Dang et al. 2006). The amount of carboxylic groups in HAI pulps was higher than expected based on the larger amount of xylan dissolved in the HAI process resulting in the loss of carboxylic acid groups bound to xylan. The higher alkali concentration in HAI may promote the rate of the stopping reactions, and this can compensate for the loss in the fiber charge by giving rise to metasaccharinic acid and other alkali-stable carboxyl groups (Sjöström et al. 1965, Laine et al. 1996). An increase in the carboxylic groups associated with stable metasaccharinic acid at the reducing ends of the molecules was previously reported in cellulose due to stopping reactions (Johansson and Samuelson 1975, Mozdyniewicz et al. 2013). It can therefore be assumed that the carboxylic acid groups formed in the HAI cooking trials were in glucomannan and cellulose chains rather than in the xylan.

Refinability

WRV and Schopper-Riegler degree

The cooked and oxygen delignified pulps were refined to different degrees in a PFI-mill. After oxygen delignification, the water retention value increased due to the greater carboxylic acid content – Figure 3a. The two oxygen delignified pulps presented a similar WRV behavior after refining, as well as the two cooked pulps.

The Schopper-Riegler value was also higher after oxygen delignification – Figure 3b. The REF and HAI pulps shared a similar development during refining. The increase in the case of the oxygen pulps was probably due to the greater fibrillation and greater fines content generated by the beating, cf. Bäckström et al. (2013), Esteves et al. (2021b).

Tensile properties

Oxygen delignification had a positive effect on the tensile index, as shown in Figure 4a. The increase is more obvious for pulps refined to higher levels (4000 revolutions). The unrefined HAI-cooked pulp had a tensile strength similar to that of unrefined oxygen delignified pulps, but after refining, the increase in tensile index was greater for the oxygen delignified pulps.

Esteves et al. (2020) showed that, compared to the tensile index of cooked pulp at a similar lignin content, the
increase in tensile index as a result of oxygen delignification, was dependent on the extent of the increase in fiber charge resulting from oxygen delignification. It has previously been shown that, up to a certain point, the charge groups increase with increasing degree of delignification during the oxygen stage (Esteves et al. 2020). A greater degree of delignification is achieved with a longer time and a higher alkali charge. It is not therefore improbable that a greater increase in fiber charge results in a more uniform distribution of the charges on the surface and inside the fiber wall. Refining a pulp with a more uniform distribution of charges within the fiber wall would probably lead to an increased swelling, greater fiber flexibility, greater fibrillation and higher tensile strength.

In the present study, oxygen delignification resulted in a fiber charge increase of 68% for HAI-pulps and 81% for REF pulps – Table 3. The increase in tensile index was smaller for oxygen delignified HAI pulps than for oxygen delignified REF pulps, Figure 4a. At 4000 PFI-revolutions, REF-K50_O25 had a tensile index 18% higher than that of K26, but the value for HAI-K51_O26 was only 10% greater than that of HAI_K28. At similar structural densities, the increase in the tensile index by oxygen delignification of REF pulp was 6% but only 2% for HAI pulp – Figure 4b.

The stiffness was quite unaffected by the refining process, but the HAI-cooked pulp had the highest stiffness at 0 and 1000 PFI-revolutions than the REF-cooked pulp – Figure 5.

The development of elongation-at-break with refining was similar for the REF and HAI pulps – Figure 6.

Morphology

Figure 7 shows the tensile strength and density as a function of curl. The curl index increased with the refining of the cooked pulps, while the curl index decreased slightly
as a result of the oxygen delignification, which is in agreement with Esteves et al. (2021b).

At a given curl index, the HAI-cooked pulp had a significantly higher tensile index and a higher structural density than the REF-cooked pulp. The higher glucomannan content in HAI pulps probably enhances the conformability of the fibers, also seen as a higher swellability (Figure 3).
The HAI-cooked pulps appear to have a lower degree of deformation at a given density than the REF-cooked pulps (ca. 1.5 % units lower – Figure 7b).

HAI pulps had fibers with a slightly greater width than the REF pulps – Figure 8. Scallan and Green (2007) proposed that the decrease in width is related to the inward contraction of the fiber wall as lignin and hemicellulose are removed. In the present study, the HAI fibers had a greater width at a given lignin content. The difference is mainly in the glucomannans content, suggesting that the glucomannan contributes to the greater fiber width.

It should be pointed out that the mechanical and morphological properties presented in this work were obtained by laboratory trials, in industrial fiberlines the pulp properties can diverge.

Conclusions

High alkali impregnation led to a higher yield after kraft cooking than a standard impregnation, due to greater glucomannan preservation. The higher yield accomplished with HAI was preserved after oxygen delignification. Oxygen delignification of HAI-cooked pulp resulted in higher preservation of glucomannans and xylan than the oxygen delignification of REF-cooked pulp.

Pulps obtained with a higher alkali impregnation had a lower xylan content than that after standard impregnation. Despite the lower xylan content, the HAI pulps had total fiber charge values similar to those of pulps obtained by standard impregnation.

The development of tensile strength properties was similar for HAI and REF cooked pulps. Regardless of the method of impregnation used in the cooking, oxygen delignification led to an increase in the tensile index due to the higher fiber charges.

HAI-cooked pulp had a higher tensile strength and a higher structural density at a given curl index than the REF-cooked pulp. HAI pulps had a lower curl index and a greater fiber width.

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