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Chemical pulping

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Consequences in a softwood kraft pulp mill of initial high alkali concentration in the impregnation stage

Abstract: Impregnation with high initial concentration is fast and efficient, leading to a homogeneous delignification in the subsequent cook, resulting in improved screened pulp yield. To obtain high initial alkali concentration, the white liquor flow needs to be significantly increased. The moisture content of the wood chips and the alkali concentration of the white liquor limit the initial alkali concentration of the impregnation liquor that can be reached. It is therefore of interest to evaluate the possibility to implement high alkali impregnation (HAI) industrially and the consequences this would have on the mill system. The effect of HAI on mass and energy balances in a kraft pulp mill has been studied using mill model simulations. The sensitivity to disturbances in important parameters for process control has been compared to impregnation scenarios used industrially. It was shown that high initial alkali concentration can be achieved on industrial scale by increased white liquor flow. HAI has a positive effect on recovery flows and reduces the need for make-up chemicals. The HAI concept is less sensitive to variations in process parameters, such as chip moisture and white liquor concentration, thus diminishing the risk of alkali depletion in chip cores.

Keywords: effective alkali; homogeneous delignification; impregnation; Kraft cooking; mill simulation model; softwood; yield.

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Introduction

When fossil-based raw materials will be replaced by biobased resources, it is increasingly important that processes for refining biomass are efficient. Although a renewable resource, the amount of wood harvested annually is limited, which makes it important to have as high yield as possible in the converting processes. In pulping, a thorough impregnation of chips with cooking liquor prior to delignification improves the yield as the amount of insufficiently delignified chip cores is decreased, i. e. the amount of screening rejects is decreased. Insufficient delignification of chip cores is caused by alkali depletion. Diffusion of chemicals ensures that all parts of the chips are supplied with sufficient amounts of active cooking chemicals. However, as hydroxide ions diffuse through the chips, they react with wood components. Most of these reactions are unavoidable and even desired, such as deacetylation, as this improves the diffusion paths within the fiber wall and reduces the alkali consumption in the cooking stage. However, for hydroxide ions to reach all the way to the chip core it is important that the amount is adequate so that consumed hydroxide ions are replenished by an in-flow of hydroxide ions. Diffusion rate can be increased by increasing the alkali concentration in the impregnation liquor. This has been shown to lead to a faster and more thorough impregnation (Gullichsen et al. 1995, Määttänen and Tikka 2012, Brännvall and Bäckström 2016, Brännvall and Reimann 2018) and resulting in a higher screened pulp yield (Gullichsen et al. 1995, Brännvall and Bäckström 2016, Brännvall 2018). An even impregnation by high alkali concentration has been shown to improve screened pulp yield by decreasing the extent of alkaline hydrolysis and thereby the extent of secondary peeling (Brännvall 2018), which is the main reason for yield loss of cellulose (da Silva and van Heiningen 2015, Paananen and Sixta 2015). Performing impregnation at high alkali concentration can also enhance yield by improved retention of cellulose and glucomannan (Brännvall and Bäckström 2016) as the stopping reaction is enhanced in comparison to the peeling reaction at elevated alkali concen-

trations (c. f. Lai and Ontto 1979, Paananen et al. 2010). In the EnerBatch® process, white liquor (WL) together with spent impregnation liquor is used (Wizani et al. 1992). The process was reported to reduce kappa number variations and gives higher screened pulp yield. The initial effective alkali concentration however, has been reported to be only 0.8 M (Wizani et al. 1997), which is not as high as 1.7–2.0 as proposed for efficient impregnation (Brännvall and Bäckström 2016, Brännvall and Reimann 2018, Brännvall 2018). The alkali concentration of the white liquor and the moisture content of the chips limit the maximum value of the initial alkali concentration achievable in industrial conditions. The question thus arises whether it is possible to implement the high alkali impregnation (HAI) concept in a mill and what consequences it would have on the mill system e. g. Na/S-balance.

For process runnability and efficient utilization of the raw material, process control is vital. To be able to control the cooking process, certain parameters need to be known, such as the amount of dry wood and concentration of active chemicals in the white and black liquors. However, in practice, fluctuations in important parameters such as moisture content (MC) in chips and wood density cannot always be monitored and are either taken to be constant or depend on regular laboratory analysis which do not catch variations occurring at faster rate. In liquor-to-wood (L/W) control in pulp mills, the moisture content is often taken as a constant (Pietilä et al. 2015) although considerable variations are found, ranging from 39 to 45 % (Watson and Stevenson 2007, Hart 2009) and variations as high as 40 %-units between chips in the same batch have been reported (Smith and Bordeau 1998). Seasonal variations cause differences in MC (Watson and Stevenson 2007), but also fast and unpredictable changes can occur. Rain or sunny, hot weather may lead to MC variations within chips, with higher respectively lower MC on the chip surface compared to chip core (Marrs 1989, Smith and Bordeau 1998). Assuming a constant MC of chips affects the diffusion and reaction rates during impregnation and cooking. According to Smith and Bordeau (1998), two thirds of the variation in kappa number of the produced pulp can be attributed to MC variations. A higher MC, which is not taken into consideration, will dilute the concentration of active chemicals in the cooking liquor. However, a high initial effective alkali concentration can diminish the effect of variations in chip characteristics, such as MC, amount of over-thick chips etc. The aim of the present study was to assess the possibility to implement high initial effective alkali concentration on industrial scale and evaluate the consequences on the mill operations. This has been addressed by mill model simulations, which take into account the mass and energy balances of individual unit processes, and by comparing how different impregnation scenarios are sensitive to disturbances in process parameters.

Materials and methods

Dried industrial softwood chips (70 % Pinus sylvestris and 30 % Picea abies) with a moisture content of 8 % were used. The chips were screened and the fraction 4–8-mm in chip thickness was used after removing bark and knots by hand. For impregnation and cooking, NaOH pastilles of puriss grade (VWR International AB, Radnor, PA, USA) and Na_2S technical grade flakes (VWR International AB) were dissolved in deionized water to obtain stock solutions of NaOH and Na_2S.

Impregnation was performed in steel autoclaves with a volume of 2.5 dm^3 with batches of 150.0 g o. d. chips. Air was removed from the chips by vacuum suction for 30 min. Impregnation liquor was prepared from the stock solutions to obtain desired hydrosulphide and hydroxide ion concentrations. The liquor was sucked into the autoclaves; the liquor-to-wood (L/W) ratio was 3.5 l/kg wood at 1.0–1.7 M alkali concentration and 8 l/kg wood in the extended impregnation case. The autoclaves were placed in a steam-heated glycol bath at 105 °C. The heating time to required temperature was 10 min, after which the actual impregnation time started. Residual alkali was determined according to SCAN-N 33:94 in duplicate. To determine the alkali concentration in the bound liquor, $[OH^-]_{bound}$, after completed impregnation, the free liquor was drained and 2000 ml deionized water was added to the chips and the entrapped liquid was leached out for 48 h after which the alkali concentration was determined.

A process simulation model of a theoretical mill developed by RISE has been used to assess the effects of implementing a HAI in a kraft mill. The model uses the process simulation program WinGEMS 5.0, designed for use in the pulp and paper industry to calculate the steady-state distributions of fibre, water and some process chemicals. The reference mill in the model represents a state-of-the-art softwood kraft mill, producing 700 000 ADt/year of bleached pulp (Berglin et al. 2011). Black liquor is extracted via a single-stage flash and sent to a 7-effect evaporation plant where it is concentrated to 80 % dry content. High-pressure steam is produced in both the recovery boiler and the power boiler at 100 bar(g) and 505 °C. The power production in the back-pressure turbine is more than sufficient to satisfy the power demand of the refer-

ence mill process. The excess back-pressure power and significant amounts of condensing power are exported. Bark is combusted in both the lime kiln and the power boiler.

Results and discussion

Assessment of high alkali impregnation (HAI) by mill simulation model

In the mill model, mass and energy balances over unit processes are made. In the reference case, the alkali charge is split while in the HAI cases, alkali is charged only to the impregnation. The in-put data to the mill simulations are shown in Table 1. The alkali concentration of the white liquor (WL) is set by the recovery cycle and in the simulations, a value of 115 g/l is used. The value for the moisture content in wood, MC, is set to 50 %. A 1.5 %-unit yield increase is assumed for the HAI cases compared to the reference, based on earlier studies of HAI, where impregnations with high $[OH^-]_{init}$ (1.7 and 2.0 M) were compared with lower $[OH^-]_{init}$ (1.3 and 1.0 M) (Brännvall and Bäckström 2016, Brännvall 2018). The yield increase is valid in the kappa number range of 35–50. The alkali consumptions during HAI impregnation are based on previous studies (Brännvall and Bäckström 2016, Brännvall 2018) as is the alkali consumption in the cooking stage at

a kappa number of approx. 40 (Brännvall and Bäckström 2016). A higher alkali consumption during the impregnation stage is assumed to lead to less alkali consumed in the cooking stage, as previously shown (Andrews et al. 1983, Tolonen et al. 2010, Tavast and Brännvall 2017, Brännvall and Bäckström 2016).

On an industrial scale, there are factors limiting the maximum level of the initial effective alkali concentration that can be achieved. In Equation 1, the resulting initial effective concentration is shown as mol/l.

$$[OH^-]_{init} = \frac{n_{EA}}{V_{tot}} = \frac{V_{WL} \cdot c_{WL} + V_{BL} \cdot c_{BL}}{(V_{WL} + V_{BL} + V_{CM}) \cdot M_{NaOH}}$$
$$= \frac{V_{WL} \cdot c_{WL} + V_{BL} \cdot c_{BL}}{L/W \cdot m_{dry} \cdot M_{NaOH}} \quad (1)$$

where

$[OH^-]_{init}$ = initial effective alkali concentration, mol/l
n_{EA} = mol NaOH
V_{tot} = total volume of liquor charged to digester, l
V_{WL} = volume of white liquor, l
c_{WL} = concentration of NaOH in white liquor, g/l
V_{BL} = volume of black liquor, l
c_{BL} = concentration of NaOH in black liquor, g/l
M_{NaOH} = molar weight of sodium hydroxide, g/mol
V_{CM} = volume of moisture in chips, l
L/W = liquor-to-wood ratio, l/kg o. d. wood
m_{dry} = amount of wood, o. d. kg

Table 1: In-put data to the mill model.

	Reference	A1	A2	B1	B2
			Impregnation		
EA, g/kg o. d. wood	182	200	240	217	223
EA in WL, g/l	115	115	115	115	115
EA from WL, g/kg o. d. wood	150	200	240	200	200
EA from recirculated liquor, g/kg o. d. wood	32	0	0	17	23
WL flow, l/kg o. d. wood	1.3	1.7	2.1	1.7	1.7
Recirculated liquor flow, l/ kg o. d. wood	2.0	0	0	0.6	0.8
Chip moisture content, %	50	50	50	50	50
L/W, l/kg	4.3	2.7	3.1	3.3	3.5
Consumption, g/kg o. d. wood	100	120	120	110	110
Residual alkali, g/kg o. d. wood	71	80	120	107	113
Residual alkali, mol/l	0.4	0.7	1.0	0.8	0.8
			Cook		
L/W, l/kg o. d. wood	5.0	5.0	5.0	5.0	5.0
EA$_{start}$, g/kg o. d. wood	129	74	100	82	82
Consumption, g/kg o. d. wood	70	44	44	54	54
Residual alkali, g/kg o. d. wood	59	30	56	28	28
Residual alkali, g/l	12	6	11	6	6
Total alkali consumption, g/kg o. d. wood	170	164	164	164	164

In Case A, high initial alkali concentration is obtained by a higher white liquor (WL) flow. In Case B, part of the impregnation liquor is recirculated. Cases A1 and B1 have lower L/W ratio and lower EA charge, g/kg o. d. wood, compared to Cases A2 and B2. EA is given as NaOH.

Table 2: Outcome of mill model simulation.

	Reference	A1	A2	B1	B2
Impregnation $[OH^-]_{init}$, mol/l	1.0	1.9	1.9	1.7	1.6
NaOH make-up, kg/ADt	8.4	8.2	8.2	8.2	8.2
CaO to lime kiln, kg/ADt	7.9	7.4	8.0	7.4	7.4
Purged ESP dust, kg/ADt	13.1	12.5	12.1	12.5	12.5
Purged lime mud, kg/ADt	17.5	16.4	17.8	16.4	16.4
Total amount of reburned lime, kg/ADt	237	230	273	230	230
Power consumption, kWh/ADt	722	710	724	711	711
Sold power, kWh/ADt	881	697	576	722	727

The basis is air-dry ton (ADt) fully bleached softwood kraft pulp.

According to Equation 1, high initial alkali concentration is obtained by high V_{WL} and low L/W ratio. In Table 1, this represents Case A. The EA charge is lower in Case A1 compared to Case A2, 200 and 240 g/kg o. d. wood, respectively. The L/W ratio is lower in Case A1, 2.7 l/kg, compared to 3.1 l/kg in Case A2. The alkali concentration, however, is quite high at the end of the impregnation after HAI and it is desirable to use this alkali by recirculating the spent liquor to the beginning of the impregnation stage. In Case B1 and Case B2, 1 l/kg o. d. wood has been recirculated and two L/W ratios, 3.3 and 3.5 l/kg o. d. wood, have been used when calculating the resulting $[OH^-]_{init}$. In the reference mill, black liquor is recirculated to the impregnation. The outcome of the simulation is presented in Table 2. The $[OH^-]_{init}$ obtained in the impregnation was 1.9 M, for Cases A1 and A2. For Case B1 and B2, the $[OH^-]_{init}$ was lower, 1.7 and 1.6 M, respectively. The level, however, is probably sufficiently high to improve the diffusion rate during impregnation and thus the pulp yield. As shown by Brännvall (2018), a yield increase of 1–1.5 %-units was obtained when impregnation was performed with an $[OH^-]_{init}$ of 1.7 M.

Implementing the HAI concept by increasing the alkali charge by higher WL flow, as for Case A1 and A2, did not increase the need for make-up noticebly. Only 0.1 % higher demand for CaO to lime kiln when a higher EA charge was used as in Case A2. Implementation of the HAI concept by recirculating part of the impregnation liquor (B-cases), the model showed no or little effect on the mill process. There was lower purges of ESP dust and lime mud per air dry ton (ADt) bleached pulp due to the higher yield which will lower the makeup of NaOH and CaO per ADt as well. The energy balance of the mill will be affected by an increased yield, which means that less organics will end up in the recovery boiler. This resulted in less steam production which decreased the mills energy surplus in form of less sold power. Increasing the white liquor also affected the mills energy balance. More steam is needed to evaporate the additional white liquor charged and less steam is
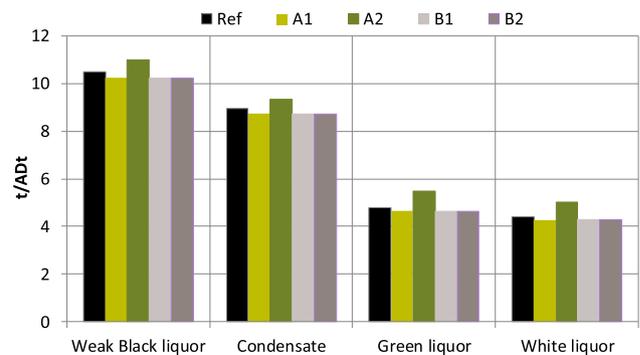

Figure 1: Flows in recovery area t/ADt for the different cases simulated.

produced in the bark boiler since more bark is needed for the lime kiln. This results in less sold power for the mill of about 160–300 kWh/ADt.

Figure 1 shows the flows in the recovery area in t/ADt for the simulated cases. The highest alkali charge (Case A2) increased the flows in the recovery area; weak black liquor by 5 %, condensate by 4 % and green liquor and WL by 15 %, since more WL is needed. For case A1 and B cases, the flows per ADt decreased somewhat, approx. 3 %, due to the higher yield.

Calculations and simulations show that is possible to perform impregnation with high initial alkali concentration. A sufficiently high concentration can be obtained also with recirculation of the impregnation liquor and the effect on material and energy balances is minor. To implement the HAI concept with recirculation of the impregnation liquor in an existing mill would however require practical considerations and depend on the mill set-up.

With a yield increase of 1.5 %-units, a mill with the HAI concept would produce 10 500 ADt of bleached pulp more annually compared to the reference case. With a pulp price of €1000/ADt, this would amount to an increased revenue of 10.5 M€/year. The loss in sold power

amounts to M€4.2–7.9/year, if a price of €37/MWh is assumed. The price of power includes a green power certificate of €7/MWh. Implementing the HAI concept with recirculation of the impregnation liquor, Case B, would increase the pulp mills revenue by approx. M€6/year.

Effect of process variations

Process control on an industrial scale is complex and needs to catch variations in important parameters and take correcting measures to minimize the effect of transient conditions. To obtain correct conditions during the process, such as chemical charges, certain parameters are monitored, and others are considered constant. It is quite common to assume constant flow of dry wood and chip moisture. However, this can lead to significant difference between assumed values of for example liquor-to wood ratio, L/W, and concentration of chemicals. The WL charge is based on the desired EA charge, kg alkali/ton wood, Equation 2.

$$V_{WL} = \frac{m_{dry} \times m_{EA}}{c_{WL}} \left[\frac{m^3}{ton} \right] \qquad (2)$$

where V_{WL} is volume of WL, m_{dry} the weight of dry content in chips, c_{WL} the concentration of WL.

The dry wood content in a given volume of wood depends on MC and density of wood, Equation 3 and Equation 4.

$$MC = \frac{m_{green} - m_{dry}}{m_{green}} = \frac{m_{moisture}}{m_{green}} \left[\frac{ton\ moisture}{ton\ fresh\ wood} \right] \qquad (3)$$

$$m_{dry} = V_c \times \rho_{dc} \qquad (4)$$

where m_{green} is the weight of fresh chips, V_c is the volume of the chips and ρ_{dc} the basic density, t/m^3.

To obtain a certain L/W ratio, the WL is diluted with a certain volume, V_{dil}, Equation 5. The dilution liquor is usually weak black liquor.

$$V_{dil} = L/W \times m_{dry} - m_{moisture} - V_{WL} \qquad (5)$$

Figure 2a shows how the initial EA concentration in the liquor entering the impregnation decreases with increasing MC for the three cases with either high (1.7 m) or normal (1.0 and 1.3 M) alkali concentration at an assumed L/W ratio of 3.5 l/kg and for an extended impregnation case with lower alkali concentration (0.8 M) at a L/W ratio of 8 l/kg. The decrease in alkali concentration with higher MC is quite large for the HAI and reference cases. If the target initial alkali concentration is low to begin with, as in the reference case, a higher MC of the wood may result in critically low initial alkali concentration and a risk of insufficient impregnation. In the HAI case, an increased MC is not as detrimental as the resulting initial alkali concentration is still on a satisfactorily high level for good impregnation to take place. A high L/W ratio, as used in extended impregnation, levels out the impact of MC and the higher MC of the wood entering the digester has only a minor effect on the in initial alkali concentration. The initial concentration of EA in the impregnation liquor is also decreased if the concentration of EA in the white liquor is lower than what is assumed, as seen in Figure 2b.

The concentration in the cooking liquor will also be lower than assumed if the basic density is higher. No significant effect on the effective alkali concentration is obtained if wood density changes, Figure 3a, thus not affecting the diffusion rate. However, if density increases, the amount of dry wood per volume of wood charged into the digester will increase and, as illustrated in Figure 3b, the amount of alkali available per ton dry wood decreases.
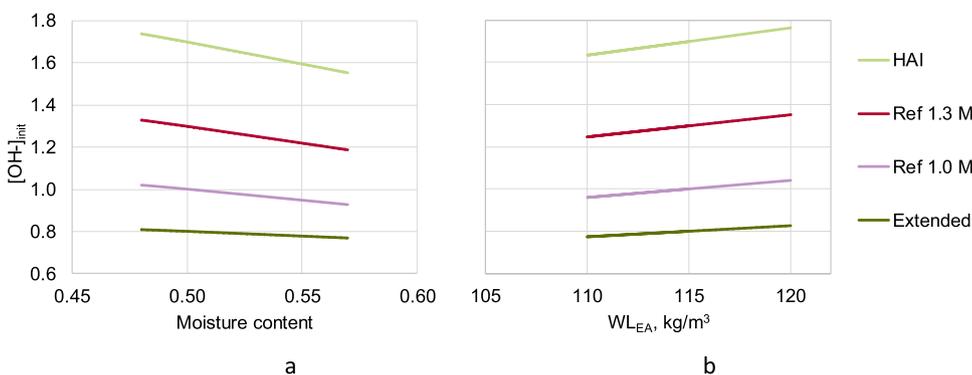

Figure 2: The L/W ratio for HAI and reference cases is 3.5 l/kg and for the extended impregnation case 8 l/kg. Effect on initial effective alkali concentration of variation in (a) moisture content (at a WL$_{EA}$ = 115 g/l) and (b) effective alkali concentration in white liquor (at a moisture content of wood = 0.50 g moisture/g green wood and basic density = 410 ton/m^3).
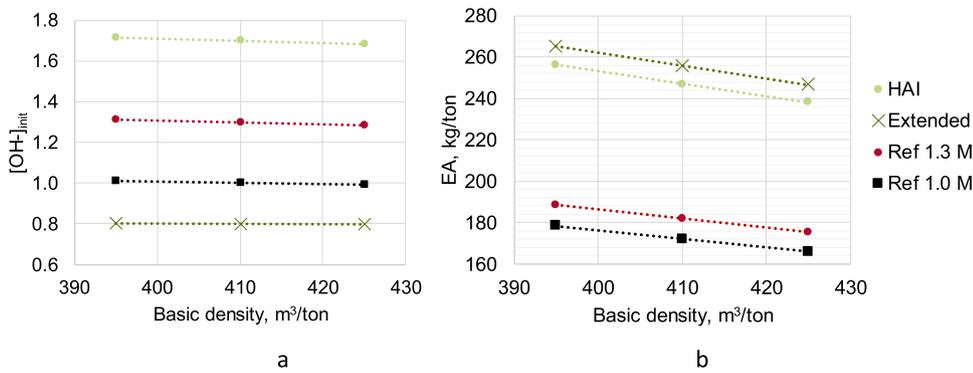
Figure 3: Effect of variation in wood density on (a) initial alkali concentration and (b) effective alkali charge (kg/ton wood). MC = 0.5 and WL_{EA} = 115 g/l.

Figure 4: The L/W ratio is 3.5 l/kg o. d. wood for assumed $[OH^-]_{init}$ = 1.0 and 1.7, 4.3 l/kg o. d. wood for $[OH^-]_{init}$ = 1.0 and 8 l/kg o. d. wood for $[OH^-]_{init}$ = 0.8. In-put values for the different cases: lowest: WL_{EA} = 105 g/l, MC = 57 %, ρ_b = 425 ton/m^3; aimed for: WL_{EA} = 110 g/l, MC = 50 %, ρ_b = 410 ton/m^3; highest: WL_{EA} = 115 g/l, MC = 48 %, ρ_b = 395 ton/m^3.

Since the major part of alkali is consumed in the impregnation, this may give a critically low alkali concentration at the end of the impregnation. The higher amount of dry wood will consume a higher amount of alkali. If this is combined with a decrease in EA concentration of the WL, the risk of alkali depletion in the chip center is increased. This has been high-lighted by Lampela (2013), who pointed out that errors in alkali-to-wood arise mainly from disturbances in the amount of wood coming in to the digester and WL concentration. Also the amount of bark and decayed wood will affect alkali consumption as they consume alkali but yield very few useful fibers (Hart 2009). The bark content can vary from 1 to 3 % (Hart 2009).

In Figure 4 an attempt has been made to demonstrate the extent of variation in EA concentration in the impregnation liquor. The EA concentration which is aimed for in the different cases assumes a certain c_{WL}, ρ_{dc} and MC. A higher concentration is obtained if c_{WL} and ρ_{dc} are

higher and the MC is lower. Similarly, the concentration of the impregnation liquor will be lower when WL concentration and wood density are lower and MC of wood higher than assumed in the process control. As can be seen in Figure 4, the HAI concept with an assumed $[OH^-]_{init}$ of 1.7 M, may in the worst case drop to 1.5 M, which still is much higher what is conventionally used. With variations in process parameters leading to higher $[OH^-]_{init}$, resulted in 1.8 M. This is not expected to present a problem, since higher alkali concentration in the impregnation liquor has no adverse effect if impregnation is performed at low temperature, <110 °C (Brännvall and Reimann 2018). The reference cases with assumed $[OH^-]_{init}$ of 1.0 and 1.3 M, dropped to 0.9 and 1.1 M, respectively. With a consumption of 100 g EA/kg o. d. wood, the concentration after completed impregnation would be 0.2 and 0.4 M, respectively. The origin of rejects is insufficiently delignified parts of the chips, normally the chips cores and knots. Complete
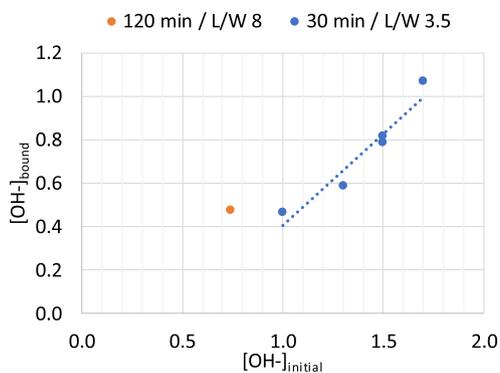
Figure 5: Experimental results on the average hydroxide ion concentration in bound liquor depending on initial effective alkali concentration.

alkali depletion in the chip core, results in condensation of lignin and creation of highly resistant lignin structures. Rejects have been shown to be very resistant towards delignification, as the lignin content remains almost as high as in wood although the liberated softwood pulp fibers have been delignified down to kappa number 20 (Tikka et al. 1993). Process models have shown that alkali depletion causes residual phase delignification reactions to set in at higher lignin content (Pu et al. 1991).

The effect of $[OH^-]_{init}$ on the average effective alkali concentration in the liquor entrapped in the chips (in lumen and in cavities within the fiber wall), was studied experimentally. Naturally, the lower the $[OH^-]_{init}$ at a given L/W, the lower the concentration in the bound liquor. If the cause of the lower $[OH^-]_{init}$ is a dip in c_{WL}, then also lower amount of alkali is available. With slower diffusion rate of cooking chemicals and if the impregnation time is not extended, there is a risk that no or insufficient amounts of alkali have reached the chip core at the beginning of cooking stage. Consequently, the pulp obtained will contain higher amounts of rejects. A comparison is made in Figure 5 with extended impregnation, where a high L/W results in low initial concentration, although the EA charge is high, thus with sufficient amount of alkali available.

The HAI and extended impregnation concepts thus handle disturbances in process parameters and avoids alkali depletion in chip cores better compared to the reference cases with low L/W.

Conclusions

To address the question if it is possible to implement a high initial alkali concentration in the impregnation stage and what consequences it would have on the mill system, a mill model simulation program was used. Results from the process simulation confirm that it is possible to reach an initial EA concentration of 1.9 M in the impregnation stage if only WL is used. With recirculation of impregnation liquor, it would be possible to reach 1.7 M. The simulations show that implementing an impregnation with high initial EA has a positive effect on the recovery flows, decreasing the need of make-up chemicals.

The effect of disturbances caused by variations in crucial process parameters on the initial effective alkali concentration in different impregnation scenarios has been calculated. If impregnation is performed with low L/W ratio and low EA concentration there is a significant risk of insufficient impregnation and alkali depletion in chip cores, resulting in low yield and high reject content. Impregnation with high EA concentration, as in the HAI concept, or high EA charge, as in the extended impregnation concept, decreases the risk of alkali depletion during impregnation.

The higher yield obtained by the HAI concept, improves the profitability of the mill, despite less sold power.

Conflict of interest: The authors declare no conflicts of interest.

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