Effects of impregnation time on hardwood kraft pulp characteristics and papermaking potential – a mill study

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ABSTRACT: We conducted a series of trials in a commercial-scale pulp mill producing fully bleached birch hardwood kraft pulp to study the effects of impregnation time. Retention time in an impregnation vessel was altered between 5 min and 60 min. Cooking kappa number target and brightness targets were held constant and the rest of the fiber line was operated according to normal practices. After analyzing collected pulp samples to monitor changes in characteristics and papermaking properties, we found that impregnation time has a significant effect on the consumption of effective alkali in the impregnation stage, but the total alkali consumption was not affected when a cooking kappa number was held constant. Reduced impregnation time required higher cooking temperatures to reach the kappa number target. Shorter impregnation times clearly increased the amount of rejects. Viscosity, carbohydrate composition, and fiber saturation point were not significantly influenced by the impregnation time, but some fiber damage occurred with longer impregnation times. The tensile strength and tensile were higher with the shorter impregnation time, whereas tear strength at a given tensile index did not correlate with impregnation time. An obvious reason for the affected papermaking properties was not found.

Application: This study showed that a long impregnation time is not necessarily advantageous for certain pulp properties. The results can be used as a reference in which the effects of impregnation time are studied on a commercial scale.

Mass transfer limitations often are the cause of nonuniform kraft cooking [1]. Process temperature affects the rate of chemical reactions and the rate of diffusion during pulping, and thus pulping uniformity. With increasing temperature, consumption of the cooking chemicals increases more than the corresponding diffusion rate. This reduces the flux of active cooking chemicals and therefore decreases cooking uniformity. The rate of alkali-consuming reactions is highest at the beginning of kraft cooking [2]. Relationships between the diffusion and consumption of the active cooking chemical are discussed by Sixta [3].

The limitations in mass transfer can be eased with an impregnation stage in which cooking liquor penetrates into chips before the temperature is raised to cooking temperatures. Impregnation affects cooking uniformity and thus the amount of rejects in the unbleached pulp. Good impregnation also facilitates the cooking process and allows for a lower H-factor to achieve the target kappa number [4].

The influences of pulp nonuniformity on papermaking are not extensively studied and for some parts the results are controversial. Nonuniformity impairs bleachability and reduces the strength potential [5]. Studies also suggest that delignification gradients in chips cause yield and strength losses in softwood pulps [6]. However, other reports have concluded that nonuniformity has no effect on pulp strength [4,7].

An opportunity to study the effects of impregnation time on pulp quality resulted when a Finnish pulp mill was facing runnability problems with its digester. To solve the problems, the mill decided to try a unique effort to improve the digester operation: the height of the chip column in the impregnation vessel was varied to improve digester operation. Because the height of the chip column is directly proportional to the retention time at a given production rate, the modification also changed the impregnation time.

This study describes a series of mill trials in which impregnation times between 5 min and 60 min were used in a commercial-scale fiber line producing fully bleached hardwood pulp. The goal of the study was to investigate the effects of the modified impregnation on fiber characteristics and papermaking properties of produced pulp.

EXPERIMENTS

The mill trials were conducted at a commercial-scale kraft pulp mill. The mill uses a separate impregnation vessel followed by a continuously operating digester with a conventional concurrent cooking zone and a counter-current high-heat washing zone.

In the experiments, the fiber line was operated with three different impregnation times designated as full (58 min), intermediate (29 min), and minimum (5 min). The minimum impregnation time of 5 min is an estimation obtained by measuring the descent rate of a fully penetrated chip in water.

Impregnation times were adjusted by changing the height of the chip column in the impregnation vessel. Because the production rate was maintained between 83% and 93% of the maximum, the correlation between impregnation time and the height of the chip column was almost linear. During the trials, a cooking kappa number target, residual alkali concentration in the digester, and brightness targets in bleaching stages were kept constant.

The cooking temperature and white liquor addition to the transfer circulation between the impregnation vessel and the digester were used to control the kappa number and the residual alkali concentration with the different impregnation times. Chemical charges in oxygen delignification and bleaching stages were adjusted to achieve brightness targets. Local birch hardwood was used as raw material in all the trials.

During the trials, we collected pulp and liquor samples from several points along the fiber line. The samples were collected within 5 h periods, which were synchronized with a process delay model. The model calculated the retention times in reaction vessels according to measured flow rates, consistency, and sizes of the vessels.

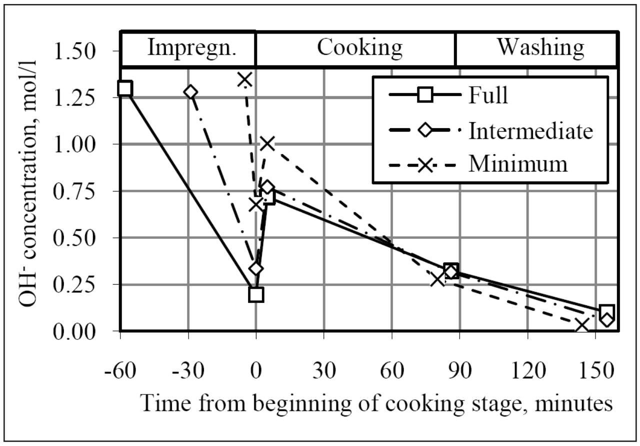
All unbleached and oxygen delignified pulps were thoroughly washed and centrifuged before analyses. Bleached pulp samples for refining studies were not centrifuged or pretreated. For paper properties analyses, we used a Voith-Sulzer refiner at 4.0% consistency and with a specific edge load of 1.0 J/m. **Table I** lists the standard methods used in the pulp and paper analyses.

Fiber lengths and curl factors were analyzed with a Metso FS300 analyzer, and fiber saturation point measurements were carried out [8]. Carbohydrate composition was determined by liquid chromatography after total hydrolysis following the standard protocol according to TAPPI T-249 cm-85. The analysis procedure is described in detail by Hausalo [9]. Hexenuronic acids were analyzed as described by Hausalo [9] and by Tenkanen et al. [10].

We determined the profiles of hemicelluloses in cell walls by peeling superficial fiber layers with desired thicknesses and analyzing the hemicelluloses composition from the peeled fractions [11]. A more detailed description of the methods used has been reported elsewhere [12].

Quantity	Standard
Effective alkali	SCAN-N 30:85
Kappa number	ISO 302:2004
Viscosity	ISO 5351:2004
Brightness	ISO 2469:2007
Preparation of laboratory sheets	ISO 5269, part 1
Density of sheets	ISO 534
Tensile strength	ISO 1924-2
Tear index	ISO 1974

I. Standard methods used in pulp and paper analyses.



1. Effective alkali profile in mill trial runs. Negative time values refer to the impregnation time. White liquor was added before impregnation and between impregnation and cooking stage.

RESULTS

Process conditions

A change in the impregnation time affected the consumption of effective alkali, i.e., hydroxyl ions, in the impregnation vessel and in the digester. **Figure 1** presents the hydroxyl ion profiles during the trial periods. In the impregnation stage, alkali consumption was high, especially at the beginning of impregnation. About half of the hydroxyl ions were consumed in chemical reactions or adsorbed into the chips during the first 5 min.

The results clearly demonstrate that the lower alkali consumption in the impregnation vessel was compensated by a higher alkali consumption in the digester when a given kappa number was targeted. Therefore, the total alkali consumption remained practically unchanged in all the trial periods. The role of the adjusted cooking conditions on the alkali consumption cannot be evaluated based on the results. **Table II** presents values for the alkali charges and consumptions on wood in the impregnation stage and cooking stage.

Reactions during an impregnation stage are exothermic [13]. In this study, temperature increased in the impregnation stage (Table II). The alkali consumption, and thus the rise in temperature, strongly correlated with the impregnation time. We noticed that the minimum impregnation required a higher cooking temperature and a higher H-factor. The reason for the high H-factor was not clearly identified, but might be related to packing degree changes in the digester. The lower packing with less impregnated chips results in a shorter retention time in the digester, which requires a higher cooking temperature.

Pulp characteristics

The first of the main goals of this study was to investigate whether the impregnation time has an effect on fiber characteristics. **Table III** presents the main fiber characteristics. Amounts of brown stock rejects were measured with an online measurement and in laboratory screening with and without wet disintegration. As expected, the amount of rejects was higher with shorter impregnation times (Table III). The

Alkali charges / consumption	Full	Int.	Min.
Impregnation EA charge	14.5	14.3	14.9
Impregnation EA consumption	12.3	10.5	7.4
Total EA charge	25.8	24.3	24.9
Total EA consumption	21.1	20.6	21.2
Temperatures	**Full**	**Int.**	**Min.**
Final impregnation temp.	118.7	117.0	110.7
Δ Temperature in impregnation	11.2	9.9	5.8
Cooking temperature	155.6	155.6	163.4
H-factor*	429	419	740

* Including impregnation and washing zones

II. Alkali charges and consumption and temperatures in impregnation and cooking stages. Alkali charges in percent of effective alkali (EA) on wood and temperatures in °C.

Screening reject, % on pulp	Full	Int.	Min.
Online measurement	1.9	3.7	6.8
Lab. screening, without WD*	2.8	7.0	15.9
Lab. screening, with WD *	0.2	0.2	3.1
Pulp after digester	**Full**	**Int.**	**Min.**
Kappa number	15.4	15.5	17.8
Viscosity, mL/g	1150	1180	1200
Brightness, % ISO	41.2	38.2	32.9
HexA, mmol/kg	54	57	53
Xyl:Glu	0.39	0.38	0.38
Fiber length, mm	0.94	0.96	0.95
Pulp after O_2 stages	**Full**	**Int.**	**Min.**
Kappa number	9.3	9.2	9.5
Viscosity, mL/g	835	845	878
Brightness, % ISO	60.0	59.2	52.5
Pulp after bleaching stages	**Full**	**Int.**	**Min.**
Viscosity, mL/g	775	763	789
Brightness, % ISO	90.2	89.9	88.5
HexA, mmol/kg	55	56	53
Xyl:Glu	0.38	0.38	0.37
FSP, g/g	1.18	1.25	1.21
Fiber length,** mm	0.89	0.91	0.94
Curl, %	15.9	12.3	13.8

*WD: wet disintegration, 30,000 revolutions.

** Length-weighted average.

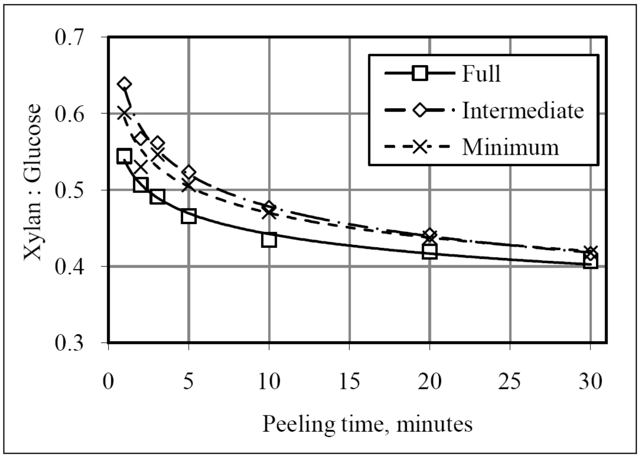
III. Pulp and fiber characteristics.

amount of rejects depended on the analysis method, but all the screening methods confirmed the effect of impregnation time on the amount of rejects. The wet disintegration had a significant effect on the amount of rejects. Based on practical experience at the studied mill, we assume that the online measurement gave the most accurate results for the real amounts of rejects.

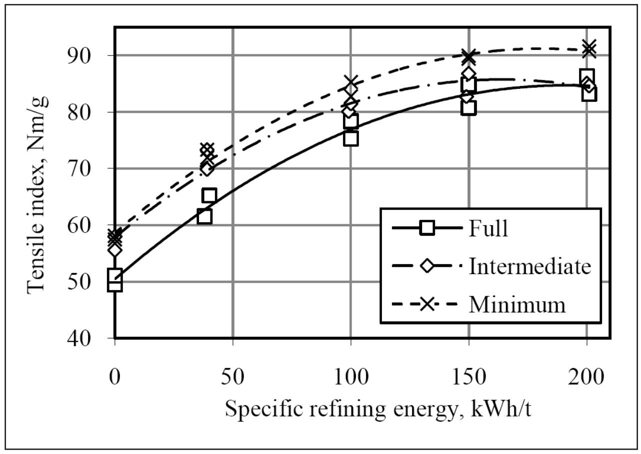
The variation in the kappa number might explain a part of the results related to the amount of rejects. A cooking kappa number of 16.5 was targeted, but a slight variation in cooking kappa number occurred (Table III). In particular, the kappa number of the minimum impregnation period was higher despite the more intensive cooking conditions. No major differences were found in hexenuronic acid contents.

For unbleached pulps, shorter impregnation times resulted in a slightly lower viscosity and a lower brightness. However, the variation in the kappa numbers might explain these observations. Oxygen delignification reduced differences among the pulp, but the minimum impregnation pulps still had a lower brightness value compared with the full and intermediate impregnation pulps. In general, pulps produced with a shorter impregnation time were more difficult to delignify by oxygen and to bleach, even when higher chemical charges were used for those pulps. In the oxygen delignification stage, a typical total oxygen charge was around 20 kg/a.d. ton, and in bleaching a typical chlorine dioxide charge of 34 kg/a.d. ton-41 kg/a.d. ton. Chemical charges and process conditions for oxygen delignification and bleaching can be found in detail elsewhere [12].

Fiber dimensions are of great importance for papermaking properties. The average length-weighted fiber lengths varied marginally between the trial periods. The average fiber length was reduced to some extent in oxygen delignification and bleaching. Long impregnation time apparently contrib-



2. Xylose:glucose ratio in surface selectively peeled fraction.



3. Development of tensile index as function of refining energy.

uted to the fiber length reduction. Fiber length distribution curves confirmed that no softwood fibers were present in any trial periods [12]. The widths of the distribution were practically identical between the studied periods. Fiber curl varied slightly between the periods and was highest with full impregnation.

We determined the composition of hemicelluloses in the pulps by carbohydrate analysis. For simplicity, the results are expressed as xylose:glucose ratios only, and individual carbohydrate results were reported previously [12].

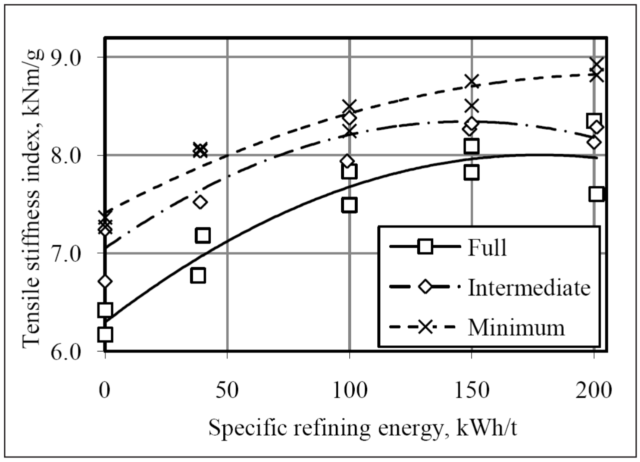
Xylan is the main hemicellulose in birch. In this study, the xylose:glucose ratio was not affected by the impregnation time. Small amounts of mannose were found in the minimum and intermediate impregnation pulps. The amount was highest with the minimum impregnation, 0.7%, on the hydrolysis residual. Similar to the average contents of hemicelluloses, no significant differences were found in the fiber saturation points or hexenuronic acid contents.

The distribution of xylan across the cell wall was different for the different pulps (**Fig. 2**). For all the pulps, the content was higher in the superficial fiber layers. The content of surface hemicelluloses was lower for the full impregnation pulp than for the intermediate and minimum impregnation pulps. However, the contribution of the impregnation time on the content of the surface hemicelluloses is challenged by the facts that the difference between the minimum and intermediate samples was marginal, and xylan content was highest with the intermediate impregnation.

Papermaking properties

The latter goal of this study was to monitor the effects of impregnation time on papermaking properties. The bleached pulps produced with the different impregnation times were refined with specific refining energies (SRE) ranging from 0 kWh/ton to 200 kWh/ton. **Figure 3** presents the development of tensile strength in refining.

The tensile strength increased with specific refining energy. Minimum impregnation pulps showed a better tensile strength development compared with full impregnation pulps.



4. Tensile stiffness index vs. specific refining energy.

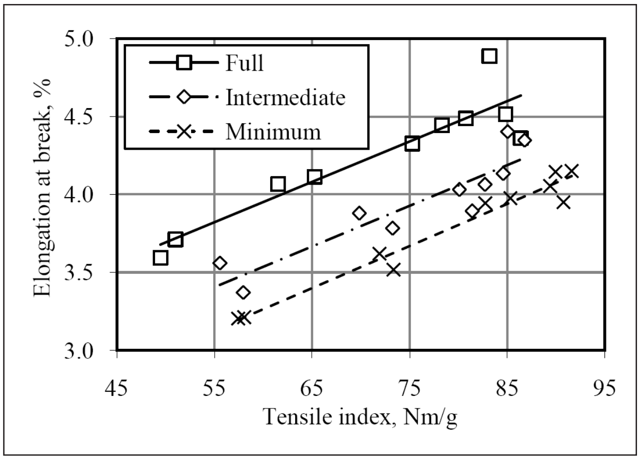
Interestingly, the tensile strengths of the intermediate pulps were similar to the minimum impregnation pulps, whereas at higher SRE values they behaved more like the full impregnation pulps. The development of tensile strength was similar when the tensile indexes were plotted against SR-degrees.

Sheet density increased linearly with increasing SRE. At a level of 50 kWh/ton, the interpolated densities were 0.79 g/cm^3, 0.80 g/cm^3, and 0.78 g/cm^3 for the full, intermediate and minimum impregnation pulps, respectively.

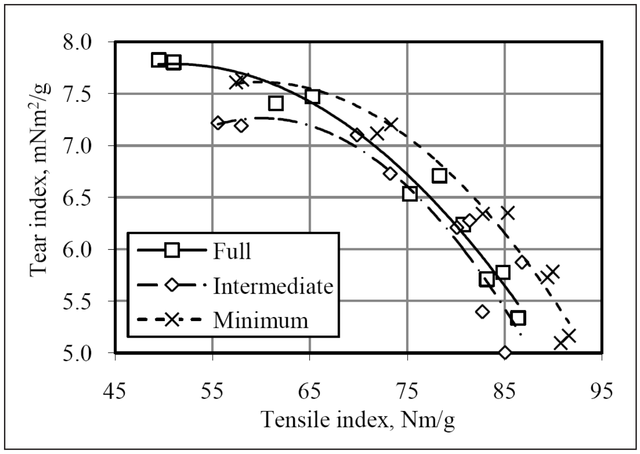
As with tensile strength, tensile stiffness increased with increasing SRE and sheet density. **Figure 4** presents the development of specific tensile stiffness against SRE.

Tensile stiffness index, i.e., specific elastic modulus, increased when the pulps were further refined. Like the tensile index, the tensile stiffness index at a given SRE value was higher with shorter impregnation times. At an SRE of 50 kWh/ton, the intermediate impregnation pulps had 9% higher tensile stiffness index compared to the full impregnation pulps, and minimum impregnation pulps had 12% higher tensile stiffness index.

A lower tensile stiffness for full impregnation pulps was in accordance with a higher elongation at a break (**Fig. 5**). At



5. Elongation at break vs. tensile index.



6. Tear index vs. tensile index.

the tensile index of 70 Nm/g, the intermediate impregnation pulps had 7% and the full impregnation pulps had 17% higher elongation at their breaking points compared with the minimum impregnation pulps.

Tear strength at a given tensile strength is typically used to characterize pulp strength. As shown in **Fig. 6**, the tear-tensile curves demonstrate no obvious dependency on the impregnation time. The minimum impregnation pulps had the highest tear strength at the tensile index of 70 Nm/g. At the same tensile index, the tear index of the intermediate impregnation pulps was the lowest.

DISCUSSION

A pulp mill is a more challenging environment than a laboratory to conduct research, and it is never possible to recognize or control all of the factors influencing the results. On the other hand, many phenomena important for commercial-scale production occur only in full-scale pulp mills. Therefore, mill-scale studies are important to confirm laboratory results, and to recognize new factors that should be modeled in laboratory studies.

To a great extent, our results are in line with previous results referred to in the introduction. The results confirm the high consumption of effective alkali in the first minutes of an impregnation stage, whereas the total consumption of alkali was not significantly affected between the trial periods. We recognized clear evidences of exothermic reactions in the impregnation stage. As in previous studies [4], our results indicated that a higher cooking temperature is required with shorter impregnation times.

The results also confirmed that a sufficient impregnation time is essential for cooking uniformity and for a low amount of rejects. Surprisingly, we did not find significant differences in the average carbohydrate composition of the studied pulps. This might be explained by the facts that the cooking kappa number was kept constant, and the total consumption of effective alkali did not vary significantly. Impregnation time did not contribute the viscosity of fully bleached pulps.

We found clear indication that the impregnation modification affected some of the analyzed papermaking properties, despite the fact that we did not find differences in chemical compositions of the studied pulps. Tensile strength was higher with shorter impregnation time. Significant differences also could be seen in the tensile stiffness and elongation at break; a short impregnation resulted in clearly stiffer paper sheets. We could not deduce the reason for this from the obtained results, but introduce new hypotheses to explain the results.

Yield effect

The papermaking potential of pulp is dependent on the coarseness of the fibers [14,15]. Because pulping yield affects the coarseness, the yield can have a direct influence on pulp properties. In practice, a lower coarseness increases the number of fibers in a given amount of pulp. The yield also correlates with other fiber characteristics, e.g., cell wall elasticity, and thus has effects on paper properties [16]. In this study, harsher cooking conditions and an increased amount of rejects probably reduced the cooking yield with the shorter impregnation times. Unfortunately, we could not determine the exact yield in the continuous process and therefore the hypothesis remains unanswered.

Effect of nonuniformity between fibers

Another interesting hypothesis is that the noticed differences were caused by pulping nonuniformity. Most of the pulp analyses are based on average values for the measured quantity, and they cannot determine the distribution of analyzed quantity within the analyzed samples between single fibers. Rayel et al. [17] and Malkov et al. [18] have shown that kappa number varies between individual fibers, and that the width of the distribution can be affected by process conditions. On the other hand, Aurell, for example, has reported [19] that the hemicelluloses content of birch pulp is dependent on the delignification degree and the alkali charge. Therefore, we assumed that besides the single fiber kappa variation, the content of single fiber hemicelluloses also varies within a nonuniform pulp sample. Because of relatively good selectivity in oxygen delignification and bleaching stages, we also assumed that the variation in single fiber hemicelluloses remains in bleached pulps even if the variation in the single fiber kappa number is marginalized.

The difference in the amounts of brown stock rejects is a strong indication that the pulping uniformity was different between the trial periods. The nonuniformity also might occur in the content of hemicelluloses between individual fibers. Unfortunately, we can only speculate whether the distribution of hemicelluloses can influence, for example, the fiber activation in paper sheets. However, variation in both cell wall elasticity and fiber swelling/shrinking characteristics could have an effect.

Effect of nonuniformity within individual fibers

Procter et al. [20] and Saka et al. [21], for instance, have reported that delignification in a cell wall is not uniform during delignification. At the beginning of delignification, most of the lignin is dissolved from inner cell wall layers. The dissolution of lignin in outer cell wall layers and middle lamella becomes faster in the later parts of delignification.

The nonuniform delignification in a cell wall may be coupled with nonuniform degradation of cellulose and hemicelluloses. This might result in the situation in which the composition of the superficial layers differs from the average composition. Other researchers have suggested that the superficial fiber layers contribute to papermaking properties. Sjöberg et al. [22] reported that surface hemicelluloses are of special importance for tensile strength, tensile stiffness, and elongation at break.

In this study, the tensile strength and stiffness and the elongation at break were different between the trial periods, but the influence of the surface hemicelluloses was not obvious. The surface hemicelluloses likely would not explain the differences between the intermediate and the minimum impregnation pulps, because the determined difference was marginal. However, they might contribute to the differences between the full impregnation pulps against the intermediate and minimum pulps.

Besides the degradation of hemicelluloses, cellulose degradation is not necessarily uniform in a cell wall. Berggren et al. [23] have reported that at a given viscosity level, the molecular size distribution of cellulose might be different depending on the cooking conditions. Similarly, Sjöholm et al. [24] have shown that the zero span strength is not solely dependent on viscosity. Therefore, in this study, the intrinsic fiber strength might have been affected despite the fact that we found no difference in the viscosities.

Chip column pressure

Of note, the difference between full and intermediate periods was at least as significant as the difference between intermediate and minimum impregnation. If this is explained by a chemical reaction, the rate of the reaction during the impregnation should be a constant. Such a zero-order reaction is difficult to find in kraft pulping processes. However, the height of the chip column was linearly dependent on the impregnation time. It is tempting to explain the results by the compression pressure caused by the chip column. High compression pressure may create fiber damages, e.g., fiber curling, that materializes in the later parts of the fiber line and reduces tensile properties in paper sheets. What is more, we found indications of reduced fiber length and more severe curling with longer impregnation times. The fiber damages might explain the higher reduction in the fiber lengths with increasing impregnation times. Unfortunately, this hypothesis cannot be confirmed by the obtained results.

CONCLUSIONS

We conducted a series of mill trials with varied impregnation times in a commercial-scale fiber line and monitored the effects of the modification on fiber characteristics and papermaking potential. The results clearly demonstrated that sufficient impregnation is essential for good pulping uniformity measured as an amount of rejects. Poor impregnation also called for more intensive cooking conditions.

The average carbohydrate composition was not noticeably affected by the process modification. This is probably because the cooking kappa target and the brightness targets were kept constant in the trials. The viscosities also were the same between the bleached pulp samples. Therefore, it was a surprise to notice that some of the papermaking properties were significantly affected between the periods with different impregnation times. The tensile stiffness of the laboratory sheets was higher with the shorter impregnation times. At the same time, the elongation at break was reduced. We could not see the contribution of the impregnation times on tear versus tensile strength curves.

Analytical results did not provide a clear mechanism for the affected tensile stiffness properties. Consequently, new hypotheses must be introduced to explain the results. The results might be explained by a decreasing screened yield with a decreasing impregnation time. In addition, delignification uniformity might affect the sheet properties. Nonuniformity in the amounts of hemicelluloses between single fibers might influence fiber activation during the drying of a paper sheet, and thus affect sheet stiffness. The modified impregnation also might affect the cooking uniformity in the cell walls of fibers. Finally, the chip column pressure might contribute to the results by creating fiber damages, which materialize as impaired papermaking properties. **TJ**

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An interesting possibility to study effects of impregnation came up when a pulp mill was testing new ways to operate its cooking plant. The main focus of this study was to ensure that papermaking properties were not impaired as a result of the new operation models of the cooking plant.

Although impregnation has been investigated in laboratory studies, this time it was possible to conduct the experiments on a commercial scale. The large scale and mill conditions were more difficult to control. However, we were able to find periods for our experiments when the process was relatively stable.

To our surprise we found out that some of papermaking properties were improved with shorter impregnation times. The results suggest that the design of the impregnation stage might significantly affect papermaking properties of pulp.

We hope that our study also helps to recognize new factors that could be studied in coming laboratory studies.

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