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Towards complete impregnation of wood chips with aqueous solutions

Part 2. Studies on water penetration into softwood chips

Abstract

Importance of the chip impregnation stage is widely recognised in chemical pulping processes. However, there is limited amount of direct data on mass transfer during the impregnation stage. Most methods used for studying the liquid penetration process were based on indirect approaches.

A new laboratory impregnator with a special weight sensor was developed, which allowed the direct measurements of the chip weight during impregnation process. First part of the research included testing of the system under various process conditions and method development. Experiments with water showed that the impregnator could be successfully used in studying the effect of different factors on the liquid penetration into wood chips. The new system can also be used as a tool to investigate different "penetration aid" techniques, including presteaming, evacuation, addition of surfactant, and various mechanical and biotreatments of wood chips.

Tiivistelmä

Kohti puuhakkeen täydellistä impregnointia. Osa 2. Veden imeytyminen havupuuhakkeeseen

Imeytyksen merkitys sellukeiton onnistumiselle on ollut jo pitkään tiedossa. Kuitenkin asiasta on vain hyvin vähän suoraa mittaustietoa ja tutkimusmenetelmät ovat olleet pikemminkin epäsuoria.

Työssä kehitettiin uusi hakkeen painomittauksen sisältävä imeytysreaktori, jolla voidaan suoraan mitata höyrytyksen ja imeytymisen aineensiirtoa. Tutkimustemme esillä olevassa ensimmäisessä osassa kehitettiin uusi imeytyksen tutkimus- ja laskentamenetelmä, joka kokeiltiin erilaisissa höyrytys- ja vesipenetraatio-olosuhteissa. Uusi menetelmä soveltuu hyvin tutkimus- ja kehitystyöhön pyrittäessä parempaan imeytykseen ja tasaisempaan keittoon käyttäen esimerkiksi höyrytystä, ilmanpoistoa, apuaineita ja erilaisia hakkeen mekaanisia sekä biologisia esikäsittelyjä.

Introduction

The mass transfer of reactive chemicals into the core of a wood chip is of great importance in chemical pulping processes. Chemical transport into the chip voids is accomplished by two primary mechanisms. The first mechanism, liquor penetration, refers to the flow of liquor into the air-filled voids of the wood chip, under the pressure gradient. The second mechanism refers to diffusion of ions or other soluble matter through water under the influence of the concentration gradient /1/.

Numerous studies concerning the penetration of various liquids into wood have been carried out during the 1950s and 1960s /2–10/. Most researchers have been investigating the effect of various factors, including experimental conditions, wood structure, and liquor properties, on the penetrability of wood. The influence of "penetration aid" techniques, such as presteaming, purging, evacuation, and addition of surfactants, has also been examined /11/.

The penetrability of wood has been studied by different techniques and methods. Special penetration clamps have been used for permeability test /2,3/. This method is based on measurements of the liquid flow-rate through wood blocks while applying pressure differential. Another popular technique was a sinkage test /4,5/, where wood blocks were immersed in a liquid for a certain time and the weight increase was determined. These methods have been primarily used to get information about the permeability properties of wood.

Canadian researchers /6,7/ have used a quartz spiral balance to continuously measure the penetration of various liquids into a single wood chip. By measuring the increase in chip weight, they were able to calculate the penetration rate and to study the effect of different process conditions and presteaming. This method was much closer to simulating the liquor penetration into a wood chip, which takes place in a closed cooking digester. While some of the techniques gave an estimation of the total amount of liquid that had penetrated into the wood, others provided information about the ways how liquid penetrated into the wood voids. These included methods based on colour reactions /8,9/ and application of radioactive tracers /10/.

Nowadays, the importance of thorough impregnation prior to cooking is widely recognised in the pulping industry. Several attempts have been made to model various impregnation and cooking processes /12–15/. Some of the suggested models are diffusion models that assume infinite penetration /12–14/. Under industrial conditions this assumption is far from reality; some entrapped air is usually present within the impregnated chips. When modelling impregnation or cooking of wood chips, it is important to take into account not only diffusion but also the bulk flow of liquid. From this viewpoint, there is a great need for modelling of the penetration process.

Several new methods have been recently developed to obtain more detailed information on the penetration process, on the factors effecting it and ways to improve its efficiency. For example, nuclear magnetic

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resonance (NMR) imaging can be used to study the spatial distribution of water in wood /16,17/. Some methods are based on the penetration of cooking liquor into the chip, freezing the penetrated chip and analysing microtomed slices with the scanning electron microscopy (SEM) technique /12,18/.

The objective of the present work was to use a special laboratory impregnator for studying the penetration of water into wood chips. Hand-made chips of exact dimensions and homogeneous properties were prepared from the heartwood and sapwood fractions of three softwood species. The effects of wood type, chip dimensions and process conditions, on the penetration efficiency of water into softwood chips were studied. In addition, the effect of "penetration aid" techniques, such as presteaming and evacuation, was examined.

Method

Materials

Three softwood species: pine (*Pinus silvestris*), spruce (*Picea abies*) and larch (*Larix sibrica*) were chosen for chip preparation. Pine and spruce logs were delivered from southeastern Finland, while larch wood samples came from Baikal'sk, Russia. Chips were prepared separately from sapwood and heartwood. With pine, separation of the heartwood and sapwood fractions was straightforward because of the clear border between them (Fig. 1). For spruce and larch, the transition from heartwood to sapwood was gradual. To avoid errors in the separation of heartwood and sapwood, the "transition" area was removed. The chips did not contain bark or knots.

The softwood chips prepared for the experiments had exact dimensions. The width of the chips (tangential direction) was 15 mm, and their lengths (longitudinal direction) – 15, 25, and 35 mm, respectively. Their thickness dimensions (radial direction) were 4, 8, and 12 mm. The moisture content and basic density of the sapwood and heartwood chips were determined according to the standard methods SCAN-CM 39:94 and SCAN-CM 43:89. The properties of the chips are shown in Table 1.

Bags with chips were stored in a freezer at -20°C to minimise moisture losses and to prevent biological degradation. There was likely to be some ice crystal formation in the chip sample while it was kept in a freezer. This could have blocked the liquor penetration into the wood matrix. To avoid this undesirable phenomenon the chip sample was taken out of the freezer three days be-
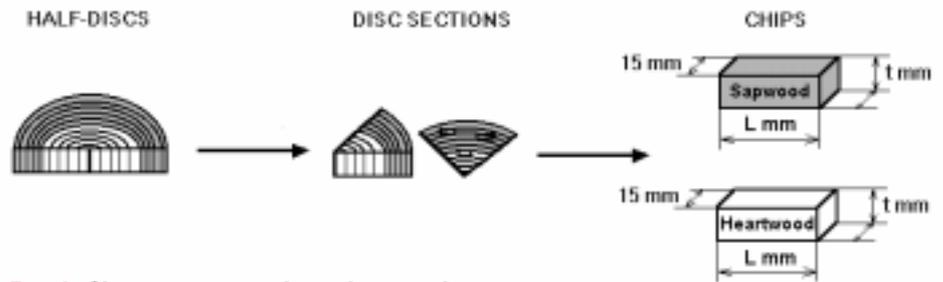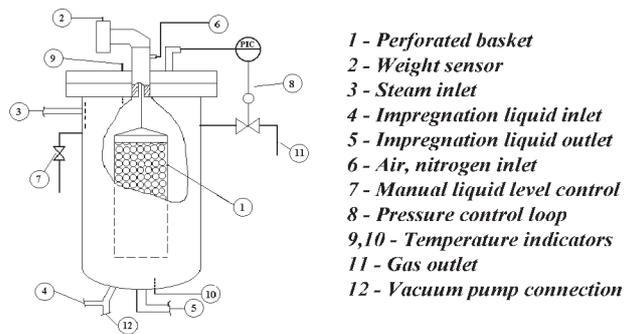

Fig. 1. Chip preparation from the pine discs.

1 - Perforated basket
2 - Weight sensor
3 - Steam inlet
4 - Impregnation liquid inlet
5 - Impregnation liquid outlet
6 - Air, nitrogen inlet
7 - Manual liquid level control
8 - Pressure control loop
9,10 - Temperature indicators
11 - Gas outlet
12 - Vacuum pump connection

Figure 2. Schematic view of the laboratory impregnator.
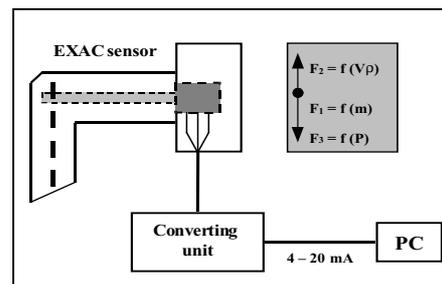

Fig. 3. Weight measuring system.

fore the experiments and kept at a temperature of + 5°C.

Experimental system

Experimental work was carried out using a special laboratory impregnator (Fig. 2). A perforated basket containing the chip sample was connected to a weight sensor, which allowed continuous measurements of the chip sample weight throughout the experiment. The vessel was equipped with several valves for inlet and outlet streams. Temperature was maintained by using a conventional jacket system. Two temperature indicators were used to monitor the temperature profiles in the lower and upper parts of the impregnator.

The heart of the experimental system is the weight-measuring unit (Fig. 3), in which the EXAC sensor serves as the primary sensing element. The EXAC sensor's operation is

based on level measurement, which is done through the strain-sensitive element. The three forces that balance weight sensor readings during the penetration experiments are shown in Fig. 3. Force F_1 is a downward force and a function of the mass of the basket and the chip sample. The buoyancy force, F_2, is an upward force and a function of the immersed volume and density of the liquid. Force F_3 is a downward force and a function of applied pressure.

When the exact volumes of the basket and chip sample, the density of the liquid, and the correlation between the weight sensor reading (WSR) and pressure are known, the reference value (REF) can be calculated. Reference value can be defined as a hypothetical weight sensor reading, in case if no liquid has penetrated into the chips. The weight of the chip sample is continuously monitored based on the weight sensor readings and reference value. Fig. 4 shows an example of the weight sensor reading profile throughout the steaming and penetration stages. The five experimental phases are:

1- Before presteaming: Sensor reading is zero at initial moment.

2- After presteaming: Weight of chips increased due to steam sorption into the wood voids and condensation of steam on surfaces. As a result, sensor reading is above zero.

3- Liquid introduction: Sensor reading becomes negative due to buoyancy effect of the immersed basket and chip sample.

Table 1. Properties of the softwood chips.

Wood species	Pine		Spruce		Larch	
Chips	HW	SW	HW	SW	HW	SW
Basic density, g /cm³	0.395	0.425	0.345	0.385	0.525	0.415
Moisture content, %	22.5	57.0	25.5	58.0	26.0	54.5

The amount of water initially present inside the wet chips (V_{water0}) and its adjusted value due to the different penetration temperature can be calculated based on known water density values:

$$V_{water0} = (W_{chip} * (1 - DMC / 100)) / \rho_{water0} \quad \text{(Eq. 4)}$$

$$V_{water\ adjusted} = V_{water0} * (\rho_{water0} / \rho_{water}) \quad \text{(Eq. 5)}$$

where

ρ_{water0}, **g/cm³** - density of water at initial temperature of the chip,

ρ_{water}, **g/cm³** - density of water at penetration temperature.

The amount of water that has penetrated into the chip ($W_{penetrated\ water}$) is calculated from the weight sensor reading and reference value. By dividing the weight by water density, the volume of penetrated water ($V_{penetrated\ water}$) can be determined:

$$V_{penetrated\ water} = W_{penetrated\ water} / \rho_{water} \quad \text{(Eq. 6)}$$

Finally, the penetration degree from the theoretical maximum of the initial (PD_0) and penetrated chips (PD) can be calculated from the volume of voids and volume of water inside the chip:

$$PD_0 = (V_{water\ 0} / V_{voids}) * 100\ \% \quad \text{(Eq. 7)}$$

$$PD = ((V_{water\ adjusted} + V_{penetrated\ water}) / V_{voids}) * 100\ \% \quad \text{(Eq. 8)}$$
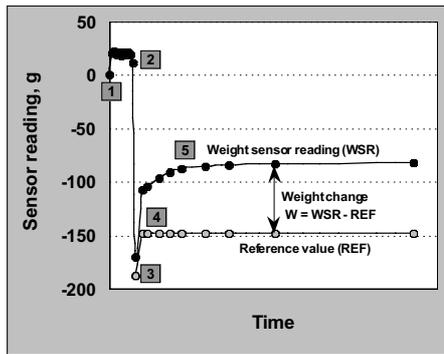
Fig. 4. Profile of sensor readings.

4- Overpressure is applied: Sensor reading becomes less negative due to the effect of applied pressure.

5- Penetration phase: Sensor reading changes with the increased weight of the chip sample due to the penetration of water.

Experimental procedure

Small chip samples of about 50g oven-dry wood were used in the penetration trials. During presteaming, saturated steam was introduced into the upper part of the impregnator. Steam temperature was 110°C, which corresponded to about 1.4 bar pressure. Condensate formed during the steaming procedure was continuously removed from the outlet valve.

A vacuum pump was used for evacuation procedure. Evacuation of the chip sample was carried out under a vacuum of -1 bar during a certain time interval.

After completion pretreatment, water of required temperature was introduced from the bottom of the vessel. The level of liquid was controlled manually. The liquid-to-wood ratio was 25. Pressurised nitrogen was used to reach the required pressure. Pressure was controlled throughout the penetration experiment by a pressure controller.

Evaluation of results

The results of the penetration experiments were evaluated in terms of the penetration degree from the theoretical maximum, i.e. the fraction of the chip voids filled by liquid. To simplify calculation of the penetration degree, the following assumptions were made:

- Volume of chips is constant during penetration.
- Water is incompressible.
- Losses of wood substance are negligible.
- Density of wood substance (ρ_{wood}) is constant: 1.500 g/cm³.

Volume of the chips (V_{chip0}), volume of the wood substance (V_{wood0}), and volume of voids within the chips (V_{voids0}) can be determined from the initial chip properties:

$$V_{chip0} = (W_{chip} * DMC) / (100 * \rho_{chip}) \quad \text{(Eq. 1)}$$

$$V_{wood0} = (W_{chip} * DMC) / (100 * \rho_{wood}) \quad \text{(Eq. 2)}$$

$$V_{voids} = V_{chip0} - V_{wood0} \quad \text{(Eq. 3)}$$

where

DMC, % - dry-matter content of the chips,

ρ_{chip}, **g/cm³** - basic density of the chips,

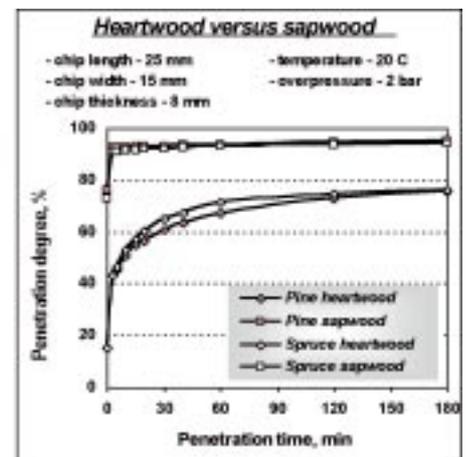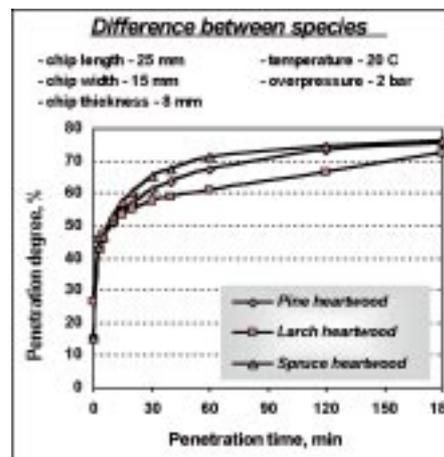W_{chip}, **g** - initial weight of the wet chips.

Fig. 5. Effect of wood structure on penetration efficiency.

Results and discussions

The set of water penetration trials, with and without pre-treatment, was carried out by using a new laboratory impregnator. The impacts of numerous factors, such as wood species, wood structure, chip dimensions and process conditions, on penetration efficiency were examined. Table 2 shows a list of data from the penetration experiments.

Effect of wood structure
It is widely recognized that the capillary structure of wood has a major influence on the efficiency of liquid penetration into wood chips /1,3,6,19/. Geometry and dimensions of individual wood capillaries vary between wood species and between different types of wood (heartwood and sapwood).

The penetration of water into the wood chips as a function of time is shown in Fig. 5. The graph on the left shows the penetration into heartwood of different softwood species, while the graph on the right compares the penetration into heartwood and sapwood chips.

The efficiency of water penetration into heartwood chips varies depending on the wood species. Penetration into larch heartwood chips has the lowest efficiency, which is probably caused by the difference in the structure of wood capillaries and wood density. Penetration into heartwood chips was less efficient than penetration into sapwood chips due to the difference in initial air con-
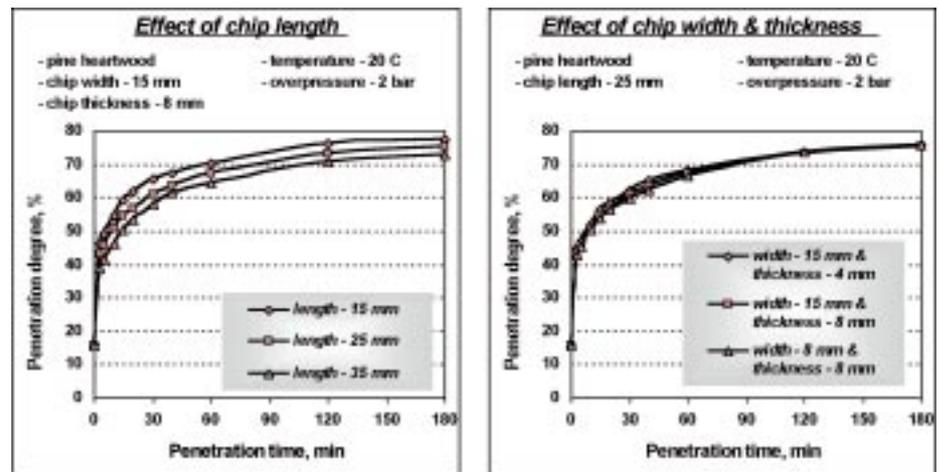

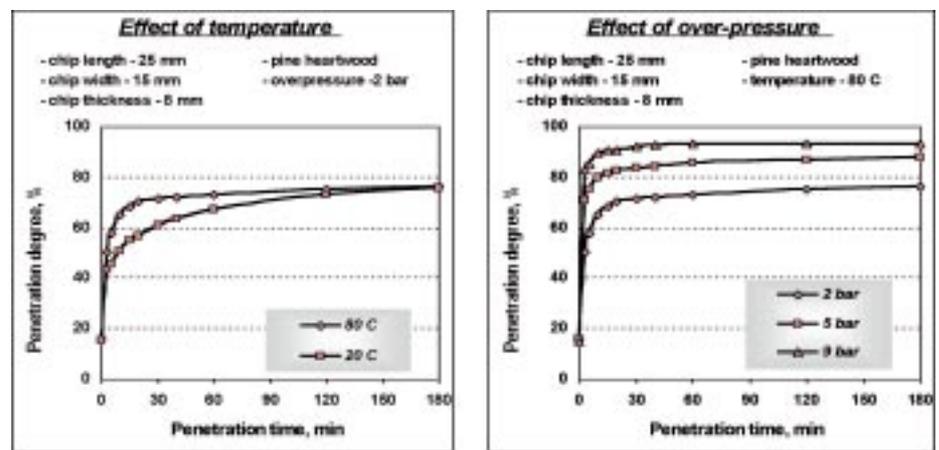**Fig. 6.** Effect of chip dimensions on penetration efficiency.

Fig. 7. Effect of process conditions on penetration efficiency.

Table 2. Selected results from penetration experiments with water.

Species	Larch	Spruce	Pine	Spruce	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine	Pine
HW / SW	HW	HW	HW	SW	SW	HW	HW	HW	HW	HW	HW	HW	HW	HW	HW	HW
Chip length, mm	25	25	25	25	25	25	25	15	35	25	25	25	25	25	25	25
Chip thickness, mm	8	8	8	8	8	8	4	8	8	8	8	8	8	8	8	8
Chip width, mm	15	15	15	15	15	7	15	15	15	15	15	15	15	15	15	15
Temperature, °C	20	20	20	20	20	20	20	20	20	80	80	80	80	80	20	20
Overpressure, bar	2	2	2	2	2	2	2	2	2	2	5	9	2	2	2	2
Steaming time, min.	-	-	-	-	-	-	-	-	-	-	-	-	5	10	-	-
Evacuation time, min.	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5	10
Time moment																
Initial	26.8	15.3	15.6	72.9	76.3	15.5	15.7	15.9	15.8	15.8	15.7	14.9	16.1	15.8	16.3	16.0
After pretreatment	-	-	-	-	-	-	-	-	-	-	-	-	30.4	41.2	16.3	16.0
3 minutes	45.7	43.2	43.3	90.9	92.4	42.6	44.7	46.2	39.1	50.2	70.8	83.2	61.0	66.8	51.0	51.9
5 minutes	47.6	46.7	45.6	91.2	92.6	45.1	47.0	49.0	41.4	57.7	75.0	85.2	67.6	73.2	53.8	55.3
10 minutes	51.4	52.9	50.6	91.5	92.8	50.2	52.2	54.6	46.3	65.2	80.1	89.4	75.1	79.5	60.6	61.1
15 minutes	53.3	57.2	54.8	91.7	93.0	53.8	56.1	59.2	50.4	68.3	81.6	90.4	79.3	84.7	64.6	66.2
20 minutes	55.2	60.3	56.8	91.9	93.1	56.3	58.5	61.9	53.5	70.4	82.3	90.7	82.5	85.8	67.5	70.2
30 minutes	57.4	65.1	60.8	92.3	93.3	59.7	62.3	65.4	58.0	71.4	83.4	91.8	85.6	87.8	72.0	74.8
40 minutes	58.9	67.3	63.5	92.5	93.5	62.3	65.0	67.5	61.5	71.9	84.3	92.6	86.5	88.8	75.4	77.6
60 minutes	60.8	71.2	67.5	92.9	93.9	66.5	68.1	70.2	64.4	72.9	85.9	93.1	88.4	90.7	80.0	82.8
120 minutes	66.5	74.7	73.2	93.9	94.9	73.7	73.6	76.1	70.6	75.3	86.9	93.1	89.3	92.6	85.7	87.4
180 minutes	72.2	76.4	75.5	94.4	95.2	75.7	75.7	77.6	72.8	76.4	88.0	93.3	90.4	93.7	87.9	89.6

tent as well as the difference in wood structure. In contrast to the sapwood chips, the compression level is not easily reached during penetration into heartwood chips. It is believed that the obstruction of liquid flow within the heartwood is caused by pit aspiration and resinification of interconnecting capillaries /5,19,20/.

Effect of chip dimensions

Fig. 6 shows penetration curves for pine heartwood chips with different length and thickness dimensions. Reducing the thickness of the chips from 8 mm to 4 mm did not result in any noticeable changes in water penetration. Similarly, the reduction of chip width did not improve the penetration. On the other hand, reducing the length of the chips led to a significant improvement in the efficiency of water penetration. Since the longitudinal flow in softwoods is 50–200 times faster than the tangential and radial flows /19/, it is clear that the length of the chips should have a more pronounced effect on the efficiency of penetration than other dimensions.

Effect of process conditions

Optimisation of process conditions, such as temperature and pressure, can be used as a primary tool for improving liquid penetration into wood chips. The pressure gradient plays a primary role in the penetration process. The temperature influences the properties of liquid as well as structure of the wood and, therefore, affects the penetration.

Fig. 7 shows the effect of temperature and pressure during water penetration into pine heartwood chips. Warm water (80 °C) penetrates more effectively than cold water and the level of compression is reached much faster. Cold water penetrates slowly at the beginning of the process and it takes quite a long time to reach the level of compression. Of course, the low viscosity of warm water has a positive effect on penetration efficiency. Softening of resin compounds present within the pine heartwood capillaries by warm water can reduce the obstruction of liquid flow, and therefore contribute to improved penetration.

There is a clear difference in the final penetration degrees achieved under three pressure levels. Higher pressure results in higher compression of air within the chip voids, thus facilitating water flow into the wood capillaries. The final penetration degrees achieved after three hours were higher than those calculated from air compression (dashed lines), which indicated partial removal of air from the chips during the penetration process.

Effect of "penetration aid" techniques

The presence of air within the wood voids is considered to be the main obstacle to penetration of liquid into the chip /1–5,21/. So, it is vital to remove the entrapped air as completely as possible prior to subjecting the wood chips to liquor. Several "penetration aid" techniques, including evacuation and presteaming, can be used to achieve this goal /2,4,7/.

Fig. 8 shows the effect of presteaming on the efficiency of water penetration into heartwood pine chips. The penetration degree, 75.5 %, was achieved with no presteaming. A much higher degree of penetration, 90.4 %, was achieved when the heartwood chips were presteamed for five minutes. Extension of the presteaming time to 10 minutes resulted in a further increase in penetration degree, up to 93.7 % of the theoretical maximum.

The primary effect of presteaming is a removal of entrapped air from the chip cavities. Part of the air is removed due to thermal expansion. Furthermore, the increase in the partial pressure of water vapour within the chip due to elevated temperature causes the remaining air to be expelled from the chip voids. In addition, when dealing with pine heartwood, resinification and pit aspiration lead to limited mass transfer between tracheids. It has been suggested that steaming treatment results in hydrolysis of some components in the pit membrane /22/. These chemical changes might be responsible for the improved penetration, since they reduce the effectiveness of pit aspiration.

The weight of the chip sample increased significantly during presteaming (Table 2). This can be explained by the following phenomena: steaming of the chip was accompanied by condensation of steam on its colder surfaces. When presteaming was completed and temperature of the chips dropped, there was condensation of water vapours within the chip voids. The vacuum caused in this way facilitated the uptake of the condensate from the chip surface into the voids, thus increasing the moisture content of the chip.

Fig. 9 shows the effect of chip evacuation on the efficiency of water penetration into heartwood pine chips. Evacuation of the chip sample for 5 minutes prior to water penetration under 2 bar pressure resulted in a penetration degree of 88 %. By extending the evacuation, it was possible to achieve a small improvement in penetration. Surprisingly, the moisture content of the chip sample did not change during evacuation.

Summary

A new laboratory impregnator was developed in the Laboratory of Pulping Technology of the Helsinki University of Technology. Penetration experiments showed that the impregnator could be successfully used in studying the penetration of various liquids into hand-made chips. Process conditions, such as pressure and temperature, can be applied within the limitations imposed by the weight sensor. The new system can also be used as a tool to investigate different "penetration aid" techniques, including presteaming, evacuation, surfactant addition, and bio-treatments of various wood chips.
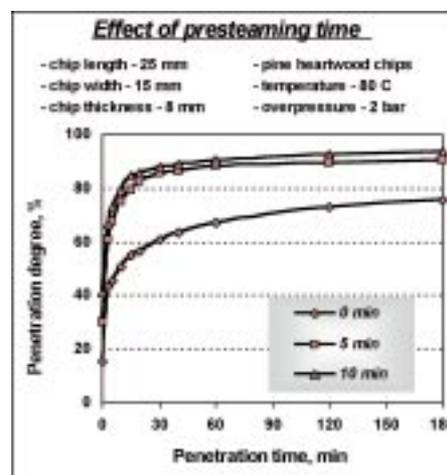
We believe that the data obtained from

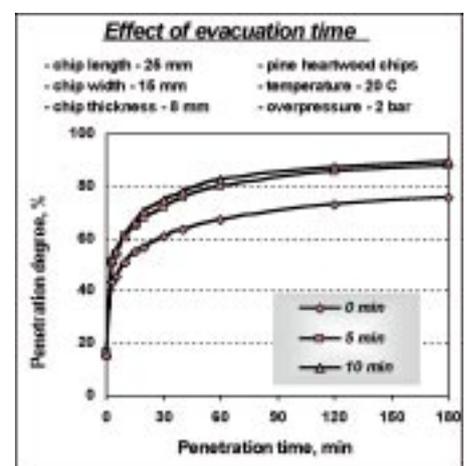**Fig. 8.** Effect of presteaming on penetration efficiency.

Fig. 9. Effect of pre-evacuation of chips on penetration efficiency.

the penetration experiments will shed more light on the phenomena, which take place during the penetration of liquid into wood voids. In addition, the obtained data will be of great importance for testing available models of penetration processes.

Acknowledgements

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