



EXPERIMENTAL DETERMINATION OF THE DIFFUSION OF MONOVALENT CATION INTO WOOD: EFFECTS OF MICRO-CRACKS, WOOD STRUCTURE, IMPREGNATION TIME AND TEMPERATURE ON LOCAL CONCENTRATION PROFILES

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ABSTRACT A thorough understanding of the key phenomena that take place during the chemical transport of reactants into a wood matrix is critical for the success of today's chemical pulp mills as well as future biorefinery operations. In the present article, our previous experimental methodology has been further developed to minimize the effect of micro-cracks present in wood pieces. The effects of wood structure (sapwood and heartwood), impregnation time, and temperature on local concentration profiles in Norway spruce wood were investigated using LiCl as a tracer substance. For experimental conditions of neutral/low pH, Li⁺ ion concentrations in the eluate of slices taken from the outer surface layers of an impregnated wood piece were found to be higher than the native anionic group content (i.e., the cationic exchange capacity) of the wood piece. The method gave reasonable results, but defects in the wood piece (micro-cracks) still existed and were detectable.

INTRODUCTION

Increased global energy demands and the limited availability of fossil resources coupled with increased social concern about greenhouse gas emissions have prompted humankind to look for sustainable alternative resources. Petroleum or fossil energy resources are considered to be nonrenewable carbon resources because of their long geologic production cycles (~200 million years) as compared with biomass feedstocks (~<1 to 80 years), which are renewable and carbon-neutral resources [1]. In recent years, several types of potential biomass feedstocks have been identified, and it has been proposed that each biomass type be processed in a specific way to produce green materials, chemicals, and energy [2]. Wood lignocelluloses are the only type of biomass that is seasonally independent, and lignocelluloses are the most abundant biomass on earth, accounting for an annual production of ~170 billion metric tonnes in the biosphere. The "biorefinery" is the most popular concept in this context for converting forest

biomass to different forms of energy, materials, and chemicals. The biorefinery (with renewable biomass as feedstock) is analogous to the petroleum refinery (with nonrenewable petroleum as feedstock) whereby a single feedstock is fractionated into a multitude of commodity products depending on economic and social requirements. Today, the major uses of wood include direct use as a building material, making paper products, and generating heat through combustion. The pulping industry is the most developed chemical technology for processing wood, and to some extent it is already a biorefinery in which heat, electric power, and cellulosic fibres are produced from wood. The biorefinery concept establishes additional improvements in this area: the decreasing competitiveness of traditional pulp and paper mills has increased the opportunities for and the urgency of transforming chemical pulp mills into integrated forest biorefineries (IFBR) to produce higher value-added products



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such as ethanol, polymers, carbon fibres, and diesel fuel in addition to pulp [3]. Therefore, it is believed that the biorefinery will play a significant role in forest clusters worldwide. Enormous research efforts in the biorefinery field are underway around the world today, targeting the production of value-added products [4–14]. An illustration of this is the extraction of hemicelluloses such as xylan and glucomannans from wood chips before chemical pulping and their conversion into barrier films through acid hydrolysis, autohydrolysis, hot-water extraction, steam explosion, or alkali extraction, leading to a potentially profitable use.

In many cases, chemicals must be transported into wood before reaction. Therefore, the wood must be impregnated with the reactants. The phenomena involved can be divided into primary (penetration) and secondary (diffusion) transport mechanisms. Penetration is defined as the flow of liquor into gas- or steam-filled voids in the wood samples under a pressure gradient, and diffusion is defined as the movement of ions or molecules through a liquid with concentration gradients as the driving force. Penetration occurs very fast in the beginning if there are any gas- or steam-filled pores, but if the pores are completely filled with liquid, diffusion is the mechanism involved and is much slower. The role of penetration is to fill fibre cavities with liquid, which enables faster and more uniform diffusion of chemical ions into wood [15]. For these reasons, proper impregnation of wood with chemicals will be of great importance for an economically viable and efficient biorefinery process because homogeneous impregnation increases treatment uniformity and reduces reaction times. Therefore, the complex chemical transport phenomena of reactants into the wood matrix are of critical importance and must be better understood to ensure the success of biorefinery operations. The most relevant investigations have been conducted in the context of measuring chemical diffusion into wood, but no standard method exists. For a complete list of literature references, the reader is referred to our previous

article [16].

From the literature review, most investigations were based on either an average flux measurement of the diffusing substance or an electrical conductivity measurement of the impregnated wood samples. These methods have limitations, e.g., these methods cannot be used to determine the concentration profile at various separate locations within a single wood piece. Many aspects of chemical diffusion into wood have not yet been extensively studied. For example, most research on cation diffusion measurements into wood has been conducted with substances such as NaCl (sodium chloride), KCl (potassium chloride), and NaOH (sodium hydroxide). In cases in which NaCl/KCl is used as the diffusing substance, determinations of cation diffusion into wood may be inaccurate due to the high original concentration of Na⁺/K⁺ ions in wood. In cases in which NaOH is used as the diffusing substance, the OH⁻ (hydroxyl) ions react with wood components and mask the pure diffusional effects of Na⁺ ions. Moreover, some observations clearly indicate that almost all wood pieces have micro-cracks, which may be too small for direct visual detection, but which can change surface-layer capillarity and thus influence the interaction with the surrounding liquor and/or re-open aspirated pits and thus make the layer structure more open. These micro-cracks are due to surface-layer damage resulting from mechanical preparation of the wood pieces (e.g., sawing) [17]. These differences influence the behaviour of a wood piece in diffusion measurements.

For these reasons, in our previous work [16], an attempt was made to develop an experimental methodology to determine the concentration profiles of cations in a wood piece as a function of wood piece dimensions, impregnation time, wood structure, and temperature. To overcome some of the difficulties mentioned above, LiCl (lithium chloride) was chosen as the diffusing substance, and a direct method consisting of slicing the wood piece and measuring the Li⁺ ion concentration with flame atomic

emission spectroscopy (FAES) was used. Even though wood contains very small amounts of naturally occurring Li⁺ and Cl⁻ ions, the effect of the original presence of these ions on cation diffusion into wood can be considered as negligibly small. Su *et al.* [18] have also demonstrated that Li⁺ has low affinity towards wood components. Furthermore, in a previous study, we observed that the micro-cracks present on the surface layers of wood are best indicated through variation in the spatial resolution of penetration (concentration profiles) and are otherwise not visible. Therefore, in the present paper, previous experimental methodology [16] has been further developed to minimize the effect of micro-cracks on measurements of local cation concentration profiles within a wood piece. The effects of impregnation time and temperature on Li⁺ ion concentration profiles in sapwood (Sw) and heartwood (Hw) samples are also discussed.

MATERIALS AND METHODS

Samples

A stemwood disc 23 cm in diameter (without bark) from a 31±1-year-old Norway spruce (*Picea abies* L.) was investigated. Both Sw and Hw samples were carefully prepared using a vertical band saw and were kept preliminarily in an airtight polyethylene (PE) bag at 1°C. It was assumed that a stemwood disc contains 50% Sw and 30% Hw and that the remaining 20% is an intermediate between Sw and Hw [19]. Only Sw and Hw were investigated and not the intermediate material. Only rot- and other deformation-free samples were selected and cut into rectangular prototype pieces using the same vertical band saw mentioned above. The dimensions of the prepared wood pieces were 100 × 25 × 8 mm³ (L × W × T). The material was then stored in an airtight PE bag in a freezer at -18°C. Defrosting the wood pieces took 24 h. To minimize surface damage to the wood pieces caused by rough sawing, the wood pieces were carefully planed on all four vertical surfaces to peel off surface layers approximately 0.5 to 1.5 mm in thickness using a hand plane

(Stanley Hand Tools, Stanley Canada, Mississauga, ON L5N 7K6). Further steps in the experimental procedure, equipment details such as cutting machine specifications, and information about the chemicals used can be found in our earlier paper [16].

Water Impregnation Cycles

The wood pieces were water-impregnated in a vacuum-pressure cycle in a small polypropylene (PP) beaker placed in an autoclave filled with deionized water (at ~22°C, i.e., at r.t.). Then a vacuum was applied for 30 min, and the autoclave was pressurized with N₂ at 0.5 MPa for 1 h. This procedure was repeated until no floating pieces were observed after complete water impregnation (maximum 3–5 cycles for Sw and 5–15 cycles for Hw). To eliminate the small bubbles left on the surfaces (which constitute possible barriers against chemical diffusion), the pieces were kept in the final vacuum for 30 min before the subsequent experimental steps.

Impregnation with LiCl

The pieces were then dabbed with filter paper and immersed in a solution of 1 M LiCl at a wood-to-liquor ratio of 1:125 (Fig. 1). The impregnation vessel was made of PP and placed in an unstirred water bath (Sub Aqua 26 Plus, Grant Scientific, Grant Instruments (Cambridge) Ltd., SG8 6GB, England). The impregnation vessel was equipped with a liquor mixer fitted with a three-blade impeller to avoid concentration gradients in the impregnation vessel and with a digital thermometer

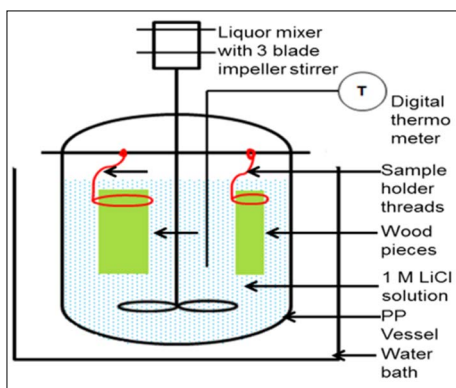


Fig. 1 - Experimental setup for impregnation with LiCl solution at different temperatures.

(Traceable® Digital Thermometer, Control Company, Friendswood, Texas 77546 USA) to monitor and control temperature in the impregnation vessel. After impregnation at time intervals of 4 and 12 h, at each of the specified temperatures of 40°C and 60°C, the pieces were removed and placed in liquid N₂ (-180°C) to stop (minimize) further migration of Li⁺ ions. The frozen pieces were lyophilized for approximately two weeks. The drying procedure was controlled by means of parallel samples, the weights of which were measured.

Flame Atomic Emission Spectroscopy (FAES)

Each impregnated wood piece was cut into small cubes (Fig. 2) using the vertical band saw. The target cube dimensions were approximately 6 x 6 x 6 mm³ in all the impregnation experiments. Each cube was microtomed in the tangential direction to slices of ~0.3 mm thickness, which were

oven-dried at 105°C for 1 h. The dried slices were kept in desiccators containing blue gel salt and at r.t. The slices were acid-leached at r.t. with 2% HNO₃ for approximately 24 h. A set of leaching experiments was also conducted for 72 h, and there was no difference in the release of Li⁺ ion between the 24-h and 72-h leaching experiments. At the end of the leaching period, the leaching liquor was collected with a syringe connected to a 0.45 μm PVDF (polyvinylidene fluoride) membrane filter. The leaching liquor was analyzed for Li⁺ ion concentration using FAES. An air-acetylene torch was the flame source, and the emission was measured at 670.8 nm. The Li⁺ ion concentration was adjusted to an optimal working concentration range of 0.02 to 5 μg ml⁻¹.

RESULTS AND DISCUSSION

The uniformity of the bulk LiCl concentration in the impregnation vessel was

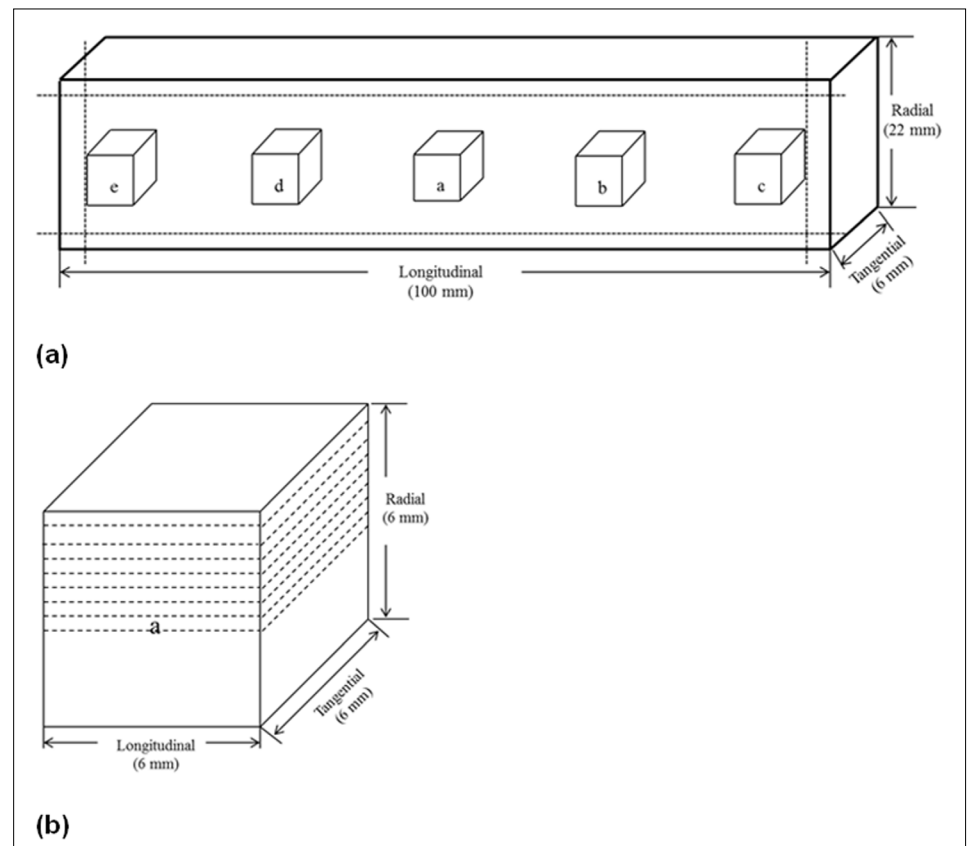


Fig. 2 - (a) Sampling of small cube samples (6×6×6 mm³) from an idealized impregnated wood piece (100×22×6 mm³); (b) microtoming of small cube into slices of ~0.3 mm thickness for Li⁺ ion concentration profile measurements.

measured at four time intervals during all the chemical impregnation experiments. The results indicated that, in all experiments with 4 and 12 h treatment times and for experiments both at 40°C and 60°C, the wood pieces were exposed to a bulk concentration of 7 ± 1 (g Li⁺ L⁻¹) [1 M LiCl = 6.98 g Li⁺ L⁻¹]. In a similar way, the temperature inside the impregnation vessel was also continuously monitored and recorded at four time intervals during the chemical impregnation experiments. Observations indicated that the temperature inside the impregnation vessel was maintained at $40 \pm 1^\circ\text{C}$ and $60 \pm 1^\circ\text{C}$ for the experiments at 40°C and 60°C respectively.

Because the concentration of chemical agent at the centre of the wood piece is a reasonable measure of the completeness of the wood treatment, most of the data reported in the present article are taken from cube a, which is from the center portion of the piece (Fig. 2). To understand the precise effects of various factors on local concentration profiles, in all the concentration profiles presented in this study, the concentration mol/Kg (i.e., moles of Li⁺ (kg dried wood)⁻¹) is plotted against the thickness of the impregnated wood piece. Indeed, this experimentally measured concentration is the total concentration of Li⁺ ion on wood and in the solution in the pores. The following abbreviations are used in the following text: NWP (normal wood pieces) means wood pieces that were carefully prepared from a stemwood disc, using a vertical band saw; SsWP (surface-smoothed wood pieces) means wood pieces that were carefully planed on all four vertical surfaces to peel off the

damaged surface layer of NWP to a depth of approximately 0.5 to 1.5 mm using a hand plane. In all the following sections, except for that on the effect of micro-cracks, SsWP are used to minimize the effect of micro-cracks.

Error analysis of the experimental methodology

To verify the reproducibility of the experimental methodology used, concentration profiles were measured for three Sw pieces with similar dimensional specifications ($\sim 100 \times 22 \times 6$ mm³) after 12 h of chemical impregnation experiments at 40°C. Figure 3 shows a good example of one advantage of local measurements because it is obviously easy to detect various defects, such as cracks in the wood sample. Clearly, two of the three experimental series show good similarities in concentration profiles, with an experimental error of approximately 11%. The third series (wood piece 1), however, has several deviant data points, which were attributed to cracks in the wood structure. If all data points (wood pieces 1, 2, and 3) are considered when evaluating the experimental error, it becomes quite large, approximately 21%. However, if some data points that may be considered erroneous due to cracks in all three wood pieces are excluded from the error calculations, the error decreases to approximately 8%.

Effect of micro-cracks

This section presents several illustrations of the influence of damaged surface layers on Li⁺ ion concentration profiles in Norway spruce wood samples. In Fig. 4,

a higher Li⁺ ion concentration in the outermost surface layers can be seen in NWP than in SsWP. This clearly indicates that the rough surface layers, which were formed during rough sawing, may have altered the influence of mass transfer on Li⁺ ion diffusion in NWP more than in SsWP. Figure 4 shows that the outermost surface layer, which is 1–2 mm thick, may have different properties from the bulk of the wood piece. Therefore, it seems that the formation and behaviour of the damaged surface layers depends on the method of preparing the wood pieces. Moreover, the effect of the damaged surface layer on Li⁺ ion diffusion in Norway spruce Sw was more pronounced than in Hw.

Effect of wood structure

Differences in Li⁺ ion concentration profile were observed between Sw and Hw samples of Norway spruce at different temperatures and for different treatment times (Fig. 5). Higher Li⁺ ion concentrations were observed in Sw than in Hw for eluates of slices taken at similar depths. This observation became more pronounced as the treatment time increased from 4 to 12 h and the temperature increased from 40°C to 60°C. These differences in Sw and Hw concentration profiles are due to differences in the structural and chemical compositions of Sw and Hw, e.g., a) Hw is usually much less permeable than Sw due to pit aspiration and incrustation [20], and b) the extractives content in Hw is higher than in Sw. However, different concentration profile shapes were observed for extended treatment times (Fig. 5 A) b)) as well as at higher temperatures

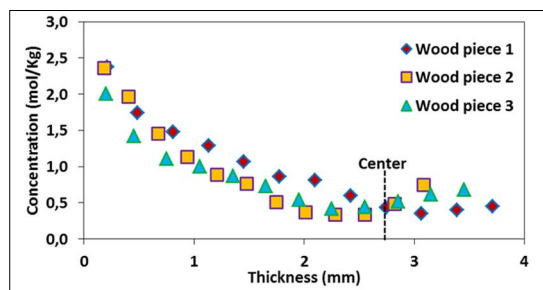


Fig. 3 - Measured concentration profiles of Li⁺ ion in Norway spruce Sw of three different wood pieces of similar specifications after 12 h treatment time at 40°C.

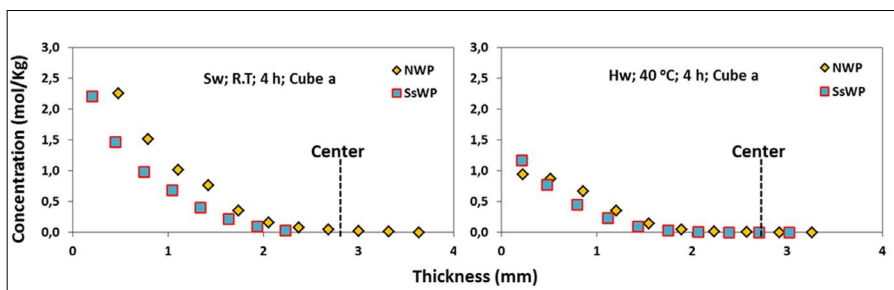


Fig. 4 - Measured Li⁺ ion concentration profiles in Norway spruce Sw and Hw samples, at room temperature and for 4 h treatment time (as indicated): NWP vs. SsWP.

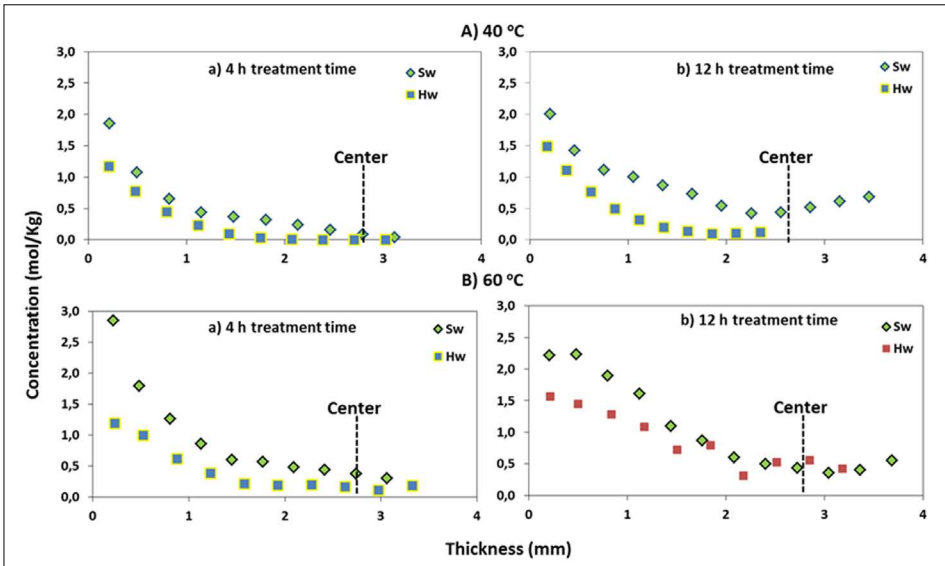


Fig. 5 - Measured Li⁺ ion concentration profiles in Norway spruce SsWP at A) 40°C and B) 60°C: Sw vs. Hw; for treatment times of a) 4 h, and b) 12 h.

(Fig. 5 B) b)). This is probably due to the remaining effect of micro-cracks on Li⁺ ion diffusion in the wood samples.

Effect of Impregnation Time

In Figs. 6 A) and 6 B), a shift in concentration profiles towards the centre of the wood piece is visible as the treatment duration increases from 4 to 12 h. This observation is more pronounced in Sw than in Hw and for increased temperature from

40°C to 60°C. In the case of Hw, at 40°C, the concentration profiles remained within experimental error as the treatment time increased from 4 to 12 h. This difference in behavior occurred because Hw is less permeable than Sw. On the other hand, at 60°C, as the impregnation time increased from 4 to 12 h, higher Li⁺ ion concentrations were observed. In addition, different concentration profile shapes were observed for both Sw and Hw as treatment

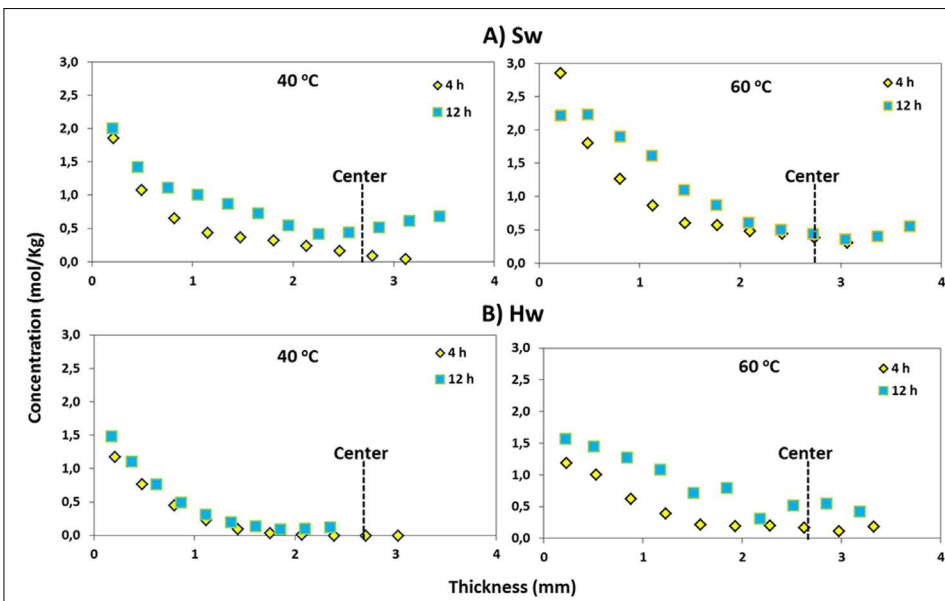


Fig. 6 - Measured concentration profiles of Li⁺ ion in Norway spruce after different treatment-time intervals: 4 h vs. 12 h, and for different temperatures (as indicated) for A) Sw and B) Hw samples.

time increased at 60°C. Moreover, the effect of micro-cracks on concentration profiles apparently still exists to some extent and contributes to the different concentration profile shapes at higher temperatures and extended treatment-time intervals.

Effect of Temperature

Minor differences in Li⁺ ion concentration profiles were found as the temperature increased from 40°C to 60°C (Fig. 7). As expected, the Li⁺ ion concentration in a given wood piece increased with temperature. This observation was more pronounced in Sw than in Hw after a 4 h treatment period. These differences may have been due to differences in chemical composition, e.g., the extractives content in Hw is higher than in Sw. As the temperature increased, the observed penetration depth also became deeper in Sw than in Hw. However, after a 12 h treatment period, the differences in concentration profiles at 40°C and 60°C were appreciable, and different concentration profile shapes were observed.

As has been noted in this experimental study, the Li⁺ ion concentration in a wood piece increased as the treatment time increased from 4 to 12 h and as the temperature increased from 40°C to 60°C. The effects of extended treatment time and increased temperature not only occurred in a predictable manner as predicted by diffusion theory, but also different concentration profile shapes were observed as well. Therefore, it is evident that Li⁺ ion transport in wood material may be influenced by other phenomena such as surface diffusion, adsorption/desorption, and Donnan equilibrium. More importantly, for the experimental conditions studied (i.e., relatively neutral or low pH), in all concentration profiles, the observed Li⁺ ion concentrations were much higher than the concentrations of carboxylic acid groups (0.086 ± 0.007 mol/Kg) (Werkelin *et al.*, 2010; Shoulaifar *et al.*, 2012) present in the native Norway spruce wood samples. In wood samples, at neutral or weakly acidic conditions, mainly carboxylic sites are predominantly ionized, and therefore

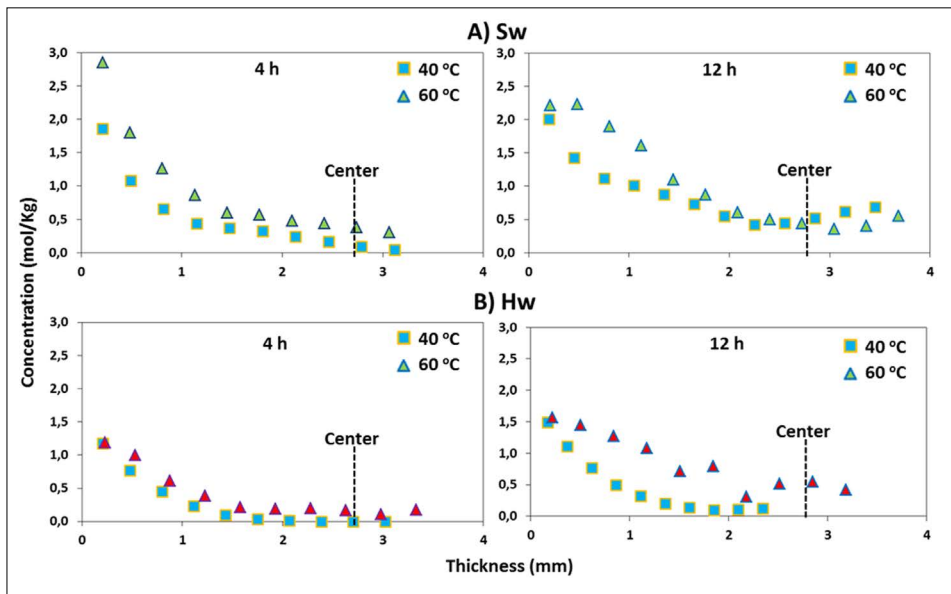


Fig. 7 - Measured Li^+ ion concentration profiles in Norway spruce at different temperatures: 40°C vs. 60°C, and after treatment intervals of 4 h and 12 h (as indicated) for both A) Sw and B) Hw samples.

they exist in anionic forms, and the Li^+ cations can bind to the carboxylic sites.

Concentration Profiles at Various Locations

Figure 8 shows concentration profiles for different locations in the wood piece (a, b, c, d, and e in Fig. 2). It is evident that the concentration profiles for cube a and cube c (cube c is symmetric to cube e) are

different. This phenomenon occurs because in the case of cube a, Li^+ ion diffusion was influenced only by mass transport in the transverse direction. However, in the case of cube c (cube e), Li^+ ion diffusion was influenced by mass transport in both longitudinal and transverse directions. This effect is evident in both Sw and Hw samples even at extended treatment-time intervals and temperatures, but with

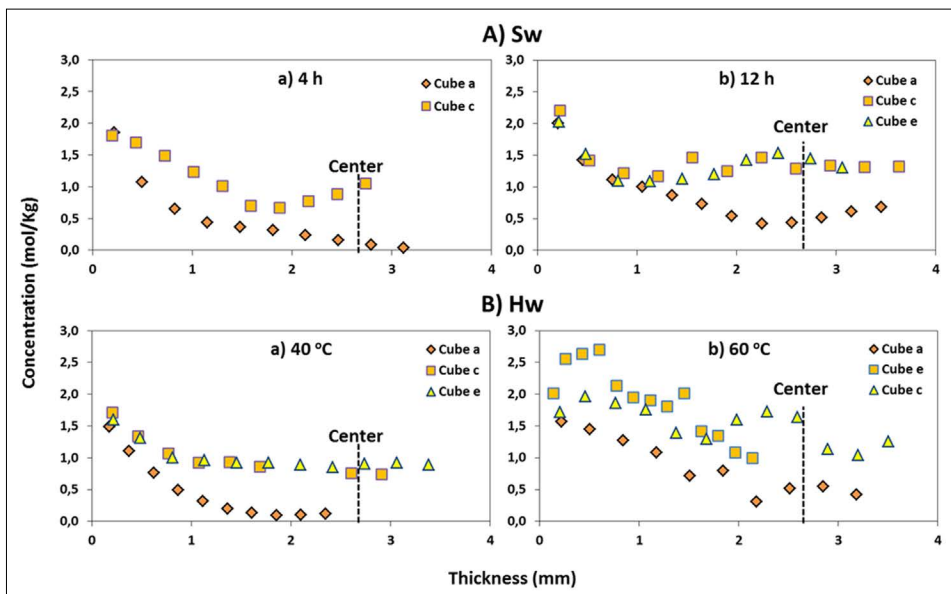


Fig. 8 - Measured concentration profiles of Li^+ ion in Norway spruce wood pieces at various locations within a single wood piece of A) Sw sample, after intervals of a) 4 h and b) 12 h treatment at 40°C, and B) Hw sample, for a) 40°C and b) 60°C after 12 h treatment time. Cubes a, b, c, and e refer to sampling within the wood piece (as shown in Fig. 2).

different concentration profile shapes (Figs. 8 A) & B) b)).

CONCLUSIONS

Our previous experimental methodology was further developed to minimize the effect of micro-cracks on Li^+ ion concentration profile measurements in Norway spruce wood pieces. The proposed methodology was also verified with three replicates, thus confirming that the method provides a good prediction of concentration profiles and showing that the effect of micro-cracks varies among wood pieces. Effects of treatment-time duration and temperature on Li^+ ion concentration profiles in both Sw and Hw pieces were investigated. For the experimental conditions chosen, concentrations of Li^+ ion much higher than the cationic exchange capacity of Norway spruce wood were observed in the slices taken from the outer surface of the wood pieces. The improved method gave reasonable results, but defects in the wood pieces (micro-cracks) remained and were detectable.

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