# Water-uptake studies of dry, mountain pine beetle-infested, grey-stage lodgepole pine sapwood chips

THOMAS Q. HU, MICHELLE ZHAO, JAMES DRUMMOND, AND PAUL WATSON

**ABSTRACT:** Water-uptake studies of dry, mountain pine beetle-infested, grey-stage lodgepole pine (LPP) sapwood chips showed that it was possible to increase the moisture content of these chips significantly (e.g., from 20.5% to 37.0% for early-grey chips) by soaking the chips for 8 min–10 min at optimal conductivity (~200 µS/cm) and pH (~6.0). The environmental scanning electron microscopy (ESEM)-energy-dispersive spectroscopy chemical-tracer technique provided qualitative information on water movement within 10 min of water soaking of both the green and the beetle-infested, late-grey LPP blocks. ESEM studies also showed the presence of fissures, fungal hyphae, and incipient decay in the late-grey blocks.

***Application:*** TMP mills that use dry, mountain pine beetle-infested, grey-stage lodgepole pine chips can significantly increase the moisture content of the chips before TMP pulping by extending chip washing and soaking time to 8 min–10 min and by controlling the conductivity and pH of the chip wash water.

The outbreak of the mountain pine beetle epidemic in British Columbia has led to the infestation of 80% of the lodgepole pine (LPP) forest in the province [1]. There are three stages of beetle attack: 1) green-stage attack, in which adult beetles have found a new host tree and tunneled underneath the bark to lay their eggs. The tree dies soon after, but the needles stay green for several months; 2) red-stage attack, in which the needles have turned red as a result of the beetles' killing the tree by mining the layer between the bark and the wood, thus cutting off the tree's supply of nutrients; and 3) grey-stage attack, in which the needles have fallen off the tree and only the bare branches remain [2]. Standing trees that have been infested by the beetle and have been dead long enough (two years or more) to lose their foliage and acquire a grey appearance are classified as grey-stage wood. Besides the presence of blue-stain fungus that is associated with both red-stage and grey-stage beetle infestations, the beetle-infested, grey-stage LPP sapwood chips have moisture contents (MCs) lower than their fiber saturation point [3]. The fiber saturation point, typically ~30% (dry basis) (~23% on a wet basis), is defined as the wood MC at which the cell walls are filled with bound water, with no free water in the lumens [4, 5]. When wood MC falls below its fiber saturation point, most of the physical and mechanical properties of the wood begin to deteriorate [6, 7]. For beetle-infested, grey-stage LPP, the material that will comprise the longer-term wood chip supply in British Columbia, stem MCs as low as 9% (wet basis) have been measured [8]. Such low stem MCs make the material of questionable utility for solid lumber applications because the loss of moisture from the cell wall is accompanied by shrinkage and by defects such as internal checking. Use of this wood therefore depends heavily on the pulp and paper industry.

According to the limited information available, strength properties were reduced when spruce chips with ≤ 15% MC were refined in pilot plants [9, 10]. There have also been reports suggesting that chip pre-steaming and water impregnation using compression screw feeders could improve remoistening of chips with low MC [11, 12, 13, 14] and thus improve pulp properties [11, 12, 13]. A TMP mill in British Columbia that uses dry, beetle-infested LPP chips is considering the costly option of raising water temperatures for chip pre-steaming, chip washing, or both to remoisten the chips. How water penetrates into dry, grey-stage LPP chips during water soaking or washing is of significant interest to this mill and to others that are or will be using such chips.

Water absorption by Sitka spruce heartwood blocks has been studied by measuring the weights of the oven-dried blocks, soaking the blocks in an aqueous medium at various temperatures for different times, removing the free water on the surfaces of the blocks rapidly with towels, and finally measuring the wet weights of the blocks [15]. This procedure is time-consuming when the kinetics of, or various factors affecting, the water uptake need to be determined. Recently, a device that measures the water-absorption rates of oven-dried chips within 35 seconds of immersing the chips in water has been reported [16, 17]. This device may enable study of factors affecting water uptake of dry, grey-stage LPP chips. A possible way to study water movement into dry, grey-stage LPP chips is the use of ESEM-energy-dispersive spectroscopy (EDS), in which the movement of a heavy element dissolved in the penetration liquid may be traced using EDS and visualized using ESEM [18].

This paper describes the results of our studies of the factors affecting water uptake by dry, grey-stage LPP chips using the Weyerhaeuser device, as well as of our preliminary ESEM-EDS studies of the movement of water into grey-stage LPP blocks using silver nitrate, $AgNO_3$, as the tracer compound.

## MATERIALS AND METHODS

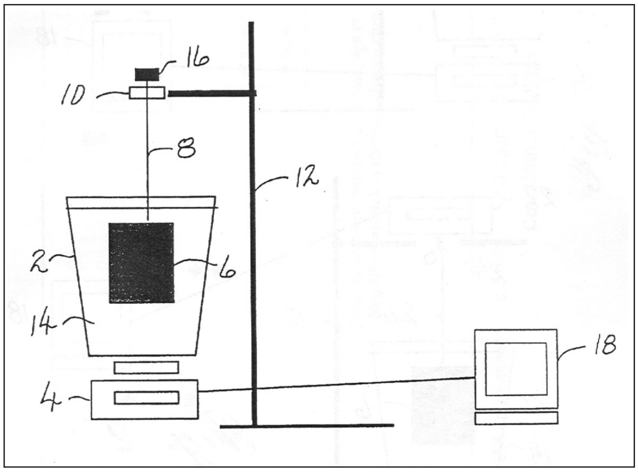
### Chip preparation

Green (uninfested) LPP and beetle-infested, early-grey and late-grey LPP trees were harvested from sites situated in an area with moist climate and with similar stand qualities (site index = 18) [19] in the interior of British Columbia. Selected bolts were debarked and then segregated by a portable sawmill to produce the mature (over 40 years) sapwood sections. The times since death of the early-grey and the late-grey woods were estimated to be ~2 and 6–7 years respectively by dendrochronology [19]. The mature sapwood sections of the samples were chipped individually using a CM&E 10-knife disc chipper. No significant difference in the chip-size distributions were found for the green, early-grey, and late-grey chips, although both the early-grey and the late-grey samples had slightly higher fines content and lower accepts chip content than the green sample [3]. Each chip sample was screened on a Burnaby Machinery and Mill Equipment Ltd. two-deck laboratory chip classifier to remove oversize (>31 mm) and fine (<8 mm) material. All the moisture content (MC) values are wet-basis {MC = [(wet weight of the chips – oven-dry weight of the chips) / wet weight of the chips] × 100%}. The average MCs and standard deviation ($\sigma$) (from three replications) of the screened green, early-grey, and late-grey chips were 53.8% ($\sigma$ = 1.0%), 21.4% ($\sigma$ = 0.7%), and 17.0% ($\sigma$ = 0.2%), respectively. A portion of the screened green chips was placed evenly on a number of plastic mats and air-dried in a dark room at 24 °C and 33%–41% relative humidity for ~7 hours to 16.9% MC ($\sigma$ = 1.1% from three replications).

### Water-uptake studies using the Weyerhaeuser device

All the equations and abbreviations used in the water-uptake studies are provided in Appendix A. A sample of the chips (~100 g wet weight) was pre-steamed in a laboratory bench-top autoclave (Brinkmann 2540M) at various temperatures for a total time of 30 min and 55 min respectively (during which the chips were inside the autoclave). Typically it took 11 min–15 min and 16 min–17 min for the autoclave to reach ~100 °C (or 123 °C) and 130 °C, respectively, which corresponded to pre-steaming pressures of ~101 kPa (or 220 kPa) and 280 kPa. The wet weight of the pre-steamed chips was recorded.

The pre-steamed or control chips (without steaming) were placed inside a covered wire-mesh basket, lowered, and immersed in an aqueous solution (or DI $H_2O$) at room temperature (~23 °C) for 30 min in the Weyerhaeuser device (**Fig. 1**). After the basket was lowered, the weight of "basket + chips" was measured immediately, and then continuously every second for 30 min on the analytical balance. The weight so measured decreased continuously because of water uptake by the chips. Based on the working principle of the device, the amount of water uptake by the chips at any given soaking time is equal to the weight of the "basket + chips" measured immediately after the lowering of the basket minus that measured at the given soaking time (Eq. 1).



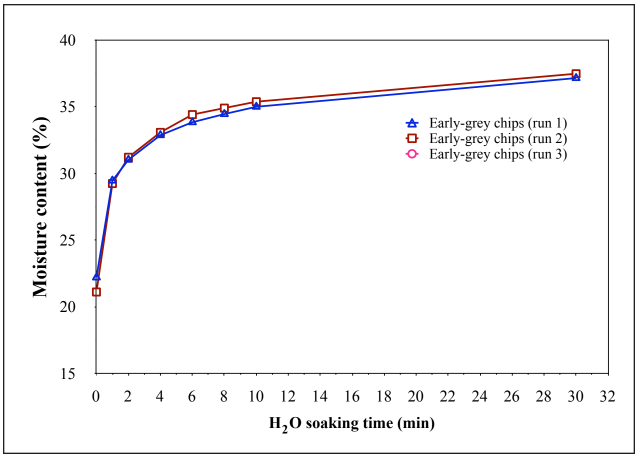

***1. Schematic of Weyerhaeuser device (2 = water container; 4 = balance; 6 = covered wire-mesh basket; 8 = rod; 10 = clamp; 12 = stand; 14 = water; 16 = stop; 18 = computer) [16].***

The soaked chips were then dried in a 108 °C oven for ≥17 h, and the oven-dried weight of the chips was measured. The MCs of the initial or control chips, the pre-steamed chips, and the chips at a soaking time of t minutes at room temperature were calculated according to Eqs. 2–4.

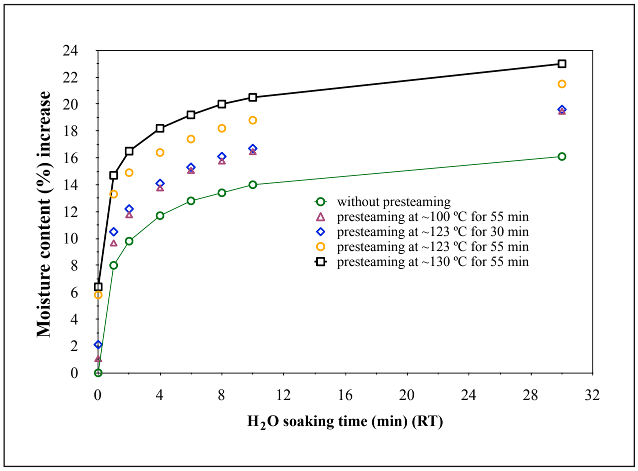
For soaking at 50 °C–80 °C, experiments were first done with ~100 g of plastic balls, and the amounts of vapor loss at a given temperature over 30 min were recorded at one-second intervals. The MCs of the chips at a given temperature and a soaking time of t minutes were calculated according to Eq. 5. For conciseness, only the MC data (all of which are average values from two or three runs) from soaking times of 0 min, 1 min, 2 min, 4 min, 6 min, 8 min, 10 min, and 30 min are presented.

### Water penetration studies using ESEM-EDS with $AgNO_3$ as the tracer compound

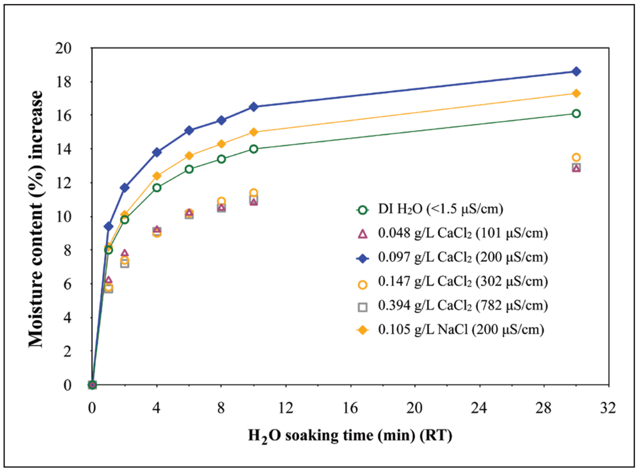
Wood blocks having 1.0 cm (radial) × 1.0 cm (tangential) × 2.0 cm (longitudinal) dimensions were prepared by hand from mature sapwood sections of late-grey and green LPP disks using a chisel and razor blades. Six late-grey and three green blocks were used to determine the average MCs of the blocks and their standard deviations ($\sigma$). The MC values




*2. MC of early-grey chips as determined by the Weyerhaeuser device versus chip soaking time in DI $H_2O$ at RT.*




*3. Increase in MC of early-grey chips after pre-steaming and during subsequent DI $H_2O$ soaking at RT.*




*4. Effect of the conductivity of, and the specific cations in, the salt added to the soaking water on water uptake by early-grey chips at RT; data for the 0.048 g/L, 0.147 g/L, and 0.394 g/L $CaCl_2$ experiments were from a single run.*

were 8.5% (σ = 0.6%) and 45.3% (σ = 3.7%) for the late-grey and green blocks, respectively. Two or three late-grey or green blocks (their oven-dry (o.d.) weights calculated based on their wet weights and the MC value of 8.5% or 45.3%) were used for each pre-steaming (at 123 °C for 55 min) and/or soaking in 10% or 0.03% $AgNO_3$ solution at RT for 5 min or 10 min. After soaking, the liquid on the surface of the blocks was removed with a blotter and the wet weights of the blocks recorded. One or two blocks were oven-dried after soaking to determine the MCs. The amount of $AgNO_3$absorbed by a block was estimated by subtracting the calculated o.d. weight of the block before soaking from the measured o.d. weight of the block after soaking. All the values are averages from two experiments.

One block from each soaking experiment was immersed in liquid nitrogen for 10 s, placed in a glass vial, stored in a freezer, and freeze-dried in a VirTis AdVantage Freeze Dryer before being placed inside a sealed vial and stored in a dessicator. Each of the dessicator-stored blocks was split along the tangential plane in the center to expose the interior, tangential, midline face. The split sample was placed on an SEM stub, and the sample was placed in a FEI Quanta 400F field-emission ESEM equipped with an EDAX Genesis EDS system incorporating a 10 mm² thin-window SiLi detector. Samples (without application of coating) were examined in low-vacuum (LV) mode at 1.0 torr chamber pressure. The intensity of Ag at various locations along the longitudinal and the tangential midlines of the split sample was measured by EDS. X-ray spectra were collected from a ~0.14-mm by 0.12-mm area at each location. Spectrum collection time was 100 s at a 20-kV acceleration voltage. Working distance was ~10 mm for all samples.

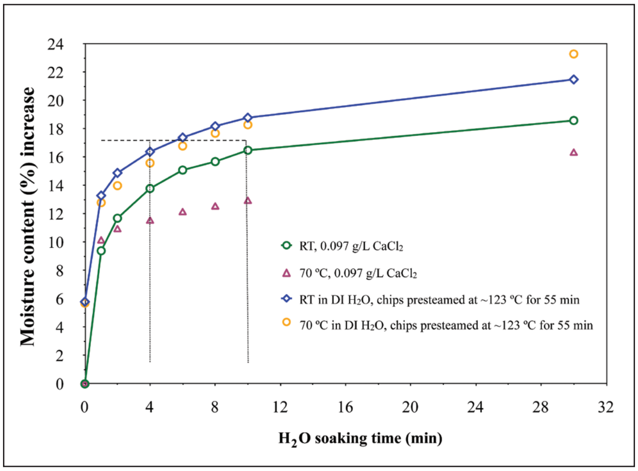
## RESULTS AND DISCUSSION

### *Water-uptake studies using the Weyerhaeuser device*

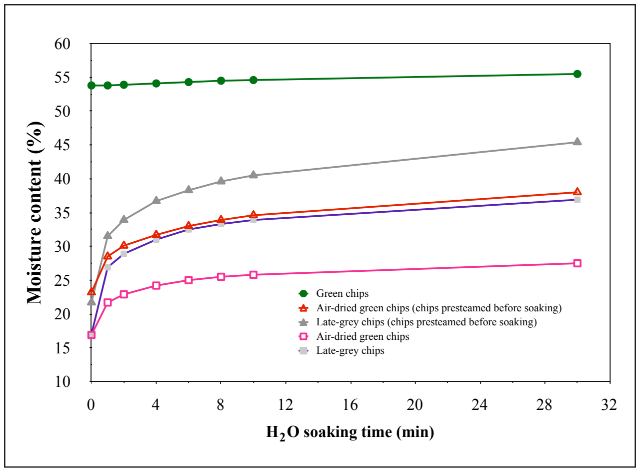
The Weyerhaeuser device (Fig. 1) has been used for estimating kraft pulping yields based on water absorption rates of oven-dried chips within 35 s of immersing the chips in water [16, 17]. The device uses a variant of Archimedes' principle. Its main advantage is its ability to monitor water uptake by the o.d. chips continuously and precisely over 35 s. An enclosed pressure device based on the same principle has been used to study and model the process of water penetration into softwood chips [20, 21].

We first tested the capability of the device and the reproducibility of the results for longer time periods and wet chips by measuring DI $H_2O$ uptake of the early-grey, LPP chips with MCs of 21.0%-22.2% over 30 min. The MCs of the chips increased rapidly over the first 5 min from ~21.6% to 34.0%, gradually over the next 5 min to 35.4%, and then slowly for the next 20 min to 37.5% (**Fig. 2**). Excellent reproducibility was found from one run to another (total three runs).

Next, we evaluated the effect of pre-steaming of the chips (MC = 19.5%–21.4%) on their subsequent water uptake, as well as the effects of the composition, conductivity, temperature and pH of the chip-soaking solution. Both the




***5. Combined effect of temperature and optimal conductivity of the soaking water or chip pre-steaming on water uptake by early-grey chips.***




***6. Change in the MC of green, air-dried green, and late-grey chips during RT DI $H_2O$ soaking; pre-steaming of the chips done at ~123°C for 55 min.***

pre-steaming temperature and time had a positive effect on water uptake during pre-steaming and subsequent DI $H_2O$ soaking (**Fig. 3**). The effect of pre-steaming and subsequent soaking was either additive (pre-steaming at 123 °C or 130 °C) or slightly synergistic (pre-steaming at 100 °C).

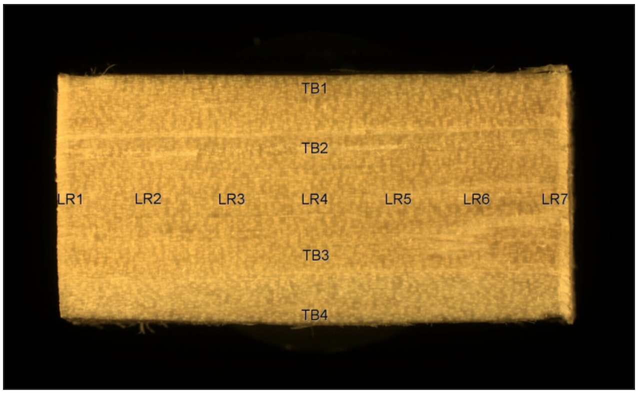
The conductivity of, and the specific cations in, the salts dissolved in the soaking water affected water uptake significantly. A conductivity of 200 µS/cm was optimal for water uptake, and the divalent cation, $Ca^{2+}$, was more beneficial than the monovalent cation, $Na^+$ (**Fig. 4**). In separate experiments, it was found that the optimal pH of the soaking water was ~6.0; changing the pH to 8.0 or 4.5 resulted in a small or a significant decrease respectively in the water uptake. Increasing the soaking-water temperature from RT to, for example, 70 °C appeared to have no or even a negative effect on the water uptake, particularly during the first 12 min (**Fig. 5**). Closer inspection of the data shown in Fig. 5 revealed that soaking of the chips in a medium with an optimal conductivity (0.097 g/L $CaCl_2$ solution, conductivity = 200 µS/cm) for 10 min increased their MC to the same level as pre-steaming of the chips at ~123 °C for 55 min and then soaking in a medium of nonoptimal conductivity (DI water) for 4 min. These results suggest that a more economical approach to raising the MC of dry, grey-stage LPP chips before pulping in TMP mills is to control the conductivity of the chip soaking water (wash water in the mills) and to use a chip washing time of 8 min–10 min instead of increasing the chip pre-steaming temperature or the chip wash water temperature. Most TMP mills have a chip-washer retention time of 4 min–6 min.

When the green chips were soaked in DI $H_2O$ at RT for 30 min, their MC increased only slightly, from 53.8% to 55.5% (**Fig. 6**). Interestingly, the air-dried green chips (MC = 16.9%) showed a much lower water-uptake rate than the late-grey chips with the same starting MC. The MC of the pre-steamed, air-dried green chips could reach only 38.0% after 30 min, compared with 45.4% for the pre-steamed late-grey chips. Possible reasons for the higher water-uptake rate for the late-grey chips will be discussed later.

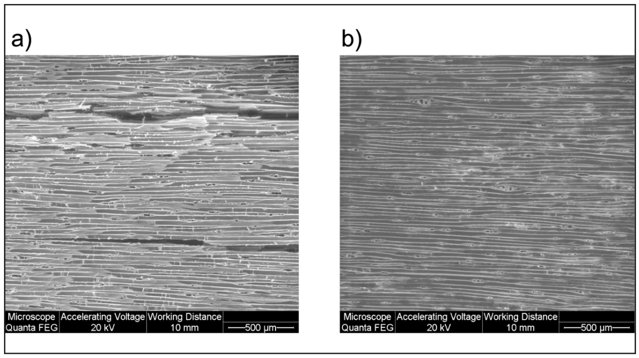
The MCs of the early-grey chips after 30-min RT soaking in DI $H_2O$, in 0.097 g/L $CaCl_2$ solution, and in DI $H_2O$ with chip pre-steaming at 123 °C for 55 min were 37.5%, 39.1%, and 42.5%, respectively (Figs. 3 and 4), when the Weyerhaeuser device was used. These values appeared to be somewhat lower than expected. Another experiment was then done on the early-grey chips using the device. The MC of the chips after 30-min RT DI $H_2O$ soaking was "measured manually" by removing the water on the surface of the chips with paper towels and recording the wet weight of the chips before subjecting the chips to an additional soaking experiment and measuring their o.d. weight. The "manually measured" MC of the chips was 45.2%, compared to the 37.8% value obtained using the Weyerhaeuser device. The additional soaking experiment showed that the difference between the "manually measured" MC and the MC value obtained using the Weyerhaeuser device was partly due to the swelling of the chips during soaking. When the swelling of the chips was taken into account, the MC of the chips after 30 min soaking could be calculated as 39.7% (instead of 37.8%). It is possible that "manual measurement" gave higher MC values due to the incomplete removal of water on the surface of the chips. Further work is needed to determine whether this is the case and whether other factors contribute to the difference. Nonetheless, the general conclusions drawn from the water-uptake data (Figs. 3–6) appeared to be valid because the differences in the MC values from different soaking experiments could still be the same even if swelling were accounted for.

### *Exploratory water-penetration studies using ESEM-EDS*

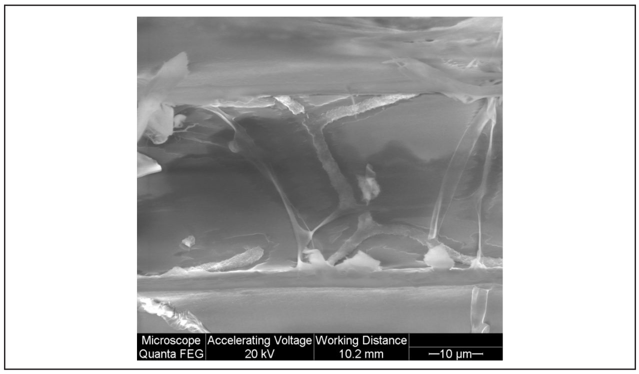
To explore the possibility of using ESEM-EDS for the monitoring of water movement into dry, grey-stage LPP, late-grey wood blocks were pre-steamed, soaked in $AgNO_3$ solution, or both. The weight changes of the blocks during




*7. Locations on the exposed interior face of the $AgNO_3$-soaked block for Ag analyses by ESEM-EDS.*




*8. ESEM micrograph of late-grey a) and green b) blocks after soaking in 10% $AgNO_3$ for 5 min.*




*9. ESEM image of late-grey block after soaking in 10% $AgNO_3$ for 5 min.*

*10. Late-grey block after pre-steaming and soaking in 10% $AgNO_3$ for 10 min.*

soaking in $AgNO_3$ solution (without pre-steaming), qualitatively support most of the observations on the water uptake behavior of the green and early- (or late)-grey chips using the Weyerhaeuser device. For example, practically all the water uptake by the late-grey blocks occurred over the first 5 min of soaking; the MC of the late-grey blocks increased from 8.5% to 60.0% and 59.8% respectively at 5 min and 10 min of soaking in 10% $AgNO_3$. This is similar to the results for the effect of soaking time obtained using the Weyerhaeuser device (Fig. 6). Soaking of the late-grey blocks in 0.03% $AgNO_3$ solution (conductivity = ~200 µS/cm) gave slightly more weight gain and a higher MC (MC = 61.2%) than soaking in 10% $AgNO_3$ (with a much higher conductivity); these results are similar to those on the effect of conductivity obtained using the device (Fig. 4). Only a small amount of the liquid was taken up by the green blocks during soaking in 10% $AgNO_3$ solution, with their MCs increasing from 45.3% to 48.8% and 50.6% respectively at 5 min and 10 min of soaking. This is similar to the results obtained for green chips using the device (Fig. 6).

However, pre-steaming of the blocks appeared to inhibit their subsequent liquid uptake in 10% $AgNO_3$ solution, which is different from the results obtained using the Weyerhaeuser device (Fig. 6). The reason for this difference is not clear at this time. The amounts of $AgNO_3$ absorbed by the late-grey blocks after soaking in 10% $AgNO_3$ solution were all somewhat less than what was expected from the amounts of liquid that would have been taken up by the blocks if the blocks had been equally penetrable to $H_2O$ and to $AgNO_3$. This could indicate a lower penetrability of the blocks to the $AgNO_3$ molecules than to the $H_2O$ molecules.

**Figure 7** shows the points (LR1–LR7 in the longitudinal direction, TB1–TB4 in the tangential direction) on the exposed interior face at which the Ag intensities were determined. All the blocks, after 10 min soaking in 10% $AgNO_3$ solution, showed decreasing Ag intensity towards the block center (LR4), except for the LR6 point of the late-grey block and the LR3, TB2, and TB3 points of the green block. Overall, Ag intensities at LR2 and LR6 for these blocks were also higher than those at TB2 and TB3 for the same blocks, confirming that the movement of liquid through lumens in the longitudinal direction was faster than that through the cell wall. Consistently with our water-uptake results obtained using the Weyerhaeuser device, the green blocks soaked in 10% $AgNO_3$ solution showed much lower absorption of Ag than the late-grey blocks along the longitudinal or tangential lines.

The late-grey block soaked in 10% $AgNO_3$ solution for 5 min showed a nonuniform Ag distribution, most likely due to the fissures in the block. Indeed, an ESEM micrograph of this sample shows large fissures that extend to the center of the block (**Fig. 8a**). No such fissures were found in the green block soaked in 10% $AgNO_3$ solution for 5 min (**Fig. 8b**), and this sample showed the expected decrease of Ag intensity towards the center (LR4) of the sample. Future work on the monitoring of water movement into the grey-stage blocks or chips by ESEM-EDS analysis of Ag should consider selection of samples without fissures. This may be difficult to achieve with dry wood samples. The presence of fissures in the beetle-infested, late-grey wood is likely responsible for the higher water-uptake rate for the late-grey chips than for the air-dried, green chips, as observed in the water-uptake studies using the Weyerhaeuser device (Fig. 6).

A close-up ESEM image of one of the late-grey samples showed the presence of fungal hyphae in the lumen of a fiber (**Fig. 9**). There was also some indication of incipient decay in the late-grey blocks (**Fig. 10**). The lighter areas seen in the block may be due to decay. This image can be directly compared with Fig. 7, which shows a green sample. As with fissures, decay in samples is difficult to avoid, particularly with samples that have a long time since death.

## CONCLUSIONS

Water-uptake studies using the Weyerhaeuser test device indicated that a significant increase in the moisture content of dry early- and late-grey LPP chips could be achieved by soaking the chips for 8 min–10 min in an aqueous medium at optimal conductivity (~200 µS/cm) and pH (~6.0). The ESEM-EDS chemical tracer technique provided qualitative information on the movement of water within 10 min of water soaking of green and late-grey LPP blocks. ESEM studies also showed the presence of fissures, fungal hyphae, and incipient decay in the late-grey LPP blocks. **TJ**

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## ABOUT THE AUTHORS

Approximately 14 million hectares of lodgepole pine (LPP) trees in British Columbia have been infested and killed by the mountain pine beetle. For beetle-infested, grey-stage (time since beetle infestation ≥ 2 years) LPP, the material which will comprise the longer-term wood chip supply to pulp mills in British Columbia and other regions of the Pacific Northwest, stem moisture contents as low as 9% have been measured. Determination of the factors controlling the water uptake (rewetting) of dry, grey-stage LPP chips is of critical importance to the use of such chips for TMP production.

Using the Weyerhaeuser chip-fissure test device to study the water uptake of dry, beetle-infested, grey-stage LPP sapwood chips, we have gained insights into the factors affecting the water uptake of such chips. Our results show that the moisture content of the chips can be significantly increased (e.g., from 20.5% to 37.0% for early-grey chips) by soaking the chips for 8 min to 10 min in water that has an optimal conductivity of ~200 µS/cm (preferably in the presence of calcium ions) and a pH of ~6.0. This method of increasing the moisture content may represent a more economical approach to raising the moisture content of dry, beetle-infested, grey-stage LPP chips before TMP pulping than increasing the chip pre-steaming temperature or the chip wash water temperature.

Environmental scanning electron microscopy studies of the dry, beetle-infested, late-grey LPP blocks also reveal the presence of fissures, fungal hyphae, and incipient decay in the late-grey samples.

The next step is to develop practical solutions to overcome the low brightness of TMP pulps made from dry, beetle-infested, grey-stage LPP chips.

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## APPENDIX A

Equations and abbreviations used in the studies of water uptake using the Weyerhaeuser device:

$$W_{H_2O\text{-}uptake,\,t} = W_{\text{“basket + chips”},\,0} - W_{\text{“basket + chips”},\,t} \tag{1}$$

$$MC_{initial} = \frac{W_{chips} - W_{o.d.chips}}{W_{chips}} \times 100\% \tag{2}$$

$$MC_{steamed} = \frac{W_{steamed\ chips} - W_{o.d.chips}}{W_{steamed\ chips}} \times 100\% \tag{3}$$

$$MC_{soaking,t} = \left(1 - \frac{W_{o.d.chips}}{W_{H2O-uptake,t} + W_{steamed\ chips(or\,W_{control\ chips})}}\right) \times 100\%$$

$$= \left(1 - \frac{W_{o.d.chips}}{W_{\text{"basket + chips"},0} - W_{\text{"basket + chips"},t} + W_{steamed\ chips(or\,W_{control\ chips})}}\right) \times 100\% \tag{4}$$

$$MC_{soaking,t,T} = \left(1 - \frac{W_{o.d.chips}}{W_{H2O-uptake,t} - W_{vaporloss} + W_{steamed\ chips(or\,W_{control\ chips})}}\right) \times 100\%$$

$$= \left(1 - \frac{W_{o.d.chips}}{W_{\text{"basket + chips"},0} - W_{\text{"basket + chips"},t} - W_{vaporloss} + W_{steamed\ chips(or\,W_{control\ chips})}}\right) \times 100\% \tag{5}$$

$W_{H2O\text{-}uptake,\,t}$ = amount of water uptake by the chips at a soaking time of t

$W_{\text{“basket + chips”},\,0}$ = weight of “basket + chips” immediately after lowering the basket

$W_{\text{“basket + chips”},\,t}$ = weight of “basket + chips” at a soaking time of t

$MC_{initial}$ = MC of initial or control chips

$W_{chips}$ = wet weight of the chips

$W_{o.d.\ chips}$ = oven-dry (o.d.) weight of the chips

$MC_{steamed}$ = MC of the pre-steamed chips

$W_{steamed\ chips}$ = wet weight of the pre-steamed chips

$MC_{soaking,\,t}$ = MC of the chips at a soaking time of t at room temperature

$W_{control\ chips}$ = wet weight of the chips (without pre-steaming)

$W_{vaporloss}$ = amount of vapor loss at a given temperature between 50 °C and 80 °C

$MC_{soaking,,\,t,T}$ = MC of the chips at a soaking temperature of T and soaking time of t