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Determination of the Spatial Distribution of Trace Elements in Jack Pine, *Pinus banksiana* Lamb., by Imaging Microprobe Secondary Ion Mass Spectrometry

J.H.E. BAILEY and D.W. REEVE

Imaging Microprobe Secondary Ion Mass Spectrometry (SIMS) has been used in determining the spatial distribution of trace elements in jack pine, Pinus banksiana (Lamb.). Trace elements were found to be concentrated in specific morphological features, namely: the torus, middle lamella, cell corners and ray parenchyma wall. Middle lamella calcium ion enrichment was particularly great in the radial middle lamella near the latewood/earlywood transition. There is also evidence of calcium enrichment in the inner layer of the secondary wall in the latewood cells. Analysis of a ray cell sectioned tangentially indicates enriched concentration of the above elements in the ray parenchyma wall, however not in the ray tracheid wall.

INTRODUCTION

In our previous paper, we described the use of Imaging Microprobe Secondary Ion Mass Spectrometry for ion imaging a sample of black spruce wood [1]. Calcium, manganese, iron, chromium and zinc were found in high concentration in several morphological features, namely: the torus, middle lamella, cell corner, ray cell wall and a deposit located in the ray cell. This was the first report to describe the distribution of manganese in different morphological features of a softwood fibre. Elemental profiles of the metal ion intensity across a double cell

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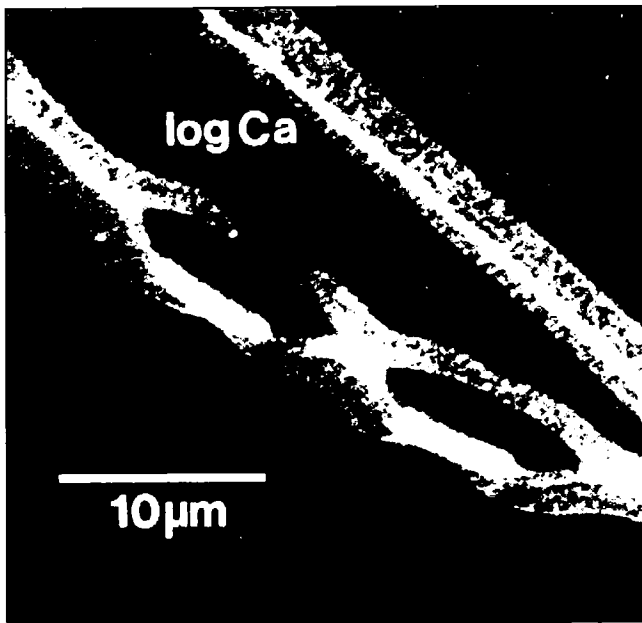200 College St.
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wall confirmed the enriched concentration of calcium and the trace elements in the middle lamella.

The total ash content of wood is 0.1 to 0.5% of the oven-dry weight of wood. Calcium, magnesium and potassium constitute 70–80% of the ash while a variety of elements are responsible for the remaining 20–30%. There are numerous reports on the ash content of the various wood species [2–13]. In contrast, reports on the distribution of inorganic constituents within the morphological features of a fibre are rare [14–18] and discussed in more detail previously [1].

The SIMS technique provides a unique combination of high spatial resolution, high sensitivity and low detection limit required for this study. SIMS was used to generate elemental maps representative of the morphological features of samples prepared from jack pine sapwood. Sapwood samples were used as they are most representative of the chips used in the manufacture of mechanical pulp. It is our goal to determine the spatial distribution of metal ions that catalyze hydrogen peroxide decompositions in order to better understand and to improve hydrogen peroxide brightening of mechanical pulps.

SAMPLE PREPARATION

Three samples of jack pine sapwood, JP#1, 2, and 3, were prepared by the method previously described [1]. Each sample of jack pine sapwood originated from a different tree. The impregnation of fibres was achieved with the use of water, ethanol and propylene oxide. Both ethanol and propylene oxide were used as received from Aldrich

Chemical Co. A Barnstead Nanopure II system was used to supply the water required. In the final step, the samples were infiltrated with Spurr resin, purchased from Marivac Ltd., Nova Scotia. Mounting, trimming, and facing of samples were performed on a Porter Blum MT-2 ultramicrotome. A DIA-TOME 3 mm diamond knife was used and glass knives were prepared as required. Just prior to analysis, the sample surface was coated with a thin carbon film at 1.33 Pa for 60 s. An Edwards 402 carbon coater was used.

ANALYTICAL METHODS

Bulk analysis was performed using both NAA (Neutron Activation Analysis) and ICAP-AES (Inductively Coupled Argon Plasma – Atomic Emission Spectroscopy). Imaging Microprobe SIMS analysis was performed on a Perkin Elmer PHI 5500 multitechnique instrument. The analyzer was a Balzers 16 mm quadrapole mass spectrometer. An FEI liquid gallium ion gun was chosen for its capability to focus to a small spot size and its high sensitivity for the elements of interest. The ion beam was maintained at 4×10^{-15} J and 300 nA, the spot size was 130 nm. SIMS facilities are located in the Institute for Microstructural Sciences at the National Research Council of Canada, Ottawa, ON.

would prefer something coarser in spatial resolution, i.e., whole ring

RESULTS AND DISCUSSION

The samples of jack pine sapwood were tested for Ca, Mn, Cu, and Zn by NAA and/or ICAP-AES and for Fe, K, Al, Cl, Mg, Sr and Cr by ICAP-AES. The results are shown in Table I. The samples were tested for several other elements; however these

Fig. 2. Mn ion image from the tangential section of Fig. 1 with a perimeter tracing from the Ca ion image overlayed.

Fig. 1. Ca ion image from tangential section of jack pine sapwood showing two intertracheid pit pairs containing tori and a double cell wall in a 33 × 33 μm square field of view.

were either not detected or were less than 1 ppm. Both Harder and Wong have studied the bulk inorganic content of jack pines [19,20]. Although differences in environmental conditions during growing can result in large variations in the concentration of trace elements, our values are within experimental error of those reported by both Harder and Wong.

A calcium image, obtained by the SIMS technique from a tangential section of jack pine sapwood (JP #l), is shown in Fig. 1. The image is 256 × 256 pixels square and is 33 μm wide. The concentration of the metal ion of interest is directly related to the image brightness. The intensity scale is logarithmic because of the high count rate found in the tori. Calcium is found throughout the secondary wall; however elevated concentrations are clearly noted in the tori which, in jack pine, are predominately on the radial wall. Enriched concentrations were also found in the middle lamella. All of these findings correlate well with those reported previously for black spruce [1].

Pit membranes, including the torus, are used as a transport medium and a means of filtering out undesirable material such as toxic substances. The main pathway for the transport of fluids, in softwoods, is through the pit network [21,22]. Bamber found that, although the cell walls of fibres and tracheids are lignified, the membranes of the pits contain no lignin [23]. It has been demonstrated by enzymatic degradation of softwoods that the pit membrane region has a high pectin concentration [24–26]. Clarkson and Hanson reported that Ca^{2+} is the major cation integral to the protein-pectin "cement" of the middle lamella [27]. Also Terashima et al. used an isotope tracer technique to deter-

mine that pectic substances are found mainly in the compound middle lamella [28]. However, it has also been reported that the pectin content of mature cell walls was considerably less than in cell walls in the early stages of development [29]. The metal ions may be present in two forms, associated with the functional groups of lignin and pectin.

Figure 2 shows the manganese image of the same region as in Fig. 1. The field of view is 33 μm. An outline of the perimeter of the calcium image from Fig. 1 has been overlayed onto the manganese image. The spatial distribution of manganese follows the same pattern as that seen for calcium, enrichment in the torus and middle lamella. Elevated concentrations of iron, chromium and zinc are also found in these regions. These results are similar to those found in the study of black spruce.

A sample containing a tangentially sectioned ray cell was prepared (JP #2). The logarithm of the calcium image is displayed in Fig. 3, 80 × 180 μm field of view. Two ray parenchyma and two ray tracheids are displayed. Although the ray parenchyma cell wall has collapsed to one side, it can clearly be seen that enrichment is lo-
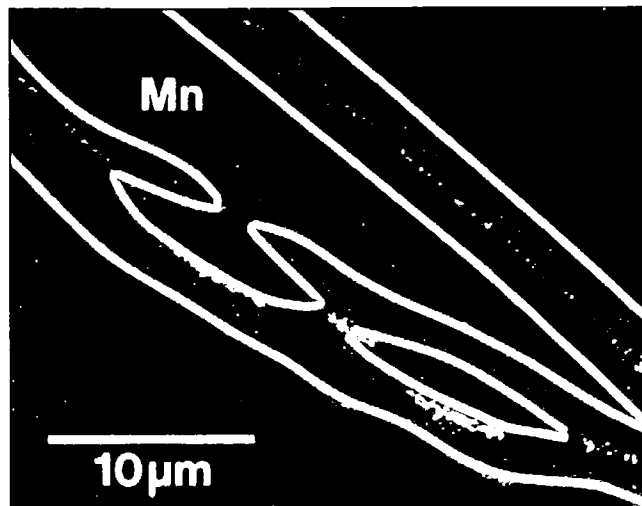
calized in the ray parenchyma cell wall, but not in the ray tracheid. It has been reported that the rays are more permeable than other cell tissue and therefore are more readily penetrated by liquids [21]. If this is the case then, perhaps during cell growth, fluids penetrate the ray cells more readily and deposit metal ions in these areas. The metal ions may be associated with lignin or pectin or both. The trace elements Mn, Fe, Cr and Zn follow the same pattern as that found for calcium.

Images were obtained for a transverse section of jack pine sapwood (JP #3) which exposed the earlywood/latewood boundary region. The counts in the calcium image were averaged using a 10 × 10 pixel matrix. The grey levels were plotted against pixel number for a profile running radially (A–B)

TABLE I
TRACE ELEMENT CONCENTRATION IN JACK PINE, BY NEUTRON ACTIVATION ANALYSIS (NAA) AND INDUCTIVELY COUPLED ARGON PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICAP-AES)

Element	Technique	Concentration (ppm)		
		JP sap #1	JP sap #2	JP sap #3
Calcium	NAA	670	576	459
	ICAP	770	543	420
Manganese	NAA	55	55	30
	ICAP	53	53	29
Iron	ICAP	11	n.d.	n.d.
Chromium	ICAP	1.7	n.d.	n.d.
Copper	NAA	n.d. [3.4]	3.9	3.3
	ICAP	1.2	4	4
Zinc	ICAP	17	6	9
Potassium	ICAP	210	300	300
Aluminum	ICAP	14	6	6
Chlorine	ICAP	87	120	33
Magnesium	ICAP	160	180	110
Strontium	ICAP	3.7	4.7	3

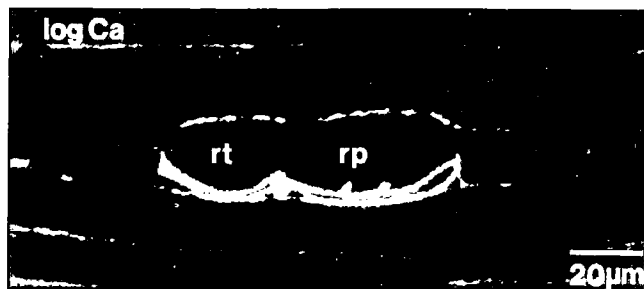n.d. – not detected
[ ] – detection limit

Fig. 3. Ca ion image from a tangential section of jack pine sapwood showing a ray cell containing two ray parenchyma (rp) and two ray tracheids (rt), 80 × 180 μm field of view.

Fig. 4. Ca ion image from a transverse section of jack pine sapwood showing the earlywood/latewood boundary, 85 × 95 μm field of view.

and tangentially (C–D). The image and profiles are shown in Figs. 4 and 5, respectively. Figure 4 shows a highly enriched concentration of calcium in the middle lamella and cell corners at the earlywood/latewood transition. This concentration decreases in the latewood cells with increasing distance from the boundary. A high concentration of calcium can also be found in the torus of a bordered pit pair in the latewood region. It should also be noted that, in the latewood cells, elevated concentrations of calcium can be seen in the inner layer of the secondary wall.

From Figs. 4 and 5 it is evident that a higher concentration of calcium is found in the radial wall as compared to the tangential wall. This is also true for the metals manganese, iron, chromium and zinc. In jack pine, bordered pits are predominately on the radial walls of the fibres, unlike most conifers [30]. Becker concluded that the higher moisture content in the radial cell walls as compared to the tangential walls provides for easier pathways for different liquids, and hence results in better penetration of liquids [21]. Ruch and Hentgartner showed that the level of lignification in the middle lamella is variable around the circumference of the cell, being greatest at the cell corners and greater on the radial as compared with the tangential

walls [31]. They also found a correlation between the age of the cell and the amount of lignin present. In Fig. 6a and b, a profile extended across the double cell wall, starting at the cambium, shows the varying levels of lignification as found by Wardrop for Monterey pine [32]. Figure 6a shows the amount of lignin from cells 38 to 39 (numbered from the cambium) whereas 6b displays the cells 44 to 45, further from the cambium. The elemental profile (C–D), shown in Fig. 5 shows a similar pattern to that found by Wardrop in Fig. 6a. The profile A–B, in Fig. 5, indicates a gradual change in the lignin content as the distance from the earlywood/latewood boundary increases. Saka and Thomas also studied the lignification as the cell wall matures [33]. Using SEM-EDXA, they looked at loblolly pine and found a similar pattern with increasing distance from the cambium. Allen used Ru-red staining techniques to determine areas of

greater lignification [34]. They found that the staining was greater on the radial as compared with the tangential walls, therefore indicating greater lignification on the radial wall. This is in agreement with Wardrop's findings of greater lignification in the radial walls [35].

As mentioned, Fig. 4 also shows elevated concentrations of calcium in the inner layer of the secondary wall, S3. This may be associated with higher concentrations of lignin in this area. Lange concluded from microspectrophotometric measurements that the concentrations of lignin in the S3 layer in spruce was about 73% [36]. Figure 6b also
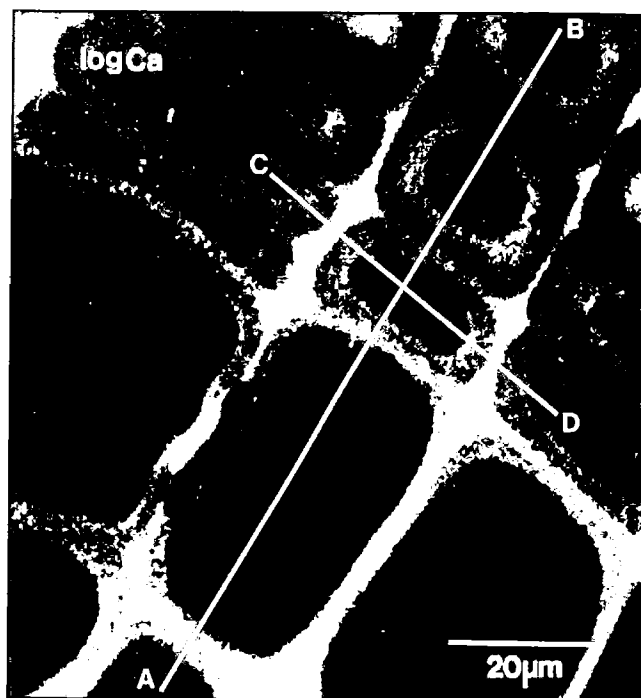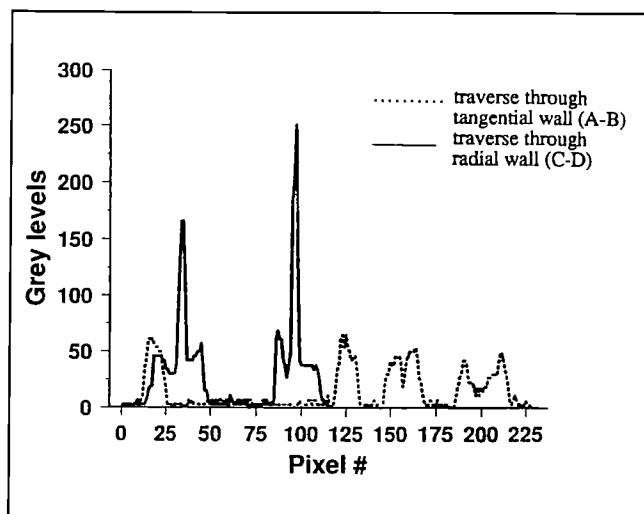

Fig. 5. Ion intensities for a calcium profile running both radially and tangentially from the transverse section of Fig. 4.
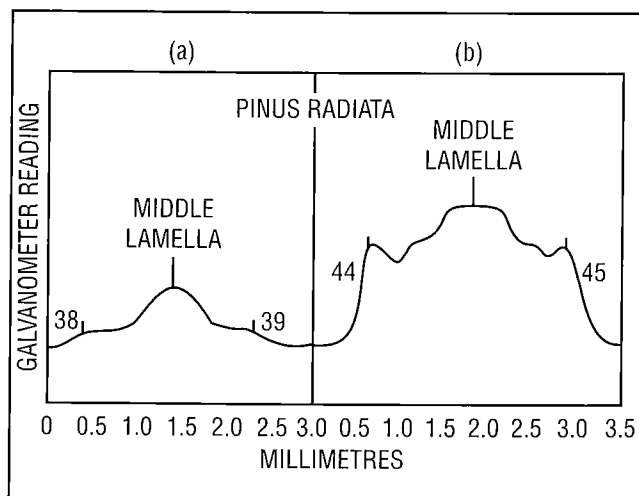

Fig. 6. Microphotometer traces through ultraviolet photomicrographs showing the absorption between tracheids of Monterey pine of increasing maturity [32].

shows evidence that supports the association of an elevated concentration of calcium in the inner layer of the secondary wall with the presence of lignin.

CONCLUSIONS

SIMS has again proven successful in determining the spatial distribution of trace elements in wood. Trace elements are concentrated in the torus, ray parenchyma cell wall, cell corners and middle lamella of a jack pine sapwood sample. These metal ions are also concentrated in the middle lamella and cell corners at the earlywood/latewood boundary and decrease with increasing distance from the boundary. Calcium is detected in the inner layer of the secondary wall in the latewood cells and is associated with the presence of lignin. Copper is not detected by SIMS in any of the samples investigated. The distribution of metal ions across a double cell wall varies depending on the proximity to the earlywood/latewood boundary. The localization of metal ions may be related to the presence of pectic substances and lignin.

ACKNOWLEDGEMENTS

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ABSTRACT: Imaging Microprobe Secondary Ion Mass Spectrometry (SIMS) has been used in determining the spatial distribution of trace elements in jack pine, *Pinus banksiana* (Lamb.). Trace elements were found to be concentrated in specific morphological features, namely: the torus, middle lamella, cell corners and ray parenchyma wall. Middle lamella calcium ion enrichment was particularly great in the radial middle lamella near the latewood/earlywood transition. There is also evidence of calcium enrichment in the inner layer of the secondary wall in the latewood cells. Analysis of a ray cell sectioned tangentially indicates enriched concentration of the above elements in the ray parenchyma wall, however not in the ray tracheid wall.

RÉSUMÉ: Nous avons utilisé la spectrométrie de masse à ions secondaires à microsonde imageuse (SIMS) pour déterminer la distribution spatiale des éléments traces dans le pin gris, *Pinus banksiana* (Lamb.). Nous avons observé que les éléments traces étaient concentrés dans des caractéristiques morphologiques spécifiques, notamment le tore, la lamelle moyenne, les coins des cellules et la paroi du parenchyme radial. L'enrichissement de la lamelle moyenne en ions de calcium était particulièrement important dans la lamelle moyenne radiale près de l'aire de transition entre le bois de printemps et le bois d'automne. Nous avons également trouvé de traces d'un enrichissement de calcium dans les couches intérieures de la paroi secondaire des cellules du bois d'automne. L'analyse d'un rayon médullaire coupé tangentiellement a révélé la présence d'une concentration enrichie des éléments précités dans la paroi du parenchyme radial, mais non pas dans la paroi de la trachéide radiale.

KEYWORDS: ALKALINE EARTH METALS, BORDERED PITS, CALCIUM, CELL WALLS, DISTRIBUTION, MASS SPECTROSCOPY, PARENCHYMA, PINUS BANKSIANA, RAY CELLS, SAP WOOD, SPECTROCHEMICAL ANALYSIS, TRACE ELEMENTS, MIDDLE LAMELLAE.

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Compactibility of a Wet Fibre Mat Using Acoustic Radiation Pressure

P.H. BRODEUR and T.M. RUNGE

The compactibility of a wet fibre mat was investigated using acoustic radiation pressure. More precisely, the radiation force of an ultrasonic traveling wave field was used to consolidate a pulp suspension into a mat and then compact the mat. The amount of compaction was determined using an image analysis system. Compaction measurements, performed on a bleached kraft softwood market pulp beaten to various levels, were shown to correlate linearly with the apparent density and tensile strength of test handsheets.

INTRODUCTION

According to Clark [1], wet fibre compactibility (WFC) relates to the ability of fibres to conform when pressed against other wet fibres and to remain consolidated

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when dried due to the cohesiveness of the surfaces. WFC is one of the most important papermaking properties because it impacts the relative bonded area of fibres and, hence, almost every optical and physical property of paper. Factors influencing the compactibility of wet fibres include cell wall thickness, degree of internal fibrillation, and to a lesser extent fibre cross-sectional shape, fibril angle, and degree of external fibrillation. WFC is particularly sensitive to the wet resiliency ("spring-back" effect) and wet plasticity of fibres [1].

As critical as this property can be to the papermaking process, a standard testing method is not available. Some evidence about wet fibre compactibility can be obtained from wet fibre flexibility analysis. Among the several different methods to determine wet fibre flexibility, bending of individual fibres using mechanical or hydrodynamic forces provides direct measurements [2–6]. Another testing approach involves the conformability evaluation of dry fibres draped over a glass rod on a glass slide [7–12]. Determination of how much the fibres bent during drying is done by measuring the amount of optical contact between the slide and the fibres. Other types of

flexibility tests include fundamental modes of vibration [13,14] and structural methods [15]. More recently, techniques involving individual fibres passing through a slot perpendicular to a laminar flow channel have been devised to collect automated fibre flexibility measurements [16–18].

Clark [19] has proposed a test method for the wet specific volume of pulp, which is a property sensitive to wet fibre flexibility and resiliency. However, a more convenient test to evaluate wet fibre compactibility is to determine the dry specific volume (bulk) or its inverse, the apparent density, of standard test handsheets [1].

The present work proposes a direct approach to determine wet fibre compactibility through the compactibility of a pulp suspension subjected to acoustic radiation pressure. The method was developed following the exploratory study of a distributed ultrasonic standing wave field acting on cantilever-suspended single fibres in water [20]. In this work, it was hypothesized that the radiation pressure exerted by a high-frequency, high-intensity mechanical wave could be used to induce fibre bending and, hence, determine wet fibre flexibility in a non-contact manner. However, instead of