FROM FOREST TO PRODUCT: NEW SOLUTIONS FOR RAPID, COMPREHENSIVE WOOD AND FIBRE ANALYSES

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ABSTRACT

Even with the latest developments in pulp processing and papermaking technologies, wood and fibre properties remain key factors contributing to both production efficiencies and product performance. There is a need for tools to efficiently measure wood and fibre quality attributes, whether those fibres are plantation based, such as eucalyptus and hybrid poplars, or from natural forests such as boreal softwoods and North American hardwoods. In Canada, the long, fine softwood fibres are recognized as the premier reinforcement fibres in the world, and hardwoods such as maple, birch and aspen offer a diverse range of properties that allow pulps to be tailored to meet specific end-use requirements. For all these species, it is becoming increasingly important to understand and assess wood quality characteristics in order to improve product performance and develop value-added opportunities. In Brazil, the short rotation, plantation-based eucalyptus fibre supply scenario is very different, but the need to measure and deliver fibre quality remains the same. Manufacturers can benefit from rapid means of evaluating genetic material for specific growth conditions or quantifying the impact of silvicultural practices on forest productivity and fibre quality. This paper describes the suite of state-of-the-art high-throughput wood and fibre analysis technologies being applied at Paprican's EvaluTree division to support research ranging from the impact of genetics on wood quality, to understanding the end-use performance of fibres. Preliminary work is also underway in collaboration with the University of São Paulo to compare wood and fibre properties of hardwoods from around the world. EvaluTree technologies include SilviScan® for laboratory measurement of pith-to-bark radial profiles on wood cores, the latest in confocal and environmental scanning electron microscopy (ESEM) for advanced imaging of fibre performance in products, the HiRes Fibre Quality Analyzer for fibre length, width, shape and coarseness, and optical spectroscopy techniques for wood and chemical properties.

INTRODUCTION

Analyses of wood and fibre qualities in a forest stand should consider within tree variability, between tree variability within a given stand, and stand to stand variability. Hence, any meaningful study requires large sample numbers, routine in-depth (high resolution) analyses and statistical validity of data increasingly directed towards end-user applicability. An information gap between wood properties and product performance exists in part because of the time-intensive nature of many of the experimental procedures required to access more fundamental information on wood chemistry and cell wall morphology. Recent years have seen a renaissance in the development of wood and fibre analysis tools. Advances in optical based technologies including infrared and Raman spectroscopy, high resolution X-ray analytical tools and applications of advanced microscopy are enabling researchers to realize unique insights into wood and fibre quality, and to quickly assess a statistically relevant number of trees in a cost effective manner.

The forest products industry is one of the key drivers of the Canadian economy and it is becoming increasingly important to understand and assess wood quality characteristics in order to identify value-added opportunities. In recognition of this, Paprican, in collaboration with the University of Northern British Columbia and the University of Victoria, has established EvaluTree™, a state-of-the-art wood and fibre analysis laboratory in Vancouver, British Columbia. Funded by the Canadian Foundation for Innovation, the British Columbia Knowledge Development fund and Paprican, this world-class facility is equipped with technologies that include SilviScan, environmental scanning electron microscopy, advanced confocal microscopy, Hi-Res Fibre Quality Analyzer, vibrational spectroscopy, image analysis, and advanced statistical analysis capabilities.

EvaluTree™ offers collaborative research and service opportunities for wood and fibre quality researchers, and has been applying these state-of-the-art techniques to support research into tree breeding and to help assess the impact of silvicultural practices and genetics on wood quality. Other recent work includes the use of near infrared spectroscopy for field portable analysis of wood quality concerns (such as blue stain and decay) associated with British Columbia's mountain pine beetle epidemic. Preliminary work is also underway in collaboration with the University of São Paulo to compare wood and fibre properties of hardwoods from around the world.

DISCUSSION

Environmental Scanning Electron Microscope

Scanning electron microscopy (SEM) has long played a pivotal role in structural characterization for material scientists, however with these earlier instruments the high vacuum requirements in the chamber required lengthy specimen preparation techniques to remove or fix the water before imaging, raising the risk of artifacts being introduced. These problems have now been overcome, thanks to the new environmental scanning electron microscope (ESEM), which permits the imaging of wet systems with no prior specimen preparation (the term environmental refers to the instrument's capacity for higher pressures in the sample chamber, allowing the presence of an environment, usually water vapour, during imaging). Whole new classes of materials, previously undreamed of, can be imaged in their natural state. But the potential of ESEM is even greater than this. Because the sample environment can be dynamically altered, hydration and dehydration processes can be observed as they happen in the sample chamber.

In November 2005, EvaluTree installed and commissioned a FEI Quanta-400F field-emission environmental scanning electron microscope (FE-ESEM). It is an extremely flexible, high-performance microscope. The instrument has a Shottky-type field-emission (FE) source and is capable of 2nm resolution in conventional high-vacuum mode.

The instrument has a large chamber (Figure 1) with a fully motorized stage allowing 100mm x 100mm X&Y travel, 60mm of Z movement and tilt angles from +70° to -5°. The instrument is equipped with an infrared chamber camera so the interior of the chamber can be viewed when the door is closed. A wide range of accessories which make the instrument extremely versatile include:

- a Peltier cooling stage capable of controlling the specimen temperature to 0.1°C increments from ambient temperature to -20°C.
- a heating stage controllable in 1 degree increments up to 1000°C
- a 200N tensile stage for in-situ tensile testing of small specimens
- detectors enabling secondary electron imaging under all environments
- an EDAX Genesis energy dispersive x-ray spectrometer capable of x-ray mapping

Some of the benefits of this instrument are:

- Fast sample preparation: samples can be mounted and inserted in the ESEM without coating.
- Direct imaging of the sample: no coating means you are imaging (and analyzing, in the case of energy dispersive X-ray spectroscopy (EDS)) the sample without a coating obfuscating the results.
- Non-destructive imaging: the sample can be imaged in the ESEM and then removed and analyzed by other techniques. For samples smaller than 25cm x 25 cm it is possible to insert the whole sample without cutting it.
- Uncoated specimens can be imaged, removed from the ESEM, subjected to various chemical and/or mechanical treatments, reinserted in the ESEM and examined. Using the stage mapping feature of the ESEM, the same spot can usually be easily located.
- Moisture cycling experiments can be conducted: Using the ESEM mode in combination with the cooling stage, it is possible to control the specimen temperature and the chamber pressure to specific levels of relative humidity from 0 to 100%.

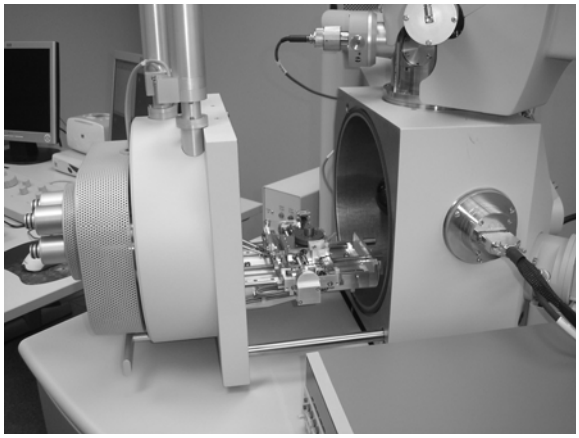

Figure 1. Chamber view of the FE-ESEM.

ESEM Applications

The ESEM enables imaging of a wide range of materials at any initial moisture content. A variety of different images have been collected with EvaluTree's FE-ESEM and no coatings were applied to any of the samples. Some examples include wood (Figure 2) and forest pests (Figure 3).

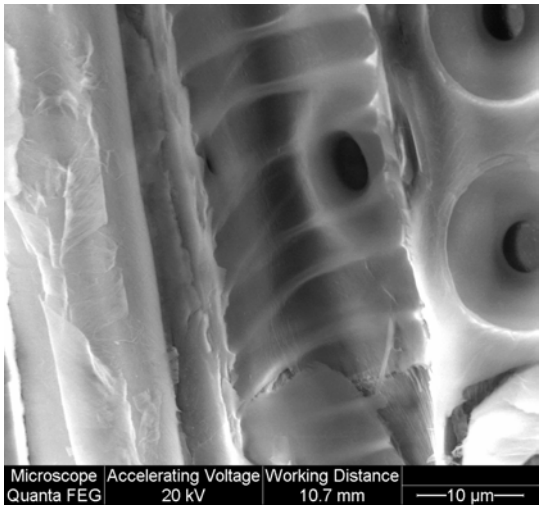

Figure 2. Uncoated ESEM image of wood.

Figure 3. Uncoated ESEM image of a mountain pine beetle.

Samples of paper sheet surfaces can be imaged in the ESEM without coating. This enables direct imaging of the sample. Figures 4 and 5 show surface images of kraft handsheets from unrefined eucalyptus (hybrid of grandis x urophylla) and birch (betula pendula). Figure 6 shows cross sections of the birch handsheet in its original state and after wetting and redrying.

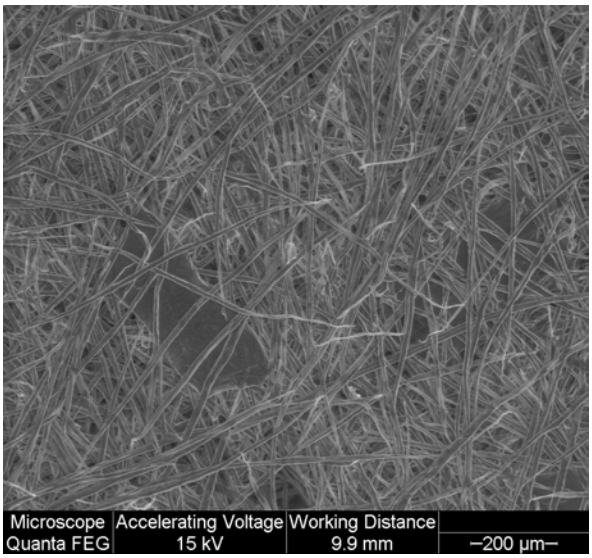

Figure 4. Uncoated ESEM image of the surface of kraft pulp handsheet from unrefined hybrid of E. grandis x E. urophylla (Brazil).

Figure 7 shows a series of images of the surface of a newsprint sheet undergoing a moisture cycling experiment. The first image shows moisture droplets beginning to condense on the surface of the sheet. The second image shows the surface almost completely covered with liquid water. The final image shows the sample after returning to room temperature and lower pressure; the sheet has dried and the considerable changes in appearance compared to the first image are readily observed.

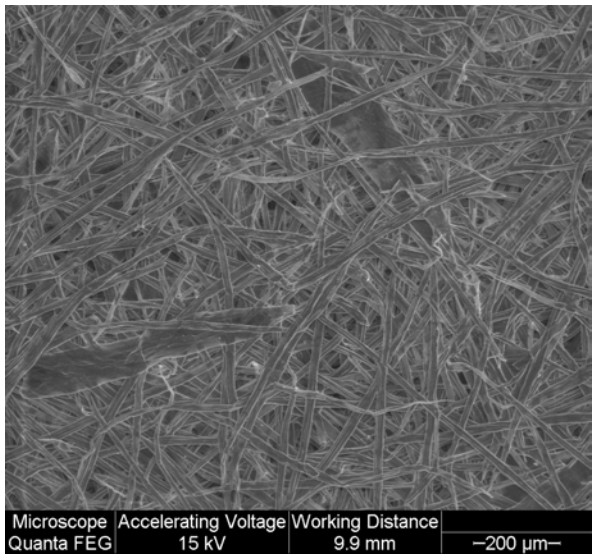

Figure 5. Uncoated ESEM image of the surface of handsheet from unrefined betula pendula kraft.

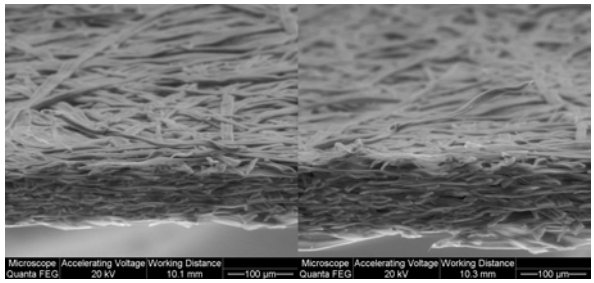

Figure 6. Cross sections of unrefined Betula pendula handsheet in its original (air-dried) state (left) and after wetting and redrying (right).

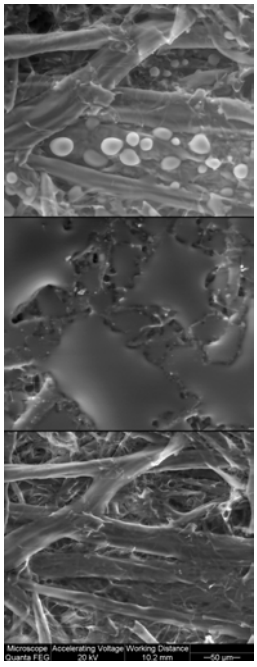

Figure 7. A series of images of the surface of a newsprint sheet undergoing a moisture cycling experiment.

Sheet surfaces can be imaged in secondary or backscattered electron mode. Figure 8 shows images of a newsprint sheet surface in back-scattered electron mode in low-vacuum. The filler distribution is clearly shown.

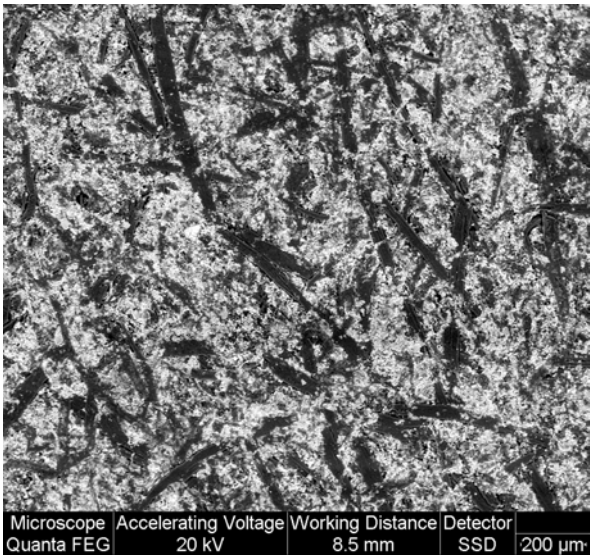

Figure 8. Newsprint sheet surface in back-scattered electron mode in low-vacuum.

In-situ tensile testing is also one of the ESEM's highlights. The 200N micro-tensile stage has been used to test small paper specimens inside the ESEM. This accessory enables collection of stress-strain data while simultaneously observing and recording the video image of the test. Single frames of a video capture of a strained-to-failure newsprint sample are shown in Figure 9. The sample can be studied in detail at the fibre level to observe the mode(s) of failure. Similar trials on wood specimen are underway.

One of the most exciting avenues that ESEM opens is the ability to do correlative microscopy. The exact same areas of a sample can be examined by light microscopy, ESEM and confocal microscopy. Figure 10 shows a sample of pen ink on paper. The same area is shown first in the light microscope and then in the ESEM. The different information available from the two techniques can be combined. The sample was analyzed with the EDAX system and the major peaks mapped to show which elements correlate most strongly with the inked area.

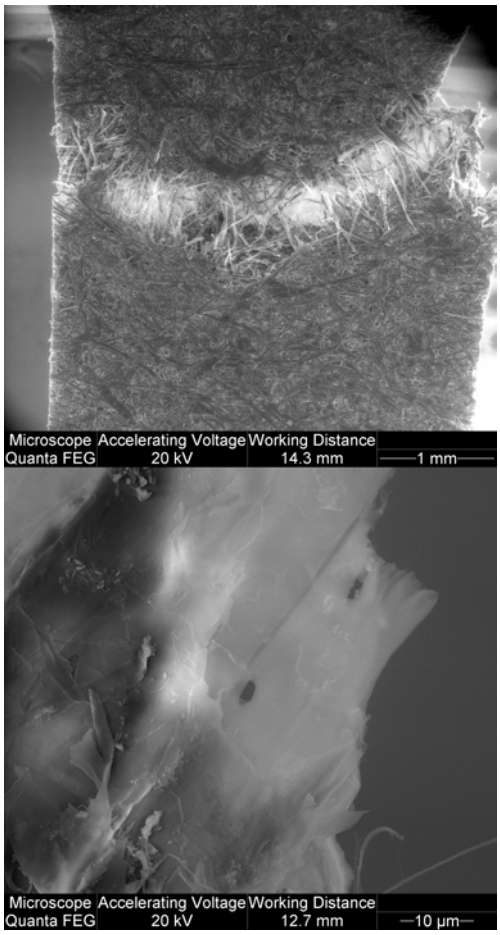

Figure 9. Single frames of a video capture of a strained-to-failure newsprint sample.

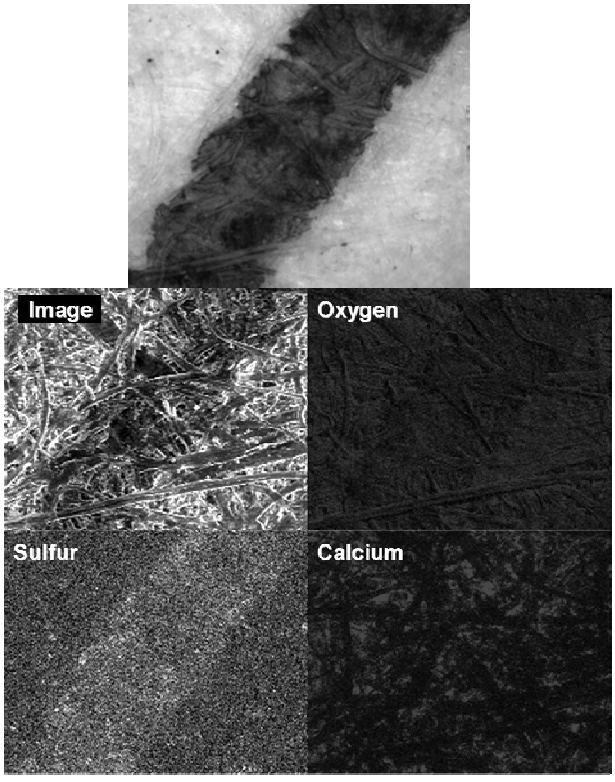

Figure 10. A sample of pen ink on paper and analyzed using the EDAX system.

Confocal and Multiphoton Microscopy

Confocal laser scanning microscopy (CLSM) is a powerful optical sectioning tool that can image the internal structures of materials without physical sectioning. It can provide single plane 2-D images or 3-D reconstructions of objects with the resulting images readily quantifiable using image analysis. Through the use of various lasers and detectors, it also allows detection of components within a material based on their fluorescent emissions.

A confocal microscope creates sharp 2-D images of a specimen that would otherwise appear blurred when viewed with a conventional light microscope. This is achieved by placing a pinhole in front of the detector to exclude light from the specimen that is not from the microscope's focal plane (Figure 11). A light source is scanned across the sample in the horizontal plane, images are taken point-by-point and reconstructed using a computer. The resulting image has less haze and better contrast than that of a conventional microscope and represents a thin cross-section of the specimen. The sample can then be imaged at varying depths or planes in the z-direction producing a stack of 2-D images (optical sectioning) which can be combined to render or create a 3-D image. It is important that the specimen is illuminated with bright, glare-free light. Lasers are used as a light source as they produce high intensity beams of parallel light and are available in a wide range of wavelengths.

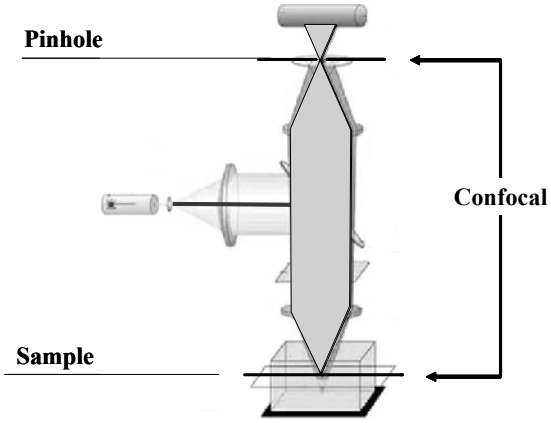

Figure 11. Schematic diagram of confocal microscope. Pinhole suppresses out of focus light.

Most confocal microscopes image either by reflecting light off the specimen or by stimulating autofluorescence or fluorescence from dyes (fluorophores) applied to the specimen. When a molecule is irradiated with light of a certain wavelength, energy is absorbed and the molecule's electrons are excited from ground to an excited state. The electrons then return to their normal energy by emitting light of longer wavelengths. Illuminated specimens emit light at many different wavelengths depending on their structure and content. Specific components within a sample will emit light with a characteristic emission spectrum; this fluorescence emission can be collected and separated into different channels or detectors through the use of dichroic filters. Each signal can be assigned a colour and recombined into a single image (Figure 12).

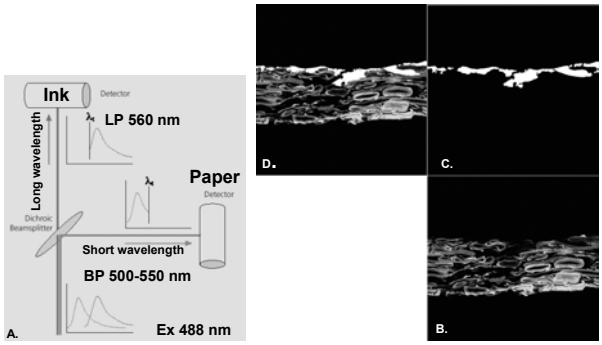

Figure 12. A. Separation of fluorescence emission from printed paper. B. Signal from paper. C. Signal from ink. D. Combined channels – single image.

Multiphoton microscopy is probably the most important development in fluorescence microscopy since the introduction of confocal imaging and provides researchers with unique possibilities for imaging deeper into samples than previously possible. It is similar in optical sectioning capabilities to conventional confocal microscopy but is based on different principles. In multiphoton microscopy, optical sectioning of a sample is achieved through the use of an infrared (NIR) laser which excites fluorescence only at the plane of focus. This eliminates the need for a pinhole in front of the detector which allows more signal to be collected and as a result, imaging is possible to greater depths within the sample than is achievable using conventional confocal microscopy.

In December 2005, EvaluTree installed a state-of-the-art Zeiss LSM 510 META NLO confocal microscopy system (Figure 13).

Figure 13. Evalutree's confocal and multiphoton microscopy facility. Zeiss LSM 510 Meta NLO confocal laser scanning microscope.

The entire system is user-controlled from a PC. Both inverted and upright microscopes have motorized x-y stages; the Axiovert is also equipped with a piezo-electric stage for rapid and accurate z direction movement to facilitate cross-sectional imaging. The large sample stage of the inverted microscope accommodates imaging of thick, unmounted specimens such as wood blocks and paper strips. In the META detector, a highly efficient optical grating separates the fluorescent emissions into 32 channels enabling the entire emission spectral range to be collected in 10nm increments. Using this spectral data plus the spectral "unmixing feature", clear separation of overlapping fluorescence signals for accurate mapping of sample components is possible. The combination of multiphoton excitation and an external detector that enhances signal collection allows optical sectioning of samples hundreds of microns deep. Additional software provides flexibility in image collection and processing:

- Surface topography analysis
- StitchArt (montaging of serial images to produce a single representation of a large sample region)
- Multi-time series for automatic image acquisition in multiple positions. Combined with the motorized XY driven stage, the sample can automatically be scanned across a large area.

Confocal and Two-Photon Microscopy Applications

CLSM has wide applications for use in the solid wood, pulp, and paper industries. It is used to characterize fibre morphology, sheet structure including surface roughness, fillers/fines distribution, and ink application. CLSM is also well suited for the evaluation of solid wood and composite wood materials. Based on technology developed at Paprican, CLSM is used to measure simultaneously the transverse dimensions, fibril angle, and collapse index of individual fibres, enabling characterization of fibre properties in pure species as well as blended commercial pulps [1]. Fibre cross-sectional images are acquired from a dilute pulp suspension stained with a fluorescent dye. As shown in Figure 14, for each image obtained, the following fibre properties can be measured or calculated: fibre wall thickness, fibre cross-sectional area, fibre perimeter, lumen area and collapse index. The morphological properties of pulp fibres, particularly their transverse dimensions, influence how fibres respond to processing and strongly affect the properties of end products [2].

The fibril angle, the angle between the fibrils and the fibre axis, is one of the important factors that control mechanical properties such as strength and modulus of elasticity shrinkage of wood pulp fibres [1]. EvaluTree's innovative technology, using CLSM in combination with polarization analysis of fluorescence, easily and accurately measures fibril angle on individual pulp fibres. This technique has been successfully applied to softwood and hardwood pulps processed under different pulping conditions (i.e. mechanical, chemical) in wet or dry states [3]. Fibre collapsibility, well recognized to be important for many physical properties of paper products [4], can then be calculated.

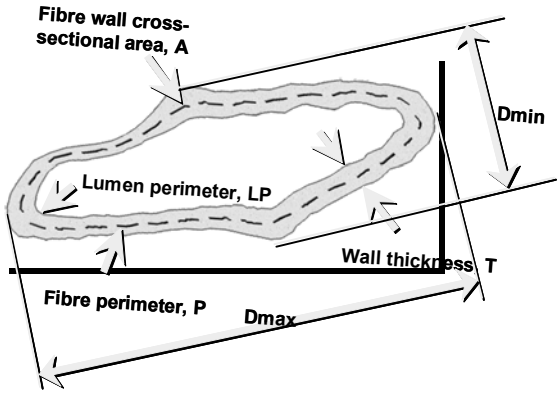

Figure 14. Schematic diagram of fibre cross-sectional image showing properties measured.

The new CLSM system has also been used to characterize: paper surface properties, internal sheet formation, ink application, and filler distribution in the z-direction. Figure 15 shows cross-sectional images of handsheets from unrefined pulps: hybrid of E. grandis x E. urophylla (Brazil) and betula pendula (Finland).

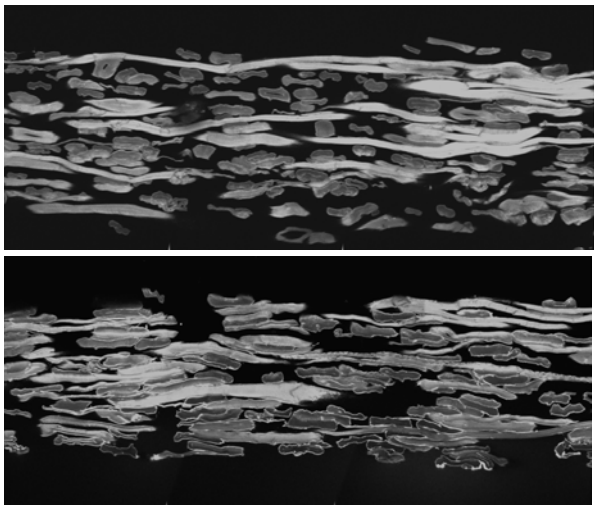

Figure 15. Confocal images showing fibre and fines distribution in z-direction of hardwood sheets. Top image: Hybrid of E. grandis x E. urophylla, Bottom image: betula pendula (birch).

A series of optical sections in the z-direction were acquired from a sheet produced from thermo-mechanical (TMP) pulp. The image stack was then combined to create a 3-D reconstruction of the surface and internal structure (to a shallow depth) of the sheet (Figure 16). The individual components; fibres, fines material, and ray cells making up the paper structure are easily visualized. Software is available to display surface topographic features in 3-D and future work will focus on development of quantitative

measurements to determine surface properties such as fibre coverage, pores distribution, and roughness. As mentioned previously, materials emit characteristic fluorescence based on their chemical composition. Sample components can then be mapped based on the various emission peaks that are detected. Figure 20 shows a paper cross-section illustrating the distribution of chemical and mechanical fibres, and fines in the z-direction of the sheet.

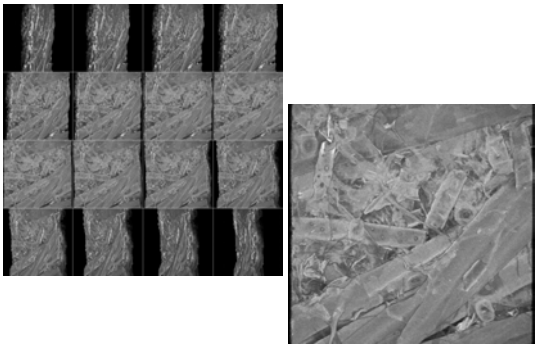

Figure 16. Optical sections acquired from a TMP pulp sheet.

The application of ink onto paper influences the final product quality, ink usage, and ink show-through. We have used CLSM to illustrate both the ink coverage and penetration into the paper base sheet. In comparison to traditional light microscopy methods, very little sample preparation was required. A section of the paper was hand cut using a single-edged razor blade and imaged at a depth away from the roughly cut edge. Excitation lasers were then applied to the paper strip and the fluorescence emissions from the sample were collected and separated into two channels. The two channels were assigned different colours and then combined into a single image which clearly demonstrated the occurrence of ink in relation to the underlying paper structure. CLSM can also be used to visualize ink-paper interactions such as paper surface roughing resulting from fibre rising during the printing process (Figure 17).

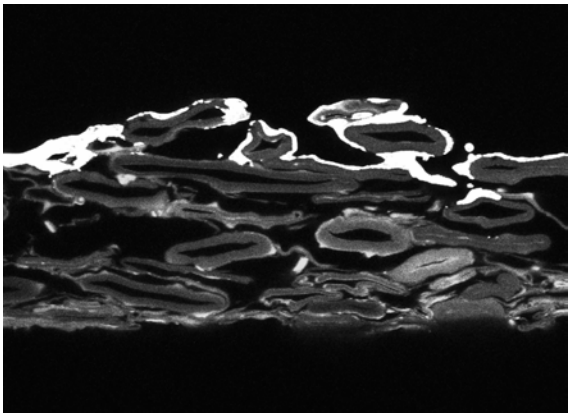

Figure 17. Ink – paper interactions. Fibre rising during printing. Ink shown in white.

CLSM has also been used to characterize filler distribution in filled papers. By separately detecting the light reflected from the filler and the fluorescence signal emitted by the fibre, we are able to visualize filler in the z-direction. Long, high-resolution paper cross-sections, with individual fibres and fillers clearly defined, can be readily generated, providing detailed analysis and statistical information.

Fibre transverse dimensions can be obtained directly from wood cross-sections and CLSM optical sectioning produces more accurate images of wood cells compared to conventional microscopy using thick sections for imaging by transmitted light microscopy [5]. High-quality cross-sectional images of large wood samples are easily generated using CLSM and the high signal to background ratio of the confocal images facilitates image analysis to measure the transverse dimensions of the cells in solid wood. As is shown in Figure 18, cell wall boundaries are easily defined due to the strong signal generated by the high lignin content of the middle lamella surrounding the cells. Integrating powerful image analysis software (Cellenger and Image Pro Plus) with confocal imaging, work is in progress to develop automated routines that will provide rapid assessment of fibre properties in solid wood.

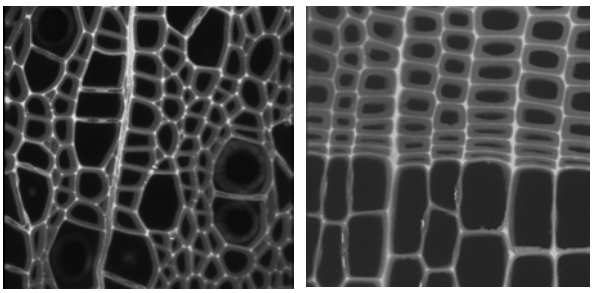

Figure 18. Confocal images hardwood (left) and softwood (right). Taken from solid wood blocks.

SilviScan

The Marcus Wallenberg prize winning SilviScan system, developed by the Commonwealth Scientific and Industrial Research Organization (CSIRO) Forestry and Forest Products Division, is a suite of instruments designed for the rapid and non-destructive assessment of wood and fibre properties.

SilviScan (Figures 19 & 20) uses a range of analytical technologies, including optical microscopy, x-ray diffractometry, x-ray densitometry, image analysis, applied mathematics, and analysis of large data sets, to give unparalleled insights into wood and fibre properties. The latest generation, SilviScan-3, can determine many wood fibre properties that govern product quality such as: fibre diameter, fibre wall thickness, wood density, coarseness, microfibril angle (MFA), and wood stiffness. SilviScan's innovative technology provides precise and distinct densitometric and diffraction measurements from earlywood/

latewood fibres within the sample, resulting in sharp definition of growth ring boundaries. Measurements generated by SilviScan are based on 2mm thick wood sample and are made several times faster than with conventional methods. SilviScan provides unequalled high throughput, high resolution data with the highest degree of accuracy [6].

The data can be used to map out the within tree variations in wood and fibre properties for process optimization, correlate wood and fibre properties to end-use product performance, quantify the effects of silvicultural treatments on wood and fibre quality, identify suitable genetic material for new plantations and generate unprecedented insight into tree radial growth as affected by environmental factors.

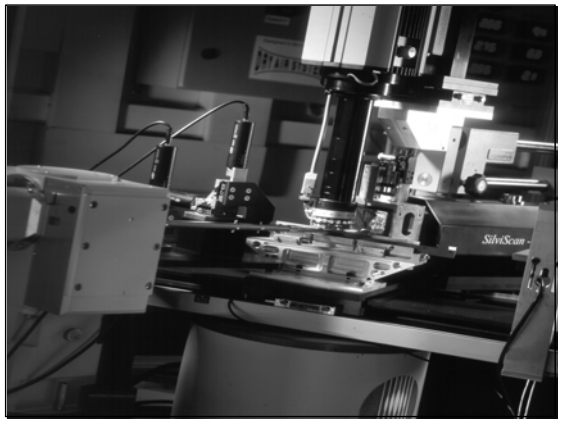

Figure 19. Illustration of the SilviScan system.

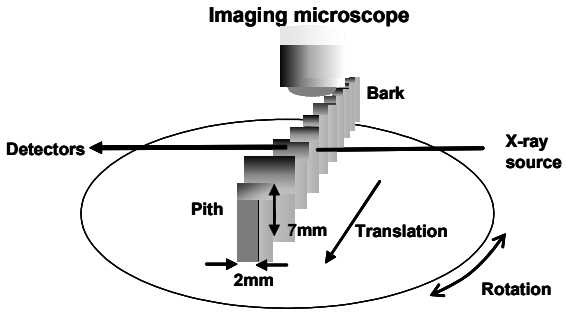

Figure 20. Schematic diagram of SilviScan's experimental design.

To prepare the sample the cross-sectional face of the wood strip is sanded and polished to reveal cell detail. A transmitted light image of the polished cross section is acquired, and processed to identify radial and tangential cell wall boundaries. Further image analysis is done to determine radial and tangential fibre diameters and estimate tracheid perimeters and cell population within a 25μm step size. From this data, the angles of the growth rings in relation to the radial side of the wood strip are determined. Using the image analysis information, the sample is rotated so that the growth rings remain parallel to the x-ray beam during the entire scan. This provides accurate and distinct density and diffraction measurements from early/latewood fibres within a sample, resulting in sharp definition of growth ring boundaries [7].
Wood density is measured by irradiating a sample with x-rays and detecting the amount of radiation that is transmitted through the sample. X-ray absorbance is related to density according to Beers Law [8].

$$I = I_0 e^{-\mu_m t \rho}$$

I_0 = incident x-ray intensity
I = transmitted x-ray intensity
μ_m = mass absorption coefficient
t = sample thickness
ρ = sample density

Density and tracheid diameter profiles are combined to calculate fibre wall thickness, coarseness, and specific surface area.

A wide-angle x-ray detector is used to record diffraction patterns from the wood, giving information on the microfibril angle (MFA), fibre orientation, and crystallite width. Longitudinal modulus of elasticity (wood stiffness) can be estimated from the density and diffraction profiles. MFA profiles are generated at client specified resolutions ranging from 0.1mm to 10mm radial steps [6].

Post data analysis of wood and fibre properties determined using SilviScan provides both unweighted and area weighted averages where the area weighting emphasizes the contributions from the outer rings of the tree. Weighting by radius is similar to weighting by the length of the perimeter corresponding to the point where the property is measured.

SilviScan Applications

Since the properties of wood strongly influence both product quality and production efficiency in wood-based industries, SilviScan technology can provide a better understanding of the role that fundamental fibre properties play in determining end-use product quality and value. Wood fibres are the building blocks of trees and can vary significantly in length, wall thickness, perimeter and cellulose microfibril orientation within a tree, within a species and between species. One SilviScan project conducted at STFI involved the evaluation of the feasibility of using fast-growing Norway spruce in plantations with optimized fertilization in Sweden. The researchers determined, from the use of SilviScan data, that with fertilization, the growth rings tended to be wider which reflected an increase in volumetric wood production (Figure 21) [9]. In addition, wood density decreased with fertilization but the net effect was an increase in the production of dry wood substance. The reason for the decrease in density, as shown from the SilviScan data,

is that upon fertilization, the wood fibre walls became thinner and the fibres become wider.

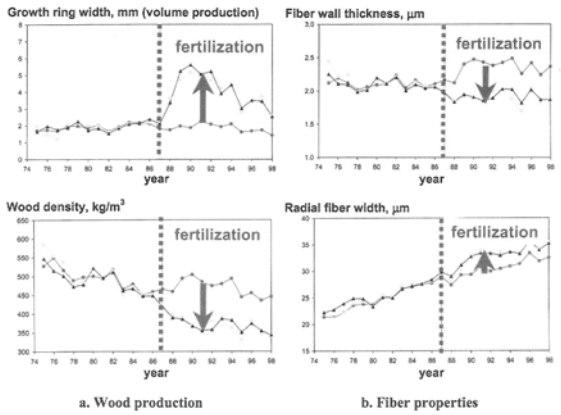

Figure 21. Effects of fertilization on wood production (left) and fibre properties (right), determined by SilviScan. With fertilization, the growth rings increased but the wood density decreased. The net effect was a considerable increase in dry wood production. The fibre walls became thinner and the fibres slightly wider, resulting in more collapsible fibres (Christina Lundgren, SLU).

In a study funded by the Canadian Forest Service, Paprican investigated the shelf life of Mountain Pine Beetle (MPB) killed trees in British Columbia (BC). The mountain pine beetle epidemic in north-central BC poses significant challenges to the forest products industry. Shelf life refers to the amount of time a standing dead tree will remain industrially useful. Thirty-nine sample sites were established across the Sub-Boreal Spruce and Sub-Boreal-Pine-Spruce biogeoclimatic zones in order to conduct a preliminary wood-quality based shelf life assessment. A Biogeoclimatic Ecosystem Classification (BEC) was completed at each site and wood cores were collected from ten trees per site. SilviScan was one of the high throughput technologies used to determine wood properties of this large sample set. Using SilviScan's x-ray densitometry, the data showed that there was no trend in density when displayed as a function of time since death, suggesting that wood density is not affected by beetle induced mortality for the sites evaluated to date. If decay fungi were present, the loss of wood mass would be expected to result in a reduction in basic density. This important project is ongoing [10].

Because of the short interval (50 microns) between density measures acquired with SilviScan, density profiles can be created. These profiles show the delineation between annual growth rings due to variation in early wood and late wood densities. Therefore, density profiles produced by SilviScan provide annual ring profiles. These profiles are illustrated in Figure 22 [10].

SilviScan technology was also used in one other study funded by the Canadian Forest Service to study the impact on pulp quality of increased pine content in spruce-pine-fir chip mixtures due to increased salvage harvesting of currently attacked and dead lodgepole pine stands. SilviScan fibre diameter measurements on wood cores from five different sample sites are shown in Figure 23. The results indicate that variation in fibre diameter between different sites is as significant as between species for this sample set [11].

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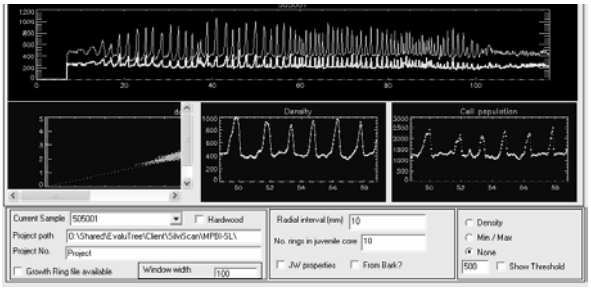

Figure 22. SilviScan density profile for one sample. Delineations between late wood and early wood define annual ring boundaries. A section of this profile is highlighted in the two right most windows below the profile. These windows show density and cell population (which correlates with density) around the 54 millimetre section of the core.

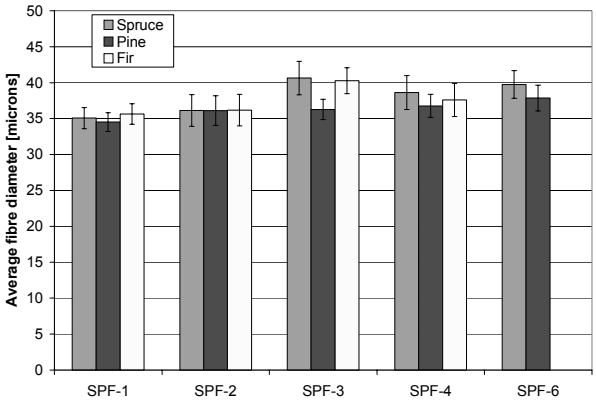

Figure 23: Average diameter of wood fibres (60 to 80 years) measured by SilviScan. The error bars represent the 95% confidence interval.

Spectroscopy

EvaluTree's in-house spectroscopy capabilities include Fourier-transform infrared (FT-IR), Fourier-transform near infrared (FT-NIR), dispersive near infrared (NIR), FT-IR microscopy, dispersive Raman and FT-Raman, as well as a range of traditional spectrophotometry techniques such as UV-Visible and fluorescence technologies.

Infrared absorption arises from molecular absorption of radiation. Of particular importance is that the

absorption is frequency specific. Each molecule and molecular bond has a specific bond-length and bond energy. To be infrared active, the bond frequency must couple or match with the specific excitation frequency for absorption to occur. Furthermore, to be infrared active, molecules must exhibit a change in dipole moment. As such, absorption peaks for each functional group and each molecule are wavelength specific. By scanning over a range of frequencies, infrared spectroscopy can provide simultaneous information on chemical compositions of samples.

The infrared spectral region encompasses wavenumbers ranging from 13,000 to 10cm^{-1}. The infrared spectrum is conveniently divided into near (12,800 to 4000cm^{-1}), the fundamental mid-IR (4000-200cm^{-1}) and far-IR (200 to 10cm^{-1}). Absorption bands in the near-infrared region are overtones or combinations of the fundamental vibration bands pertaining to that of C-H, N-H and O-H. Mid-infrared spectral region is used largely for qualitative analysis and functional group identification, based on absorption and emission spectra. The far infrared region has more utility for studying inorganic compounds due to strong Vibrational modes associated with metal atoms and, both, inorganic and organic molecules.

Raman spectroscopy is a scattering process whereby a laser photon of well-defined energy is scattered off a molecule; a small amount of energy is lost to a molecular vibration, and the resulting spectrum exhibits a shift from the laser line. This shift is referred to as the Raman shift and the corresponding plot of frequency against intensity is the Raman spectrum. For Raman spectroscopy, molecules that exhibit strong Raman signals are those that have strong polarizability. For example, molecules with no dipole moment (N2, C6H12) and those conjugated bonds (aromatic rings and carbon double bonds) have strong Raman bands [13].

Spectroscopy Applications

The traditional methods employed for measuring wood chemistry, moisture content, and many other wood properties can be time consuming and costly. NIR and Raman spectroscopies, when correlated with standardized data from traditional analyses using Chemometrics, can provide an opportunity to measure wood quality rapidly, cost-effectively and comprehensively.

Using visible-light Raman spectrometry, a method for determining the chemical composition of solid wood samples, such as total lignin content, Klason lignin, cellulose, hemicellulose and carbohydrates content, independent of species variation and fluorescence intensity, has been developed. Thermo Galactic's GRAMS/32 AI Chemometric software package and the partial least squares algorithm was used to build calibration models correlating spectral features to native wood components.

Figure 24 illustrates a typical Raman spectrum for hybrid poplar showing strong lignin absorption at 1600cm^{-1} wave numbers. Figure 25 compares the results obtained for total lignin content of 27 native poplar wood cores as measured by standard wet chemical analysis versus Raman spectrometry [15]. A strong correlation is observed with an R^2 of 0.90 and a standard error of prediction (RMSEP), for an independent validation dataset, of 0.5%. This technique was further employed for determining a dataset of 700 wood core samples for total lignin content.

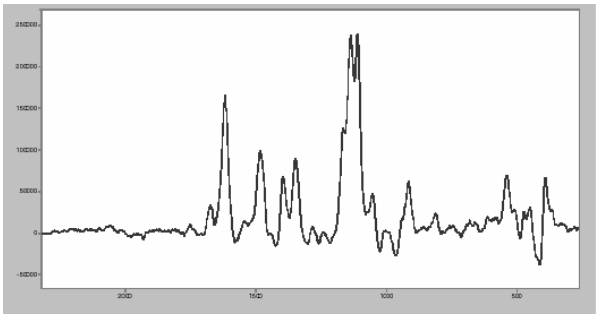

Figure 24. Raman spectrum of hybrid poplar.

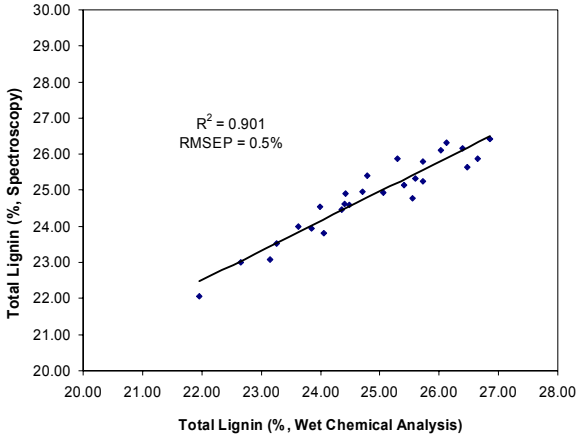

Figure 25. Validation results for total lignin content of hybrid poplar.

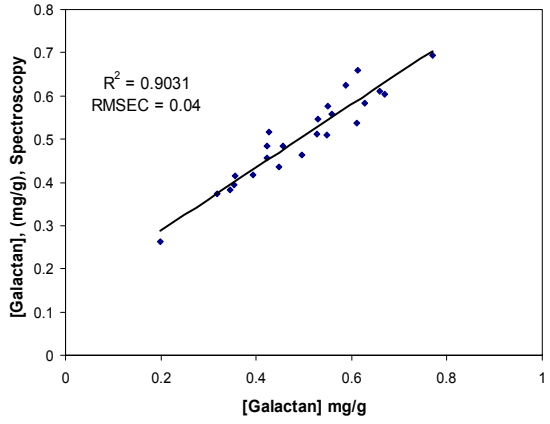

Figure 26. Calibration plot of Raman spectral data to concentration of galactan obtained with PLS2.

Figure 26 shows results obtained from native poplar wood cores of Raman measured galactan content versus galactan content measured by chemical analysis. A strong correlation is also obtained with a R^2 of 0.90 and a standard error of calibration of 0.04mg/g. Figure 27 summarizes the correlation between Raman spectrum and other wood constituents. It is conceived that, given the proper calibration, Raman spectroscopy could be used for rapid assessment of a multitude of fundamental wood chemistry properties that could provide insights into forest practices.

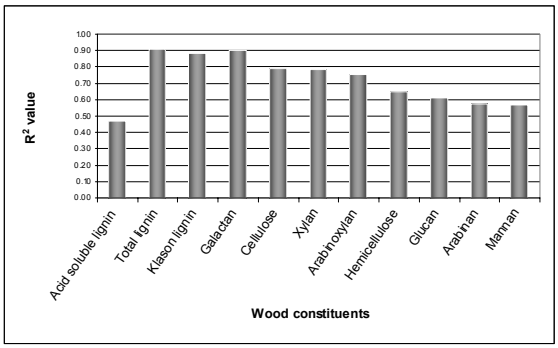

Figure 27. Correlation (R^2 values) between Raman spectrum and wood constituents.

As discussed previously, the mountain pine beetle epidemic in north-central BC poses significant challenges to the forest products industry. Visual indicators can be used to estimate time since attack for a tree or tree stand, however, they are not a reliable predictor of wood and fibre quality. Moisture content, bluestain, decay and density content are important determinants of the value of beetle infested wood. Paprican, in collaboration with colleagues in the U.S., developed a portable field tool based on visible-near infrared (Vis-NIR) spectroscopy. The tool is currently being tested to quantify parameters such as moisture content, density, blue stain, and decay in MPB infested lodgepole pine trees and decked logs. Using multivariate analysis, calibration models were developed to simultaneously measure moisture, density, blue stain and decay for field and laboratory applications. A summary of the results obtained on decked logs is shown in Table 1. The model was developed using 19 mountain pine beetle attacked trees and then validated using 8 trees (Figure 36). Strong correlations between the Vis-NIR model predictions and values obtained using standard laboratory technique for moisture content (R= 0.94), blue stain (R=0.93) and decay (R=0.88) were achieved. Furthermore, small standard errors in prediction (SEP) compared with standardized testing demonstrate that Vis-NIR spectroscopy could be used to measure these parameters rapidly and with good reproducibility and accuracy. Although density measurements using Vis-NIR spectroscopy are less accurate than the standard laboratory method, the ease of use and speed of measurement may make it useful for measuring density trends in tree stands [14].

Table I. Calibration and validation results for the vis-NIR model developed to measure wood properties on mountain pine beetle decked logs.

	R (Cal)	SEC	R (Val)	SEP	Error in Ref Method
Moisture	0.92	1.9%	0.94	1.3%	1.3% (Marrs)
Basic Density	0.53	35 kg/m^3	0.63	31 kg/m^3	5.6 kg/m^3 (TAPPI)
Blue Stain (D-b*)	0.95	1.3	0.93	1.4	0.6 (TAPPI)
Decay (1% Caustic Solubility)	0.9	2.1%	0.88	2.6%	2.0% (CPPA)

HiRes Fibre Quality Analyzer

The Fibre Quality Analyzer (FQA) was developed jointly by Paprican, the University of British Columbia, and OpTest Equipment Inc. It measures fibre length, shape (kink and curl) and average coarseness on dilute fibre suspensions made from macerated wood cores or pulp. Latest development efforts have resulted in a new high resolution (HiRes) FQA that can also simultaneously measure fibre width. With the addition of width measurement, the HiRes FQA can now measure shives and can detect vessel elements. EvaluTree's HiRes FQA is also equipped with the AutoFeed option that allows users to perform fiber analyses on up to 99 samples automatically with continuous running. When combined with a chemical maceration method (using acetic acid and hydrogen peroxide) to liberate fibres directly from wood cores, the HiRes FQA with AutoFeed makes it possible to measure fibre length efficiently on large sample sets of wood cores. Figure 28 shows fibre width distributions on five hardwood pulps, including three different eucalyptus hybrids from Brazil.

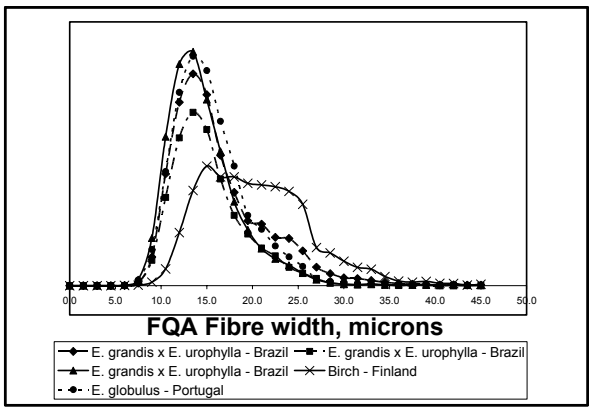

Figure 28. Fibre width distributions measured on fines-free unbleached hardwood kraft pulps using the HiRes Fibre Quality Analyzer (FQA).

CONCLUDING REMARKS

New developments in optical-based, rapid assessment techniques for wood and fibre quality make it easier that ever before to conduct high-throughput wood quality evaluations on statistically significant sample sets. In many cases the testing can be conducted on wood cores extracted from standing trees, eliminating the need to destructively sample the trees. A suite of these state-of-the-art wood and fibre analysis technologies being applied at Paprican's EvaluTree division to support research ranging from the impact of genetics on wood quality, to assessing utilization of fibres for optimal end-use performance. These technologies include SilviScan® for laboratory measurement of pith-to-bark radial profiles on wood cores, the latest in confocal and environmental scanning electron microscopy (ESEM) for advanced imaging of fibre performance in products, the HiRes Fibre Quality Analyzer for fibre length, width, shape and coarseness, and optical spectroscopy techniques for wood and chemical properties.

These combined technologies provide the capability to assess wood and fibre quality and gain new insights into wood and fibre science, including genetic and silvicultural methods to better control wood characteristics in the forest. For example, in Brazil, the well-recognized success of eucalyptus market pulps is the result of a great deal of effort over many years, to develop highly productive forests using tools such as forest genetic improvements and forest management techniques. As manufacturers look to the future, it will be important to ensure that cost and quality advantages are maintained, while at the same time monitoring possible competitive fibres such as acacia. Since superior genetic clones cannot be simply transferred to new regions, foresters can benefit from rapid means of evaluating genetic material for specific growth conditions or quantifying the impact of silvicultural practices on forest productivity and fibre quality.

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