In Situ Chemical Mapping of Wood to Visualize Changes Occurring During Viscoelastic Thermal Compression

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Research Objective

Develop an improved method for characterizing and understanding the chemical changes occurring at the wood surface after hot pressing.

Method Background

As a complex, heterogeneous, solid composite, wood is a challenging matrix to chemically analyze. As wood composites, resins, and coatings continue to gain importance and become more advanced, this challenge must be met using techniques capable of both in situ analysis and high resolution at a micrometer scale. Fourier-transform Infrared (FTIR) Microscopy is an enabling technology to support these techniques. This research utilized an FTIR microscope for cross-sectional, surface down chemical analysis of viscoelastic thermally compressed (VTC) veneers. Wood is a very strong absorber of infrared energy and chemical changes on the wood surface might be only a few micrometers deep, so performing FTIR microscopy on VTC wood to generate surface down chemical maps required considerable method development.

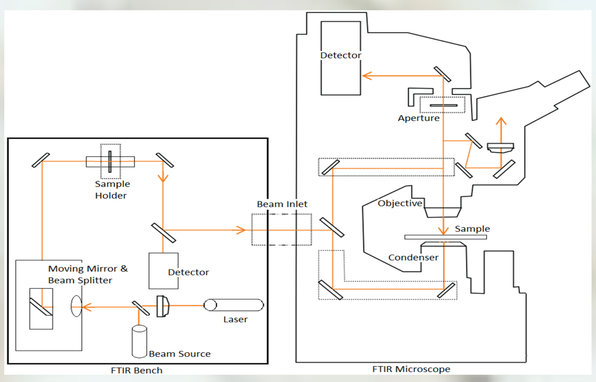


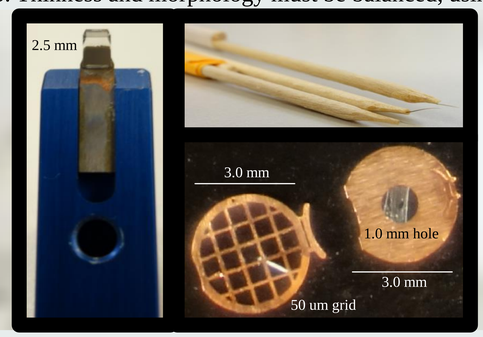
Figure 1: FT-IR microscope diagram. ThermoFischer Nicolet Continuum FTIR microscope and Thermo Nicolet Nexus 470 FTIR bench were utilized.. Using a 50 µm mercury cadmium telluride (MCT) detector and a 32X objective, the Thermo-Fisher Continuum IR Microscope is able to advance the traditional diffraction limit of 10 µm and achieve a spot size of 5 µm. The 5 µm resolution is important because the thickness of wood cell walls generally lies in the range of 3-8 µm.

Figure 2: Viscoelastic thermal compression (VTC) press. Viscoelastic thermal compression of wood involves 4 steps; steam & heat, compress, heat & cool, release. The heating range is ~ 100-250 ˚C, the steam pressure is ~ 150-250 psi, the press pressure is ~ 300-1000 psi and the time duration of processing is ~ 10-15 mins. Analysis was focused on VTC wood because it was representative of an intensive wood modification process that introduced no new chemical variables.

Thin

Samples need to be ≤ 500 nm thick (diamond knives) and thin samples have better signal/noise ratio. Thinness and morphology must be balanced, using latewood helps

Small

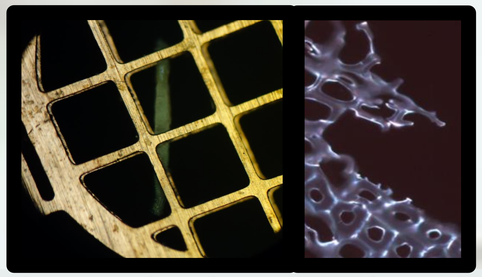


Samples can't be larger than diamond knife and must fit on transmission electron microscopy (TEM) grid - better than KBr windows. Mapping large samples at µm scales can be risky

Purity

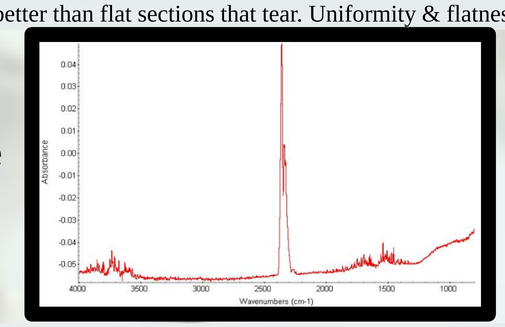
Signal/noise ratio better with less variables. Extractives add variables. Embedding and cell wall bulking add non-wood variables (Cryo-ultramicrotomy very helpful)

Morphology

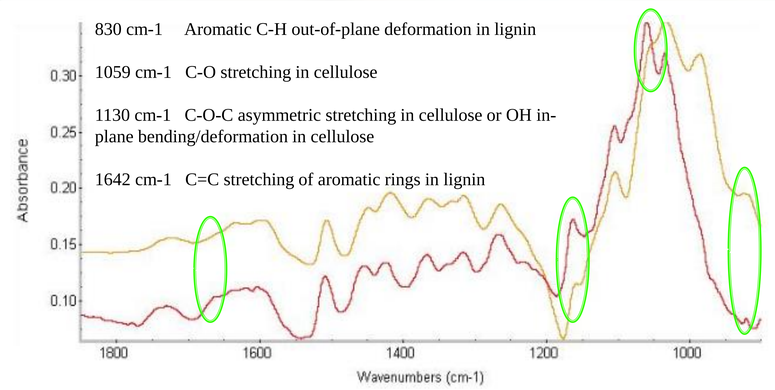


Sample shape affects absorption and signal/noise. Surface down analysis makes edges key; rolled-up sections better than flat sections that tear. Uniformity & flatness vs. energy & scale

Appropriate Reference



Must consider if any part of sample is unmodified. 2400-2300 cm-1 is good reference region for morphology related absorption. Frequent background scans important at small scale



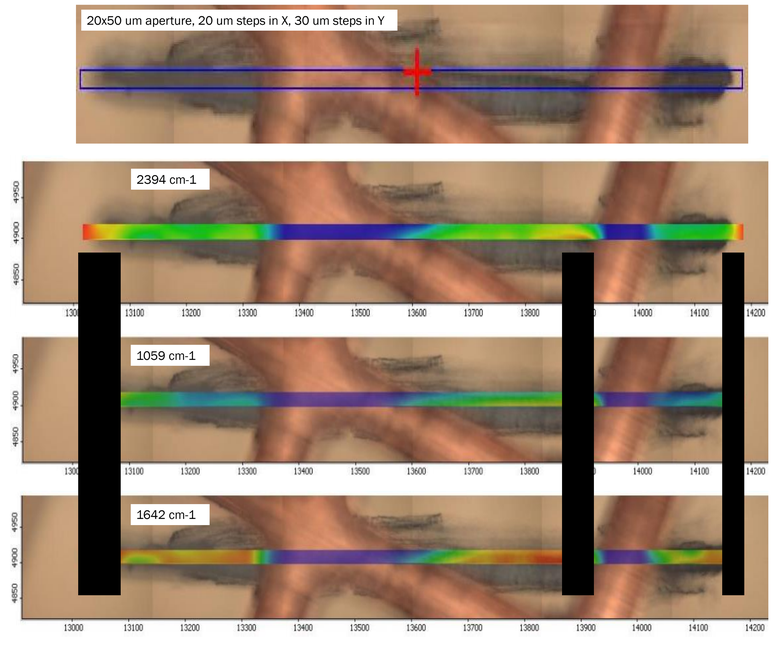


Figure 3: Images of a rolled 250 nm thick Douglas-fir section positioned inside a 50 um TEM grid. Progression (top to bottom) shows FT-IR microscopy images of the background (2394 cm-1), a cellulose specific region (1059 cm-1), and a lignin specific region (1642 cm-1). Blue = strong absorption, Red = weak absorption. Some progression from the outside in can be seen for cellulose, with the outside edges showing the weakest cellulose related absorption. Very little progression is seen with lignin, suggesting more global changes from processing.

Method Development Lessons

- Ways of reducing signal/noise are important and image is sum of these efforts.
- Oversampling does not particularly harm analysis and tends to support higher resolution maps.
- Every effort to increase beam energy is worth it and interferometer and detector alignment must be constantly checked at this scale.
- There is no perfect scenario and there are always trade-offs between sample morphology and signal/noise.
- The mid-IR beam is strongly absorbed by wood and sets practical limits on aperture size.

Acknowledgments

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