

## Thermal Compression of Hybrid Poplar: Analysis of Extractable Components after Treatment

Noridah Osman<sup>1</sup>, Armando G. McDonald<sup>1</sup> and Marie-Pierre Laborie<sup>2</sup>

<sup>1</sup>Department of Forest Products, College of Natural Resources, University of Idaho, Moscow ID 83843-1132, USA [e-mail: noridah.osman@vandals.uidaho.edu; armandm@uidaho.edu]

<sup>2</sup>Wood Materials and Engineering Laboratory, Washington State University, Pullman WA, USA

**Keywords:** GC-MS analysis, FT-IR spectroscopy, HPLC analysis, hot-pressing, hybrid poplar, thermal treatment, wood extracts

### ABSTRACT

Thermal modification or heat treatment on wood is common to wood composites industry. Nevertheless, research and development on this aspect keep on continuing to cater the demand from wood production and the environment. The study of non-durable hybrid poplar species has become important as it serves multiple purposes such as in wood-engineered oriented strand board. The chemical effects of a thermal compression treatment on hybrid poplar wood, namely extractable constituents were studied. Poplar veneers (0 and 8% moisture contents) were pressed at 3.45 MPa compression pressure at 150, 200 and 250°C for 5 minutes. Several changes in the composition of the extractives (aqueous and organic soluble) were observed by conventional chemical methods (HPLC and GC-MS) and spectroscopic analysis (FT-IR). Although the trend of effects and behaviours of heat treatment on wood chemical and structure will be very much similar to other literatures, the magnitudes and modification are expected to be slightly different. In the paper the thermal modification on hybrid poplar wood controlled by temperature and moisture content governed by hot-pressing are presented and discussed.

### INTRODUCTION

Thermal treatment is common practice in manufacturing wood-composites and other engineered wood products. Thermal treatment by hot-pressing involves heat and chemical transformation processes that modify colour, density, dimensional stability and durability of the wood as their chemical properties change (Tjeerdsma and Militz 2005, Boonstra *et al.* 2006). During mat consolidation the temperature can range between 140 and 210 °C and the quality of the panel is governed by the press cycle and moisture content. Because hybrid poplar wood is a non-durable species due to its fast and high-survival growth rates in a wide range of conditions, and multiple uses in wood-engineered industries (Englund 2008), it has been widely chosen in improving end product performance (Stanton *et al.* 2002, Semple *et al.* 2007). The chemical changes in this low-density wood depend upon the treatment applied, and a chemical composition is considerably altered by elevated temperature and during hot pressing (Winandy and Krzysik 2007). The aim of this study is to investigate the effects of thermal compression on the chemical properties of hybrid poplar wood as a function of temperature and moisture content. Chemical changes will be determined by classical chemical composition methods for extractives, lignin and carbohydrates in conjunction with FTIR spectroscopic analysis.

## EXPERIMENTAL

**Materials;** hybrid poplar wood veneers (10 cm x 10 cm, ten replicates) from clone OP-367 (crossed between *P. deltoides* × *P. nigra*; Potlatch Corporation) was conditioned to 0 and 8% moisture content (MC) prior to pressing. The veneers were pressed on Wabash (50 ton, 45 cm x 45 cm) hot-press at 150, 200 and 250°C for 5 minutes at 3.45 MPa pressure. The control and hot-pressed samples were Wiley Milled to pass through a 0.5 mm screen.

**GC-MS Analysis;** Dichloromethane (DCM) organic-soluble extracts of pressed and control veneers were analyzed by GC-MS as their trimethylsilyl (TMS) by gas chromatography-mass spectrometry (GC-MS) on a PolarisQ instrument in the electron impact mode. The temperature profile was from 40 to 300 °C at a ramp rate of 5 °C/min. Data was analyzed using the Xcalibur software package.

**HPLC Analysis;** aqueous extracts from DCM extractives-free hybrid poplar wood components were subjected to hydrolysis in 2M trifluoroacetic acid (TFA) for 3 hr at 105 °C. The hydrolyzed extracts were analyzed for monosaccharides by HPLC using two Rezek RPM columns (90 °C) in series using water as eluent (0.5 mL/min) with refractive index detection.

**FTIR Spectroscopic Analysis;** on aqueous and DCM extracts were performed in the Attenuated total reflectance mode (Smart Performer) on a ThermoNicolet Avatar 370 spectrometer and data analyzed using OMNIC v7 software.

## RESULTS AND DISCUSSION

**Analysis of water extracts** from control and hot-pressed veneers showed the presence of hemicelluloses as judged by monosaccharide composition (mainly glucose and xylose, with minor amounts of galactose mannose and arabinose) as shown in Table 1 (Aimi *et al.* 2005). The yields of the water-soluble extract were shown to increase as pressing temperature increased (Brito *et al.* 2008). This may be due to the partial hydrolysis and cell wall disruption which eased the release of xylan and glucan.

*Table 1: Hybrid Poplar Thermal Compression Wood Extractives (aqueous and organic-soluble)\**

Sample, MC %, Temp. °C	Control	0, 150	0, 200	0, 250	8, 150	8, 200	8, 250
Water Extract Yield [%] <sup>a)</sup>	2.36	2.41	2.67	3.76	2.49	3.35	3.31
Glucan [%] <sup>b)</sup>	0.83	0.87	0.33		0.97	0.13	0.1
Xylan [%] <sup>b)</sup>	0.35	0.3	0.35	1.32	0.29	0.56	1
DCM Extract Yield [%]	0.65	0.74	0.9	7.93	0.68	0.97	9.9

\* relative composition/average, <sup>a)</sup> water-soluble polysaccharides in mg/g wood, <sup>b)</sup> Sjöström, E.

**Analysis of the DCM extracts** showed that the yield increased as pressing temperatures increased (Table 1). GC-MS analysis identified several classes of compounds which were free phenolic acids, fatty acids and diglycerides (Figure 1). The main compounds identified were benzoic acid, benzaldehyde, *p*-hydroxybenzoic acid, vanillic acid and glycerol and they were derived from lignin component (Kamdern *et al.* 2000, Fernandez *et al.* 2001). Significant variations in the content of these compounds were observed after hot-pressing as shown in Figure 1 (Windeisen *et al.* 2007).

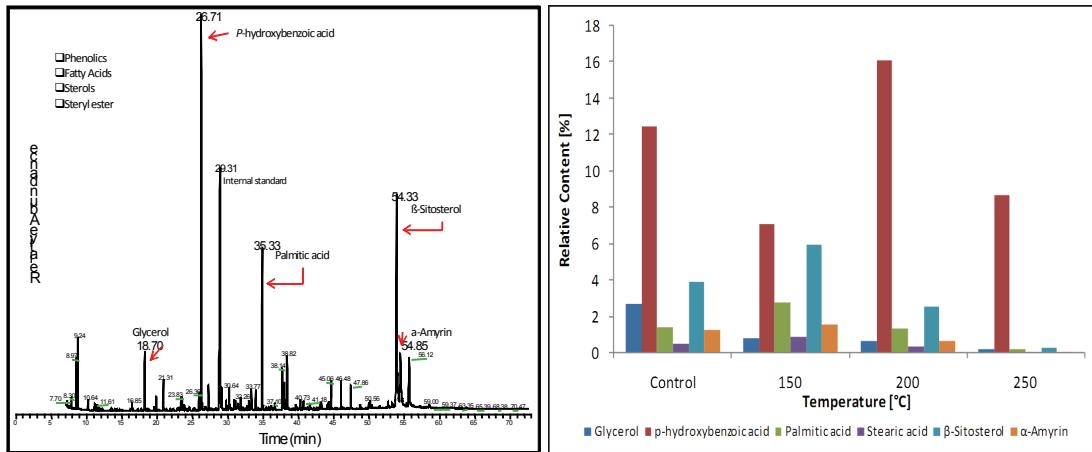


Figure 1: GC-MS chromatogram (left) and Composition (right) of DCM Wood [0%MC]

**FTIR spectroscopic analysis** of both aqueous and DCM extracts (Figure 2) showed bands in the finger print region. The water-soluble extractives spectra had peaks at 1730, 1380, 1044 and 1112  $\text{cm}^{-1}$  attributed to polysaccharides with special attention paid to the absorption of hemicelluloses which occurs around 1730  $\text{cm}^{-1}$  (Tjeerdsma and Militz 2005). The DCM extractives spectra showed peaks at 1606/1593, 1514, 1269, 1211 and 1110  $\text{cm}^{-1}$  that were assigned to wood resins and lignin. The spectra did differ significantly among the hot-pressed and control wood specimens. The low temperature samples showed minor chemical changes (e.g. reduce degree of polymerization, some degradation to form aldehydes, lignin cleavage) however, as the temperature increased under dry and moist condition major chemical changes were observed (e.g. auto-condensation, polymerization reactions occurs).

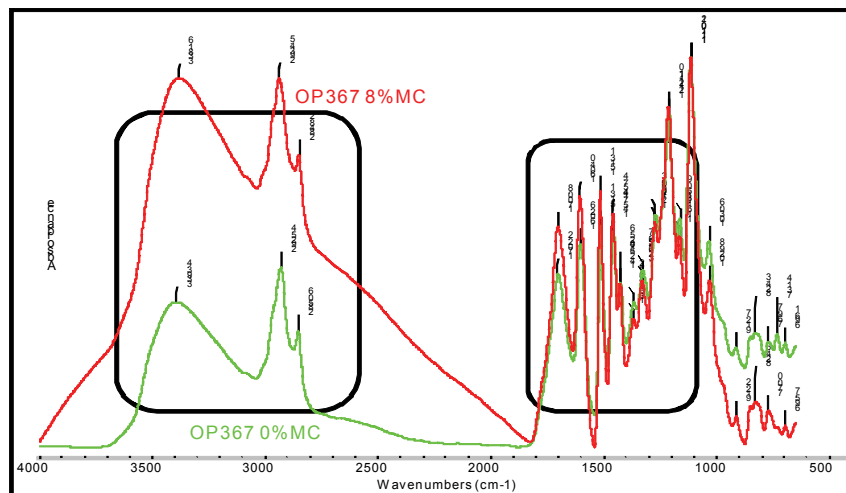


Figure 2: FTIR spectra of DCM extracts (250 °C treated-sample conditions)

## CONCLUSIONS

The temperature has a major effect on wood degradation during hot-pressing. At high temperatures the aqueous and organic-extracts found were mainly solubilized hemicelluloses (xylan and glucan) and lignin derived components, respectively.

## ACKNOWLEDGEMENT

This project was supported by the USDA-CSREES-National Research Initiative grant number 2005-35103-15277.

## REFERENCES

- Aimi, H., Matsumoto, Y. and Meshitsuka, G. (2005). Structure of small lignin fragments retained in water-soluble polysaccharides extracted from birch MWL isolation residue. *Journal of Wood Science*, **51**, 303-308.
- Boonstra, M.J., Rijdsdijk, J.F., Sander, C., Kegel, E., Tjeerdsma, B., Militz, H., van Acker, J., and Stevens, M. (2006). Microstructural and physical aspects of heat treated wood. Part 2. Hardwoods. *Maderas Ciencia y Tecnologia*, **8**, 209-217.
- Brito, J.O., Silva, F.G., Leao, M.M. and Almeida, G. (2008). Chemical composition changes in eucalyptus and pinus woods submitted to heat treatment. *Bioresource Technology*, **99**, 8545-8548.
- Englund, K. (2008). Hybrid poplar as a feedstock for wood-plastic composites. *Forest Products Journal*, **58**, 61-65.
- Fernandez, M.P., Watson, P.A. and Breuil, C. (2001). Gas chromatography-mass spectrometry method for the simultaneous determination of wood extractive compounds in quaking aspen. *Journal of Chromatography A*, **922**, 225-233.
- Kamdern, D.P., Pizzi, A. and Triboulot, M.C. (2000). Heat-treated timber: potentially toxic byproducts presence and extent of wood cell wall degradation. *Holz als Roh- und Werkstoff*, **58**, 253-257.
- Semple, K.E., Vaillant, M-H., Kang, K-Y., Oh, S.W., Smith, G.D. and Mansfield, S.D. (2007). Evaluating the suitability of hybrid poplar clones for the manufacture of oriented strand board. *Holzforchung*, **61**, 430-438.
- Stanton, B., Eaton, J., Johnson, J., Rice, D., Schuette, B. and Moser, B. (2002). Hybrid poplar in the Pacific Northwest: The effects of market-driven management. *Journal of Forestry*, **100**, 28-33.
- Sjostrom, E. 1993. Wood Chemistry: Fundamentals and applications (2<sup>nd</sup> Edition). Academic Press, San Diego, New York, Boston, London, Sydney, Tokyo, Toronto.
- Tjeerdsma, B.F., Militz, H. (2005). Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood. *Holz als Roh- und Werkstoff*, **63**, 102-111.
- Winandy, J.E. and Krzysik, A.M. (2007). Thermal degradation of wood fibers during hot-pressing of MDF composites: Part I. Relative effects and benefits of thermal exposure. *Wood and Fiber Science*, **39**, 450-461.