Optical analysis of extractive materials distribution in wood densification

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Abstract. Lignin considered as one of extractive materials in wood densification process. During preheating and in wood densification process, lignin might flow and distributed in a temperature range of 127 °C to 176 °C, and water in wood pores might increase extractive materials flowability in wood. Crossectional extractive materials distribution was measured and quantify optically. Relationships between temperature and extractive materials distribution were discussed briefly in an aim to improve densification methodology.

1. Introduction

Purpose of wood hydrothermal treatment is a modification method to increase dimensional stability; improve performance of preservatives and to reduce equilibrium moisture content [1]. All thermally treatment were used heat and steam that resulted color change in final appearances. Research on measuring color change is an interesting indicator to other wood properties. Color measurement also able to determined wood quality in a world trading. Study about color change by hydrothermal treatment [1-25] is an interesting subject because can give desireable appearence that similar to Tectona gradis [25]. Steam is use frequently because high heat capacity and resulted homogenized temperature distribution inside a wood. However, no water as heat transfer medium of wood softening were used. This paper purpose is to study effect of water impregnated into a wood pores during heat treatment process and to observed extractive material distribution as an effect on variation of preheating time. Infrared spectroscopy also measured to observed effect of hydrothermal treatment to materials characteristics of water impregnated compressed wood. With an exploration on basic interaction of wood and water in liquid form and an aim to improve wood densification methodology.

2. Materials and Methods

2.1. Sample preparation of drying, impregnation, preheating and compression

An Albizia wood (Paraserianthes falcataria (L.) I.C. Nielsen) was used as a sample with dimension of 50 mm × 50 mm × 120 mm. The sample was dried prior water impregnation in an oven (Drying and heating chamber, Model FD 23, Binder GmbH, Tutllingen, Germany) with temperature of 100 °C for 24 hours. After drying sample then impregnated by soaking in a water container inside a vessel (Wood
antiseptic injecting apparatus, Type PV5, Tokyo Clutch Door Co. Ltd, Tokyo, Japan) then supplied with 7 kg/cm² compressed air (Air Compressor, Model 2.2P – 14V, Hitachi, Ltd., Tokyo, Japan) for 24 hours.

Water in wood pores is decreased glass transition temperature \( T_g \) of hemicellulose and lignin [ ]. Melting temperature of low and high molecular weight of lignin is 127°C to 176°C [26]. Beside reduce \( T_g \), water existence in wood pore might increase wood extractive flowability of hydrothermal compressed wood.

To observed and identify above phenomena, a water impregnated wood then preheated between a hot press plates (Compression Molding Machine, NF- 50-HH, Shinto Metal Industries Ltd., Osaka, Japan) in a dies and punch with temperature of 176 °C for one, two and three hours of heat exposure time. Sample temperature was measured by a thermocouple type K and read using a panel meter (Omega, DP462, Omega Engineering, Inc., Norwalk CT, USA) in a small hole with same diameter of thermocoule wire at the center side of radial direction that parallel to compression direction. Then followed with densification until compression ratio of 0.87 and cooled in natural convection to room temperature. After cooled condition achieved the sample then removed from compression dies.

2.2. Image acquisition, measurement and data presentation
Crossectional extractive distribution was identified optically by measure color intensity using a flat-bed scanner (Canon PIXMA MP 287, Canon Singapore Pte. Ltd.). Left, right, top and bottom side of a sample without front and rear side were scanned and saved in a portable network graphics (.png) format. The grayscale distribution was measured using ImageJ software (National Institutes of Health, Bethesda, MD, USA). Measurement was taken at the centerline with direction from bottom to top surface at left side for one hour preheating and top to bottom surface at right side for two and three hour preheating. Results of maximum grayscale intensity was ploted in abssica and ordinate respectively for preheating time and its location from top to bottom direction.

2.3. Spectroscopy sample preparation, measurement, data processing and presentation
Characteristics of extractive materials was identified using FTIR spectrometer (Spectrum Two, Perkin Elmer, Waltham, MA, USA). Untreated wood, pale area and darker area of hydrothermal treated wood samples was prepared using screen mesh of 60 to 80, prior identification. Measurement for each sample were repeated with \( n = 8 \). Spectrum data of every wavelength then compared using student-t test and significance wavelength data were analyzed to identify extractive characteristics that related to color-change distribution along crossectional direction of compressed wood.

3. Results and Discussion
Hydrothermal treated compressed wood of one, two and three hour preheating are shown in a row of Fig. 1. Some darker area are appeared around center of every side. In brief, dark colored extractive appeared at top and bottom side of one hour of hydrothermal treatment. However, in two hours treatment darker area is distributed around bottom surface and for three hour treatment almost all darker area is appear at top side of hydrothermal compressed wood.

3.1. Results of one to three hours preheating
Gray value measurement as represented in Fig. 2 that indicate distribution of the darker extractive along radial direction of wood compression. Peak of intensities and indicated location are marked with ellipse for each row. Location of one hours peak intensity was measured from bottom to top side with peak intensity around 1 mm from bottom surface. However for two and three hour hydrothermal treatment were measured from top to bottom side. Location of two and three hours peak intensity is at 3 mm and 1 mm from top surface, respectively.
3.2. Extractive distribution

Relationships between hydrothermal preheating time of compressed with distribution of extractive materials is shown in Fig. 6. Increase of preheating time will increased movement of extractive materials in opposite direction to gravitational direction. This phenomena might related with combination of capilarity force and surface tension of liquid at wood vessel and pores of compressed wood. Additional heat flux inside the wood also might increase viscosity and flowability of extractive solution.

![Figure 1](image1.png)  ![Figure 2](image2.png)

**Figure 1.** Hydrothermal compressed wood  
**Figure 2.** Gray value measurement
Figure 3. Effect of preheating duration time. 0 to 4.5 on vertical axis indicated top and bottom surface, respectively.

3.3. Results of FTIR measurement

Figure 4. FTIR shift of control, pale, and dark extractive samples shown in order from top to bottom curves.

Characterization using FTIR is shown in Fig.4. Results was shown that dark extractive materials is shifted from control spectra. The area of interest is hydrogen bonding which can improve mechanical properties of a compressed wood [27] as presented in Table 1.
### Table 1. IR Absorption of a Hydrothermal Compressed Wood

| Assignment [28]                                                                 | Wave-numbers (cm⁻¹) | Control | Pale | Dark |
|--------------------------------------------------------------------------------|----------------------|---------|------|------|
| O-H out of plane bending in COH alcoholic groups                                | 665                  | 0.069   | 0.058| 0.027|
|                                                                                 | 705                  | 0.055   | 0.047| 0.022|
| C-O stretching mainly of C₆H₂-O₆H primary alcohols (secondary conformation)     | 1000                 | 0.100   | 0.085| 0.040|
|                                                                                 | 1015                 | 0.114   | 0.094| 0.042|
|                                                                                 | 1035                 | 0.124   | 0.102| 0.040|
| C-O stretching mainly of C₃O₃H secondary alcohols                               | 1060                 | 0.109   | 0.088| 0.039|
| C-O-C stretching (ring mode)                                                     | 1075                 | 0.085   | 0.068| 0.030|
| C-O stretching mainly of C₂O₂H secondary alcohols                               | 1115                 | 0.072   | 0.059| 0.027|
| C-O-C stretching glycoside                                                       |                      |         |      |      |
| Mainly antisymmetric                                                             | 1160                 | 0.048   | 0.040| 0.018|
| Symetric                                                                         | 1205                 | 0.034   | 0.029| 0.014|
|                                                                                 | 1315                 | 0.036   | 0.031| 0.016|
| O-H(…) stretching (in-plane bending) of C-O-H alcohol groups                    |                      |         |      |      |
| mainly C₂O₂-H secondary alcohols                                                | 3275                 | 0.032   | 0.028| 0.011|
| mainly C₆H₂O₆-H primary alcohols (minor conformation)                            | 3305                 | 0.034   | 0.029| 0.012|
| mainly of C₃O₃-H secondary alcohols                                             | 3340                 | 0.035   | 0.030| 0.012|
| mainly of C₆H₂O₆-H primary alcohols (dominant conformation)                    | 3410                 | 0.031   | 0.026| 0.010|
| O-H(…) stretching of alcohols establishing weak hydrogen bonds or perhaps no hydrogen bonds. These alcohols are mainly C₂O₂-H secondary alcohols. | 3450                 | 0.026   | 0.022| 0.008|

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