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Structural analysis of heat-treated birch (*Betule papyrifera*) surface during artificial weathering

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ABSTRACT

Effect of artificial weathering on the surface structural changes of birch (*Betule papyrifera*) wood, heat-treated to different temperatures, was studied using the fluorescence microscopy and the scanning electron microscopy (SEM). Changes in the chemical structure of wood components were analyzed by FTIR in order to understand the mechanism of degradation taking place due to heat treatment and artificial weathering. The results are compared with those of the untreated (kiln-dried) birch. The SEM analysis results show that the effect of weathering on the cell wall of the untreated birch surface is more than that of heat-treated samples. The FTIR spectroscopy results indicate that lignin is the most sensitive component of heat-treated birch to the weathering degradation process. Elimination of the amorphous and highly crystallised cellulose is observed for both heat-treated and untreated wood during weathering. It is also observed that heat treatment increases the lignin and crystallised cellulose contents, which to some extent protects heat-treated birch against degradation due to weathering.

1. Introduction

The weathering of untreated wood causes roughening and cracking of wood surface and damages its microstructure. Untreated wood exposed to outdoor weathering undergoes checking and surface erosion principally due to the effects of solar radiation and stresses imposed by cyclic wetting, temperature changes, environmental pollutants, and certain micro-organisms [1]. A number of researchers have examined the effect of weathering on the physical structure of wood [1–8]. Microscopic studies showed characteristic ridges on the S3 wall layer, wall checking, ray and pit degradation, and middle lamella breakdown. Several publications describe similar observations related to microscopic changes found on untreated wood surfaces which were artificially weathered by exposure to UV irradiation [9–11]. Changes observed on the wood surfaces due to artificial weathering were very similar to those caused by natural outdoor weathering [12]. The effect of weathering on the chemical structure of wood was also studied by means of FITR spectroscopy [13]. These studies generally have been carried out for untreated wood.

High-temperature heat-treated wood is a relatively new product which is heated to high temperatures in the range of 180–260 °C, depending on the species used and the desired material properties [14]. A few researchers have studied the microstructural properties of heat-treated wood by means of SEM. Boonstra et al. [15,16] found that heat treatment had an effect on the anatomical structure of wood, and the extent of this effect depended on the wood species and the treatment method and conditions used. Softwood species with narrow annual rings which had an abrupt transition from earlywood to latewood were sensitive to tangential cracks in the latewood section. Radial cracks occurred mainly in impermeable wood species such as Norwegian spruce, caused by large stresses in the wood structure during treatment. Sapwood of treated pine species revealed some damage to parenchyma cells in the rays and epithelial cells around resin canals while this phenomenon was not noticed in the heartwood section [16]. However, it was found that the anatomical structure of wood was only slightly affected during heat treatment [17]. Vessels, fibers, parenchyma, and rays were still intact after the heat treatment. The main effect of heat treatment was the presence of significant quantities of extractives deposited in the vessels, which normally disappear after heat treatment. Heat treatment changes both chemical and physical properties of wood. The decrease of amorphous polysaccharide content (hemicelluloses), condensation and demethoxylation of lignin, and the removal of certain extractives occurring due to heat treatment at high temperatures were reported by different researchers [18–20]. As a result, heat-treated wood possesses new physical properties such as reduced hygroscopy, improved dimensional stability, better resistance to degradation by insects and micro-organisms, and most

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importantly, attractive darker color. The new versatile properties make heat-treated wood popular for outdoor applications.

As explained above, various studies were carried out on the structural analysis of untreated and heat-treated wood, and weathering of untreated wood. Most of the previous studies on structural changes due to weathering of heat-treated wood were limited to the study of physical structure investigated using SEM. To the authors' knowledge, although there are several previous studies on the effect of weathering on the surface properties (such as changes in wettability) of heat-treated woods [21–23], a detailed and comprehensive study of physical and chemical structural changes of heat-treated wood including fluorescence microscopy, SEM, and FTIR are not available in the literature and structural changes of heat-treated wood due to weathering are not completely understood. Furthermore, there is no published study available on the structural analysis of wood that is heat-treated under different heat treatment conditions and then subjected to weathering. The present study aims to fill this void.

The objectives of this study are to investigate the detailed physical micro-structural variations and chemical, structural changes the different components of heat-treated wood undergo due to accelerated artificial weathering and to identify the relation between the physical structural changes and the chemical degradation of wood components. The effect of different heat treatment conditions (such as temperature) on the artificial weathering degradation process was also studied. The type of wood studied is the hardwood birch (*Betule papyrifera*). The birch wood is popular in the North American market, especially in Quebec, and the heat-treated wood has diverse outside uses.

2. Materials and methods

2.1. Materials

Birch (*Betule papyrifera*), a hardwood that is commonly used for outdoor applications in North America, was studied. Heat-treated birch boards were subjected to artificial weathering. Untreated wood boards, kiln dried to a final moisture content of about 12%, were also exposed to artificial weathering along with heat-treated specimens for comparison purposes. Specimens were arbitrarily selected for a complete statistical randomization. They were stored in a room at 20 °C and 40% relative humidity (RH) until they were exposed to artificial weathering and then characterized using the tests described below.

Specimens of 70 mm × 65 mm cross-section on longitudinal surfaces and 20 mm in length were cut from sapwood of heat-treated and untreated wood, and then planed to have smooth surfaces. The prepared specimens were used in artificial weathering tests. The color evaluation was carried out on both longitudinal tangential surface (LT) and longitudinal radial surface (LR) of the wood samples.

2.2. Artificial weathering tests

Artificial weathering tests were conducted at the Laval University in collaboration with FPInnovation. The samples were exposed to UV light in a commercial chamber, Atlas Material Testing Technology LLC (USA) Ci65/Ci65A Xenon Weather-Ometer. A controlled irradiance water-cooled xenon arc with a CIRA inner filter and a Soda outer filter was used as the source of radiation to simulate sunlight. Tests were performed according to Cycle 1 of Standard ASTM G155: 102 min Xenon light, 18 min light and water spray (air temperature is not controlled) without dark cycle to simulate rain in natural weathering. The black panel temperature was set to 63 ± 3 °C and the irradiance level was 0.35 W/m^2 at 340 nm. Heat-treated samples and untreated control samples were exposed to UV light. The irradiation was interrupted after 72, 168, 336, 672, 1008, and 1512 h of exposure, and two samples for each set of experimental conditions were taken out for evaluation of changes in chemical and physical structure.

2.3. Fluorescence microscopy analysis

Structural properties of untreated and heat-treated wood before and after weathering were investigated. Transverse sections cut through wood at a thickness of 7–20 μm were examined and photographed with the Nikon eclipse E600 microscope. Some sections were also stained with 0.05% aqueous toluidine blue prior to examination with the photomicroscope. Light microscopy was also undertaken on sections that had been stained with 1% aqueous osmium tetroxide (OsO4) for 10–30 min at room temperature. Sections for scanning electron microscopy were cut from the same blocks from which the sections for light microscopy were taken.

2.4. Scanning electron microscopy (SEM) analysis

Small wood blocks measuring 20 mm × 20 mm on the weathered tangential face were cut from heat-treated and untreated boards after artificial weathering for different times (0, 336, 672 and 1512 h). For subsurface cell degradation analysis, same blocks measuring 20 mm × 10 mm on the transverse face and radial face were used. The specimens were immersed in water for 30 min. Then, one of the end-grain surfaces and one radial surface were carefully cut with a razor blade mounted onto a microtome. A new razor blade was used for each final cut. Another method of preparation for SEM analysis is to split wood samples. However, these surfaces were rough, and they usually did not allow observation of the cell lumen. The specimens were washed in distilled water to remove the bleaching agent and then air-dried at room temperature for more than two nights, and desiccated with phosphorus pentoxide for 10 days. Finally, all blocks were sputter-coated with a palladium/gold layer (20 nm) and then mounted onto standard aluminum stubs using electrically conducting paste. The samples were scanned using a Jeol scanning electron microscope (JSM 6480LV) with a magnification up to 300,000× at 10 kV of accelerating voltage. The distance between sample and electron microscope head was 20–25 mm with a spot size of 35. The specimen temperature was approximately 20 °C and the column vacuum was 6.66 × 10^4 Pa. Electron micrographs of the UV irradiated longitudinal tangential surfaces for different exposure times were taken. SEM micrographs of longitudinal radial surfaces were also taken to observe the cell damage from the radial direction.

2.5. FTIR spectroscopy analysis

The effect of sunlight irradiation on chemical compositions of both cellulose and lignin and cellulose crystallinity on wood surface were studied using Fourier transform infrared (FTIR) spectroscopy. The air-dried specimens (10 mm × 20 mm × 20 mm) were studied using Jasco FT/IR 4200 equipped with a diamond micro-ATR crystal. IR spectra were recorded in the wave number range of 550–4000 cm^{-1} at 4 cm^{-1} resolution for 20 scans prior to the Fourier transformation. The incident angle of the micro-ATR crystal was 47° corresponding to the sampling depth of the infrared radiation of 0.2–5 μm, depending on the wave number. This ensured that the recorded IR spectra of wood surfaces were sufficiently surface sensitive. Thus, changes in IR spectral features were solely caused by changes in surface chemistry, and there was no change of underlying bulk chemistry of the wood specimen. The aperture diameter was 7.1 mm. All spectra were analyzed using Jasco spectra manager software. The FTIR spectra were corrected by the FTIR software