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Wood degradation under UV irradiation: A lignin characterization

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ABSTRACT

The photodegradation of white spruce by artificial ageing was studied by several techniques: colourimetry, FTIR-ATR and FT-Raman spectroscopy. Samples were exposed at a xenon lamp for 2000 h. Two distinct colour changes were found by colourimetric analysis, yellowing and silvering. These colour modifications indicate the formation of chromophoric structures which supports previous FTIR-ATR experiments. The degradation of lignin to generate the first chromophoric group for yellowing and then the appearance of surface layer cellulose. New carbonyl compounds conjugated with double bond at 1615 cm^{-1} are probably the second chromophoric group. The crystallinity index was also calculated and showed an increase of cellulose crystallinity by prior degradation of amorphous cellulose. The FT-Raman analysis confirms the wood sensitivity to photodegradation but the most remarkable results is the increase of fluorescence as a function of time. In softwood lignin, the compound able to produce fluorescence is a free rotating 5–5′ linkage of one biphenyl structure. At native state these linkages are not free rotating, this phenomenon means the release of 5–5′ linkage of lignin structure by cleavage of both α carbon linkages (Norrish type I reaction). These data confirm also the photosensitivity of α and β carbon in lignin and the resistance of 5–5′ linkages.

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1. Introduction

Among environmental degradations of wood, photodegradation is the fastest and the strongest [1,2]. Wood compounds, especially lignin, are able to absorb light at short wavelength (from 295 to 400 nm). Lignin is a random and complex polymer which contains chromophoric structures (aromatic compounds) responsible for the wood colour (Fig. 1) [1,3–8]. Photodegradation is a surface phenomenon which starts by a photon absorption as explained by the first photochemical law [7]. The more energetic the photon is, the more important the degradation is with the respect of the second photochemical law. However, the UV's energetic photons do not penetrate wood as much as photons from visible light [3,9].

Wood compounds do not have the same sensitivity to photodegradation due to the variety of functional groups and linkages. For lignin, the most abundant linkage is β-O-4, but there are also 5–5, β − 5, β − 1, α-O-4 and 4-O-5 linkages [10–12](Fig. 1). First Electron Spin Resonance (ESR) studies of a model compound suggested that β-O-4 linkages are the most sensitive to cleavage by Norrish type I reaction [13,14], i.e. the α carbon linkage cleavage. The depolymerisation of lignin leads to the formation of free radicals able to migrate deeper into the wood, leading to degradation under the wood surface [15].

Due to the degradation of chromophoric structures and formation of secondary chromophoric ones, wood colour evolves to yellow and brown then after to silver with a general deterioration in brightness [4,7]. Without contribution of biological deterioration, this silver patina is likely due to the birefringence of cellulose. In fact, a grey colour can be observed on delignified and biological degraded wood. This relates to the silver patina according to the role of microorganism in its appearance. On a wood not thoroughly delignified a dark colouration appears after colonization.

These modifications lead to the loss of (i) aesthetic appeal in indoor applications and (ii) performance of exterior wood coatings and wood outdoor applications [1,16].

Even if the lignin is the most exposed, the other wood constituents, cellulose and hemicelluloses, are also degraded [2,6]. In fact the photodegradation of wood involves a degradation of hemicellulose and a depolymerisation of cellulose. The amorphous cellulose is particularly degraded which leads to an increase of cellulose crystallinity [17,18].

The mechanism and the monitoring of chemical photodegradation has been studied extensively [2,4,9,17–25]. Several approaches have been studied to get a better understanding of wood degradation. The two main being: 1) colour measurements of wood using CIELAB system colour scale. This method allows the following of colour modifications due to lignin degradation and chromophores generation [4,18]; 2) FTIR or FTIR-ATR analysis of the surface allows the study of chemical modifications due to photodegradation [17,23,26–28]. It is a useful technique used by several authors to study this mechanism and to know the extent of wood degradation. Significant contributions of wood compounds such as lignin or holocellulose can be tracked by this technique. As an example, a pure lignin peak can be found at 1510 cm^{-1} (aromatic skeletal vibration in lignin C = C) [29].

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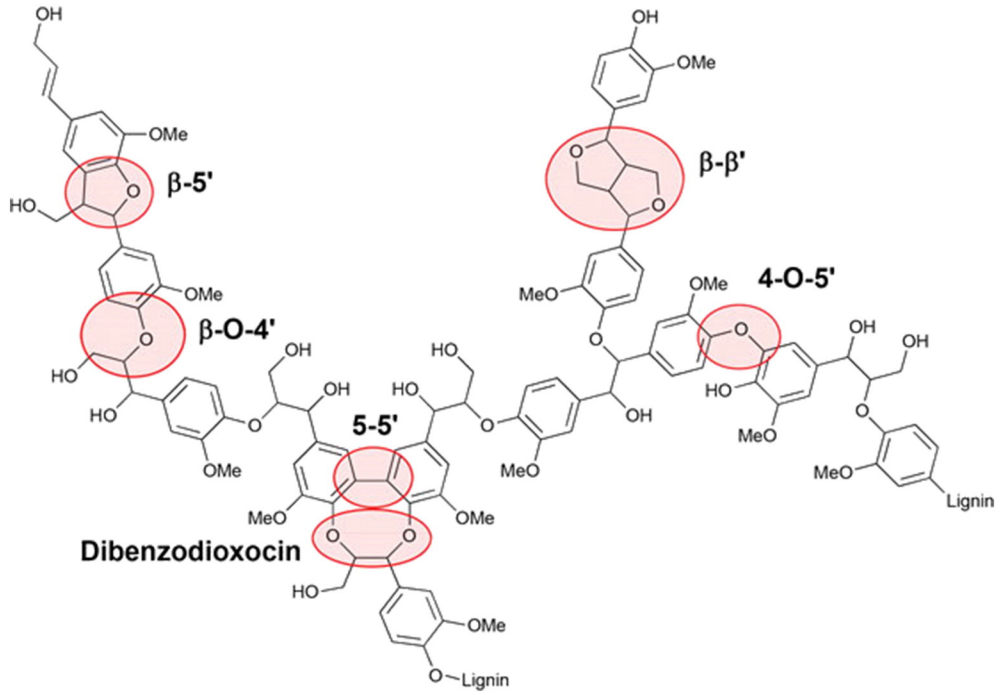

Fig. 1. The different intermolecular bonds in lignin (1).

Raman spectroscopy is a useful technique as it is non-destructive with little or no sample preparation. It was established that Raman information are complementary to the one obtained by FTIR [30] as it addresses a different part of the light spectrum. However, the major problem with Raman spectroscopy when used for plant material study is a strong presence of fluorescence. This phenomenon is due to lignin excitation by laser (laser induced fluorescence: LIF) [31] [32]. Some parts of the lignin are able to absorb photons from the laser to produce fluorescence which could hide the Raman signal.

However, the use of a near-IR laser (1064 nm) limit the excitation and of an interferometer (Fourier Transform method) allow nowadays this technique to be user friendly for plant analysis [33] [34]. Even if several studies utilize FT-Raman for plant investigation [32,33,35,36] none have been found on the analysis of wood photodegradation.

In this study, these techniques were used to study *Picea glauca* ((Moench) Voss) photodegraded wood. The purpose is to identify new approaches to follow photodegradation as well as new information on the chemical modification of wood constituents and on the appearance of the silver patina. Interests and limits of these techniques will also be discussed. Then, chemical analysis of photodegraded wood will also be presented.

2. Materials and Methods

2.1. Wood Specimens

Wood specimens were obtained from white spruce (*Picea glauca* ((Moench) Voss)) measuring 50 × 75 × 4 mm (longitudinal x radial x tangential). They were sanded using P-150 sandpaper. Four stems were used, for each stem four samples were prepared including early and late wood. Wood samples were put in a conditioning room at 8% HR and 22 °C until constant mass.

2.2. UV Irradiation

Samples were exposed in a QUV accelerated weathering tester from Q-Lab (USA) for 2000 h. Cycle 1 of ASTM G154-2012 Standard test method "Standard Practice for operating Fluorescent Ultraviolet (UV) Lamp Apparatus for exposure of non-metallic Materials" was used. The contribution of the water is taken in account by a condensation system and allows simulating dew and rain and so water specific phenomena like leaching, erosion and participation in chemical reactions. An UV-A 340 lamp was used for the irradiation at 0.89 W/m^2/nm to simulate the UV portion of the solar spectrum. The irradiance of the lamps is not homogeneous in several locations of chamber. For this reason, samples have been distributed into the chamber with a random distribution process in R studio software.

2.3. Colour Measurements

Colour changes were analysed using a reflectance spectrophotometer: BYK- Gardner (USA) - colour guide 45°/0°. CIEL*a*b* colour scale was used. The L* axis represents the lightness and varies from 100 (pure white) to zero (pure black). a* and b* are the chromaticity coordinates: +a* is for red, −a* for green, +b* for yellow and -b* for blue. Zero (0) is grey. The overall colour differences (delta E) were calculated using the following Eq. (1):

$$\Delta E^* = \left[\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}\right]^{1/2} \quad (1)$$

where ΔL*, Δa* and Δb* are the difference of initial and final values. A low ΔE* value corresponds to a low colour difference. Four measurements were performed on each sample at different locations following a pre-determined pattern. The average value was calculated and used for presentation purpose in this paper.

2.4. ATR-FTIR Spectroscopy Analysis

FTIR spectroscopy experiments were carried out using a Spectrum 400 from Perkin Elmer (UK) equipped with an attenuated total reflection accessory (ATR). The resolution was set at 4 cm^{-1}, 64 scans were recorded for each analysis and the scanning range was from 650 cm^{-1} to 4000 cm^{-1}. Four analyses were performed at four locations per sample. The information from these spectra were extracted with GRAMS software form Thermo Scientific (USA). For calculating ratios, the top

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