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Modeling color and chemical changes on normal and red heart beech wood by reflectance spectrophotometry, Fourier Transform Infrared spectroscopy and hyperspectral imaging

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A B S T R A C T

The use of beech is a key topic for Europe as it is one of the most important and abundant broad-leaf species. Physical, mechanical and esthetical features influence both the value and the usage of this wood in each commercial product. In this sense, the comprehension of the surface color modifications induced by solar irradiation is of crucial importance to define the commercial value of the beech wood.

Starting from these general remarks, the aim of this work is to study the surface modifications on beech wood with and without red heartwood by different spectroscopic techniques and to obtain a modeling of the changes validated by rigorous statistical and chemometric methods together with principal component analysis.

The artificial photo-irradiation of the wood samples was performed in a Solar Box. Reflectance spectrophotometry, Fourier Transform Infrared spectroscopy and hyperspectral imaging were used to assess artificial sunlight influence. The experimental data were statistically treated in order to evaluate their significance.

Color monitoring allowed to find that the chromatic coordinates ($L^*a^*b^*$) in normal wood and in red heartwood tended to similar values after 504 h of photo-irradiation.

Fourier Transform Infrared spectroscopy showed the rate of photo-degradation of wood surface due to lignin oxidation and the statistical analysis allowed to demonstrate that red heart and normal wood have the same behavior.

Concerning hyperspectral imaging (HSI), the detected spectral features were correlated to color changes in the Visible–Near Infrared (VIS–NIR) range (400–1000 nm) and to the variations of cellulose and lignin during accelerated aging in the Short-Wave Infrared (SWIR) range (1000–2500 nm).

The most important result is that a correlation, validated by statistical analysis, of the color changes may be derived with the photo-degradation of wood components obtained by spectral analysis. This fact suggests the possibility to choose the reflectance spectrophotometry as a non-invasive, simple standard method to monitor the state of preservation of the wood surfaces.

1. Introduction

The aim of this work is to study the surface modifications on beech wood, with and without red heartwood, by different spectroscopic techniques in order to obtain a modeling of the changes validated by rigorous statistical and chemometric methods together with principal component analysis.

The choice of beech wood was due to its wide use in Europe [1], as it is one of the most important and abundant broad-leaf species. The extent of beech forests in Europe is estimated at about 12 million hectares of which more than 550,000 in Italy [2]. In particular, beech wood from coppices in transition shows interesting qualitative characteristics, suggesting a more profitable use

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than firewood [3]. Coppices in transition originated from the conversion of large areas from coppice to high forest as a consequence of the progressive abandonment of rural and mountain zones and policies of ecological protection of the territory in the last century, leading forest managers to a less intensive use of forests [4], creating different situations in Europe [5–7]. In Central Italy, large areas were converted to coppice, as a result of changing the socio-economic conditions of the last century, the gradual rise in the cost of labor and the stagnation in the market prices of firewood. In Italy, the transitory high forests stand on about 150,000 ha [8]. In Italy, the wood sector involves about 80,000 businesses, over 500,000 work units and it is heavily dependent on foreign countries for the supply [9], as the domestic timber availability is not always appreciated due to the lack of quantitative and qualitative homogeneity. From coppices in transition to high forest, which now reached maturity and therefore are ready to renewal cuts, firewood or biomass in the form of particles are obtained, mainly for energy production [3,10–11]. However, some species are particularly valued for their technological features, valuable products also are obtained as round wood and sawn timber, despite the large amount of branched and buckled stems. Concerning the beech wood, the occurrence of red heartwood due to wounds, root dieback and dead branch constitutes a drawback. The grading rules concerning sawn timber, as EN975-1 [12], limit the presence of red heart. For this reason red heartwood is less appreciated [13–14] and causes loss of commercial value [15–17], influencing the yield of the production.

As a consequence, the study and comprehension of beech color parameters are crucial to define the potential usages of this species for higher quality production, in order to enhance the wood chain from the forest to the processing industry.

In general, color variation of wood due to light radiation is an interesting and important topic in wood science as testified by a lot of literature papers concerning the photo-oxidation of wood surfaces [18–29]. . In particular, the occurrence of red heartwood in beech is definitely a significant characteristic that often leads to debasement following classical grading rules.

For this reason, the modeling of color and chemical changes on beech wood with or without red heartwood becomes important. To do this, reflectance spectrophotometry, Fourier Transform Infrared (FTIR) spectroscopy and hyperspectral imaging were applied to the two kinds of wood. The data were correlated to check the relationship between variation of color and of the chemical components [24].

2. Materials and methods

2.1. Sample preparation and aging

Wood samples were obtained by boards of beech harvested on Terminillo Mountain (Central Italy – Leonessa Municipality) in a coppice in transition to high forest. Information on the site and sample preparation was detailed in a previous paper [3]. All samples were artificially aged in a Model 1500E Solar Box (Erichsen Instruments). The system is equipped with a 2.5 kW xenon-arc lamp and a UV filter that cuts off the spectrum at 280 nm. The samples were exposed in the Solar Box chamber from 1 to 504 h at 550 W m^{-2}, 55 °C and the UV filter at 280 nm. The experimental conditions were chosen following the specifications supplied by Erichsen, in order to simulate the sunlight exposition. Inside the Solar Box chamber relative humidity was constant and determined by the irradiation conditions. Relative humidity was monitored by a data logger positioned inside the Solar Box.

To perform the FT-IR analysis directly on wood surface, slices with a size of 10 (diameter) × 2 (thickness) mm were obtained from the specimens of beech. The dimensions of the slices were suitable for the FT-IR diffuse reflectance accessory.

2.2. Color monitoring

After exposure for a given length of time the samples were removed from the Solar Box chamber and the color was measured using an X-Rite CA22 reflectance spectrophotometer according to the CIELAB color system. The characteristics of the color measuring instrument are the following: light source D65; standard observer 10°; fixed geometry of measurement 45°/0°; spectral range 400–700 nm; spectral resolution 10 nm; aperture size 4 mm [24,25,30–32]. The wood samples were conditioned before color measurements at 22 °C and 50% RH.

Measurements were taken at the following hour intervals: 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 24, 48, 72, 96, 120, 144, 168, 216, 312, 408 and 504 h.

Sixty color measuring points were chosen: thirty for red heartwood and thirty for normal beech wood. Three measures for each point were performed, so that one hundred and eighty measurements were obtained at each exposure time, then average values and standard deviations were calculated.

2.3. Fourier Transform Infrared spectroscopy

Infrared spectra were obtained using a Nicolet Avatar 360 Fourier transform spectrometer. For each sample 128 scans were recorded in the 4000 − 400 cm^{-1} spectral range (2500–25,000 nm) in diffuse reflection modality (DRIFT) with a resolution of 4 cm^{-1}. As background the spectrum of the KBr powder was used.

Spectral data were collected with OMNIC 8.0 (Thermo Fisher Scientific Inc.) software.

FT-IR spectra were recorded at the following time intervals: 0, 6, 12, 24, 48, 72, 96, 120, 144, 168, 216, 312, 408 and 504 h.

Peak heights were measured using OMNIC software according to the method described in the literature [33].

2.4. Hyperspectral imaging

Hyperspectral analyses were carried out in two steps. A 1st step was addressed to analyze the degraded wood surfaces in the wavelength interval 400–1000 nm (VIS–NIR). A 2nd step was finalized to perform investigations in the wavelength interval 1000–2500 nm (SWIR).

For the 1st set of acquisitions (400–1000 nm), the ImSpector V10E™ (Specim, Finland) was installed on a Leica M205C™ stereomicroscope. The energizing source was constituted by a MI-150 Dolan Jenner fiber optic device equipped with a dichroic lamp. The magnification was 7.8×, corresponding to a spatial resolution of 40 micron. The spectral resolution was 5 nm. For the 2nd set of acquisitions (1000–2500 nm) the SISUChema XL™ (Specim, Finland) was used, equipped with a 31 mm lens allowing the acquisition of wood samples with a resolution of 300 micron/pixel. The spectral resolution was 6.3 nm. Images were acquired through scanning each investigated sample line by line.

The calibration was performed through recording black and white reference images. Certified standards were used. Black image (B) was acquired to remove the dark current effect of the camera sensor. White reference image (W) was acquired using a ceramic tile, calibrated with a NPL Spectralone specimen, in the same condition employed for the raw image acquisition. The image correction was thus performed adopting the following equation:

$$I = \frac{I_0 - B}{W - B} \times 100,$$

where I is the corrected hyperspectral image in a unit of relative reflectance (%), I_0 is the original hyperspectral image, B is the black