High temperature treatment allows the detection of episesamin in Paulownia wood extractives

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The composition and the relative variation of secondary metabolites of Paulownia tomentosa S. wood under thermal effect is a little explored area. Wood material was previously thermo-treated at 210 °C for 3 hours using a press vacuum technology. Extractives of untreated and thermo-treated wood material achieved with Soxhlet extraction techniques were obtained. Then the extracts were chromatographed by using thin layer chromatography. Component groups in extracts were determined by gas chromatography in combination with mass spectrometry. In terms of wood change the thermo-treatment of wood induces a darkening of wood color surface ($\Delta L^* = 28.3$), an increase of mass loss (3.5 %) and an increase of the amount of extractives and lignin content as well as an increase of the chloroform soluble fraction. This work mainly describes the chemical exploration of the extract from paulownia wood, leading to the isolation and identification of episesamin.

Keywords: Paulownia; thermo-vacuum treatment; Soxhlet extraction; episesamin.

3. Experimental

Paulownia tomentosa S. wood 30 boards free of defect with dimension of 30 mm by 180 mm by 1500 mm, were supplied by a local manufactures in Basilicata Region in Italy. Half of boards was thermo-vacuum treated with press vacuum technology at 210 °C × 3 hours. More details on the technology process can be found in Ferrari et al. (2013). Other 158 boards was used as references.

Wood properties
The mass loss (ML), due to the thermal treatment, was determined by weighting each treated board immediately after the drying process (when the wood moisture content MC was 0%) and at the end of the thermal treatment. Further details are in Ferrari et al. (2013).

Color analysis was obtained by a Minolta CM-2002 Spectrophotometer (Minolta Corp, Osaka, Japan) with a spot probe of 8 mm diameter. The instrument measures the L*, a*,b* (lightness, red/green, and yellow/blue, respectively) chromaticity coordinates. Forty measurements were taken for each treatment.

**Chemical characterization**

Treated and untreated wood samples were randomly selected and reduced to a small size with a mill saw and subjected to Soxhlet extraction technique with 1:2 ethanol-toluene mixture for 7 h in a Soxhlet apparatus by using the TAPPI test method T204 (TAPPI 2004). 1 g of milled wood repeated three time was used.

The extraction apparatus consisted of a 500 mL flask, Soxhlet tube, and 300-mm Allihn condenser.

Samples were put in cellulose thimbles (33 × 80 mm) of medium porosity. After extraction, the solution was dried in a previously weighed 25 mL flask, by using a rotary evaporator connected to a vacuum pump (Vacuumbrand PC3001). The percentage of extractives was determined gravimetrically obtained by weighing the flask containing the residue and comparing the weight to that of the initial dry mass wood.

The obtained mixture was fractionated as follows: the mixture was treated with chloroform (20 mL) and filtered. The solvent was evaporated, and the residue was chromatographed by using tin layer chromatography. The eluent was 1:1 hexane - ethyl acetate.
Qualitative and quantitative measurements of the extracts were then made by analytical method using a GC-MS system. GC-MS analyses were performed on an HP 6890 (Agilent) GC system equipped with an HP 5963 MS selective detector, with a high temperature capillary column (HP-5MS, 30 m x 0.25 mm I.D., 0.25-μm film thickness; J&W Scientific, CA, USA) and helium as carrier gas. Samples were injected directly into the column at a temperature of 80 °C. After injection, the temperature was held at 80 °C for 3 min, and then heated to 250 °C at a rate of 20 °C min⁻¹ and held for 20 min. Compounds were identified by computer comparison of the mass spectra with NIST libraries and by mass fragmentation patterns.

The lignin content was determined as it follows. The sawdust was transferred to a 50 mL beaker, a cold H₂SO₄ solution (72%) (15 mL) was added, and the mixture was frequently stirred for 2 h at room temperature. The mixture was then diluted to 3% (w/w) with 560 mL of distilled water, heated under reflux for 4 h, filtered, and washed with 500 mL of water. The residue was dried at 105 °C to a constant mass. The holocellulose content was determined by difference between the residue amount after extraction and the lignin content.
Figure 1S. Mass spectrum of episesamin.

References

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