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Effects of water re-saturation conditions and associated extractives leaching on thermal softening of wet wood

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Thermal softening, or thermo-activated viscoelasticity, is involved in several industrial and/or traditional craftsmanship techniques processing of wood. More fundamentally, it represents a very useful probe of chemical differences and/or of physical histories of wood samples [1,2,3,4,5]. Thermal softening of wet (= water-saturated) wood occurs at much lower temperature than that of completely (anhydrous) dried wood [6,7]. Softening of oven-dry wood happens at temperatures above 150°C, a temperature level at which wood amorphous polymers start to degrade [3,8]. Therefore, the characterisation of wet wood is the unique method that allows thermal softening to be distinguished from thermal degradation. Thermal softening of wet wood is also easier, or at least less energy consuming, in several applied processes. In many cases, it is not always practically feasible to start from green (e.g. never-dried) wood, either for scientific experiments or in real-scale applications, so that air-dry wood may need to be re-saturated. However, it is known that water re-saturated wood behaves differently from never-dried wood [4,5,9]. In addition, soaking wood in water is susceptible to remove secondary metabolites (extractives), some of which are known to modify viscoelastic properties of various wood species [10,11]. The objective of the present work is to provide insight on how thermal softening (studied by Dynamic Mechanical Analysis – DMA) of several hardwoods is affected by conditions (duration and temperature) of water re-saturation, and by additional effects of extractives leaching during these re-conditioning processes.

The study is based on five wood types from four hardwood species, chosen for contrasted chemical composition: black locust, oak, poplar, and beech (including normal wood and tension wood). Radial specimens for DMA ($40\times4\times0.6mm^3$, R×T×L) were cut from naturally air-dried wood and prepared following a careful matching procedure. Three replicate specimens were tested for each modality of pre-treatment. Nearly 100 specimens were tested by DMA in total. Each DMA specimen was matched with a specimen designed for monitoring oven-dry weight losses due to a given pre-treatment. Pre-treatments included (see Figure 1): cold water (just after re-saturation, after 2 months and 10 months), 40°C water (4h and 96h), 70°C water (6h), 98°C water (2h), all cooled down slowly to ambient temperature again, and an overnight extraction in cold acetone followed by exchange with water. Tests were conducted in tension, using a TA Instrument Dynamic Mechanical Analyser, at frequencies 0.1, 1, 10 and 25Hz. Evolution with temperature, from 20°C to 100°C, was studied using 5 minutes isothermal steps every 5°C.

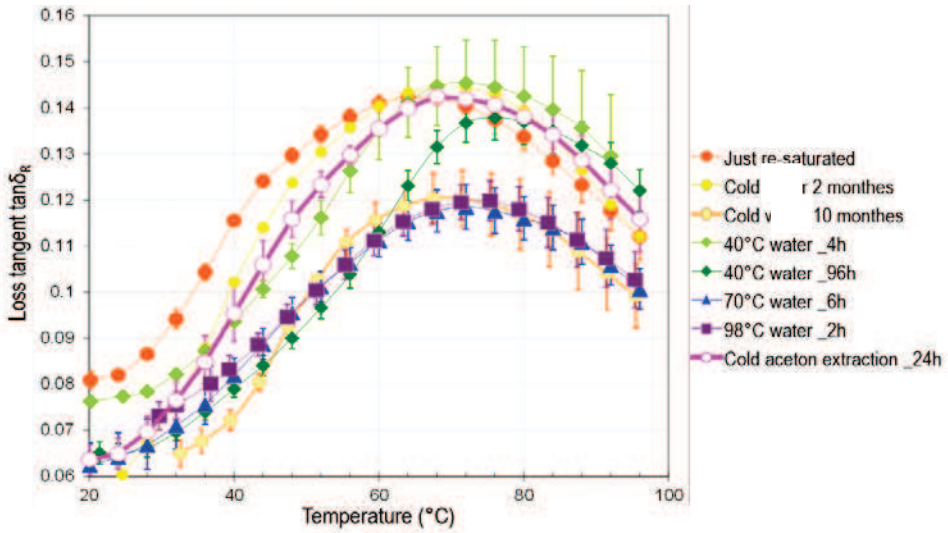

Figure 1. Illustration of the different pre-treatment modalities tested in this work, through an example of the evolution with temperature of loss tangent (tanδ, at 0.1Hz) for black locust wood. Error bars = 2×standard deviation between 3 specimens

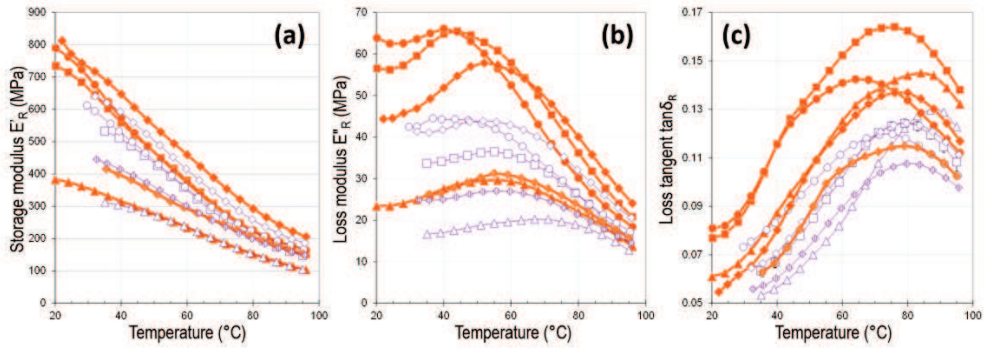

Figure 2. Comparison of the thermal softening behaviour just after re-saturation (filled symbols) and after re-conditionning at 98°C (empty symbols) of 5 different wood types: oak (squares), black locust (circles), poplar (triangles), beech (diamonds; filled: normal wood, hatched: tension wood). (a) Storage modulus E', (b) loss modulus E", (c) loss tangent tanδ, all at 0.1Hz

Results show that the thermal softening behaviour just after re-saturation appears to include a kind of "secondary transition" in the range of 50-60°C (Figure 2c for tanδ), which is consistent with previous findings [4,5]. However, the amplitude of this phenomenon is not similar for all tested wood types (see Figure 2). This disappears after re-conditioning above the glass transition temperature Tg (pre-treatments at 70°C or 98°C), which also leads to a reduction of the maximum of loss modulus and loss tangent and an apparent increase in Tg. Longer pre-treatments at lower temperature partially lead to similar results but even 10 months in cold water did not remove the "secondary transition", whereas 40°C pre-treatment did but could not reduce as much the maximum of viscosity. Pre-treatments in water removed between 2 and 8% of extractives, depending on species and conditions. Results without re-conditioning but after cold acetone extraction show species-dependant effects notably on loss tangent tanδ, which strongly suggests an additive effect of extractives, and of physical re-conditioning, on the thermal softening behaviour of hardwoods.

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