Impact of soaking pretreatment of spruce tonewood on its physical and acoustical properties

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Abstract

The research analyzed the influence of alkaline soaking pre-treatment on some acoustical properties of Norway spruce tonewood (*Picea abies* Karst.) used for stringed instruments. Basic physical properties were determined at several randomly selected tonewood boards before and after the soaking, combined by dynamic mechanical response and FTIR spectroscopy. Most changes of vibrational properties and FTIR spectra of spruce tonewood after the treatment are assigned to mass and density loss caused by reduced amount of lignin and hemicelluloses. The specific modulus of elasticity after the soaking remained unaltered, whereas sound damping in longitudinal direction slightly increased. Significantly greater changes of dynamic shear modulus and internal friction caused on the other hand higher acoustical anisotropy of spruce tonewood after the soaking.

Keywords: spruce, tonewood, soaking, acoustic properties, infrared spectroscopy.

1 Introduction

Exquisite Cremonese stringed instruments produced by Antonio Stradivari and Joseph Guarneri during the late 17th and early 18th centuries have become the benchmark to which more recent and contemporary instruments are compared. Since modern craftsmen have employed all traditional know-how of the art aided by volumes of acoustical research, may the instrument quality basics lie in the material differences caused by an ancient and forgotten practice of wood selecting, manipulation, pretreatment, preservation and finishing technologies?

The micrographs from the violins of Stradivari, Guarneri and Guadagnini revealed namely the remnants of microorganisms and also mineral deposits (Nagyvary 1988), whereas a SEM study on the spruce samples from Italian musical instruments did not found any morphological changes (Barlow and Woodhouse 1990). More than decade later, using various spectroscopic methods, researchers indisputably proved that various degradations of wood polymers in some instruments of Stradivari and Guarneri could be only explained by chemical manipulations, possibly by preservatives (Nagyvary et al. 2006; Nagyvary et al. 2009). This conclusion rests foremost on presence of few minerals which are not known to exist in detectable quantities in natural maple wood, like borates, crystals of $ZrSiO_4$, mineral fluorite (CaF_2) and various insoluble particles of salts ($BaSO_4$ and SiO_2). The quite different proportions of chemicals were found in Stradivari's and Guarneri's back plates of violins as well as in comparison to cello samples, where the later was explained by the size difference of these instruments. Due to high concentration of Ca and Mg, found at mineral composition of Stradivari maple researchers suggest that at least one soaking solution used could have been simply a local mineral water (Nagyvary 2005). The true specific formula that would have been commonly used in Cremona is apparently absent, where the mix could have contained crushed crystals of calcite, gypsum, barite, fluorite and quartz, in addition to some water soluble salts like borax and the sulfates of Zn, Cu, Cr and Fe (Nagyvary et al. 2009).

The first and undoubted conclusion on using of extraneous chemicals found in Cremonese woods is for the purpose of wood preservation. The successful practice of employing borates as a biocide is ancient, as well as current, where other found salts are commonly sprayed on trees, could have been included by design or as contaminants (Cockcroft and Levy 1973; Travis 1984). Nevertheless, the Cremona "slurry of minerals" might also been used purposely for the degradation of organic matrix of wood, found on some samples from Cremonese stringed instruments (Nagyvary et al. 2006), which might affected some physical and acoustic properties of wood. This hypothesis as known has several pros- and even more contra, since many luthiers who tried aqueous treatments reported only deleterious effects on the stiffness of wood (Haines 1979).

Contrary some positive comments exist to the quality of sound of more the 150 contemporary violins, which were treated by many variations of the aqueous processing and composite finishes (Nagyvary 2005). The improvement of wood dimensional stability by soaking of tonewood is exposed as one of positive issues in this case, due to nearly 50 % reduced equilibrium moisture content of wood (EMC) at normal climatic conditions. Reduced hygroscopicity of wood might be found at the tonewood of some Cremonese violins at the disposal of the precious material. This assumption is supported by IR- and NMR spectroscopic studies of wood slivers from some Stradivari's and Guarneri's violins where significantly lower amount of hemicelluloses (arabinan) and acetyl groups was confirmed (Nagyvary 2005). The research supports the idea that differences are likely to have originated from a regional practice of wood preservation caused oxidation and hydrolysis of wood of some Cremonese violins which might also affected its mechanical and acoustical properties.

The objective of this research is to deepen the knowledge on impact of soaking pretreatment, re-made by contemporary violin maker on some structural and physico-chemical properties of tonewood. Additionally the aspect of changed physical properties of tonewood, having influence on its acoustics is analyzed.

2 Material and Methods

2.1 Material

We investigated the tonewood of Norway spruce (*Picea abies* Karst.). In the warehouse of a commercial tonewood provider, we inspected stacks of wood for violin production and randomly collected 20 tonewood boards of AA-quality (i.e. standard commercial tonewood quality) with dimensions of 450 mm × 120 mm × 22 mm (Long. × Rad. × Tang). The selected boards were 1 month laboratory dried and equilibrated in normal climate, following the determination of mass and board dimensions.

Half of the pieces (n = 10) were afterwards placed into soaking bath by local luthier Milan Oreški and loaded to keep it in the mineral water solution (Indices: I – Innate; S - Soaked). The true specific formula of the alkaline soaking solution is the technological secret of the luthier, where used minerals are completely naturally based, and attempts to re-create the eventual treatment of great Cremonese masters (Nagyvary 2005; Nagyvary et al. 2009). The soaking process went on in a dark room for a period of 18 months, having a constant temperature of 18 ± 2 °C. After the treatment the boards were carefully dried in a stack at normal room climate (20 °C, 65 % *RH*) in a 6 months period to reach the final equilibrium moisture content (*EMC*).

2.2 Physical properties

The oriented small specimens, with dimensions of 20 × 20 × 20 mm (Long. × Rad. × Tang.), were cut out from the boards end-sections. The specimens were measured by calipers having a precision of 0.01 mm, and weighed with a precision of 0.001 g, at 3 moisture content stages: 1 and 2 – equilibrated at 20 °C and constant relative air humidity (RH_1 = 65 %, RH_2 = 33 %), and 3 – the oven-dry stage (vacuum dryer: T = 50 °C, P = 20 mbar, t = 48 h). The moisture content (*MC*) was determined gravimetrically.

The shrinkage strain ε and the shrinkage coefficient α of the small specimens were determined in radial- (ε_R, α_R) and tangential direction (ε_T, α_T) by definition equations (Eq. 1, Eq. 2). The sorption coefficient s was additionally determined by Eq. 3.

$$\varepsilon_{R,T} = \frac{L_1 - L_2}{L_0} \tag{1}$$

$$\alpha_{R,T} = \left| \frac{\varepsilon_{R,T}}{MC_1 - MC_2} \right| \tag{2}$$

$$s = \frac{MC_1 - MC_2}{RH_1 - RH_2} \tag{3}$$

2.3 IR analysis

Thin microtome slices 20 µm of thickness, were cut off from *LR*-plane of equilibrated small specimens (20 °C, 65 % *RH*) and measured at FTIR spectrometer SpectrumOne (PerkinElmer Inc.). The absorbance IR spectra in the spectral range between 800 and 4,000 cm^{-1} was obtained and analyzed by Spectrum® 3.02 software package using basic spectra pre-processing, i.e. baseline correction and spectra normalization. All the spectra were measured at spectral resolution of 4 cm^{-1} and 16 scans were taken per sample. The presented spectra are average value of the respective single spectra.

2.4 Acoustic properties

The boards from both groups were supported by loose thin silk threads located at the nodes of the 1st mode of the flexural vibration; they were exposed to free-free flexural vibration, using a steel ball. Displacement was measured at the belly of the vibration by a condense microphone (PCB–130D20), whereby the signal was acquired by NI-9234 data acquisition module (Fig. 1a). The whole experimental procedure, data acquisition and signal analysis is described elsewhere (Straže et al. 2015).

An exponentially time-decayed vibration curve of the fundamental resonant frequency f_1 was additionally fitted, using least-squares regression analysis, whereby the value of the damping coefficient ($tan\ \delta_L$) was determined. The modulus of elasticity (E_L) was determined from the fundamental resonant frequency using Bernoulli solution and used also to determine the ratio of acoustic energy radiated from the boards, defined by acoustic conversion efficiency *ACE* ($\sqrt{E_L\ /\rho^3}/tan\delta_L$). The dynamic shear modulus (G_{LT}) and loss tangent ($tan\ \delta_S$) in the LT plane were measured by the torsional vibration method (Divos et al. 1998). The specimen was supported at the middle of the narrow face where the impact of a steel ball was induced at the upper corner of the wide face. The G_{LT} value of the specimen was calculated from the 1st resonant frequency mode of torsional vibration (Eq. 4), and the loss tangent ($tan\ \delta_S$) was calculated from the signal time-decay curve.

$$G_{LT} = \left(\frac{f_{Tn} 2L}{n} \right)^2 \frac{\rho I_p}{K_t} \tag{4}$$

Where:

f_{Tn} = the n-th torsional frequency,

I_p = polar moment of inertia ($I_p = I_x + I_y$),

$K_t = c \times R \times T^3$ ($a \geq b$)

R, T = cross sectional dimensions

c = constant (c = 0.3 at R/T = 5.5)

The acoustical anisotropy of wood was additionally determined by E_L/G_{LT} ratio and by $\tan\delta_S$ / $\tan\delta_L$ value, wherefrom index of tone quality (β) was determined (Eq. 5) (Nozaki et al. 1988; Obataya et al. 2000):

$$\beta = \frac{(E_L / G_{LT})}{(\tan\delta_s / \tan\delta_L)} \tag{5}$$

3 Results and discussion

3.1 Physical properties of soaked tonewood

The discernible reduction of the wood density after the soaking, from initial mean value of 486 kg/m^3 (CV = 11.2 %) to 407 kg/m^3 (CV = 7.3 %) was in the first place noted at normal climate conditions. The mean initial density of the tested tonewood is concordantly to the literature close to the average density of Norway spruce, e.g.: 470 kg/m^3 (Wagenführ 2007). Rather lower wood density, having high stiffness and homogeneous grain is preferable in making of soundboards of stringed instruments (Bucur 2006). The reduction of wood density after the mineral soaking treatment was therefore desirable ($\Delta\rho$ = -16.3%) and came near to the median density of top plates in contemporary or ancient violins (Stoel and Borman 2008; Stoel et al. 2012).

The determined mean tangential- (0.41 %/%; CV = 3.5) and radial shrinkage coefficient (0.17 %/%; CV = 9.5) of the innate tonewood were similar to that which has been reported in earlier studies (Straže et al. 2011). Small reduction of shrinkage strain was confirmed after the soaking and attained 0.39 %/% (CV = 7.1), and 0.15 %/% (CV = 9.5) in tangential- and radial direction individually. The small lessening of the wood hygroscopicity was confirmed as well, found also on wood studies from old violins (Nagyvary 2005), where the sorption coefficient changed from 0.12 %/% (CV = 4.8) to 0.10 (CV = 11.3) after the process.

3.2 FTIR spectroscopy

The mid-range FTIR spectra of innate- and soaked spruce tonewood differ significantly. The most spectral information was found in region of 1800 – 800 cm^{-1}, where after normalization using invariable regions/peaks at 830 cm^{-1}, 1070 cm^{-1}, 1180 cm^{-1}, 1530 cm^{-1}, 1800 cm^{-1}, several differences found in spectra absorbance of these two spruce tonewood groups.

3.2.1 Cellulose

In the spectral region assigned to cellulose C-O-C bridges no shifting of the infrared band was confirmed. The bands were located at innate- and at soaked wood at 1161 cm^{-1}, which is close to crystallized cellulose band (1163 cm^{-1}), while for amorphous cellulose it is located at 1156 cm^{-1}. These values confirm that innate and soaked spruce tonewood shows high crystallized cellulose I content. No shift of the infrared band was confirmed also at CH_2 bending vibration in crystalline cellulose I and amorphous cellulose mixture, found at our samples at 1424 cm^{-1}.

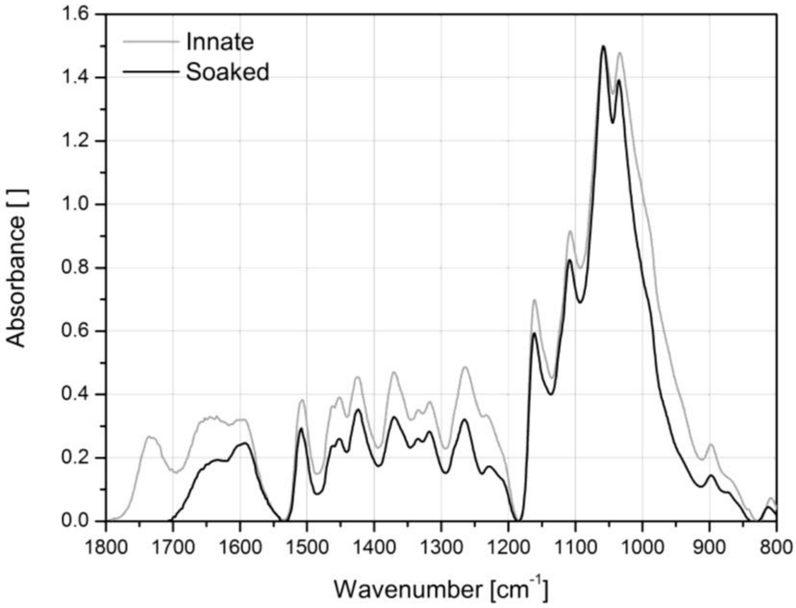

Figure 1 FTIR spectra of innate (…) and soaked Norway spruce tonewood (—) in the range from 800 cm^{-1} to 1800 cm^{-1}

The spectroscopic evolution of the doublet at 1335 – 1316 cm^{-1} assigned to the cellulosic component of wood and only appearing in celluloses with high crystallized cellulose I and/or cellulose II content was likewise confirmed at both, the innate- and soaked tonewood. Small decrease of the ratio 1335/1316 observed at soaked spruce wood (-0.005) can be interpreted as a slight increase in the crystalline cellulose I content.

Significantly reduced spectral absorption was found at band at 1633 cm^{-1} and originates from less adsorbed water molecules via hydrogen bonding in the amorphous regions of the cellulose macromolecules. The result confirms decreased hygroscopicity of soaked spruce wood, which supports also the improved dimensional stability of this material.

3.2.2 Lignin and hemicelluloses

The intensity of bands 1505 cm^{-1} and 1595 cm^{-1} as well as 1230 cm^{-1} and 1270 cm^{-1} assigned to aromatic skeletal vibration (Faix 1992), decreased considerably after the soaking process indicating the degradation of lignin polymer structure and pronounced loss of lignin aromatic content. The decreased spectral absorbance of soaked wood was confirmed also at bands centered at 1463 cm^{-1}, 1425 cm^{-1}, 1375 cm^{-1}, 1160 cm^{-1}, 1111 cm^{-}

^{1} and 1030 cm^{-1} which are assigned to characteristic bending or stretching vibrations of different groups for lignin, hemicelluloses and cellulose (Adler 1977; Chen et al. 2010; Fengel and Shao 1985).

Nevertheless, the absorbance at 1740 cm^{-1}, assigned to C=O stretching vibration of acetyl or carboxylic acid (Popescu et al. 2009; Sarkanen et al. 1967), decreased most significantly after the soaking of the spruce tonewood. Since carbonyl groups are mainly in the hemicellulose branched chain component, the intensity of the band depends on the ratio of holocellulose content to lignin content (Colom and Cariillo 2005). The question arises, whether the reduction is more outcome of lignin decrease or the lower amount of hemicelluloses? By a marvelous coincidence, the recent FTIR study of maple tone wood from one Stradivari- (dated 1717) and Guarneri del Gesu violin (dated 1741) find as well the strongest FTIR spectra absorption band changes particularly in the carbonyl region at 1730 cm^{-1} to 1650 cm^{-1} (Nagyvary et al. 2006). The change in the absorption at around 1650 cm^{-1} is accounted for the formation of quinones from lignin by oxidation (Anderson et al. 1991).

3.3 Impact of soaking process on mechanical and acoustical properties of spruce tonewood

The soaking process negatively impacted the stiffness (ΔE_L = -19.6%) and the dynamic shear modulus of spruce tonewood (ΔG_{LT} = -40.4%), adequately to the significant reduction of the wood density ($\Delta\rho$ = -16.3%) (Tab. 1). The comparable changes of wood density and dynamic modulus of elasticity (E_L) caused insignificant lessening of the specific modulus of elasticity after the soaking process ($\Delta \frac{E_L}{\rho} = -4.0\%$). Oppositely, the damping of sound increased in bending- ($\Delta tan\ \delta_L$ = +42.1%) and especially in torsion vibration of specimens after the treatment ($\Delta tan\ \delta_S$ = +77.8%). The increase of damping in bending vibration had notable bearing on reduction of Acoustic Conversion Efficiency after the treatment (ACE_S = 3905 $m^4\ s^{-1}\ kg^{-1}$; ΔACE = -17.7%).

Table 1 Comparison of mean physical, mechanical and acoustical properties of innate- and soaked spruce tonewood. Coeff. of variation (%) is presented in the brackets.

Property	Innate wood	Soaked wood
ρ [kg m^{-3}]	486 (11.2)	407 (7.3)
E_L [GPa]	15.8 (11.8)	12.7 (9.2)
E_L/ρ [GPa]	32.5 (7.8)	31.2 (6.8)
G_{LT} [GPa]	1.14 (12.0)	0.62 (10.5)
$\tan\delta_L$	9.5×10^{-3} (9.5)	14×10^{-3} (7.1)
$\tan\delta_S$	18×10^{-3} (11.3)	32×10^{-3} (10.4)
$\tan\delta_S / \tan\delta_L$	1.9 (10.7)	2.4 (12.2)
E_L / G_{LT}	15.2 (9.8)	20.5 (8.6)
ACE [m^4 s^{-1} kg^{-1}]	3.90×10^3 (15.0)	3.22×10^3 (11.5)
β	8.02 (14.3)	8.64 (12.9)

According to previous studies, the amplitude of sound variation from wooden soundboard depends on the acoustic conversion efficiency of wood along the grain (Obataya et al. 2000; Ono 1996). However, the reduced *ACE* in a case of wood soaking doesn't independently compare these two groups of wood, since change of wood density during the treatment significantly affects the result. On the other hand, highly important improvement of acoustic anisotropy to 8.64 was reached after the soaking treatment ($\Delta\beta$ = +7.8%), when both moduli- ($\Delta(E_L/G_{LT})$ = +34.8%) and the damping ratio ($\Delta(\tan\delta_s / \tan\delta_L)$ = 25.1%) in bending- and torsional vibration of tonewood were significantly improved. The increase of E_L/G_{LT} ratio can also be found in some studies of aged wood, which is also an interested material for musicians and artisans making traditional musical instruments (Noguchi et al. 2011). In these cases the reduction of dynamic shear modulus and shear strength is explained by depolymerization of amorphous matrix substances, mainly hemicelluloses, during ageing. However, the reduction of $\tan\delta_L$ during wood ageing is in contradiction to the results of this research.

The diminished bonding strength between hollocelulose and matrix, confirmed by FTIR analysis, is proposed as a possible cause of stiffness reduction and increase of internal friction after the soaking of spruce tonewood. Alike explanation is found in related studies, where acoustical properties of wood were changed either by extractives removal (Brémaud et al. 2010) or re-injection of extracts into a “neutral” wood (Matsunaga et al. 1999; Matsunaga et al. 2000; Minato et al. 2010). However, there is still no convenient explanation of greater changes in shear- comparing to bending vibration response after the soaking treatment. Since wood anisotropy is scale-related, might the answer be found on higher, i.e. wood micro scale level. Several preceding studies on dynamic mechanical properties of wood did not find any correlation between dynamic modulus of elasticity and shear modulus (Ilic 2003; Leite et al. 2012).

Some studies predicted that the higher E_L/G_{LT} and higher $\tan\delta_S$ / $\tan\delta_L$ values of wood, which was a result after the wood soaking in this research, give larger acoustic loss at higher frequencies (Obataya et al. 2000; Ono 1996). It is also known that Sitka spruce wood, the preferred material for the soundboard of pianos, records high E/G value, and that its damping in the high frequency range is greater than that of other wood species (Meinel 1957; Ono and Kataoka 1979). The popular speculation is that the skills of the Cremonese violin makers, combined with the “secret ingredient” (or undocumented technique) gave also these instruments the rich sound (Gough 2000).

4 Conclusions

Owing to the complex wood structure, most changes of vibrational properties and FTIR spectral bands of spruce tonewood after the soaking treatment cannot be directly assigned to one single component. Nevertheless, the significant decrease of mass and density of spruce wood after the soaking in mineral solution is indisputable related to the reduced amount of lignin and hemicelluloses. Similar lignin structure changes were confirmed also at maple wood of two ancient violins of Stradivari and Guarneri. These compounds, especially the latter, decreased the hygroscopicity and shrinking of tonewood after the treatment, which might improve the resonance wood service life properties, when it is used in musical instruments at changing climate conditions. Generally, most of the acoustical parameters determined in bending vibration were up to

20% lower after the treatment, whereas specific modulus of elasticity was not altered. However, due to significantly greater reduction of dynamic shear modulus, combined by two-fold increased shear internal friction, the soaking treatment had undoubtedly positive impact on the acoustical anisotropy of Norway spruce tonewood.

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