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Influence of moisture on the vibro-mechanical properties of bio-engineered wood

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Abstract In this study, changes in the vibro-mechanical properties of fungi-treated wood, during sorption and desorption at different humidity levels, were investigated. Norway spruce resonance wood (with uniform narrow annual rings and high tonal quality for musical instrument craftsmanship) was incubated with *Physisporinus vitreus* for 36 weeks. Stiffness, internal friction, and tonal performance indices of control (untreated) and fungi-treated wood were compared after exposure to a stepwise variation of relative humidity. It was demonstrated that fungal treatment increased the internal friction and decreased the specific modulus of elasticity, during reduction of wood density. Internal friction of both control and fungi-treated wood significantly increased during dynamic sorption, especially during early stages (hours) of each humidity change step. Both specific modulus of elasticity and internal friction showed a hysteretic behavior during humidity variation cycles. Hysteresis was smaller in fungi-treated wood. Also, tonal performance indices were improved after fungal treatment and showed a reduced variation at different relative humidity conditions. Dynamic vapor sorption tests and FT-IR microscopy studies revealed changes in hygroscopicity and the supramolecular structure of wood, which may explain the observed vibrational behavior. Less dependency of wood vibrational properties to the variation of the ambient humidity is important for the acoustic performance of string instruments.

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Introduction

Wood is a hierarchically structured cellular composite with exceptional vibro-mechanical properties, which despite all the recent achievements of material scientists in designing new materials, is still the most essential material for musical instruments. The characteristics of the multi-scale structure of wood, including architecture and variation in the volume fraction of chemical components in different cells and their sub-layers, determine the density, hygroscopicity, and mechanical properties of different wood species [1]. These properties are important, for assessing resonance wood quality [2].

Due to the hygroscopicity of wood, moisture exchange with the environment results in dimensional instability (swelling/shrinkage) and changes in the physico-mechanical properties [3]. Also, changes in the relative humidity (RH) alter the vibrational properties [4–7]. This explains the significance of wood reaction and adaptation to the humidity of the environment to which it is exposed. In nature, the rate and quantity of water exchange with the surrounding atmosphere are often decreased for high density wood and content of extractives [8, 9], and also as a result of different modifications that change the supramolecular structure of the wood cell wall [10–12].

Wood modification by specific white rot fungi reduces density without significantly altering the stiffness of the material, as a result of selective delignification [13], and thus improves the acoustic properties [14, 15]. The main objective of the present study was to improve the understanding of the impact of incubation with *Physisporinus vitreus* (Pers.) P. Karst on the hygroscopicity and vibro-mechanical behavior of resonance Norway spruce wood. The hypothesis was that changes in the arrangement of the molecules of lignin, hemicelluloses, and cellulose in the fungi-treated wood will change the hygroscopicity and vibro-mechanical behavior of the material. Twin samples of control (untreated) and fungi-treated resonance Norway spruce wood were exposed to a stepwise change in the RH, and the vibro-mechanical properties were measured using a free–free beam vibration method. Changes in the moisture content (MC), specific moduli of elasticity, and internal frictions in a cycle of sorption–desorption were measured. Using Fourier transform infrared spectroscopy (FT-IR) and dynamic vapor sorption (DVS) system, change in molecular architecture of the wood and hygroscopic properties was analyzed. The expectation was to elucidate the reaction and adaptation of the fungi-treated wood with fungi to the change in the RH, for further improvement of the fungal modification processes.

Materials and methods

Sample preparation and fungal treatment

Two twin plates of 120 mm (*R*, radial direction) × 2.5 mm (*T*, tangential direction) × 420 mm (*L*, longitudinal direction) were excised from a plank of Norway spruce wood (*Picea abies* L.), with narrow and even annual rings and perfectly quartered with minimal run out according to the criteria of 'master grade' resonance wood for string instruments craftsmanship. Tree was felled in autumn 2009, in Sertig, Davos region, Switzerland. Out of these twin plates, six pairs of specimens were quarter-sawn in strips of 12 (*R*) mm × 2.5 (*T*) mm × 150 (*L*) mm. While half of the specimens were used as untreated controls, the remaining twin specimens were incubated in the dark at 22 °C and 70 % RH, with *P. vitreus*, according to the European Standard EN 113 [16]. After an incubation period of 36 weeks, which allows for obtaining the required weight losses for changing the vibrational properties, specimens were sterilized with ethylene oxide and stored in a climate chamber at 65 % RH at 20 °C. Figure 1 shows the two groups of control- and fungi-treated specimens. Due to the narrow width of growth rings in Norway spruce resonance wood, identification of the transition between the sapwood, the outermost portion of a tree trunk (which makes the center part of violin top plate), and the heartwood was not straightforward.

Vibrational test setup

A free–free resonance flexural vibration test was used to determine the first resonance frequency (f_R) and internal friction (tanδ) of the samples [17, 18]. In Fig. 2, details of the test setup are presented. A thin metallic plate of less than 20 mg weight was glued to one end of each specimen, for non-contact forced-released vibration on the face of an electric magnet. The specimens were fixed using two thin silk threads, at the location of vibration nodes of the first bending mode of beam element. Vibration was emitted by means of an electro-magnetic device, and displacements at

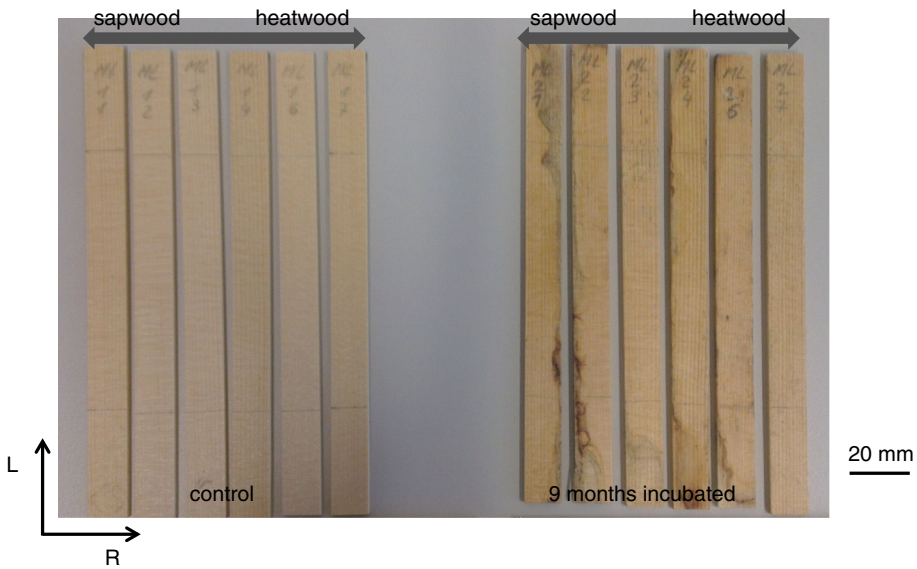

Fig. 1 Twin specimens of resonance Norway spruce wood—*left* control specimens. *Right* wood specimens after 9-months incubation with *Physisporinus vitreus*

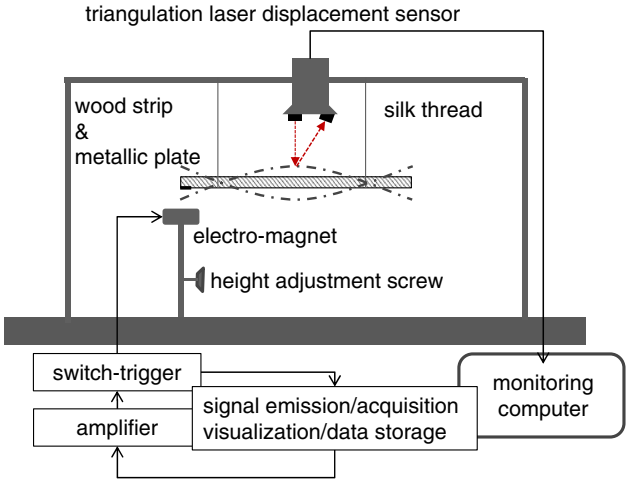

Fig. 2 Free–free flexural vibrational testing setup showing the wood specimen and the attached metallic plate, support silk threads, electro-magnet with adjustable heights, triangulation laser displacement sensor, and control algorithm for data acquisition and storage

anti-node of the first vibration mode were measured using a laser triangulation displacement sensor (Fig. 2). Vibration emission and acquisition of data were performed in an algorithm developed specifically for this purpose [19]. A broadband frequency scan was used to detect the first resonance frequency, on which a second, narrow-band scan allowed the determination of the quality factor Q (bandwidth at half-power). Then, the excitation (fixed at f_R) was stopped and the logarithmic decrement (λ) of amplitudes was recorded to compare the tanδ in both frequency ($1/Q$) and time (λ/π) domains. Given the high aspect ratio of the samples, the influence of shear and rotary inertia can be ignored. Thus, the specific modulus of elasticity was calculated by the f_R in Euler–Bernoulli equation:

$$\frac{E}{\rho} = \frac{48\pi^2 l^4}{m_n^4 h^2} f_{Rn}^2, \quad (1)$$

where l is the length of the sample, h is the thickness, f_{Rn} is resonance frequency of the mode n, and m is a constant depending on the mode order. Also, E is Young's modulus (MPa) along the specimen length, and ρ (kg m^{-1}) is the density of the material, thus the unit of the specific modulus of elasticity, E/ρ, is MPa m^3 kg^{-1}.

Experimental procedure

Before incubation of wood, vibrational properties of the designated specimens for fungal treatment were tested at equilibrium MC at 65 % RH and 20 °C. After incubation, to avoid changing of the mechanical and hygroscopic properties due to over time heating, all control and fungi-treated specimens were mildly dried in oven at 50 °C, over a week, with an instant increase to 80 °C for 2 h. Then, they were stored in a dry desiccator over silica gel particles and allowed to cool. The average dry density mass for the wood strips was 391 kg/m^3 (standard deviation of 11 kg/m^3).

Dry specimens were exposed to a stepwise variation of the RH from 30, 65, 80, 65, and 30 % at 20 °C. RH step exposures were implemented by storing the samples in different climate chambers with constant RH and temperature (20 °C). Experiments were performed inside the chamber, on a few points before and at equilibrium MC (at 1,7,14, and >28 days). The f_R and tanδ, together with the temporal change in the MC (precision balance, ±0.0001 gr) and dimensions (caliper, ±0.01 mm) of samples, were measured at each RH step, until equilibrium was completed. Each measurement was performed in triplicate, after specimens were exposed to a new RH step and after 1-, 7-, 14-, and 28-day conditioning (total of 180 measurements).

In addition to vibrational tests, changes in wood hygroscopicity after fungal treatment were investigated by a DVS test. DVS provides accurate sorption and desorption isotherms over a user set RH and a temperature range at which isotherms are recorded. Two replicate control and 36-week-incubated specimens (total of four) were tested in ten RH steps at adsorption (5–15–25–35–45–55–65–75–85–95 %) and ten at desorption. The sorption and desorption processes were run at a constant temperature of 25 °C over the full RH range. The target RH was kept constant at each step, until the sample moisture content change per minute was less than 0.001 % per minute over a 10-min period. This process allows for obtaining the equilibrium moisture content values within 0.1 % of the true equilibrium value at infinite time [11]. For small test wood specimens in DVS (less than 0.3 g of dry mass), each step lasted between 2 and 4 h, while for thin plates of wood like tested samples in free–free vibration, time of equilibrium took up to 4 weeks. Results were discussed together with the measured changes in the wood elemental composition after fungal treatment using Fourier transform infrared spectroscopy (recorded by a Tensor 27 FT-IR spectrometer, Bruker/Switzerland). For this purpose, 10 semi-thin replicate sections of wood were prepared with a rotary microtome from the control and 10 from the fungi-treated wood (thickness of 300 μm). For each specimen, the diamond crystal of an attenuated total reflectance accessory was brought into contact with the wood surface (a circle of about 1.5 mm in diameter) to be analyzed. All spectra were recorded between 4000 and 600 cm^{-1} with a resolution of 4 cm^{-1} and 64 scans per sample. A baseline correction was constructed in all spectra at four arbitrary wavenumbers (3960, 1784, 1545, and 782 cm^{-1}).

Analysis

The most important acoustical properties for selecting materials for musical instruments are the tanδ within the material, the speed of sound, c, the sound radiation, R, and the characteristic impedance, z. The tanδ and E/ρ in the control and fungi-treated specimens were evaluated for changes in the RH from Eq. 1. Also, c, with which sound travels through a material, R, and z are defined as the root of the E/ρ:

$$c = \sqrt{\frac{E}{\rho}} \tag{2}$$

$$R = \sqrt{\frac{E}{\rho^3}} \tag{3}$$

$$z = \sqrt{E\rho} \tag{4}$$

Resonance wood has both a high c and a high R [2]. R describes how much the vibration of a body is dampened due to sound radiation. Also, z is important for transmission of vibratory energy from one medium, like a soundboard, to another one, like air. Variation of these parameters in terms of change in the RH and their first derivatives (difference of successive data divided by difference of RH) were studied in the control and fungi-treated wood specimens.

Results and discussions

Variation of the vibrational properties after fungal treatment

In Fig. 3, ρ, E, and tanδ of the six pairs of twin specimens, at 65 % RH, are compared. For the fungi-treated specimens, experiments were performed once before incubation. Horizontal axes correspond to the data for sapwood on the left and heartwood on the right. Each bar represents one property of a single specimen, while the standard deviation between triplicate measurements of its stiffness, and internal friction was less than 0.03 and 0.3 %, respectively. Differences between densities of the twin specimens before treatment, according to their position in soundboard plates, were 0.5 %. Differences between the stiffness and the tanδ were 1.5 and 4.9 %, respectively. Proximity of the properties was expected as the compared specimens were matching twins, excised from wood with even grain lines. Thus, the differences between the properties of control specimens compared to their twins after treatment were assumed to be due to fungal decay.

After treatment, density and stiffness were decreased by 4.1 and 10.6 %, respectively, when compared to the prior

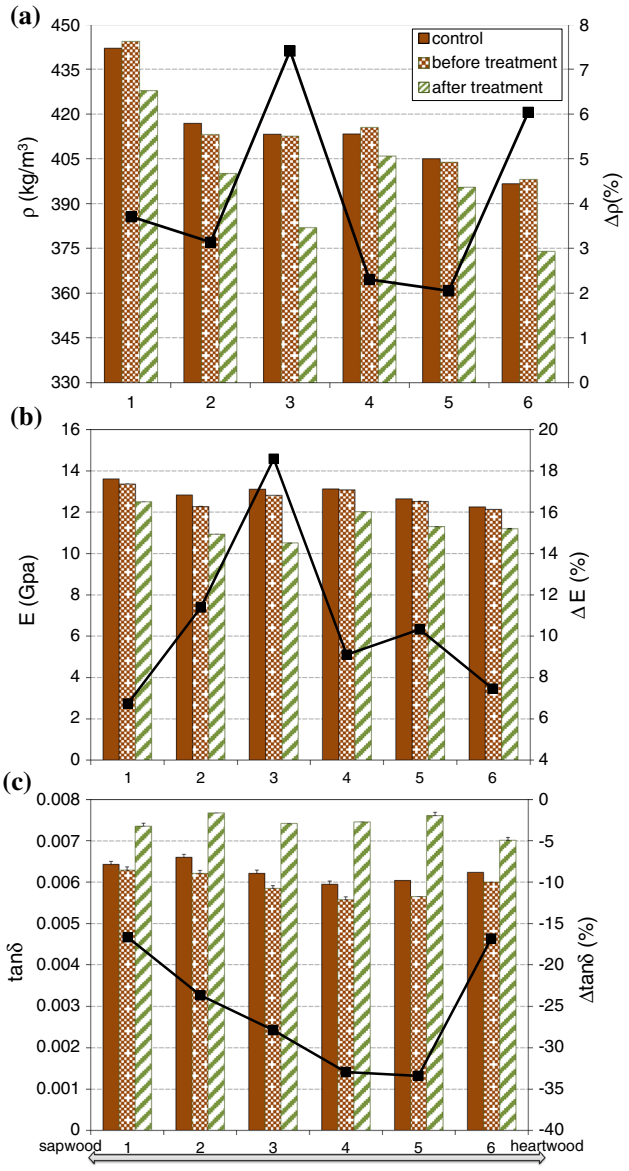

Fig. 3 **a** Density, **b** modulus of elasticity, and **c** internal frictions at 65 % RH, for six pairs of twin specimens (one control and one fungi-treated, before and after incubation)

to treatment results and 4.1 and 11.7 % when compared to the control specimens. Also, the tanδ was increased by 25 % compared to the prior to treatment results and 19.1 % when compared to the control specimens.

The control sapwood specimens had higher density, stiffness, and tanδ when compared to the heartwood. However, as the square markers (plot series on the secondary vertical axis) indicate, changes in the properties after treatment regarding the position of specimens were scattered. Former analysis on the structure of fungi-treated wood showed that *P. vitreus* acts more efficient in sapwood during decay process [20]; although in alternation of the vibro-mechanical properties, this trend was not identified.

Vibrational properties during moisture sorption

In Fig. 4, variation of the f_R and tanδ for control and fungi-treated samples, versus MC, is presented. Plots are based on the mean measured properties for six twin specimens, from sap- to heartwood, and their standard deviations for control and fungi-treated samples, as they were exposed to the stepwise variation of the RH (from dry to 30, 65 and 80 %). Measurements were performed after 1, 7, 14 and 28 days and conditioned at each RH step. Solid lines and markers with solid fill correspond to the control wood and the dashed lines and markers without filling represent the fungi-treated wood. Fungal treatment resulted in a decrease in f_R and increase in tanδ, at all adsorption ranges. During the 30 % RH step, f_R which is correlated with the material stiffness was increased by 4.5 Hz in the control and 2.9 Hz in the fungi-treated wood, while MC was increased by 1.6 and 1.5 %, respectively. Increase in f_R in sorption, below 8–10 % MC, is in good agreement with the literature [21, 22]. Contrary to f_R, tanδ was decreased by 13.8 % in the control and 10.3 % in the fungi-treated wood.

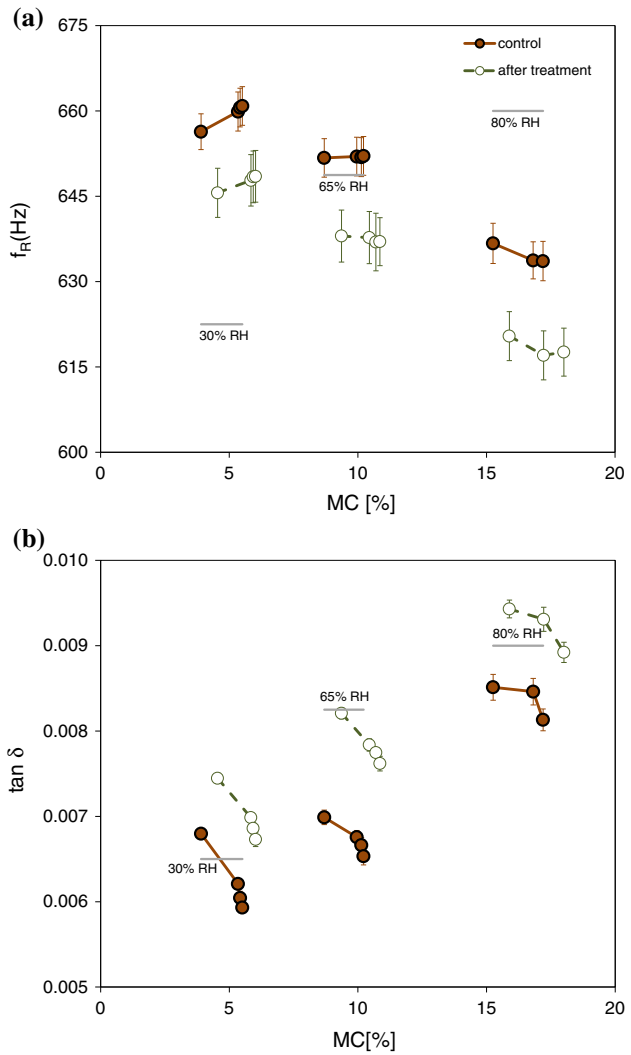

Fig. 4 **a** First resonance frequency and **b** internal friction of control and fungi-treated wood versus MC, for dynamic sorption at 30, 65, and 80 % RH steps. Successive points correspond to the measurements after 1-, 7-, 14-, and 28-day conditioning at each RH step

After the step change in RH to 65 %, and sequentially 80 %, f_R decreased while MC increased, as expected. Increase in the MC and the resulting decrease in f_R were always more significant for the first measurement (day 1) after a change in RH step. Also, tanδ showed a significant increase after the first day measurement, while it decreased gradually again during the next measurements after 7, 14, and 28 days. This relaxation was approx. 7 % for both fungi-treated and control wood at 65 % RH, and about 5 % at 80 % RH respectively. Deviations in the measured tanδ were larger at higher RH steps, while the standard deviation of f_R stayed at the same range in all RH steps. Increase in tanδ during early sorption state and its following relaxation indicate ensuing of different processes at different time scales. One process is diffusion of water molecules with a high rate into the porous structure when RH suddenly changed, while the other is a chemical reaction, formation and disruption of hydrogen bonds within the material until the equilibrium MC is gained [7]. This increases the capacity of solid wood for dissipation of vibrational energy in the first hours of RH change, while it is decreased in the course of adsorption.

Hysteresis of moisture exchange and vibrational properties

Figure 5a shows the isotherm curves of the control and fungi-treated wood. Deviation of the MC for two replicate experiments was less than 0.25 %, thus only one isotherm from each group was presented. The isotherm curves exhibited hysteresis, as the amount of water associated with the solid was greater for the desorption isotherm than for sorption. Hygroscopicity of wood was changed due to the fungal treatment. Equilibrium MC of wood at 95 % RH was 5 % higher in fungi-treated wood. An increased slope of the fungi-treated wood isotherm at high RHs indicates that a higher rate of sorption occurred after the vapor level exceeds a certain level, while below that, hygroscopicity of both materials was comparable. In Fig. 5b, the first derivative of MC, with respect to the change in RH, is shown. It highlights the higher moisture capacity of fungi-treated wood at high RHs, e.g., above 75 %.

In Fig. 6, changes in the E/ρ and tanδ of control and fungi-treated wood are compared. Each marker represents the average and deviation of the measured vibrational data, for samples that gained equilibrium after exposure to three

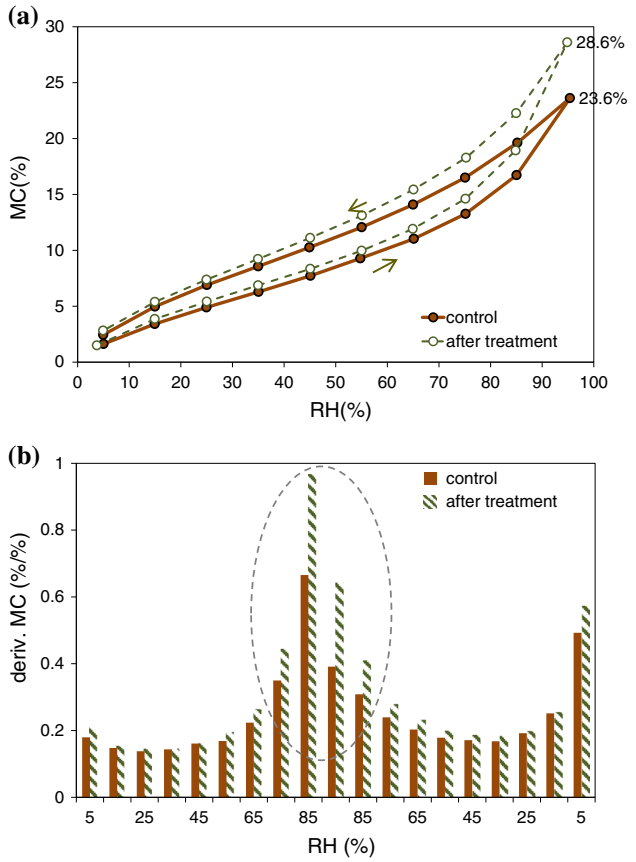

Fig. 5 **a** Moisture sorption–desorption isotherms for control and fungi-treated wood and **b** their first derivative. Differences between first derivatives of the control and fungi-treated wood are more significant at high RHs

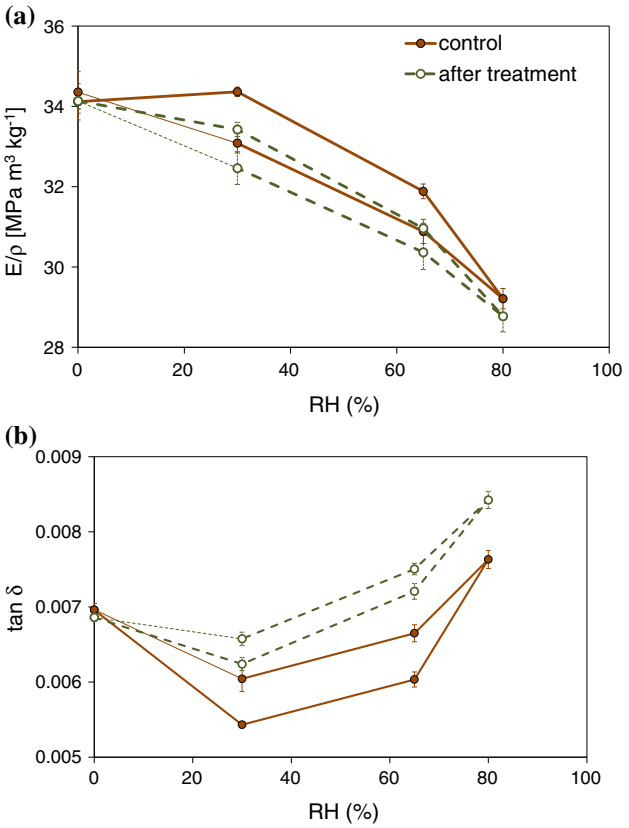

Fig. 6 **a** Specific modulus of elasticity and **b** internal friction of the control and fungi-treated wood versus RH in sorption–desorption cycle

RH steps in adsorption (0–30–65–80 %) and two in desorption (80–65–30 %). In all sorption and desorption ranges, E/ρ was lower and tanδ was higher for the fungi-treated wood when compared to the control. E/ρ decreased in sorption and increased in desorption, in line with the expected hygro-mechanical behavior of wood [22]. Also, tanδ increased in adsorption and decreased in desorption. Both E/ρ and tanδ showed hysteresis during humidity variation cycles. Unlike the dynamic vapor sorption in Fig. 5, the hysteretic behavior was more pronounced in the control wood when compared to the fungi-treated wood. While at 65 % RH, moisture hysteresis (difference between the absolute value in sorption and desorption) in the control and the fungi-treated wood was 3.1 and 3.5 % MC, respectively, and the hysteretic E/ρ was identified as 3.1 % for the control and 1.9 % for the fungi-treated wood. Hysteresis of tanδ was about 9 % for the control wood and 4 % for the fungi-treated wood. Also, the full hysteresis cycle areas under the E/ρ and tanδ were larger in the control wood, which indicates that following fungal treatment, and wood vibrational properties are less influenced by moisture exchange with the atmosphere.

In Fig. 7, c, R, and z of the control- and the fungi-treated wood, and their first derivatives (secondary vertical axis) during humidity variation cycle, are compared. Bold columns and solid lines represent the control wood, while columns with diagonal pattern filler and dashed lines represent the fungi-treated wood. Fungal treatment decreases the average c (1.1 %) and z (2.9 %) of the specimens but increases the R (0.9 %) over the whole RH steps. However, deviation of the results is quite large, due to the scattering between the properties of sap- and heartwood.

It is assumed that a high quality resonance wood possesses a high c and R but low z [2]. Thus, the fungal treatment improves R, which correlates with production of a loud sound and z which relates to transmission of vibratory energy from wood to the air.

All vibrational properties varied, versus change, in the RH. Also, the slope of the first derivative lines in the treated specimens was slower, and they were most likely above the derivative of the control wood. This indicates that the negative effect of high RH may be less significant

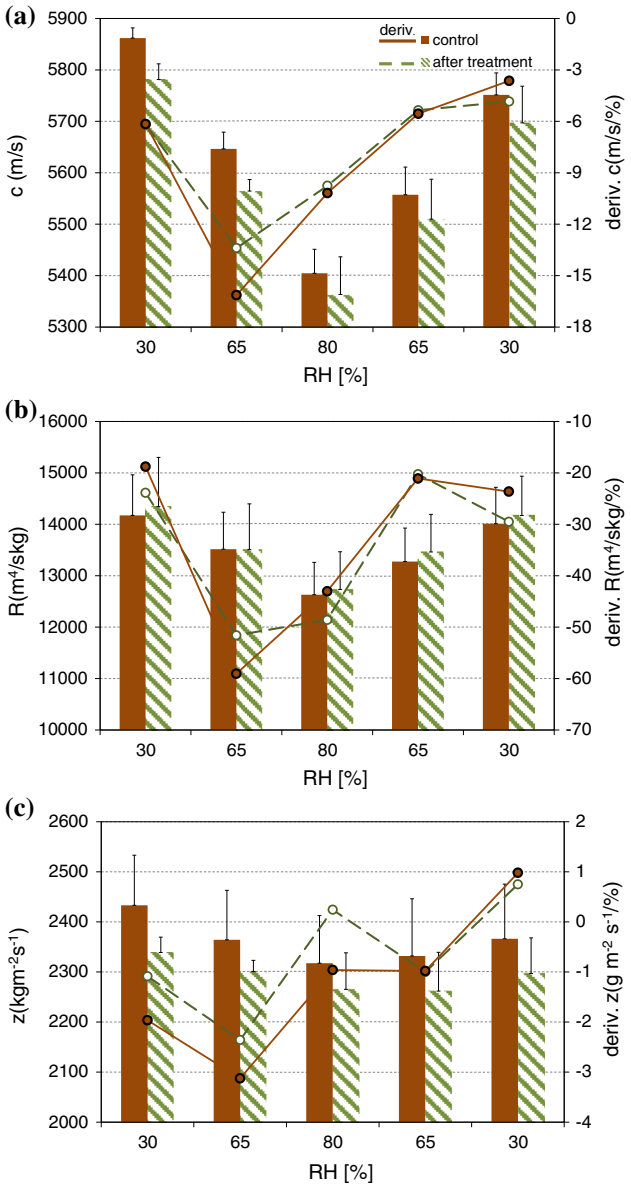

Fig. 7 Variation of the speed of sound (**a**), sound radiation ratio (**b**), characteristic impedance (**c**) and their first derivatives according to changes in RH. *Solid* and *dashed lines* represent the first derivatives for control and fungi-treated wood, respectively

in the fungi-treated wood hence the vibrational properties are less affected by the dynamic change in RH.

The fungi-treated wood showed an increased hygroscopicity but reduced dependency of the vibrational properties to moisture exchange. This contradictory behavior is possibly due to condensation of a fraction of water molecules in the cell wall voids that develop during fungal decay. The voids are within the range of 2–5 nm [23], thus based on Kelvin equation, condensation may take place even if the ambient atmosphere is not fully saturated with water vapor [3]. One observation that supports this assumption is the recorded increase in the moisture capacity of the fungi-treated wood with a higher rate, when RH exceeded a certain level (Fig. 5b). This means that not all the gained moisture during sorption is bound chemically to the cell walls, and thus does not affect the dimensional stability of wood [24]. Selective degradation of structural cell wall constituents by white rot fungi treatment can be another explanation for the observed behavior, as already evidenced with FT-IR spectroscopy [25]. Degradation of lignin and hemicelluloses occurs at the beginning of fungal colonization, while the cellulose fraction is degraded at rather later stages [14, 26]. Accordingly, in our study, FT-IR spectroscopy was used to qualitatively highlight any cellulose degradation after fungal treatment. All spectra were baseline corrected and normalized on the band peak at 1508 cm^{-1}, which purely arises due to aromatic skeletal vibration (C=C) in lignin. In Fig. 8a, FT-IR spectra of a control and a fungi-treated specimen are presented in the fingerprint region 1800–800 cm^{-1}. The spectrum of the control sample displayed the main characteristic vibrations of spruce at 1735 cm^{-1} (unconjugated C=O in hemicellulose), 1650 cm^{-1} (absorbed water and conjugated C–O), 1593 cm^{-1} and 1508 cm^{-1} (vibrations associated with the aromatic skeleton of lignin), 1462 cm^{-1} and 1423 cm^{-1} (C–H deformation in lignin and carbohydrates), 1375 cm^{-1}(C–H deformation in cellulose and hemicellulose), 1158 cm^{-1} (C–O–C vibration in cellulose and hemicellulose), 1104 cm^{-1} (aromatic skeletal and C–O stretch), 1051 and 1031 cm^{-1} (C–O stretch in cellulose and hemicellulose), and 898 cm^{-1} (C–H deformation in cellulose) (25, 10, 12).

After fungal treatment, a small decrease in the intensity of band characteristic of carbohydrate vibrations at 1375, 1158, 1051, and 898 with regards to the lignin reference band at 1508 cm^{-1} was observed. To support this observation, we have monitored the evolution of the ratio of the lignin peak height intensity at 1508 cm^{-1} against carbohydrate peak heights at 1375, 1158, 1051, and 898 cm^{-1} [25]. The baselines constructed for measuring the different peak heights are presented in Fig. 8b, and the results are summarized in Table 1. After fungal treatment, an increase of the $I_{Lignin}/I_{Carbohydrate}$ ratio has been obtained for all selected carbohydrate peaks. Since all spectra have been normalized to the band at 1508 cm^{-1}, independent from the degree of delignification in wood, the increase of this ratio confirmed the degradation of carbohydrates during the fungal treatment, which usually starts after lignin. This influences the volume fraction and accessibility of cellulose microfibrils inside the wood cell walls, and consequently the hygroscopicity and dimensional stability of wood.

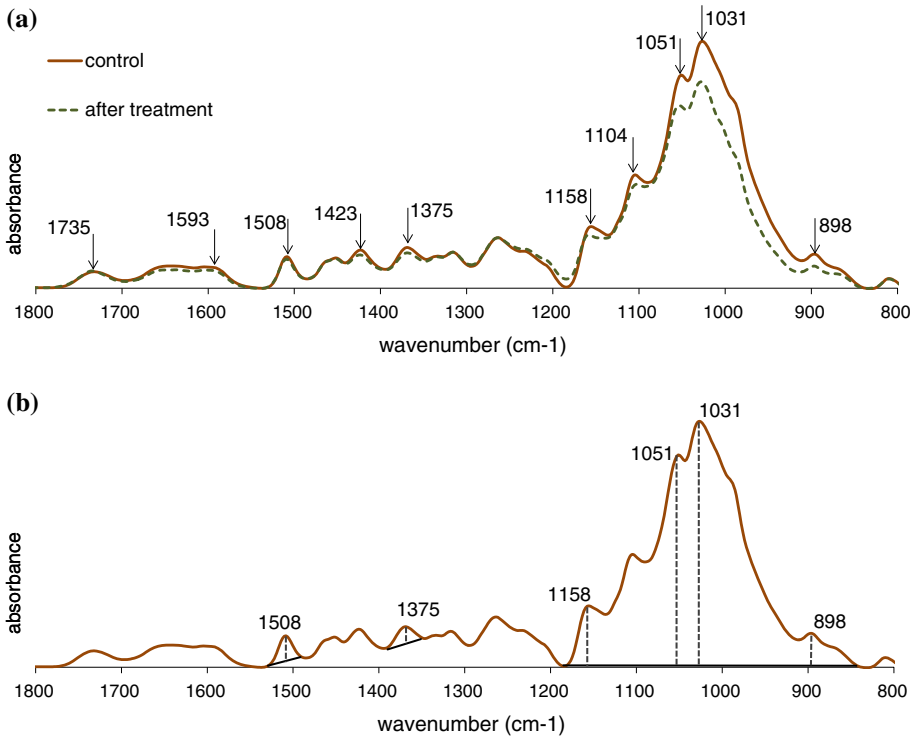

Fig. 8 **a** Qualitative comparison of the absorbance FT-IR spectra of control and fungi-treated specimens in the finger print region, **b** measurement of peak heights of 1508, 1375, 1158, 1051, 1031, and 898 cm^{-1} for control wood

Table 1 Ratios of the intensity of the lignin-associated band with carbohydrate bands

	Relative intensities, based on peak heights of lignin aromatic skeletal vibration against typical bands for carbohydrates				
	I_a/I_{1375}	I_a/I_{1158}	I_a/I_{1051}	I_a/I_{1031}	I_a/I_{898}
Control	1.375 (±0.04)	0.407 (±0.015)	0.118 (±0.003)	0.102 (±0.003)	0.739 (±0.035)
After treatment	2.000 (±0.120)	0.480 (±0.026)	0.139 (±0.007)	0.123 (±0.018)	1.131 (±0.152)

Standard deviation calculated for 10 replicates

Conclusion

Treatment of Norway spruce wood with *P. vitreus* can reduce wood density, a property for selecting resonance wood with superior tonal properties. Vibrational properties of the fungi-treated wood were compared with control twin specimens, under varying RH conditions. Moisture sorption decreased the specific modulus of elasticity and increased the internal friction in both control and fungi-treated wood. Increase of the internal friction in sorption was more significant after the first hours of change in RH, before the water exchanged with the atmosphere evolved to equilibrium. Wood decay resulted in slight degradation and changes in the molecular architecture in the lignified cell walls. Fungi-treated wood showed a greater hysteresis in moisture sorption and desorption, due to condensation of water in decay-associated nano-pores. In contrast, the specific modulus of elasticity and internal friction of fungi-treated wood were less affected by moisture in sorption and desorption. Also, tonal performance indices were improved and became less moisture dependent. This is an important prerequisite for a more reliable performance of wooden string instruments when they are exposed to changes in ambient humidity conditions. Further investigations on the relation between the initial properties of the resonance wood, e.g., density and stiffness, and the extent of variations in the tonal properties will help to standardize the modification process for manufacturing superior tonal quality string instruments.

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