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Original article

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Moisture content dependence of anisotropic vibrational properties of wood at quasi equilibrium: analytical review and multitrajectories experiments

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Abstract: This article aims at providing a synthetic view of the equilibrium moisture content (EMC) dependence of wood vibrational properties (i.e. dynamic mechanical properties in the audio-frequency range), including specific dynamic modulus of elasticity (E'/y) and damping coefficient expressing internal friction (tan δ). A series of multitrajectories experiments was designed to complete an analytical review. Literature indicates that: (1) in longitudinal (L) direction, the EMC dependence of E'/γ shows a very consistent shape (rather linear) between studies, while its shape is non-linear for $tan\delta$ and varies significantly between studies; (2) EMC dependence of $tan\delta$ is rather well documented in the L direction, in adsorption, for softwoods, but data covering EMC dependence in both L and other anisotropic directions, and sorption hysteresis, are still scarce. Experiments were conducted on a softwood (spruce) and a hardwood (maple), in L and radial (R) directions, in full adsorption from oven-dry state, full desorption from water-saturated state, and relative humidity (RH) loops without extreme conditioning. Measurements were made at conditions considered "at equilibrium" and some were monitored through time. Results indicated that $tan\delta$ was much more (×3) sensitive to EMC differences than E'/γ . R properties, especially tan $\delta_{\rm R}$, were much more $(\times 2-3)$ sensitive than L properties – resulting in strong increase of anisotropy with increasing EMC. In L direction, differences due to EMC remained moderate compared to the natural variability of wood for E'/y, while for tan δ the

EMC-induced changes were at least equal to natural variability in high-grade spruce. Vibrational properties did exhibit a hysteresis as a function of RH, but very little hysteresis as a function of EMC. The tan δ -EMC relation strongly depended on the actual time of stabilisation after reaching EMC. A related paper will address the transient, out of equilibrium effects of changing moisture conditions on the vibrational properties of wood.

Keywords: anisotropy; damping by internal friction; equilibrium moisture content dependence; sorption history; specific dynamic modulus of elasticity; viscoelastic vibrational properties.

1 Introduction

Wood, as a material, is characterised by its hierarchical structure and polymeric nature. The hetero-polymeric nature of wood induces a viscoelastic and hygroscopic behaviour. The cellular structure, and the fibre-matrix composite organisation of the cell-walls, makes wood highly anisotropic. The combination of anisotropy, viscoelasticity, and hygroscopicity is part of the complexity of the physical-mechanical behaviour of wood. The dependence of mechanical properties on equilibrium moisture content (EMC, defined as the stable moisture content reached after storage in given hygrothermal conditions) has been studied, over a range of species, for elastic moduli in the different directions and planes of anisotropy, and some analytical literature reviews provide general trends or statistical models (Gerhards 1982; Guitard and El Amri 1987). However, there is relatively fewer information for the EMC dependence of anisotropic viscoelastic properties, mostly considering that existing works are often with different methods and time/frequency scales.

Vibrational properties, i.e. dynamic mechanical properties in audio-frequency range, are an interesting mean to study the variations of wood viscoelastic behaviour,

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because they can be measured with high reproducibility, and on relatively small dimensions, that allow testing different material directions, and a reasonable time for moisture stabilisation. Vibrational measurements with the most widely used methods on free beams (e.g. Fukada 1950; Obatava et al. 2000b) can be very rapid, allowing to test numerous specimens (Ono and Norimoto 1983; Brémaud et al. 2012), and a single test provides information both on elastic (specific dynamic modulus of elasticity E'/y, where E' is storage modulus and y is the – dimensionless – specific gravity) and viscoelastic (damping coefficient tan δ which represents internal friction) behaviour. Therefore, vibrational properties are very useful for the integrated study of the biological, versus physical, sources of variation of wood mechanical behaviour. These properties are also of practical significance for several fields of utilisation of wood, an often cited use being musical instrument making (Brémaud 2012; Obataya et al. 2000b; Ono and Norimoto 1983; Yano and Minato 1992), while they are also studied for other types of uses, e.g. early wooden aircrafts (Greenhill 1942).

Existing literature data on the diversity of longitudinal viscoelastic vibrational properties (in "standard air-dry" condition) has been compiled over 450 species, and analysed as a function of biodiversity taxonomy (Brémaud et al. 2009) and/or of cultural diversity of uses (Brémaud 2012). Their anisotropy has been analytically reviewed over a hundred species to propose general trends (Brémaud et al. 2011). However, regarding their moisture dependence, information is still scattered, partly in some important and well-identified references (Obataya et al. 1998; Sasaki et al. 1988), but also in articles with a somewhat different focus or in not-easy-to-obtain references. Therefore, a preliminary goal of the present research was to compile and analyse the existing information on EMC dependence of vibrational properties, to better set the frame for the experimental work presented. At present, information on EMC dependence of vibrational properties of wood was identified within 26 references, ranging from the 1940s to recent years. As a general trend, it appears that the earlier references address both the between-species diversity and the multi-physical (moisture, temperature, frequency) dependence of vibrational behaviour (Fukada 1951; Greenhill 1942; James 1961; Kollmann and Krech 1960; Matsumoto 1962; Pentoney 1955; Sellevold et al. 1975; Suzuki 1962, 1980), while the later references tend to have a primary focus on how moisture content and vibrational properties are affected by wood modification (Ahmed and Adamopoulos 2018; Akitsu et al. 1993; Kubojima et al. 2000; Matsunaga et al. 2000; Obataya et al. 2000a, 2001; Sedighi Gilani et al. 2014; Yano and Minato 1992; Yano et al.

1986). This, of course, is only a general historical trend, and physical-mechanical behaviour and/or natural variability are still found as a main focus of studies on EMC-vibrational properties from the past 3 decades (Hunt 1997; Kubojima et al. 2005a; Jiang et al. 2012; Inokuchi et al. 1999; Obataya and Norimoto 1995; Obataya et al. 1998; Sasaki et al. 1988; Viala 2018). Table 1 summarises the information on studied species, on employed methodology (measurement and humidity conditions), if additional factors of material variability and/or of physical behaviour are studied, and which vibrational properties are studied for their EMC dependence.

These 26 references on EMC dependence of vibrational properties address mostly softwoods: 14 species in 23 references, while only six references deal with 15 hardwood species; monocotyledons (bamboo, reed) are addressed in two detailed references (Inokuchi et al. 1999; Obataya and Norimoto 1995) but their EMC-vibrational behaviour is drastically different from "standard wood". Indeed, "non-standard" chemical composition, notably peculiar extractives components in the case of untreated wood, can strongly modify both EMC, $tan\delta$, and their relation (Matsunaga et al. 2000; Minato et al. 2010; Obatava et al. 2001; Wangaard and Granados 1967). Most of the information quantitatively reviewed in Table 1 is based on wood without very atypical chemistry, thereafter considered as "standard" wood. Most references are based on measurements on beams; however, they do not all share completely comparable testing conditions or humidity changes protocols (as summarised in the third column of Table 1). Twenty-five references include data in longitudinal (L) direction for the EMC dependence of $tan\delta$, and usually of associated E'/γ (with four exceptions). Eighteen studies follow a single ad-sorption trajectory, while only two references present a comparison of ad- and de-sorption directions. Seventeen references include anhydrous/oven-dry properties, and five also include water-saturated condition. Furthermore, a few references (Gril 1988; Hunt 1997; Hunt and Gril 1996; Sasaki et al. 1988; Suzuki 1962) clearly indicate that reaching equilibrium moisture content, i.e. stable values of mass, does not necessarily indicate equilibrium, i.e. reaching stable values, in vibrational properties. But this is seldom discussed, and the duration of stabilisation varies between studies. Regarding anisotropy, not all directions and planes have been equally studied: the EMC dependence of viscoelastic vibrational properties is mostly documented in L and sometimes in R (radial) directions. For radial (R) direction, only 10 out of the 26 references detailed in Table 1 present quantitative data (Fukada 1951; Jiang et al. 2012; Kubojima et al. 2000; Kubojima et al. 2005a; **Table 1:** Summary of information contained in references from the literature on the moisture content dependence of viscoelastic vibrational properties.

References	Species ^a	Method and protocol				Vibrational properties ^f and their rates of change				Other studied parameters	
		Meth ^b	a/d ^c	nRH ^d	o/w ^e	tanð∟	<i>Ε'</i> _L /γ	tan $\delta_{ m R}$	$E'_{\rm R}/\gamma$	Var ^g	Phy ^h
Greenhill (1942)	5 species SW	≠b,f	а	3 (35–90)	_	1.5	-1.0	_	_	b	G
	5 species HW	≠b, <i>f</i>	а	3 (35-90)	_	1.1	-0.5	_	_	b	G
Fukada (1951)	1 SW + 1 HW	=	d	3 (5–65)	0	Y ^{nf}	Y ^{nf}	Y ^{nf}	Y ^{nf}	-	T, f
Pentoney (1955)	3 SW + 3 HW	=	da	8 (10-90)	0	5.9	_	12.6	_	b, ga	f
Kollmann and Krech (1960)	Picea abies	≠T,d	а	7 (10–90)	0+W	2.7	-1.2	_	_	γ	, f, G
	Quercus sp. (HW)	≠T,d	а	7 (10–90)	0+W	2.1	-1.2	_	_	γ	<i>f</i> , G
James (1961)	Pseudotsuga menziesii	≠f	d	7 (10–97)	_	2.1	-1.1	_	_	-	Т
Matsumoto (1962)	2 species SW	≠T,d	а	4 (10–75)	0	1.7	_	_	_	S	S
Suzuki (1962)	Chamaecyparis obtusa	≠b, <i>f</i> ,d	а	4 (8–61)	0	1.1	-0.9	_	-	-	tr
Sellevold et al. (1975)	Fagus sylvatica (HW)	=	а	6 (11–97)	0+W	1.8	-0.5	6.6	-1.3	-	Т
Suzuki (1980)	Chamaecyparis obtusa	≠b,f	а	5 (<i>30–97</i>)	0+W	3.3	-1.2	4.2	-1.4	b,γ	S
Yano et al. (1986)	Picea sitchensis	=	а	6 (29–85)	-	5.1	-1.4	9.5	-2.1	cm	f
Sasaki et al. (1988)	Chamaecyparis obtusa	=	а	5 (35–95)	-	3.4	-1.3	-	-	mfa	tr
Yano and Minato (1992)	Picea sitchensis	=	а	6 (30–85)	-	4.5	-1.3	9.5	-2.1	cm	f
Akitsu et al. (1993)	Picea glehnii	=	а	3 (35–85)	0	5.8	-1.4	-	-	cm	_
Obataya and Norimoto (1995)	Picea sitchensis	=	а	6 (10-90)	0+W	4.4	-1.6	-	-	-	-
	Arundo donax (M)	=	а	7 (10–90)	0+W	Y ^{nf}	Y ^{nf}	-	-	e	-
Hunt (1997)	Picea abies	≠b, <i>f</i>	a+d	6 (32–89)	-	4.1	-	-	-	-	tr,S
Obataya et al. (1998)	Picea sitchensis	=	а	9 (3–100)	0	3.8	-1.2	-	-	cw	-
Inokuchi et al. (1999)	2 Phyllostachys spp. (M)	=	а	7 (12–99)	0	Y ^{nf}	\mathbf{Y}^{nf}	-	-	w,e	-
Kubojima et al. (2000)	Picea sitchensis	=	а	5 (11–98)	0	2.2	-1.2	7.1	-2.6	w,tm	-
Matsunaga et al. (2000)	Picea sitchensis	=	а	3 (33–94)	0	5.6	-	-	-	e(b)	f
Obataya et al. (2000a)	Picea sitchensis	=	а	5 (11–92)	0	4.5	-1.4	-	-	tm	tr
Obataya et al. (2001)	Picea sitchensis	=	а	6 (11–97)	0	5.5	-1.6	-	-	e,cm	-
Kubojima et al. (2005a)	Picea sitchensis	=	а	5 (11–98)	0	-	-	15.1	-2.6		T,tr
Jiang et al. (2012)	Cunninghamia lanceolata	≠b, <i>f</i>	da	3 (30–90)	0+W	10.0	-0.7	10.2	-1.9	-	tr
Sedighi Gilani et al. (2014)	Picea abies	=	a+d	3 (30–80)	0	3.2	-1.3	_	-	bm	tr
Ahmed and Adamopoulos (2018)	4 species HW	=	da	3 (35–85)	-	1.8	-1.4	-	-	tm,cm	_
Viala (2018)	Picea abies	≠d	а	3 (33–75)	-	5.4	-0.9	15.0	-2.8	-	G,tr

^aSW: softwoods (gymnosperms), HW: hardwoods (angiosperms), M: monocotyledons (bamboo, reed). Species with no coding are softwoods. ^bMeasuring method. "=" means similar to the majority of literature, i.e. vibrations on free-free beams, frequency 100–2000 Hz, temperature 20– 25 °C, stabilisation at each condition ≥ 2 weeks; " \neq " means at least one parameter is different: (b) boundary conditions, (f) frequency range, (T) temperature, (d) duration of stabilisation.

^ca: ad-sorption protocol, d: de-sorption protocol, da: mixed directions of sorption, a+d: full protocols in both ad- and de-sorption.

^dnRH: number of RH steps tested (range of RH in brackets).

^e"o": properties tested at oven-dry (anhydrous), "w": data for water-saturated.

^fFor vibrational properties, whenever possible the "rate of change" (dimensionless, expressing % relative change in property per % absolute increase in EMC) was calculated in the respective column; "Y^{nf}" means that data are presented but did not allow to calculate a numerical figure (i.e. very different RH range, or shape of curves).

^gVar. lists studied factors of material variability: (w) within-species, (b) between-species, (γ) specific gravity, (cw) cell-wall properties, (mfa) microfibril angle, (ga) grain angle, (e) extractives, (m) wood modifications (cm: chemical, tm: thermal, bm: biological), (S) several (>2) factors. ^hPhy. lists studied factors of physical behaviour: (*f*) effects of frequency, (T) effect of temperature, (tr) transient effects, (G) shear properties, (S) several (>2) factors.

"-": no data; "numbers in italics": only RH or only EMC are presented (the figure in italics is estimated from isotherms closest to each study).

Pentoney 1955; Sellevold et al. 1975; Suzuki 1980; Yano and Minato 1992; Yano et al. 1986; Viala 2018), of which only three concern hardwoods, and eight are not readily

comparable to the other information sources (e.g. part of the humidity or vibrational parameters are not numerically presented, or the methodology is different). Eventually, it seems only one reference (Kubojima et al. 2000) provides a fully comparable dataset on both R and L directions, in ad-sorption, on a "standard" softwood.

Hence the purpose of the present research was to propose a simple, yet complete and comparable, dataset on the EMC dependence of viscoelastic vibrational properties for "standard" wood. To that effect, the presented work included a softwood and a hardwood (spruce and maple that, incidentally, are also musical instrument making woods), longitudinal and radial directions, ad- and desorption trajectories, anhydrous and water-saturated conditions, plus some considerations about the effects of measurements, of the duration of stabilisation, and of natural variability. In this paper, the focus will be put on equilibrium values of properties, including EMC, observed after a "sufficient" amount of time in given conditions of temperature (T) and relative humidity (RH), in the sense that further variations cannot be detected. This definition is somewhat vague, making the equilibrium depend on the precision of the equipment - and the patience of the observer. Here durations of one to several weeks were applied, and systematically recorded. In a related paper (Brémaud and Gril 2020), the intermediate measurements made during the transitory phases will be presented and analysed.

2 Materials and methods

2.1 Wood material and specimen preparation

The study was based on Norway spruce (Picea abies [L.] Karst.) as a softwood, and sycamore maple (Acer pseudoplatanus L.) as a hardwood. Material was selected for being straight grain and homogeneous. Specimens with dimensions of $120 \times 2 \times 12 \text{ mm}^3$ (R \times T \times L) for properties in radial (R) direction, of $12 \times 2 \times 150 \text{ mm}^3$ (R \times T \times L) for properties in longitudinal (L) direction, were cut with fine smooth surfaces using a well-sharpened planer. For the main part of the sampling, care was taken to reduce variability: material came from medium heartwood and specimens were matched following series cut along tangential (T) and L direction for L specimens, and series cut along L direction for R specimens. This homogeneous sampling, covering spruce and maple, was distributed between protocols ADS and DES (for "full Adsorption" and "full Desorption", described below). Another part of the sampling was intended at representing variability, it came from six boards of spruce pre-selected for violin making, sampling was done along the radius of boards and was used for protocols VC (for "Variability and Cycles", see below). The annual ring width ranged from 1 to 2.5 mm for both spruce and maple. The specific gravity (y, unitless) was of 0.63 ± 0.03 for maple (coefficient of variation, or COV = standard deviation/average, of 4%). For spruce, y was of 0.47 ± 0.02 (COV of 4%) for the ADS and DES sampling, and of 0.38 ± 0.05 (COV of 13%) for the VC sampling. The mass of specimens was of 2.3 g in average (from 1.6 to 3.2 g).

2.2 Vibrational measurements

Tests were done by non-contact forced-released vibrations of free-free slender beams, a most widely used method for wood vibrational properties (Fukada 1950; Obataya et al. 2000b, 2020). On this principle, measurements used a custom-built hardware and software device that reduces testing time and error (Brémaud 2006; Brémaud et al. 2012). Specimens were hung on thin flexible threads located at the position of vibration nodes of the first bending mode. Frequency sweeps were imposed without contact, through an electro-magnet facing a very thin iron plate glued on one end of the specimen. Displacement was measured at the centre of specimen by a laser triangulation displacement sensor. The setup and the geometry of specimens (very slender free-free beams) allow to precisely measure displacement at a given mode without interferences. Specific dynamic modulus of elasticity (E'/y) was calculated from the resonance frequency at the first mode of flexural vibration according to Euler-Bernouilli equation. Resonance frequencies were in the range of 100-300 Hz for R specimens and of 500-700 Hz for L specimens. Viscoelastic damping coefficient tan δ (\approx internal friction or loss factor) was obtained, through a finer frequency sweep, in the frequency domain by the bandwidth method (tan $\delta = Q^{-1}$ with *Q* being "quality factor").

2.3 Quantification of experimental errors

For this widely used vibrational method, several experimental sources of errors have been evaluated (Brémaud et al. 2012; Kataoka and Ono 1975, 1976; Kubojima et al. 2005b), and absolute values of properties have been found very reproducible by cross-comparison of literature data (Brémaud 2012; Brémaud et al. 2007, 2009). The present study evaluated some minute sources of errors that could interfere with fine measurements along humidity trajectories. Repeatability tests were conducted with 10 repetitions each time with different settings (Figure 1). The experimental error on frequency was 0.1 Hz, corresponding to an average relative error of 0.1%. The machining error, although very small in absolute value (0.03 mm in average), was of 0.8% relative to the 2 mm thickness of specimens, and had more effect on calculations of E'/y. The precision on damping/internal friction $(\tan \delta)$ determination by bandwidth $(\tan \delta = Q^{-1})$ is theoretically increased with an increase of the acquisition time (Brémaud 2006; Brémaud et al. 2012). Here the repeatability tests suggested this time to be fixed to 20 s, which gives a coefficient of variation of 1% (range of variation of 2.5%).

Figure 2a shows the relative variations in properties through time when a specimen previously stabilised (20 °C and 65%RH) is tested a few minutes after being taken out of the climatic equipment, in an uncontrolled room with a climate of circa 30 °C and 50%RH. A shortterm increase in damping was clearly seen, followed by gradual decrease due to lower ambient RH. This first increase followed by redecrease was typical of the phenomena of transient destabilisation, as evidenced by Hunt and Gril (1996) and further analysed in a related paper (Brémaud and Gril 2020). For precise measurements of wood damping, measurements should be conducted inside a climatic equipment. Furthermore, due to the relatively high temperature during this test, the experimenter's fingers were moist, and their contact with the specimen is clearly seen on Figure 2b, where it induced a transient increase in damping. Repeatability tests showed that manipulating specimens with tweezers had no effect, with dry fingers had very slight effect, but with artificially moistened fingers strongly



Figure 1: Repeatability test (10 goes) for measurement of damping coefficient (tan $\delta = Q^{-1}$) on longitudinal (L) and radial (R) spruce specimens. (Left) effect of the acquisition duration; (Right) effect of specimens unmounting and type of manipulation. COV, standard deviation/mean; ROV, (max-min)/mean.

increased the variation between repetitions (Figure 1 right). Therefore, all tests were conducted inside a climatic equipment, using 20s acquisition time, and specimens were manipulated with tweezers.

2.4 Trajectories of moisture changes

The whole wood material had been naturally air-dried for at least one year before sampling. After sampling and machining, all specimens were first measured for mass, dimensions and vibrational properties after at least 4 weeks in controlled conditions of 20 °C and 65%RH. Sub-samplings were used for studying different trajectories of changes in humidity conditions (Figure 3). The "full ad-sorption" (ADS) sampling included eight radial and eight longitudinal specimens of each species. They were first oven-dried (60 °C for 48 h followed by 103 °C for 2 h), then stored in a desiccator over silica gel (at 20 °C), then at 20 °C and 35, 50, 65, and 85% RH. The "full desorption" (DES) sampling was equivalent in specimens' number and in initially measured properties. DES trajectory started with water immersion of specimens, then going down the same RH conditions as above. The "Variability and Cycles" (VC) spruce sampling underwent RH loops without the extreme conditions (no oven-drying nor water-saturation). It was separated into a group of 34 axial and 10 radial specimens, which followed a "loop" from 65% RH, then 85% RH, then de-sorption, then up to 65% RH again (VC_{ada}). A smaller group (six axial and two radial specimens) followed the inverse loop (VC_{dad}), starting from desorption, then adsorption again up to 85%RH and back to 65%RH (Figure 3). For each humidity condition and protocol, mass, dimensions and vibrational properties of all specimens per group were measured after a minimum of 2 weeks for ADS and DES, of 3 weeks for VC, stabilisation in a given condition. Total number of vibrational measurements "at equilibrium" was of 1018, including repetitions.

For several of the humidity changes, two specimens of each direction (L and R) were used for monitoring the changes in properties through time, which was conducted for 1–5 weeks (usually about 3 weeks). To compare the changes after the exact same time-steps



Figure 2: Quantification of experimental errors on resonance frequency (f_R) and internal friction ($\tan \delta = Q^{-1}$) due to measurement's environment, when conducting tests in a room without climate control (on wood previously stabilized at 20 °C and 65%RH). Example on spruce in R direction. (a) Short-term effects (first 4 h of monitoring); (b) mid-term effects (2 days). Arrows indicate the variations in $\tan \delta$ due to the resolution of measurement (a), and to the manipulation with moist fingers (b). Dashed lines indicate general trend of change in $\tan \delta$ excluding the previous variations.

between the different experiments, data were then interpolated as a function of time, following a procedure described in a related paper (Brémaud and Gril 2020).

2.5 Collecting of literature data

Data were collected from 25 references, from 1942 to 2018, in English, German and Japanese. Information was gathered (Table 1) on the type of wood, geometry of specimens, duration of stabilisation, temperature and frequency of test, testing method and boundary conditions, studied material direction, and if additional factors of material variability and/or of physical behaviour were studied. In some cases, the main data on RH, EMC, E'/γ and $\tan\delta$ are presented as data tables. For most references, data are presented as figures, and the numerical values were extracted using the online software Webplotdigitizer®. The error due to this procedure was considered as small compared to the global sources of variability.

5



Figure 3: Samplings and trajectories of changes in humidity conditions. Numbers in the circles and squares: order of successive humidity conditions. Squares: measurement of oven-dry mass and dimensions, circles: measurement of mass, dimensions and vibrational properties. Number of days in each condition is indicated (durations might differ between specimens' groups).

3 Results and discussion

3.1 Analytical review of literature data

Information on the material, experimental parameters, and studied properties in the reviewed literature is detailed in Table 1. All values extracted from literature on the EMC dependence of L vibrational properties of softwoods and hardwoods are compared in Figure 4. For $E'_{\rm L}/y$, the trend is very similar to that observed for modulus of elasticity measured in other mechanical domains (Gerhards 1982; Guitard and El Amri 1987), with an initial plateau (or slow decrease) over the lowest range of MC (<5%), followed by a nearly linear decrease in the rest of hygroscopic range (Figure 4d). The dispersion is high between curves (60% between minimum and maximum values at "standard airdry" EMC of 10–12%), mostly reflecting variability between studied wood material. Nevertheless, once normalised as explained below, the trend is fairly consistent (Figure 4e), with a rate of change (expressed, dimensionless, as % relative change in $E'_{\rm L}/\gamma$ per % absolute increase in EMC) ranging from -0.5 to -1.6 (Table 1) with an average of -1.1. This rate of change has a ~30% coefficient of variation, defined as the ratio of standard deviation over average, which falls to ~25% when only data obtained by comparable measurement methods (see comment on protocol in Table 1) are taken into account.

For tan δ_L , EMC dependence (Figure 4b) generally follows the shape described in the most detailed reference (Obataya et al. 1998), with a peak at very low EMC, a minimum value of tan δ_L observed in average at about 6.5 (±1.7)% EMC, followed by a nearly linear increase up to about 20% EMC, slowing down at 20-30% EMC. The low-EMC peak in tan $\delta_{\rm I}$ is close to the inflexion point in the $E'_{\rm I}/$ y-EMC curve (Fukada 1951; Obataya et al. 1998; Suzuki 1962). The corresponding $E'_{\rm L}/\gamma$ or tan $\delta_{\rm L}$ were used as reference to calculate the relative values in Figure 4c, e. The strongly nonlinear shape of the tan $\delta_{\rm L}$ -EMC dependence is ascribed to the superposition of two mechanisms, one related to molecular rearrangement (drying stress or "packing effect" of water molecules) at low EMC, followed by one more directly related to the plasticising effect of adsorbed water in the main part of hygroscopic range (Fukada 1951; James 1961; Obataya et al. 1998; Suzuki 1962). The low-EMC peak is also ascribed to a transition of hemicelluloses which EMC position is shifted as a function of temperature and/or frequency (Fukada 1951; Obataya et al. 1998; Sellevold et al. 1975; Suzuki 1962). The low-EMC peak becomes predominant and followed by very slow increase at low (<0 °C) temperatures, while at high (>50 °C) temperatures the low-EMC peak gradually disappears and the tan δ increases over the whole hygroscopic range, with increasing rates of changes with higher temperatures (James 1961; Kubojima et al. 2005a).

Almost all compiled data in Figure 4 are obtained at "near ambient" temperature, and mostly in a comparable frequency range, i.e. although moduli and internal friction depend on frequency between quasi-static, dynamic and ultrasonic tests (Ouis 2002), detailed experimental results indicate a frequency-independent range of approx. 100–2000 Hz for air-dry damping (Ono and Kataoka 1979). Nevertheless, the tan δ -EMC curves exhibit a quite important dispersion (Figure 4b). The part linked to natural variability is of the same order as for E'_L/γ , but



Figure 4: Graphical synthesis of literature data on EMC dependence of longitudinal vibrational properties. (a) Relationship between EMC and relative humidity (RH); (b) damping coefficient $\tan \delta_{L}$ as a function of EMC; (c) relative $\tan \delta_{L}$: each curve is normalized by its minimal value of $\tan \delta_{L}$ (occurring at 6.5 ± 1.7% EMC); (d) Specific modulus of elasticity E'_{L}/γ as a function of EMC; (e) relative E'_{L}/γ : each curve is normalized by the value of E'_{L}/γ observed at the same EMC as the minimum in $\tan \delta_{L}$. Circles: softwoods; squares: hardwoods; filled symbols with plain lines: comparable methodology; open symbols with dotted lines: partly differing experimental parameters (see Table 1).

measurement methods appear to have a bigger effect on absolute values of $\tan \delta$ (notably different boundary conditions). Once normalised (Figure 4c), the $\tan \delta$ -EMC curves still appear quite highly dispersed. The average rate of change in $\tan \delta_L$ is of +3.7 (% relative variation in $\tan \delta$ per % absolute change in EMC), but ranges from +1.1 to +10.0 (Table 1), with a coefficient of variation of 60% (twice that of E'_L/γ) and still of 40% between the fully comparable methods. Most studied species in Figure 4 and Table 1 have low/standard extractives composition, otherwise extractives can completely modify EMC, $\tan \delta$, and their relation (Wangaard and Granados 1967, Obataya and Norimoto 1995, Matsunaga et al. 2000, Minato et al. 2010).

The limited information on hardwoods (six references) and on R direction (10 references) prevents from statistical analysis. It seems that rates of changes would be about twice higher in R than in L direction, and would tend to be lower for hardwoods (+2.6 and -0.9 for tan $\delta_{\rm L}$ and $E'_{\rm L}/\gamma$ vs. +3.7 and -1.2 on softwoods, respectively). However, these average values, compiled from a small number of

references with some methodological variability, can hardly be generalised.

3.2 Experimental EMC versus RH (sorption isotherms)

EMC at different relative humidity (RH) following ADS and DES protocols were similar to the general case (Glass and Zelinka 2010). Spruce and maple wood behaved in a very similar way (Table 2) and the degree of hysteresis between full desorption and full adsorption was comparable to literature (Sasaki et al.1988). On spruce (Figure 5), in partial sorption cycles VC the maximum reached EMC meets the curve for full adsorption, and the minimum reached EMC meets the curve for full desorption. It seems that a loop starting by increasing RH (VC_{ada}) results in larger hysteresis than a loop starting by decreasing RH (VC_{dad}). For ADS and DES protocols, moisture content in desiccator was of 1.4% for spruce (1.8% for maple) and

Table 2:	Equilibrium moisture content (EMC) of spruce and maple at
different	RH conditions.

Protocol	Species		EMC(%) at:				
		35% RH	50% RH	65% RH	85% RH		
Adsorption (from	Spruce	5.5	7.9	10.4	17.4		
anhydrous)	Maple	5.6	8.1	10.3	17.3		
Desorption (from water-	Spruce	7.1	10.1	13.1	20.5		
saturated)	Maple	6.9	10.1	13.3	21.1		

Protocols ADS and DES (see Figure 3).

that in water-saturated condition of more than 100% for both species.

3.3 EMC dependence of anisotropic vibrational properties in full ad- or desorption: softwood versus hardwood

Figure 6 summarizes The EMC dependence of anisotropic vibrational properties of spruce and maple in the ADS and DES protocols. With higher EMC, the specific modulus (Figure 6a–c) decreased in L direction, and markedly more so in R direction, resulting in a marked increase in the ratio of anisotropy, which is consistent with literature on EMC dependence of elastic orthotropy (Gerhards 1982, Guitard and El Amri 1987, Ozyhar et al. 2013), including for spruce and maple (Palka 1973, Sonderegger et al. 2013). The rate of change (expressed dimensionless for % relative variation in property per 1% absolute change in EMC), in adsorption, was -1.1 for $E'_{\rm L}/y$ and -2.7 for $E'_{\rm R}/y$ (+2.4 for ratio of anisotropy) in spruce, -1.3 and -2.8 (+2.2) in maple. Despite similar EMC dependence, maple had overall a lower $E'_{\rm L}/y$ than spruce, and a higher $E'_{\rm R}/y$, i.e. markedly lower (-35%) ratio of elastic anisotropy. The studied maple being straight-grained (not wavy), this smaller anisotropy might be due to a combined effect of wider microfibril angle, and of stronger reinforcement by rays (Alkadri et al. 2018).

The two species were less distinct in damping, than in moduli. After a minimal value at about 5.6% EMC, damping increased along with EMC (Figure 6d–f), in line with literature on L direction (Figure 4, Table 1). Damping increased dramatically more so in R direction, with a rate of change of +9.9 for spruce in adsorption (+7.0 for maple) compared to +2.4 (+1.7 for maple) in L damping. Thus, the anisotropic ratio of tan δ showed a much stronger EMC dependence (+5.8 in spruce, +4.4 in maple) than did the anisotropic



Figure 5: Partial isotherm of sorption (35–85% RH) for spruce wood. Triangles with bold lines: complete adsorption (filled symbols: samples ADS starting from oven-dry) and desorption (open symbols: samples DES starting from water-saturated); diamonds with thin lines: samples neither oven-dried nor water-saturated but submitted to cycles starting from 65% RH and subsequently in adsorption-desorption-adsorption (VC_{ada}), or in desorption-adsorption–desorption (VC_{dad}). Textured grey curve: average trend on wood (Glass and Zelinka 2010).

ratio of E'/y. This seems an interesting result, because, although it is intuitively known that R damping should be much more sensitive to moisture and/or chemicals entering the cell walls (Norimoto et al. 1986, Yano and Minato 1992, Minato et al. 2010), there are not many comparable published data on the EMC dependence of anisotropic damping, as stated in the introduction. From the literature reviewed in Table 1, the rates of changes in $\tan \delta_R$ appear to be from 1- to 4-fold higher than those in $\tan \delta_L$. In the present results, averaged over the whole ADS and DES sampling, they showed a 3.8-fold difference.

The shapes of curves (Figure 6) were consistent with literature on the L direction. Values for water-saturated wood appeared in the continuity of the trends in hygroscopic range, in line with a few previous works on vibrational properties above and just below fibre saturation point (Kollmann and Krech 1960, Sellevold et al. 1975, Obataya and Norimoto 1995, Jiang et al. 2012). At very low EMC, the plateau in E'/γ was not much pronounced in the present results in L direction (Figure 6), which is similar to many curves in Figure 4d,e, but this plateau was clearer in R direction. The low-EMC maximum in tan δ occurred at



Figure 6: EMC dependence of specific modulus of elasticity (E'/γ) : (a, b, c) and damping coefficient (tan δ : d, e, f), in longitudinal (a, d) and radial (b, e) directions, and degrees of anisotropy (c, f). Triangles: spruce; circles: maple; filled symbols: full adsorption (ADS); empty symbols: full desorption (DES); for water-saturated (WS) data on specific modulus, hatched symbols are raw experimental data and open symbols are data corrected for the contribution of the mass of free water. Error bars: standard deviation between eight specimens per group.

1.6% EMC and had a value 1.29 higher (L direction), or 1.21 higher (R direction) than the minimum value of $tan\delta$ (Figure 6). This high value of $tan\delta$ at very low EMC (between 0 and 5%) is in line with literature (Figure 4b,c), where, in average, a peak occurs at 1.7% EMC and the above ratio for tan $\delta_{\rm L}$ is 1.21 (1.06–1.36). Above 5.5% EMC, for the well-documented L direction, it appears that average results of ADS and DES protocols were very consistent with literature values regarding the rates of change in $E'_{\rm L}/y$ (-1.14 in Table 1 compared to -1.16 in Figure 6), whereas the average rates of changes in $tan \delta_{\rm L}$ from ADS and DES protocols (+2.2, for spruce +2.5) were slower than the average from literature (+3.7 for softwoods, +2.6 for hardwoods, although literature trends are highly dispersed). ADS and DES trajectories were similar for E'/y(only slightly below in full de-sorption) and $tan \delta_{R}$, but $\tan \delta_{\rm L}$ appeared lower in *de*-sorption that in *ad*-sorption, which is counter-intuitive. These discrepancies suggest studying how the shape of the EMC dependence of damping can be affected by factors such as the history of specimens (conditions and duration of previous drying and/or wetting), hysteresis in the hygroscopic range, and natural variability.

3.4 Effect of stabilization time on "E"MC dependence of vibrational properties

In the literature studies on wood viscoelastic vibrational properties, specimens are stabilised at least a few days, most usually from 1 week up to 1 month, in a given RH condition (or sometimes "until weight is stable"), but the effect of the duration of stabilisation is seldom quantified. Although a perfect equilibrium is never reached when stable conditions are imposed, neither for moisture content nor vibrational properties, significant differences of kinetics are observed until a reasonable level of equilibrium is reached. When changing humidity, the damping coefficient does not change linearly nor at the same speed as MC. Tan δ first undergoes a strong transient increase (that can be called destabilisation), followed by re-decrease (Suzuki 1962, Sasaki et al. 1988, Hunt 1997). This second phenomenon, i.e. slow reach towards a new equilibrium after a destabilisation, is called physical ageing (Hunt and Gril 1996). Present experiments of monitoring through time confirmed this two-stage behaviour and are analysed in a related paper (Brémaud and Gril

2020). Here, they were used to calculate the apparent relation between $tan\delta$ and MC, assuming different fixed duration for each step of RH. First, within a given step, a linear relation was drawn between values of $tan\delta$ and of MC both considered at equilibrium (i.e. after 22 days corresponding to the average 3 weeks employed in the literature), on which the actual change in MC after Ndays (4, 7, 12 and 22) were plotted (on x-axis). Then the measured destabilisation (transient increase) in $tan\delta$ through time was added to these relations (on yaxis). For each RH step, the next initial point would start from this Ndays calculation, the next final point would tend towards the measured values at 22 days (supposed equilibrium) - but stop at the relative changes for Ndays – and so on. Monitoring experiments were not always sufficient to calculate this on all samples and protocols, but the general trends should be comparable to the results shown in Figure 7 for ADS protocol on spruce in L direction.

The position on the *x*-axis (MC) was little affected by times of stabilisation above 7 days. On the contrary, the position on the *y*-axis (tan δ) was notably shifted to lower values with longer duration of stabilisation. This means that equilibrium was sooner reached for MC, than for tan δ . Therefore, the reaching of equilibrium in MC is a poor indicator of the reaching of equilibrium in viscoelastic properties. Rates of changes (per % MC, as defined above) increased from +2.4 to +2.9 between 4 and 22 days. The apparently low (+2.5) rate of change of this actual ADS experiments (with varying durations between steps, Figure 7) was mostly due to a very long (101 days) stabilisation time for the last step at 85% RH. This also suggests that 3 weeks stabilisation (considered as a standard) is sufficient to obtain consistent and comparable trends, but not sufficient to reach real equilibrium in tan δ . The detailed analysis and discussion of these different kinetics in reaching equilibrium in moisture content, and in tan δ , is developed in a related paper by the authors (Brémaud and Gril 2020).

3.5 Is there a hysteresis in vibrational properties along cycles of RH?

In the results of Figure 6, a very slight hysteresis against EMC was observed for E'/γ (more pronounced when plotted against RH), and a clear but counter-intuitive (i.e. $\tan \delta$ lower in de-sorption than in ad-sorption) EMC hysteresis of damping was observed (less pronounced when plotted against RH). However, in ADS and DES trajectories, the effect of history could not be ruled out. Destabilisation due



Figure 7: Effect of the duration of stabilisation at each RH, on the apparent relation between damping coefficient and "equilibrium" moisture content. Sampling ADS, spruce in L direction. Filled symbols: experimental data on the whole group of specimens, actual number of days of stabilisation at each RH (35, 50, 65, 85%) are shown (shaded dot: data in desiccator, with short stabilisation time). Open symbols: curves of $\tan \delta$ versus MC, calculated from experiments of monitoring through time on two specimens from the same group, assuming the stabilisation time between RH steps would be of 4, 7, 12 or 22 days.

to drying, but also to water re-saturation, are observed in DMTA – Dynamic Mechanical Thermal Analysis (Furuta et al. 1995, Kojiro et al. 2008), and complex reversible/ irreversible effects of ageing, seasoning, heating and moistening on EMC and vibrational properties are acknowledged (Obataya et al. 2000a, Obataya and Tomita 2002, Obataya 2017, Obataya et al. 2020). Water immersion could also remove extractives and reduce damping, as found in reed and bamboo (Obataya and Norimoto 1995, Inokuchi et al. 1999), maybe also in maple (Roohnia et al. 2015), but not in spruce (Sonderegger et al. 2008).

In protocols VC (VC_{ada} and VC_{dad}), most RH steps lasted about 5 weeks (Figure 3) and samples were cycled without being oven-dried nor wetted. A hysteresis was indeed found as a function of relative humidity (Figure 8a,b), both in specific modulus of elasticity and in damping coefficient, and this hysteresis was more pronounced in R direction. However, as a function of internal EMC (Figure 8c,d, VC_{ada}), there was nearly no observable hysteresis for vibrational properties in L direction, and only a very weak one in R direction. Residual EMC hysteresis seemed even smaller in the VC_{dad} cycle starting by desorption. A reduction in damping is found along repeated humidity cycles (Akahoshi and Obataya 2015, Zhan et al. 2015).

This result seems interesting as, to the best of the authors' knowledge, there are very few information on the





adsorption/desorption asymmetry in vibrational properties, and they suggest contrasted trends (Hunt 1997, Sedighi Gilani et al. 2014). The present results on damping, i.e. hysteresis as a function of RH but not of EMC, go in the opposite direction of those of (Hunt 1990) on longitudinal shrinkage, where hysteresis is found as a function of EMC but not of RH, which suggests different fine mechanisms are involved. The existence – or not – of a hysteresis in hygro-mechanical properties, and its physical meaning, is discussed (Hunt 1990, Ishimaru et al. 2001). Ishimaru et al. (2001) suggest that hysteresis might be an effect of time and of very slow reach to equilibrium state, and that it might eventually disappear (even in sorption isotherms) after a very long period.

3.6 EMC dependence versus material variability of wood vibrational properties

The analytical review of literature highlights a quite high dispersion of curves (Table 1 and Figure 4). A first question is the respective effects of EMC dependence, and of material variability. By using a same methodology and

sampling, in the present results, between 34 longitudinal spruce specimens (VC_{ada}, Figure 9a,b), the natural variability amounted to 39% (ROV: range of variation= [max–min]/average) for $E'_{\rm L}/\gamma$ and 29% for tan $\delta_{\rm L}$. The variability induced in a given specimen by 7-17% EMC amounted to, in average, 15% for $E'_{\rm L}/y$ and 32% for tan $\delta_{\rm L}$. Here the effect of EMC was much lower than that of natural variability for $E'_{\rm L}/y$, but of the same amplitude for tan $\delta_{\rm L}$. On a wider within-species variability, on hinoki (Chamaecyparis obtusa) with microfibril angles ranging from 3° to 20° (Sasaki et al. 1988), the ROV due to natural variability is of 124% for $E'_{\rm L}/\gamma$ and 67% for tan $\delta_{\rm L}$, but the equivalent EMC-induced variability is as in the present results: 14% for $E'_{\rm L}/\gamma$ and 32% for tan $\delta_{\rm L}$. Therefore, $E'_{\rm L}/\gamma$ is much more sensitive to natural variability than $tan \delta_L$, and $tan \delta_L$ is much more sensitive to moisture-induced variability than $E'_{\rm L}/y$. This, however, holds true only for "standard woods" with similar chemical composition, as particular extractives components of some species can completely modify both the "air-dry" damping, its EMC-dependence (Obataya and Norimoto 1995, Inokuchi et al. 1999, Matsunaga et al. 2000, Obataya et al. 2001) and its anisotropy (Minato et al. 2010).

A second question raised is that of the respective effects of methodologies, and of material variability, in the



Figure 9: Variability induced by EMC dependence versus natural variability. Thin plain lines (in [a, b]) and data symbols (in [c]): 34 longitudinal (in [a, b, c]) and 10 radial (in [c]) specimens of "high quality" spruce wood, sampling VC_{ada}. Thick dotted curves: data on hinoki (*Chamaecyparis obtusa*) with a wide range of microfibril angle (Sasaki et al. 1988). (a, b) EMC dependence of longitudinal *E* '/ γ (a) and tan δ (b); (c) relation between tan δ and *E*'/ γ measured at different RH (R and L directions).

dispersion of the rates of change relative to EMC variations. Between the 34 VC_{ada} spruce specimens (Figure 9a,b), rates of change showed a COV (=standard deviation/average) of 12% for $E'_{\rm L}/\gamma$ and 18% for tan $\delta_{\rm L}$. When isolating literature data obtained with comparable methodologies and only on *Picea* species, the COV in rates of change are of 12% for $E'_{\rm L}/\gamma$ and 28% for tan $\delta_{\rm L}$ (for a range of natural variability similar to that of VC_{ada} sampling for $E'_{\rm L}/\gamma$ but 1.6-fold higher for tan $\delta_{\rm L}$). As discussed in preceding sections, tan δ

is much more sensitive to minute variations in conditions, but even in similar conditions rates of change relative to EMC are always more variable for tan δ than for $E'_{\rm L}/y$. This raises a third question, about the determinants of variability of rates of change.

It is well known that hygroscopicity and viscoelasticity are determined by components of cell-wall matrix, while elastic stiffness is driven by cellulose microfibrils (Norimoto et al. 1986). With increasing microfibril (or grain) angle, the hygro-viscoelastic behaviour of matrix should have more impact on macroscopic behaviour (Obataya et al. 1998, Obatava et al. 2000b, Brémaud et al. 2011). This is in line with the much higher rates of change in vibrational properties along EMC in R than in L direction (Table 1, Figure 4, Figure 6, Figure 8). In L direction, specific modulus of elasticity is a good indicator of microfibril or grain angle (Norimoto et al. 1986, Brémaud et al. 2011, Brémaud et al. 2013). However, when plotting rates of change of VC_{ada} sampling against initial air-dry $E'_{\rm L}/\gamma$ (65%) RH), no correlation was found. With decreasing $E'_{\rm L}/\gamma$, individual slopes decreased for $E'_{\rm L}/y$ and increased for tan $\delta_{\rm L}$ (Figure 9a,b), but rates of change, relative to individual specimen, were not explained by initial values of $E'_{\rm L}/y$. With the much wider within-species variability studied by Sasaki et al. (1988), rates of change in $E'_{\rm L}/y$ are very strongly correlated to variability of air-dry $E'_{\rm L}/y$, and more strongly so with MFA, but rates of change in tan $\delta_{\rm L}$ have no significant correlation. On average values from nine references in Table 1 having the most comparable methodologies on Picea spp., a significant correlation is found between air-dry $E'_{\rm L}/\gamma$ and rate of change of both tan $\delta_{\rm L}$ and $E'_{\rm L}/\gamma$: the lower the air-dry $E'_{\rm L}/\gamma$, the higher the rate of change per % EMC. On the contrary, the usually lower rates of change (as found in average in the literature) for hardwoods, despite their usually lower $E'_{\rm L}/\gamma$ than softwoods, appears counter-intuitive with the tendencies above.

As tan δ and E'/γ both depend on microfibril (or grain) angle, they are linked by a strong, species-independent relation in the form of a power-law (Ono and Norimoto 1983, Brémaud et al. 2012). On this curve (Figure 9c), orientation of wood elements determines the position on the *x*-axis, while chemical differences cause a vertical shift from the reference curve (Sasaki et al. 1988, Obataya et al. 2000b, Brémaud et al. 2012, Brémaud et al. 2013). As EMC induces variations in both tan δ and E'/γ , it has been proposed (Gril 1988) to use this representation as a kind of abacus of the respective effects of orientation and of moisture, allowing to switch microstructural unknowns and physical uncertainties. Present results on L and R variability of vibrational properties in the range of 35–85% RH were in very good agreement with the trends from a wider variability in the single L direction obtained by Sasaki et al. (1988) on a different softwood species. Therefore, these trends could be useful to estimate the vibrational properties at different RH/EMC based on an initial value of specific modulus of elasticity.

4 Conclusions

The goal of this article was to provide a synthetic view of the "equilibrium" moisture dependence of anisotropic vibrational properties of wood. A complete experimental campaign was conducted and compared to an analytical review of literature. Main results were as follows:

- Tanδ was much more sensitive to EMC than *E'*/*y*, and R properties much more than L ones. Anisotropy increased strongly with EMC. Rates of changes per % EMC were in average +2.8 (L) and +8.1 (R) for tanδ, and -1.2 (L) and -2.4 (R) for *E'*/*y*. Hardwoods could have smaller rates of change than softwoods despite their lower anisotropy.
- Rates of changes were rather well conserved for *E'*/*y*, but they varied (significantly in the present work, widely in literature) for tanδ (very sensitive to minute experimental and physical factors).
- EMC-induced changes in E'/γ were smaller than its natural variability, while for tan δ EMC-induced changes were at least as important as the usual amplitude of natural variability.
- Identifying structural indicators for rates of changes was not obvious. However, the curve relating $\tan \delta$ and E'/γ was consistent between studies, and its shifts as a function of EMC could serve as a kind of abacus.
- Very little if any ad-/de-sorption hysteresis in vibrational properties was found as a function of internal EMC.
 Oven drying or water saturation could cause shifts in tanδ resembling a hysteresis, but most likely they rather expressed some kind of destabilisation.
- Equilibrium in moisture content was a bad indicator of stabilisation in tanδ, which had a slower reach to equilibrium. The actual duration of stabilisation at different humidity strongly influenced both nominal values, and rates of change of tanδ.

Several of these observations indicate that $\tan \delta$ can be affected by various "destabilisation" phenomena. Nonequilibrium destabilisation in vibrational properties when changing humidity will be analysed in a related paper extending the present research. Experiments and literature analysis indicate that $\tan \delta$ is much more sensitive than E'/γ to various physical factors, but proportionally less to structural determinants. But both EMC and $\tan\delta$ can be strongly modified by non-standard chemical composition. It would be enlightening to pursue the study of EMC dependence of vibrational properties to a diversity of species with very atypical chemistry. Such studies should contribute to linking biological diversity, and complexity of physical behaviour of wood. These results also have important practical implications, both for scientific experiments and for real-life wood usages, notably in musical instruments but not only, and the collected dataset could also be useful for modelling studies.

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References

- Ahmed, S.A. and Adamopoulos, S. (2018). Acoustic properties of modified wood under different humid conditions and their relevance for musical instruments. Appl. Acoust. 140: 92–99.
- Akahoshi, H. and Obataya, E. (2015). Effects of wet-dry cycling on the mechanical properties of *Arundo donax* L. used for the vibrating reed in woodwind instruments. Wood Sci. Technol. 49: 1171–1183.
- Akitsu, H., Norimoto, M., Morooka, T., and Rowell, R.M. (1993). Effect of humidity on vibrational properties of chemically modified wood. Wood Fiber Sci. 25: 250–260.
- Alkadri, A., Carlier, C., Wahyudi, I., Gril, J., Langbour, P., and Brémaud, I. (2018). Relationships between anatomical and vibrational properties of wavy sycamore maple. IAWA J. 39: 63–86.
- Brémaud, I. (2006). Diversity of woods used or usable in musical instruments making, PhD In: Mechanics. University Montpellier 2, Montpellier, France, p. 302.
- Brémaud, I., Thibaut, B., and Minato, K. (2007). A database linking woody species, vibrational properties, and uses in musical

instruments of the world. In: *ISMA2007 (International Symposium on Musical Acoustics)*. Barcelona, Spain, paper 3-S2-4.

Brémaud, I., Minato, K., and Thibaut, B. (2009). Mechanical damping of wood as related to species classification: a preliminary survey.
In: 6th Plant Biomechanics Conference PBM09. 16–21 November 2009, Cayenne, French Guyana, pp. 536–542.

Brémaud, I., Gril, J., and Thibaut, B. (2011). Anisotropy of wood vibrational properties: dependence on grain angle and review of literature data. Wood Sci. Technol. 45: 735–754.

Brémaud, I. (2012). Acoustical properties of wood in string instruments soundboards and tuned idiophones: biological and cultural diversity. J. Acoust. Soc. Am. 131: 807–818.

Brémaud, I., El Kaïm, Y., Guibal, D., Minato, K., Thibaut, B., and Gril, J. (2012). Characterisation and categorisation of the diversity in viscoelastic vibrational properties between 98 wood types. Ann. For. Sci. 69: 373–386.

Brémaud, I., Ruelle, J., Thibaut, A., and Thibaut, B. (2013). Changes in viscoelastic vibrational properties between compression and normal wood: roles of microfibril angle and of lignin. Holzforschung 67: 75–85.

Brémaud, I. and Gril, J. (2020) Transient destabilisation in anisotropic vibrational properties of wood when changing humidity. Holzforschung. https://doi.org/10.1515/hf-2020-0029.

Fukada, E. (1950). The vibrational properties of wood I. J. Phys. Soc. Jpn. 5: 321–327.

Fukada, E. (1951). The vibrational properties of wood II. J. Phys. Soc. Jpn. 6: 417–421.

Furuta, Y., Yano, H., and Kajita, H. (1995). Thermal-softening properties of water-swollen wood I. The effect of drying history *(in Japanese)*. Mokuzai Gakkaishi 41: 718–721.

Gerhards, C.C. (1982). Effect of moisture content and temperature on the mechanical properties of wood : an analysis of immediate effects. Wood Fiber 14: 4–36.

Glass, S.V. and Zelinka, S.L. (2010). Moisture relations and physical properties of wood. In: *Wood handbook: wood as an engineering material: chapter 4. Centennial*. Dept. of Agriculture, F.S., Forest Products Laboratory Madison, WI, USA, pp. 4.1–4.19.

Greenhill, W.L. (1942). The damping capacity of Timber. J. Council Sci. Indust. Res. 15: 146–153.

Gril, J. (1988). Une modélisation du comportement hygro-rhéologique du bois à partir de sa microstructure, PhD In: Physics. Université Paris 6, Paris, p. 268.

Guitard, D. and El Amri, F. (1987). Modèles prévisonnels de comportement élastique tridimensionnel pour les bois feuillus et les bois résineux. Ann. Sci. For. 44: 335–348.

Hunt, D.G. (1990). Longitudinal shrinkage-moisture relations in softwood. J. Mater. Sci. 25: 3671–3676.

Hunt, D.G. and Gril, J. (1996). Evidence of a physical ageing phenomenon in wood. J. Mater. Sci. Let. 15: 80–82.

Hunt, D.G. (1997). Dimensional changes and creep of spruce, and consequent model requirements. Wood Sci. Technol. 31: 3–16.

Inokuchi, Y., Fushitani, M., Kubo, T., and Sato, K. (1999). Effects of water extractives on the moisture-content dependence of vibrational properties of Bamboo (*in Japanese*). Mokuzai Gakkaishi 45: 77–84.

Ishimaru, Y., Arai, K., Mizutani, M., Oshima, K., and Iida, I. (2001). Physical and mechanical properties of wood after moisture conditioning. J. Wood Sci. 47: 185–191. James, W.L. (1961). Effect of temperature and moisture content on internal friction and speed of sound in Douglas-fir. For. Prod. J. 11: 383–390.

Jiang, J., Lu, J., and Cai, Z. (2012). The vibrational properties of Chinese fir wood during moisture sorption process. Bioresources 7: 3585–3596.

Kataoka, A. and Ono, T. (1975). The relations of experimental factors to the vibration and the measuring values of dynamic mechanical properties of wood. I. The experimental errors due to the measuring apparatus. Mokuzai Gakkaishi 21: 543–550.

Kataoka, A. and Ono, T. (1976). The relation of experimental factors to the vibration and the measuring values of dynamic mechanical properties of wood. II. On the shape and size of specimen. Mokuzai Gakkaishi 22: 1–7.

Kojiro, K., Furuta, Y., and Ishimaru, Y. (2008). Influence of histories on dynamic viscoelastic properties and dimensions of waterswollen wood. J. Wood Sci. 54: 95–99.

Kollmann, F. and Krech, H. (1960). Dynamische Messung der elastischen Holzeigenschaften und der Dämpfung Ein Beitrag zur zerstörungsfreien Werkstoffprüfung. Holz Roh Werkstoff 18: 41.

Kubojima, Y., Okano, T., and Ohta, M. (2000). Vibrational properties of heat-treated green wood. J. Wood Sci. 46: 63–67.

Kubojima, Y., Suzuki, Y., and Tonosaki, M. (2005a). Vibrational properties of green wood in high-temperature water vapor. Holzforschung 59: 446–450.

Kubojima, Y., Tonosaki, M., and Yoshihara, H. (2005b). Effect of additional mass on the Young's modulus of a wooden beam. J. Test. Eval. 33: 278–282.

Matsumoto, T. (1962). Study on dynamic Young's modulus and logarithmic decrement of wood by transverse vibration method *(in Japanese)*. Bull. Kyushu Univ. For. 36: 1–86.

Matsunaga, M., Obataya, E., Minato, K., and Nakatsubo, F. (2000). Working mechanism of adsorbed water on the vibrational properties of wood impregnated with extractives of pernambuco (*Guilandina echinata* Spreng.). J. Wood Sci. 46: 122–129.

Minato, K., Konaka, Y., Brémaud, I., Suzuki, S., and Obataya, E. (2010). Extractives of Muirapiranga (*Brosimun* sp.) and its effects on the vibrational properties of wood. J. Wood Sci. 56: 41–46.

Norimoto, M., Tanaka, F., Ohogama, T., and Ikimune, R. (1986). Specific dynamic Young's modulus and internal friction of wood in the longitudinal direction *(in Japanese)*. Wood Res. Tech. Notes: 53–65.

Obataya, E. and Norimoto, M. (1995). Acoustic properties of cane (*Arundo donax* L.) used for reeds of woodwind instruments I. The relationships between vibrational properties and moisture content of cane (*in Japanese*). Mokuzai Gakkaishi 41: 289–292.

Obataya, E., Norimoto, M., and Gril, J. (1998). The effects of adsorbed water on dynamic mechanical properties of wood. Polym 39: 3059–3064.

Obataya, E., Norimoto, M., and Tomita, B. (2000a). Moisture dependence of vibrational properties for Heat-treated wood *(in Japanese)*. Mokuzai Gakkaishi 46: 88–94.

Obataya, E., Ono, T., and Norimoto, M. (2000b). Vibrational properties of wood along the grain. J. Mater. Sci. 35: 2993–3001, 6317.

Obataya, E., Minato, K., and Tomita, B. (2001). Influence of moisture content on the vibrational properties of hematoxylinimpregnated wood. J. Wood Sci. 47: 317–321. Obataya, E. and Tomita, B. (2002). Hygroscopicity of heat-treated wood, 2: reversible and irreversible reductions in the hygroscopicity of wood due to heating *(in Japanese)*. Mokuzai Gakkaishi 48: 288–295.

Obataya, E. (2017). Effects of natural and artificial ageing on the physical and acoustic properties of wood in musical instruments. J. Cult. Herit. 27: S63–S69.

Obataya, E., Zeniya, N., and Endo-Ujiie, K. (2020). Effects of seasoning on the vibrational properties of wood for the soundboards of string instruments. J. Acoust. Soc. Am. 147: 998–1005.

Ono, T. and Kataoka, A. (1979). The frequency dependance of the dynamic Young's modulus and internal friction of wood used for the soundboard of musical Instruments II. The dependance of the Young's modulus and internal friction on frequency, and the mechanical frequency dispersion *(in Japanese)*. Mokuzai Gakkaishi 25: 535–542.

Ono, T. and Norimoto, M. (1983). Study on Young's modulus and internal friction of wood in relation to the evaluation of wood for musical instruments. Jpn. J. Appl. Phys. 22: 611–614.

Ouis, D. (2002). On the frequency dependence of the modulus of elasticity of wood. Wood Sci. Technol. V36: 335.

Ozyhar, T., Hering, S., and Niemz, P. (2013). Moisture-dependent orthotropic tension-compression asymmetry of wood. Holzforschung 67: 395–404.

Palka, L.C. (1973). Predicting the effect of specific gravity, moisture content, temperature and strain rate on the elastic properties of softwoods. Wood Sci. Technol. 7: 127–141.

Pentoney, R.E. (1955). Effect of moisture content and grain angle on the internal friction of wood. Comp. Wood. 2: 131–136.

Roohnia, M., Kohantorabi, M., and Tajdini, A. (2015). Maple wood extraction for a better acoustical performance. Eur. J. Wood Wood Prod. 73: 139–142.

Sasaki, T., Norimoto, M., Yamada, T., and Rowell, R.M. (1988). Effect of moisture on the acoustical properties of wood *(in Japanese)*. Mokuzai Gakkaishi 34: 794–803. Sedighi Gilani, M., Heeb, M., Tingaut, P., and Schwarze, F.W.M.R. (2014). Influence of moisture on the vibro-mechanical properties of bio-engineered wood. J. Mater. Sci. 49: 7679–7687.

Sellevold, E.J., Radjy, F., Hoffmeyer, P., and Bach, L. (1975). Low temperature internal friction and dynamic modulus for beach wood. Wood Fiber 7: 162–169.

Sonderegger, W., Alter, P., and Niemz, P. (2008). Investigations on selected properties of tonal wood of spruce from Grisons (Untersuchungen zu ausgewählten Eigenschaften von Fichtenklangholz aus Graubünden). Holz Roh Werkstoff 66: 345–354.

 Sonderegger, W., Martienssen, A., Nitsche, C., Ozyhar, T., Kaliske, M., and Niemz, P. (2013). Investigations on the physical and mechanical behaviour of sycamore maple (*Acer pseudoplatanus* L.). Eur. J. Wood Wood Prod. 71: 91–99.

Suzuki, M. (1962). The effects of water-sorption and temperature on dynamic Young's modulus and logarithmic decrement of wood. Mokuzai Gakkaishi 8: 13–18.

Suzuki, M. (1980). Relationship between specific gravity and decrement of dynamic Young's modulus with water (*in Japanese*). Mokuzai Gakkaishi 26: 299–304.

Viala, R. (2018). Towards a model-based decision support tool for stringed musical instrument making, PhD In: Mécanique. Université Bourgogne-Franche Compté, Bourgogne-Franche-Comté, p. 314.

Wangaard, F.F. and Granados, L.A. (1967). The effect of extractives on water-vapor sorption by wood. Wood Sci. Technol. 1: 253–277.

Yano, H., Norimoto, M., and Yamada, T. (1986). Changes in acoustical properties of Sitka spruce due to acetylation *(in Japanese)*. Mokuzai Gakkaishi 32: 990–995.

Yano, H. and Minato, K. (1992). Improvement of the acoustic and hygroscopic properties of wood by a chemical treatment and application to the violin parts. J. Acoust. Soc. Am. 92: 1222–1227.

Zhan, T., Jiang, J., Lu, J., and Peng, H. (2015). Dynamic viscoelastic properties of Chinese fir under cyclical relative humidity variation. J. Wood Sci. 61: 465–473.