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Identification of anisotropic vibrational properties of Padauk wood with interlocked grain

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Abstract: Grain deviations and high extractives content are common features of many tropical woods. We aimed at clarifying their respective impact on vibrational properties, referring to African Padauk (*Pterocarpus soyauxii* Taub.), a species selected for its interlocked grain, high extractives content, and uses in xylophones. Specimens were cut parallel to the trunk axis (L), and local variations in grain angle (GA), microfibril angle (MFA), specific Young's modulus (E'_{\perp}/ρ , where ρ stands for specific gravity) and damping coefficient ($\tan\delta_L$), were measured. GA dependence was analyzed by a mechanical model which allowed to identify the specific Young's modulus (E'_{\parallel}/ρ) and shear modulus (G'/ρ) along the grain (3), as well as their corresponding damping coefficients ($\tan\delta_3$, $\tan\delta_G$). This analysis was done for native and then for extracted wood. Interlocked grain resulted in 0-25° GA and in variations of a factor 2 in E'_{\perp}/ρ and $\tan\delta_L$. Along the grain, Padauk wood was characterized, as compared to typical hardwoods, by a somewhat lower E'_{\parallel}/ρ and elastic anisotropy (E'/G'), due to a wide microfibril angle plus a small weight effect of extracts; and a very low $\tan\delta_3$ and moderate damping anisotropy ($\tan\delta_G/\tan\delta_3$). Extraction affected mechanical parameters in the order: $\tan\delta_3 \approx \tan\delta_G > G'/\rho \gg E'_{\parallel}/\rho$. That is, extractives' effects were nearly isotropic on damping but clearly anisotropic on storage moduli.

Keywords: Anisotropy; Damping coefficient; Extractives; Grain angle; Interlocked grain; Microfibril angle; Vibrational properties; Young's modulus

Introduction

Viscoelastic vibrational properties of wood are mainly affected by the orientation of wood elements (grain angle GA and microfibril angle MFA) and by chemical composition. Due to the highly anisotropic nature of wood, the actual orientation of grain inside a wood piece will strongly affect its apparent mechanical properties (e.g. Bodig and Jayne 1982). Wood grain inside a trunk can be straight, but also spiralled, interlocked or wavy. At least one kind of deviation may be found in almost any tree (Harris 1989). “Grain” represents the orientation of all axial elements (Ogata et al. 2003) and “Grain angle” usually refers to the tangential-longitudinal plane. Interlocked grain oscillates alternatively to the left- or right-hand along the radius. Wood pieces cut along the main axis of a trunk will thus systematically include GA, either cross-grained or not.

In straight-grained woods, microfibril angle is recognized as the primary factor affecting axial vibrational properties (specific modulus of elasticity E'/ρ and damping coefficient $\tan\delta$), and their axial-to-shear anisotropy (Obataya et al. 2000; Ono and Norimoto 1983; Norimoto et al. 1986). Cellulose microfibrils govern elastic response, while viscoelastic damping depends on the “matrix” of lignins and hemicelluloses. Wood, however, is not only composed of polymers, but also contains secondary metabolites, variable in their amounts as in their nature, known as extractives. In some species these are found to modify damping coefficient by as much as a 2-fold order, either decreasing it (Matsunaga et al. 1999; Minato et al. 1997, 2010; Yano 1994; Yano et al. 1995) or increasing it (Obataya et al. 1999; Sakai et al. 1999).

Resulting variations in vibrational properties are of practical importance, notably in musical instruments, where different functions are associated to preferred ranges of properties. For xylophones, where the primary vibrating body is the wood piece itself, a very low $\tan\delta$ is the most important factor in the judgement of woods (Aramaki et al. 2007; Hase 1987). While for top plates of string instruments, a very high specific modulus E'/ρ predominates (associated to moderate values of $\tan\delta$). Axial to shear anisotropy of modulus and of damping is also very important for the frequency response in flexural vibration (Obataya et al. 2000; Ono and Kataoka 1979).

Our work aimed at clarifying how these vibrational properties are influenced by, respectively, grain angle deviations and extractives, which are notable features of most tropical woods. It refers to the species *Pterocarpus soyauxii* Taub. (African Padauk) which was selected on the basis of its interlocked grain and predominant uses in xylophone-like instruments. We previously found that its extractives strongly affect damping (Brémaud et al. 2010a). In the present paper, after assessing local variations in grain and microfibril angles, the grain angle dependence of vibrational properties was analysed through a mechanical model. This served to identify mechanical parameters along the fibres and in shear for Padauk wood, and to compare their values with hardwoods average and with the same wood after extractives removal.

Material and methods

Material and preparation of specimens

Wood material of African Padauk (*Pterocarpus soyauxii* Taub.) from one tree (provenance Cameroon) was obtained from CIRAD sawmill. Sampling is schematized on Fig.1. Two

adjacent flat-sawn planks (12×80×800 mm, R×T×L) with contrasted orientations were selected in medium heartwood. They were then cut into series of specimens for vibrational tests (12×2×150 mm, R×T×L), all parallel to the main axis of the trunk. Total number of specimens was 158. Then, 6 representative specimens were cut, each one into 6 smaller specimens (1×2×20 mm ; R×T×L) for X-Ray measurements, successively in radial direction.

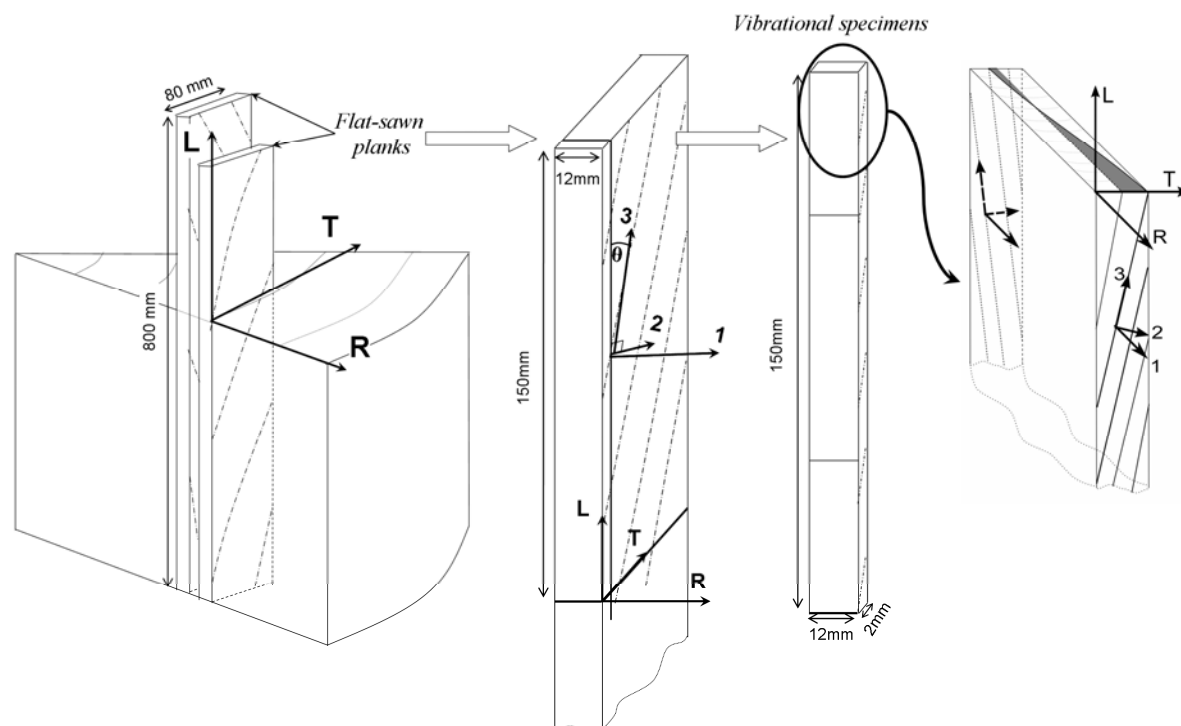


Fig.1 Schematic view of the sampling procedure and of the systems of axis related to the trunk and specimens [R,T,L] and to the grain direction [1,2,3]

Measurement of surface grain angle (GA)

Grain angle has been recorded on each tangential plane of planks before specimen cutting, basically following vessels. Records were always viewed as “from outside the trunk”. A grid was affixed to pictures of LT planes of planks, so as to materialise the location of specimens prepared from them. For each box of the grid, 12 measurements (accuracy $\pm 0.1^\circ$) of local angles were made using the image analysis software ImageJ (Rasband 1997-2008). Global pathways of vessels were also traced all along the LT planes, and their intersecting angle with the centre of the boxes/specimens measured. Both gave very similar results. Angles recorded on the LT planes were called α , and the mean of absolute values of angles for a given vibrational specimen was called θ .

Microfibril angle (MFA) and GA measured by X-Ray diffraction

Experiments were performed on a 4-circle diffractometer (Oxford Diffraction Gemini S) equipped with a 1024x1024 CCD camera. CuK $_{\alpha}$ radiation was generated by an X-ray generator operating at 50 kV, 25 mA. Specimens ($R \times T \times L = 1 \times 2 \times 20 \text{ mm}^3$) were mounted perpendicular to the beam and with $\leq 0.6^\circ$ positioning error for verticality. Images were recorded during 100 seconds and integrated along the whole 360° azimuthal interval to plot the intensity diagram of the (200) plane. An automatic procedure detected (200) peaks and their inflexion points. The T parameter as defined by Cave (1966), is measured as the half distance between intersections of tangents at inflexion points with the baseline. The average MFA of

each sample was estimated as $MFA = 0.6 \times T$ (Cave 1966). As MFA were moderate, (200) peaks were monomodal. Thus the tilting angle of the fibre axis to the sample axis, *i.e.* the grain angle, was measured as the deviation angle of the maximum intensity peaks to the horizontal.

Vibrational properties

Samples were kept for at least 3 weeks in controlled conditions of $20 \pm 1^\circ\text{C}$ and $65 \pm 2\% \text{RH}$, before being tested by non-contact forced vibrations of free-free bars (e.g. Obataya et al 2000). Specimens were made to vibrate through a tiny iron piece glued on one end, facing an electric magnet. Their displacement was measured using a laser sensor. Vibration emission and detection were computer-driven through a semi-automated interface that we developed using Labview® software (Brémaud 2006). E'/ρ was deduced from the first resonant frequency according to the Euler-Bernoulli equation. Damping coefficient $\tan \delta$ was measured both through the quality factor and through logarithmic decrement. Measurement frequencies were in the range of 200-400 Hz. 3 repetitions were made for each specimen and mean error was $\leq 5\%$.

Removal of extractives

After first vibrational tests, 5 groups (26 specimens each) with not significantly differing ranges of grain angle and properties were defined. Each group has been exhaustively extracted in a given solvent and its properties after extraction measured again (see Brémaud et al. 2010a). Solvents were: diethyl-ether (ET), methylene dichloride (MD), acetone (AC), methanol (ME) and hot water (HW, c. 90°C).

Identification of anisotropic parameters

The grain angle dependence of vibrational properties was analyzed by a mechanical model based on transformation formula for elastic solids (*i.e.* Bodig and Jayne 1982) applied to complex compliances (Brémaud et al. 2010b). This allows expressing the storage (E') and loss (E'') Young's moduli along the specimen's axis (L) at a given grain angle θ , as a function of anisotropic properties in the fibre-related system of axis (3, 2, see Fig.1):

$$E'_L(\theta) \approx E'_3 \frac{(1+u)^2}{1+a'u+b'u^2} \quad \text{and} \quad E''_L(\theta) \approx E'_3 \tan \delta_{33} \frac{(1+u)^2 (1+a''u+b''u^2)}{(1+a'u+b'u^2)^2} \quad (1)$$

With the parameters:

$$u = (\tan^2 \theta) \quad \text{and} \quad \begin{aligned} a' &= E'_3 / G'_{32} - 2\nu'_{32} & \text{and} & \quad a'' = a' \frac{\tan \delta_{G32}}{\tan \delta_3} (1+q) \\ b' &= E'_3 / E'_2 & & \quad b'' = b' \frac{\tan \delta_2}{\tan \delta_3} \end{aligned} \quad (2)$$

Where E'_3 and E'_2 , and $\tan \delta_3$ and $\tan \delta_2$, are respectively the storage modulus, and the damping coefficients, along, and tangential to, the fibre direction; G'_{32} and $\tan \delta_{G32}$ are storage modulus and damping coefficient in shear (longitudinal-tangential to the fibres); ν'_{32} is Poisson's ratio (about 0.46), and the term q amounts to 0 to 8% depending on Poisson's loss factor (for which no data are available). Grain angle dependence of damping coefficients was given by:

$$\tan \delta_L(\theta) = E''_L(\theta) / E'_L(\theta) \quad (3)$$

The radial gradient of GA inside specimens (see Fig.1) was approached by linear extrapolation between the angles α on both tangential faces and 12 discrete, absolute values of angle were defined (i.e. every mm across the width). Apparent moduli $E'_L(\theta)$ and $E''_L(\theta)$ and damping coefficient $\tan\delta_L(\theta)$ were calculated for each of these points using eq.1 and 3, and the properties along the specimen axis were obtained by integration over these 12 values.

The root of the mean square error (rmse) between calculation and experimental measurements of both E'_L/ρ (expressed in GPa) and of $\tan\delta_L$ (in ‰) were calculated. Then the model parameters (eq.2) were fitted to minimize the sum of both rmse, using the solver of Microsoft Excel.

As, in the range of GA under study, parameters b' and b'' have secondary influence, they were assigned plausible values (11.5 for b' and 40 for b'' , based on data on another species of the same genus (Caldersmith and Freeman 1990)) for the identification of other parameters. For analysis on extracted material, they were slightly adjusted by admitting an analogy with the results of (Yano et al. 1995).

Results and discussion

Grain angle measurements

Surface angles α ranged from -26° to $+33^\circ$ (mean $+9^\circ$), with different profiles: about one half of specimens had only positive angles up to $+33^\circ$ and the other was cross-grained with a distribution from -20° to $+20^\circ$. At a given longitudinal position, GA differences along 12 mm radial distance ranged from 1.5° to 30° (mean 15°). GA varied also axially: variations along 150mm ranged from 0.5° to 25° (median 5.5°). In tangential direction GA varied from 2° to 23° (median 8.5°).

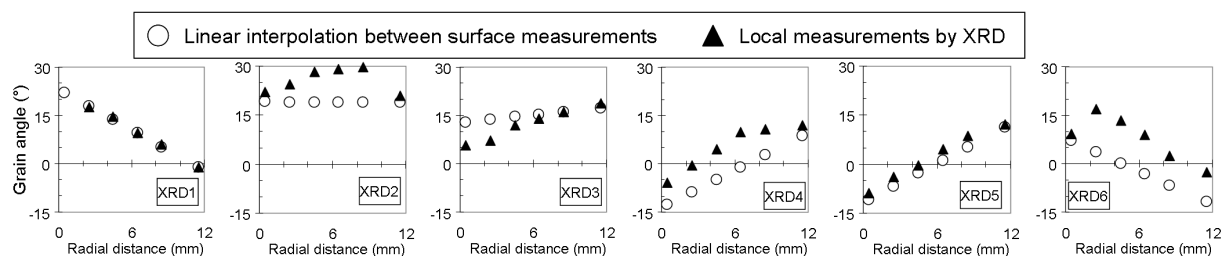


Fig.2 Trends in grain angle across the width (LR plane, R direction) of 6 vibrational specimens

When comparing (Fig.2) surface records (based on vessel alignments) and local XRD measurements (which should represent mainly fibres orientation) of GA inside vibrational specimens, overall trends were very similar. The relation between the two types of measurements was close to unity and had a high (87%) coefficient of determination, which tends to confirm the validity of XRD for GA assessment. However, where discrepancies occurred, surface GA showed bigger absolute values. This might be related to observations by (Ogata et al. 2003) on interlocked grain of *Acacia mangium*, where the orientation of vessels tends to show higher peak values than fibres.

Local variations of microfibril angle

The range in microfibril angle (MFA) was from 13° to 21° (mean 17.2°) according to Cave's (1966) relation. On the whole set of samples, there was no apparent relationship between local values of MFA and of GA (both determined by XRD on the same small

specimens) (Fig.3). But within each group (coming from different locations), there was a tendency of MFA to decrease with increasing GA, which is significant for 4 of the 6 groups. In addition, according to Sarén and Serimaa (2006), in the XRD conditions that we used, actual MFA values for specimens with wide GA shall be lower than the apparent ones (shown in Fig.3). This would then reinforce a tendency of MFA decreasing with increasing GA.

This might be related to a theory (Schulgasser and Witzum 2007) about the mechanism of spiral grain formation. A model developed by these authors predicts leftward GA for very high MFA, progressing towards right-hand angles when MFA diminishes (to about 19°, which seems consistent with our results). However, this hypothesis is designed for the case of spiral grain and is difficult to extrapolate to more complex phenomena such as interlocked grain. This issue seems to remain open. Co-occurrences of oscillations in MFA and in GA are also observed in Spruce (Sarén et al. 2006), and in tension wood of an *Acacia sp.* (Hillis et al. 2004), but variations in MFA and in GA are not clearly synchronous.

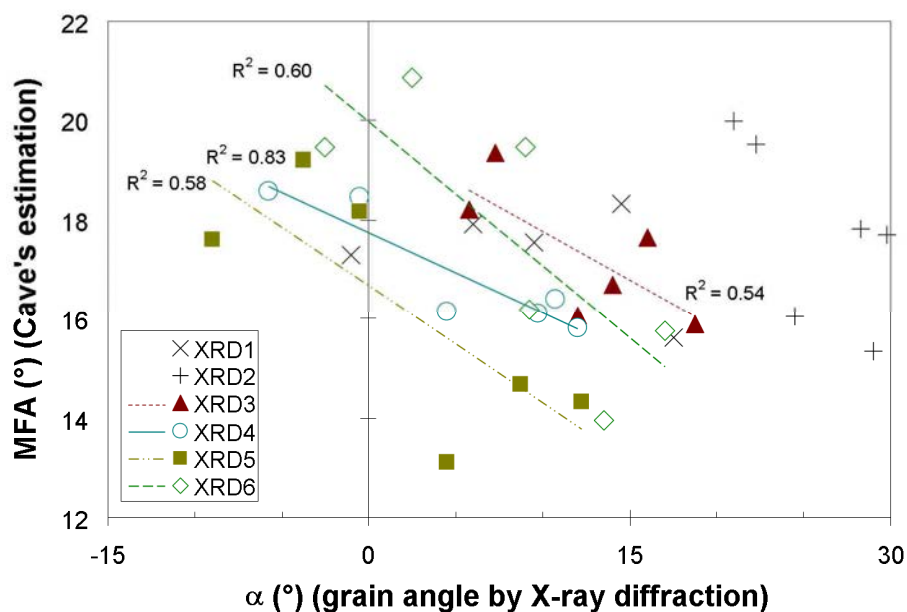


Fig.3 Local values of microfibril angle plotted against local grain angles, for 6 groups = initial location. Trend lines are shown for groups where the relation is significant

Be it as it may, the range in MFA strongly overlapped between groups, and their average values were not statistically different. Also, the amplitude of variations in MFA (8°) was 5 times smaller than that for GA variations (40°). Thus, the main affecting factor for mechanical properties will be grain angle, in addition to which MFA variations would bring modulations of secondary importance.

Vibrational properties and grain angle dependence

The samples had homogeneous specific gravity (0.76 ± 0.02). The mean grain angle θ inside vibrational specimens ranged from 0 to about 25°. Resulting variations in specific modulus of elasticity E'_L/ρ and in damping coefficient $\tan\delta_L$ were of a more than 2 fold order (8 to 18 GPa and 4.4 to 9.9 ‰ respectively). In contrast, the specific loss modulus E''_L/ρ varied only of about 25% (72 to 89 MPa). Calculations based on the fibre-scale related properties identified for Padauk (see table 1) described, respectively, 82% and 77% of the experimental variations in E'_L/ρ and of $\tan\delta_L$ (along the trunk and samples axis) as a function of mean grain angle θ (Fig.4). Residual dispersion was mainly linked to different initial locations, i.e. local variations in GA and MFA profiles should contribute. When comparing

(Fig.4), the grain angle dependence for Padauk wood (P) and for properties corresponding to a “mean hardwood” MH (Brémaud et al. 2010b), Padauk had a lower than average apparent specific storage modulus at small grain angles, yet correction (P_{corr}) for the contribution of extractives to specific gravity reduced the difference. Padauk $E'_{L/\rho}$ decreased slower than for MH over angles up to 15° but for bigger GA trends were similar. Loss properties of Padauk were more atypical. Specific loss modulus $E''_{L/\rho}$ of Padauk was nearly 2 times smaller over small angles, and it decreased very slightly over GA up to 25° , while for a mean hardwood a pronounced decrease starts from about 10° GA. Damping coefficient $\tan\delta_L$ of Padauk increased less steeply as a function of GA than for MH and trends evolved in parallel for both types of materials, Padauk values being much lower (about one third). When plotting (Fig.4d) the evolutions in $\tan\delta_L$ against those in $E'_{L/\rho}$, dispersion was very much reduced: uncertainties on GA profiles inside specimens are here sidestepped. For Padauk heartwood, $\tan\delta$ along the grain was nearly half that of a mean hardwood of similar $E'_{L/\rho}$, and remained systematically lower with increasing GA, curves for both type of material being parallel.

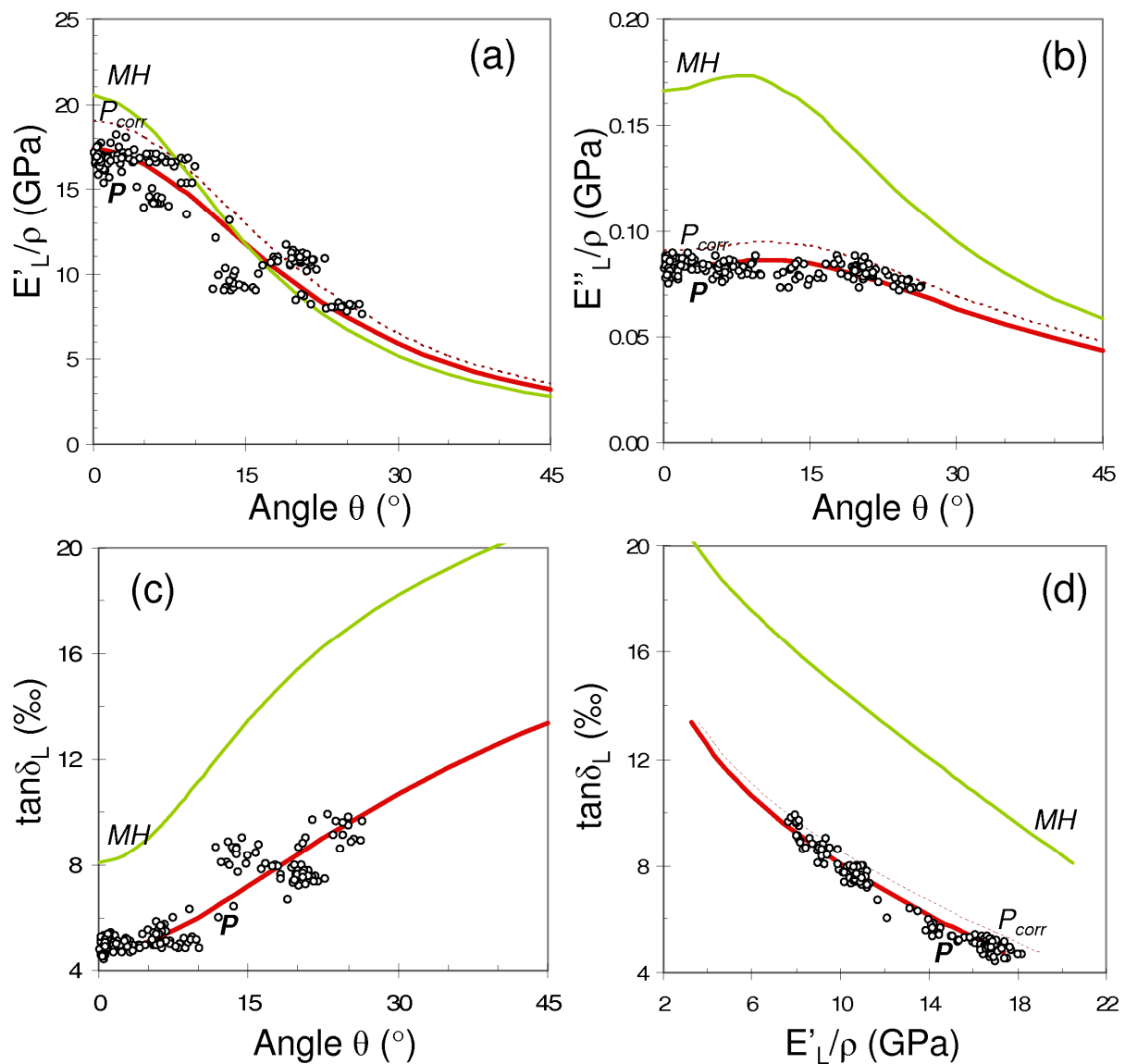


Fig.4 Experimental (circles) and calculated properties for untreated Padauk heartwood (bold line: apparent values P; thin dashed line: values corrected for weight of extractives P_{corr}) and for a “mean hardwood” (gray curve, MH). Trends in specific storage modulus (a), specific loss modulus (b) and damping coefficient (c) at the global scale, as a function of grain angle. (d): evolution of $\tan\delta$ plotted against that of $E'_{L/\rho}$

Identification of properties in the fibre-related system of axis (table 1), obtained by optimization of the model to fit the data, showed that Padauk had quite lower $E'_{3/\rho}$ (along the fibres) and elastic anisotropy than a “standard hardwood” of equivalent density, for which $E'_{3/\rho}$ would be about 21-22GPa, and a' about 13 (Guitard and ElAmri 1987). The relatively low value of $E'_{3/\rho}$ is partly linked to the contribution of extractives to specific gravity: it increased of 1.6 GPa after correction for this factor. Yet these lower values of E'/ρ along the fibre, and of anisotropy ratios, are also linked to the quite high microfibril angles. In this range of MFA, microstructural models predict a comparable decrease in $E_{3/\rho}$ and in 3-2 anisotropy (Gachet and Guitard 2006; Obataya et al. 2000). On the other hand, damping coefficient $\tan\delta_3$ for Padauk (<5‰) is much lower than those indicated in the literature (about 8‰) for similar MFA (Norimoto et al. 1986).

Table 1: Properties in the fibre-related system of axis obtained by optimization of model parameters to fit experimental data. rmse: root mean square error.

| Padauk heartwood (Untreated) | $E'_{3/\rho}$ (GPa) | $E''_{3/\rho}$ (MPa) | $\tan\delta_3$ (‰) | a' (see eq. 2) | a'' | $\tan\delta_{G32}$ (‰) | $\frac{\tan\delta_{G32}}{\tan\delta_3}$ | b' (see eq.2) | b'' | rmse $E'_{L/\rho}$ (GPa) | rmse $\tan\delta_L$ (‰) |
|---|------------------------|-------------------------|-----------------------|---------------------|-------|---------------------------|---|--------------------|-------|--------------------------------|-------------------------------|
| Apparent values (P) | 17.3 | 82 | 4.8 | | | | | | | | |
| Corrected for mass of extractives (P_{corr}) | 19.0 | 90 | 4.8 | 8.8 | 18.6 | 10.1 | 2.13 | 11.5 | 3.5 | 1.4 | 0.8 |

Anisotropic vibrational properties as affected by extractives

Extractive removal (2.3, 3.1, 8.8, 13.4 and 0.6% respectively for solvents ET, MD, AC, ME and HW) affected global-scale storage modulus only marginally as compared to grain angle (Fig.5a). On the contrary, for specific loss modulus $E''_{L/\rho}$, methanol (ME) extraction caused variations as important –but opposite- as that for a rotation of 45°. Damping coefficient was strongly dependent both on orientation and on extractives. The variation after ME extraction for damping along the fibres was close in amplitude to that resulting from a rotation of 20°. Curves between calculated $\tan\delta$ and E'/ρ (Fig.5d) all had very similar shape, while differences in extractives content shifted their $\tan\delta$ values.

The amplitudes of modifications (Fig.6) of axial and shear properties (in the fibre-related system of axis) caused by extractive removal ranked as $\tan\delta_3 \approx \tan\delta_{G32} \approx E''_{3/\rho} \geq G'_{32/\rho} \geq G''_{23/\rho} \gg E'_{3/\rho}$. Anisotropic effects of Padauk extractives can be summarized as follows: in the direction of fibres, the presence of extractives in the cell walls of native wood decreased strongly the loss properties $E''_{3/\rho}$ and $\tan\delta_3$, while they little affected –positively- the specific storage modulus. On the contrary, concerning shear properties, the storage modulus $G'_{32/\rho}$ was clearly higher (> 20 %) before extraction, indicating a role of extracts in reinforcing the matrix substances in native wood. The effect of extractives on damping coefficient was similar both along the fibres and in shear. This sounds consistent with the analyses of (Obataya et al. 2000; Norimoto et al. 1986) stating that the elasticity of matrix substances would be of little significance when the viscous matrix and the elastic reinforcement of the microfibrils are in parallel, while the influence of matrix properties would increase as the reinforcement makes a bigger angle. Perhaps, the slight effect of extractives observed here on storage modulus along the fibres would be barely noticeable in a wood with smaller MFA. On the other hand, loss coefficient is governed by matrix viscosity and as such can be affected by changes in extractives content of the cell-wall, whatever the orientation.

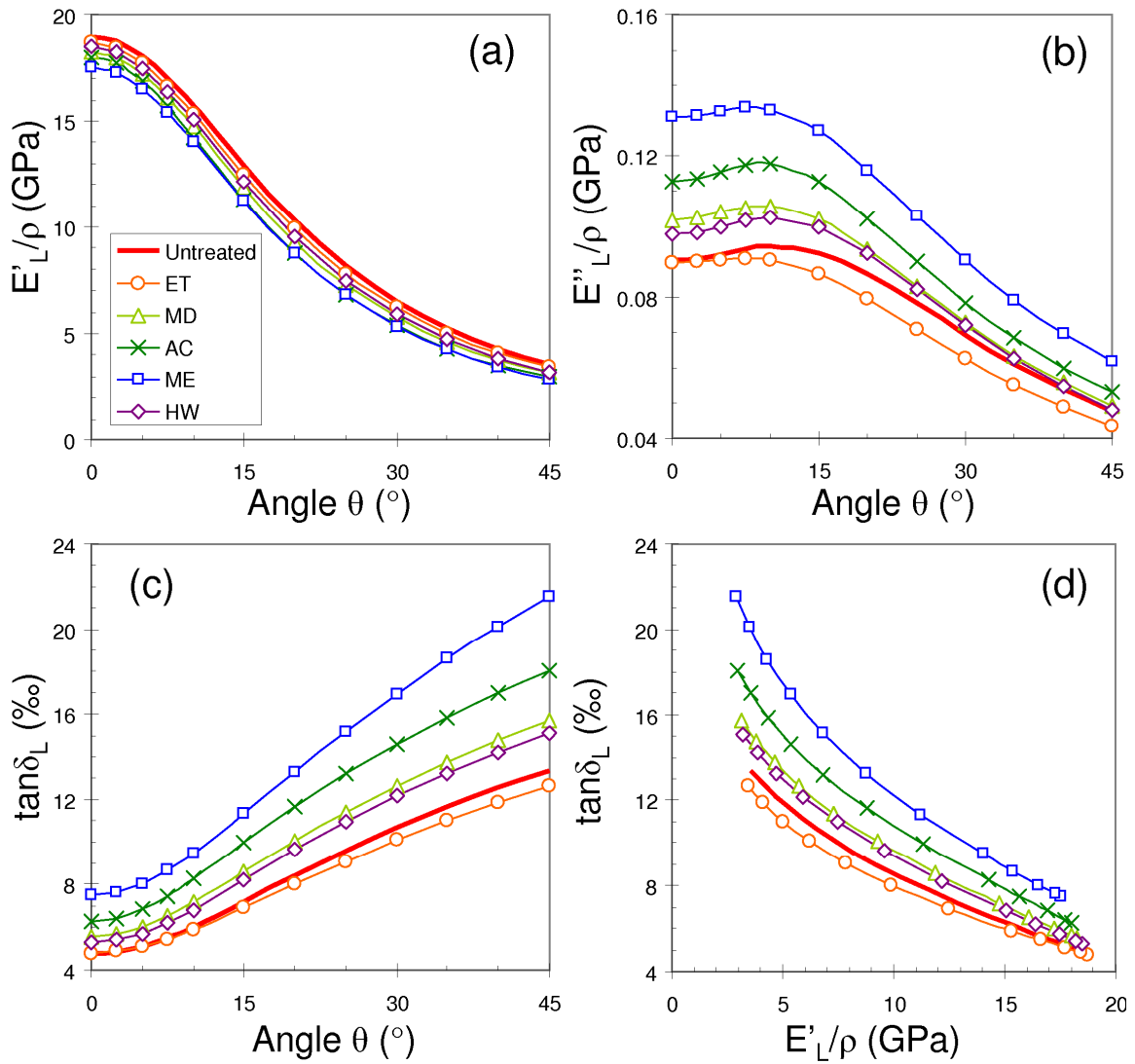


Fig.5 Trends in mechanical properties as a function of grain angle, calculated with parameters in the fibre-related system of axis identified on Padauk heartwood before and after extractions (properties are corrected for the contribution of extractives to specific gravity)

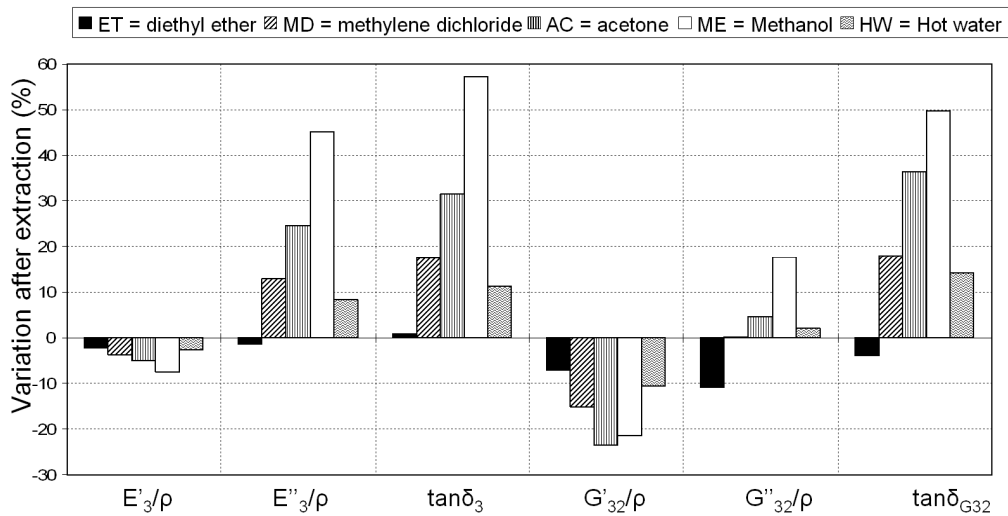


Fig.6 Variations after extractions of identified mechanical properties (variations in G' and G'' were calculated from parameters a' and a'' assuming that changes in Poisson's ratios would be negligible)

Extractives soluble in ET (diethyl-ether) were mainly lumen located (Brémaud et al. 2010a) and had different effects: no change in $\tan\delta_3$ (along the fibre) and their presence slightly increased shear damping. This may be interpreted as: (i) lumen-located compounds could only increase the loss coefficient of wood, as was proposed by (Akitsu et al. 1993), this being modulated by their characteristic properties. (ii) For rather small amounts of lumen filling or lining, such an effect would be barely noticeable in the fibre direction, as compared to the cell-wall properties, but may become more significant in shear or transverse directions.

From a point of view of application, as compared to the same wood without extractives, native Padauk heartwood could be considered as a “natural chemical modification”, and had: a E'_3/ρ slightly higher (+7.5%), a $\tan\delta_3$ much lower (-36%), a lower ratio E'_3/G'_{32} (-13%) and a slightly higher ratio $\tan\delta_{G32}/\tan\delta_3$ (+5%). The combination of these latter two ratios was proposed by Obataya et al. (2000) as a descriptor of frequency response and “tone quality” of vibrating wooden pieces: higher E/G anisotropy results in higher loss and weaker radiation at high frequencies (Ono and Kataoka 1979). In the case of xylophone bars, for which Padauk is a favourite material, the best descriptor of sound qualification is reported to be a low damping of the first mode, associated to few spectral components (Aramaki et al. 2007; Hase 1987). Thus, Padauk extracts, as they decrease its damping along the fibres about 4 times more than they decrease the axial to shear viscoelastic anisotropy, must participate in making this wood suitable for xylophone-like instruments. Of course, it shall also be reminded that special care should be taken concerning local grain orientation when preparing pieces of wood from this species and others for which interlocked grain is a frequent feature.

Conclusion

This study aimed at clarifying the respective influence of the orientation of wood elements, and of extractives, on vibrational properties, referring to a selected tropical hardwood with interlocked grain. After assessing local variations in grain and microfibril angles, properties were analysed by a mechanical model. Mechanical parameters in the fibre-related system of axis [1,2,3] were identified for untreated and extracted Padauk wood. Results showed that:

- Mean grain angles up to 25° naturally occur. Resulting variations in E'/ρ and in $\tan\delta$ are well described by the model, yet with residual dispersion due to local profiles in grain and microfibril angle.
- For native Padauk E'_3/ρ and elastic anisotropy are quite low, which is partly due to the mass contribution of extracts, and mostly to rather high microfibril angle ($13\text{-}21^\circ$).
- Loss modulus E''_3/ρ is much lower than average and its anisotropy very reduced; $\tan\delta_3$ is also very low, while its GA dependence is parallel to the general case.
- Extractives affect mechanical parameters in the rank: $\tan\delta_3 \approx \tan\delta_{G32} \approx E''_3/\rho > G'_{32}/\rho \geq G''_{23}/\rho \gg E'_3/\rho$. Extractives' effects are nearly isotropic on $\tan\delta$ but clearly anisotropic on storage moduli: they significantly increase shear modulus, but only slightly along the grain. Damping along the grain is only affected by cell-wall located extracts, while lumen ones have a significant and opposite effect on shear damping.

From a practical point of view, results also confirm that special caution should be taken when preparing specimens from the many woods with grain deviations such as interlocked grain.

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