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Radial variations of vibrational properties of three tropical woods

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Abstract The radial trends of vibrational properties, represented by the specific dynamic modulus (E'/ρ) and damping coefficient ($\tan\delta$), were investigated for three tropical rainforest hardwood species (*Simarouba amara*, *Carapa procera*, and *Symphonia globulifera*) using free–free flexural vibration tests. The microfibril angle (MFA) was estimated using X-ray diffraction. Consistent patterns of radial variations were observed for all studied properties. E'/ρ was found to decrease from pith to bark, which was strongly related to the increasing pith–bark trend of MFA. The variation of $\tan\delta$ along the radius could be partly explained by MFA and partly by the gradient of extractives due to heartwood formation. The coupling effect of MFA and extractives could be separated through analysis of the $\log(\tan\delta)$ versus $\log(E'/\rho)$ diagram. For the species studied, the extractive content putatively associated with heartwood formation generally tends to decrease the wood damping coefficient. However, this weakening effect of extractives was not observed for the inner part of the heartwood, suggesting that the mechanical action of extractives was reduced during their chemical ageing.

Key words Tropical woods · Dynamic modulus · Damping coefficient · Microfibril angle · Extractives

Introduction

Diversity in the mechanical properties of wood depends on genetic and environmental factors and is found at all levels between gymnosperms and angiosperms, tropical and temperate trees, species of any of these groups, populations of a given species, trees of a given population, and finally sampling locations within a single tree. The within-tree variability is of special interest because it is often found to be larger than the between-tree variability, and cannot easily be reduced or manipulated since it results from ontogenetic gradients. Understanding this diversity and its link with wood structure and chemical composition is an important issue to find optimal uses for each kind of wood.

Wood is a natural composite material composed of cellulose, hemicelluloses, lignin, and a small amount of extractives. A large part of the cellulose is present in a crystalline state in the form of microfibrils. The microfibrils are considered to be elastic and wind spirally around the cell wall axis at an angle termed the microfibril angle (MFA). MFA has been found to be an important microstructural factor influencing the mechanical properties of wood.[1] Hemicelluloses and lignin mix together and are distributed inside the cell wall. This mixture forms a matrix that is considered to be viscoelastic and is the main source of energy dissipation in wood. Extractive compounds are present mainly in the heartwood and are found in lower amounts than the other wood structural constituents. These secondary constituents are determinant for some wood properties, such as color and durability. Additionally, they have been shown to influence wood viscoelastic behavior,[2–5] although this effect differs from case to case: some extractives induce a strong decrease of the damping coefficient, some others slightly increase it,[6] and some have limited effect.[7]

The microfibrils represent about 45% of wood by weight and are by far the stiffest constituent. Much work has been carried out on the relation between MFA and mechanical properties.[8–10] It is found that the mechanical properties of wood in the grain direction are improved when the MFA decreases. In fact, the wood cell wall can be considered a fiber-reinforced composite;[11] therefore, the mechanical properties along the microfibril direction are superior to those in other directions. On the other hand, MFA is also an important factor in determining wood viscoelastic properties: in dry wood, a positive relation is generally found between MFA

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Table 1. Specific gravity and weight fraction of components of the species studied

Species	Specific gravity	Cellulose content (wt%)	Hemicellulose content (wt%)	Lignin content (wt%)	Extractive content[a] (wt%)
Simarouba amara	0.391	48	13	33	2.0 + 2.5
Carapa procera	0.655	40	N.A.	33	3 + 3.5
Symphonia globulifera	0.709	47	24	24	5.0 + 1.0

Data from Mariwenn database[23]
N.A., data not available
[a]The first value is the result of alcohol-benzene extraction, and the second value is the result of water extraction

and the damping coefficient ($\tan\delta$) obtained by dynamic tests,[12] and in quasi-static creep tests, the creep compliance is found to be strongly related to MFA.[13,14] In fact, it is the usual methodology to compare directly the dynamic modulus and damping coefficient in evaluating the acoustic properties of woods. A strong negative correlation has repeatedly been observed between E'/ρ and $\tan\delta$.[15–17] A "standard relation" was established to describe the statistical relation between these properties[15] based on more than 1000 specimens taken from 20 softwood species. The validity of this relation has also been demonstrated in hardwood species.[3] Recently, using selective extractions, Brémaud et al.[3,18] showed that wood extractives modified this "standard relation" by changing the intercept of the line, but not its slope.

A large part of the within-tree diversity in wood properties occurs in the radial direction.[19] In fact, variations along the radius result from the superposition of two gradients. The first gradient is related to the effect of cambial age.[20] A piece of wood near the pith was produced by a younger cambium compared to that near the bark, which brings about changes in microstructure, particularly in MFA. The second gradient is related to the distinction between sapwood and heartwood.[21] Peripheral wood is physically young and remains functional in terms of contributing to the conduction of sap. When it gets physically older (i.e., wood in inner locations), it loses its functional hydraulic properties and is subjected to the deposition of extractives, becoming heartwood. From outside rings to inside rings in the heartwood, there is a chemical ageing of the extractives and their bioactivity is known to decrease strongly for old trees: inner heartwood is much less resistant to rot than outer heartwood.[22]

In the present study, wood specimens were prepared from three tropical rainforest hardwood species at various positions along the radial direction of the trunks in order to include a large diversity of microstructures (radial position) and secondary extractive content (heartwood/sapwood). X-ray diffraction tests and free–free flexural vibration tests were undertaken and the vibrational properties were analyzed with respect to MFA and sample locations, aiming at a better understanding how the variation of the MFA and extractives affect the viscoelastic properties of wood.

Materials and methods

Wood materials

The three tropical rainforest hardwood species chosen for this study were *Simarouba amara*, *Carapa procera*, and *Symphonia globulifera*, abbreviated to Sa, Cp, and Sg, respectively. Cp is widely used for cabinetwork in South America and Sa is used traditionally by the Marons people for making objects because, when dried, it has a good resistance to termites. The average specific gravity, ρ, and weight fraction of the components of the three species obtained from the Mariwenn database[23] are listed in Table 1. One tree of each species, with a diameter at breast height ranging from 50 to 75 cm, was taken from the Paracou experimental forest (Kourou, French Guiana) in October 2008. The wood logs, after cutting from trees, were conditioned for six months at 26°C and 65% relative humidity before the tests. The moisture content measured after vibrational tests was 10.7% on average, without significant variations between species.

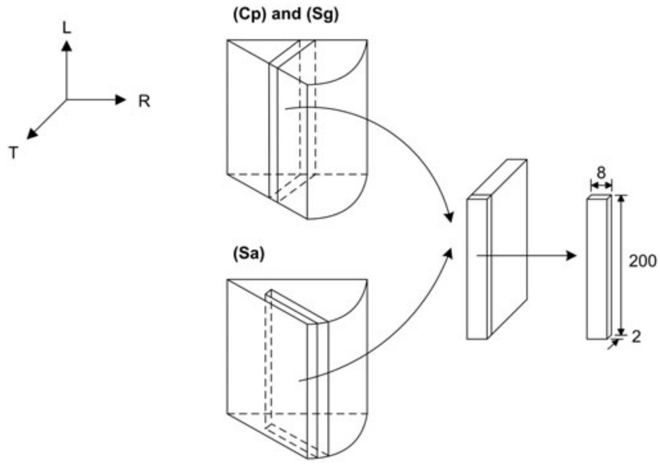


Fig. 1. Geometric description showing the direction and dimensions (in millimeters) of the two sampling schemes. Cp, *Carapa procera*; Sg, *Symphonia globulifera*; Sa, *Simarouba amara*

Wood specimens ($2 \times 8 \times 200$ mm) were machined as close as possible ($< 5°$) to the grain direction. Specimens were taken at different radial positions according to two sampling schemes, as shown in Fig. 1. Specimens of Cp and Sg were machined from tangential boards taken at different distances from the pith. They were grouped as heartwood or sapwood and were further classified as inner, middle, or external subgroups according to their original positions on the stem cross section, as shown in Fig. 2. This sampling method provides information on the radial position of wood specimens, which suggests not only the microstructural variations (MFA was evaluated afterwards) but also the chemical differences. The separation between heartwood and

Table 2. Numbers of tested specimens in free–free flexural vibration tests for each group

Species	Abbreviation	Heartwood			Sapwood	
		Inner	Middle	External	Inner	External
Carapa procera	Cp	16	20	24	16	18
Symphonia globulifera	Sg	20	–	21	23	
Simarouba amara	Sa	35				

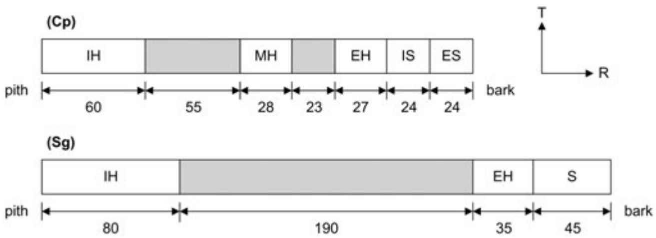


Fig. 2. Sample locations for Cp and Sg. *IH*, inner heartwood; *MH*, middle heartwood; *EH*, external heartwood; *S*, sapwood; *IS*, inner sapwood; *ES*, external sapwood. All dimensions are in millimeters

sapwood offers a valid background for discussing the relations between wood properties and putative extractive content. For species Sa, the heartwood could not be separated from sapwood by visual assessment. Therefore, specimens were taken at different positions from a single radial board (Fig. 1), without separation into different groups. It should be noted that, although both sampling schemes yield longitudinal specimens giving the properties along the grain direction, those of Cp and Sg woods are in the L-R plane, while those of Sa are in the L-T plane. Table 2 gives the numbers of specimens tested in this study.

Vibrational property measurements

The vibrational properties, represented by the specific dynamic modulus and the damping coefficient, were experimentally characterized. The specific dynamic modulus, E'/ρ, is the ratio of dynamic elastic modulus to density. It represents the amount of wood stiffness generated by a given amount of cell-wall material, and is therefore a measure of cell-wall quality (depending only on the chemistry and microstructure, not on the amount of cell wall present in wood). The damping coefficient, $\tan\delta$, is representative of the dissipative behavior of wood. These properties were measured by a free–free flexural vibration method,[24] using an apparatus previously described.[5,25] E'/ρ was calculated from the resonance frequency of the first vibration mode based on Euler-Bernoulli beam theory as:

$$\frac{E'}{\rho}=\frac{48\pi^2 l^4}{m^4 h^2}f^2 \tag{1}$$

where l and h are the length and thickness of the specimen, respectively, f is the resonance frequency of the first vibration mode, and m is the corresponding vibration constant, which is 4.73 for the first vibration mode. The resonance frequencies excited in this study ranged from 230 to 340 Hz. The value of $\tan\delta$ was calculated using linear regression analysis of the decay curve. Three measurements were performed for each specimen to give the average value.

Mean microfibril angle (MFA) measurements

Selected wood specimens were characterized by X-ray diffraction (XRD) to determine the MFA. Wood sections (2 mm thick) were mounted on a sample holder and inserted into an XRD instrument (Gemini-S, Oxford Diffraction) equipped with a Cu source (monitored at 50 kV and 25 mA) and a CCD camera. XRD patterns were recorded along the longitudinal direction of the sample perpendicular to the incident beam with an exposure time of 15 s. The azimuthal pattern of the diffraction arc associated with the (200) crystal plane of cellulose was obtained by integrating the full pattern for angles $2\theta = 20°–24°$. The MFA was calculated from these azimuthal patterns using Cave's method: the so-called "T-angle" was determined as half the span separating the intersections of the tangents at the inflexion points of the peak with the baseline,[26,27] and the MFA was estimated as $0.6 \times$ T. Several authors[28–30] have shown that this estimation is well correlated to the real mean MFA of wood measured with optical methods. However, the relation between this estimated value and the real value is not always 1:1 and is sometimes nonlinear; in addition, the calibration coefficients depend on the species. For one of the species studied (Sa), the calibration was found in the literature,[30] but for the other species, because no calibration data could be found, we used the raw value of Cave's estimation. Considering the MFA variability within a specimen, four sections were cut from each selected specimen and the average value was used to represent the estimated mean MFA of the specimen.

Results and discussion

Diversity in vibrational properties

Although all the trees studied grew in similar conditions, large variations in wood properties were found: the specific dynamic modulus E'/ρ ranged from 16 to 35 GPa, and the damping coefficient $\tan\delta$ ranged from 5×10^{-3} to 9.5×10^{-3}. This variability was due to variations in wood microstructure and chemistry between species and within trees. A general comparison of the three species is given in Fig. 3.

Analysis of variance showed that the differences between species were significant ($p = 0.022$ for E'/ρ and $p < 0.001$ for

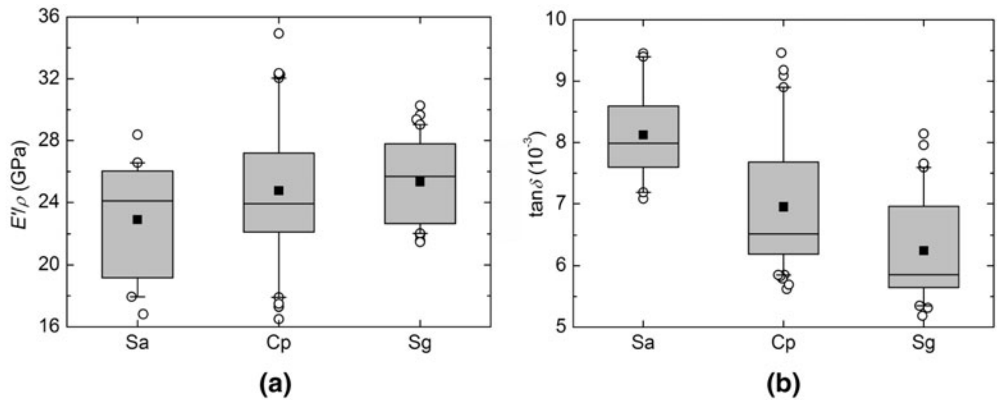


Fig. 3. Comparison of vibrational properties, **a** specific dynamic modulus (E'/ρ) and **b** damping coefficient ($\tan\delta$), among the three species studied. *Black squares* indicate the average values, the *box* indicates the median and quartiles, *whiskers* indicate 5% and 95% quartiles, and *circles* indicate outlying values

$\tan\delta$). Large variations of measured properties were observed within all three species, which were mainly due to the differences in sample cutting locations. This within-tree variability represented 83% of the total variance for E'/ρ and 47% for $\tan\delta$. It is noteworthy that the species were clearly different by observing the $\tan\delta$ data, whereas they could not be distinguished so evidently regarding the E'/ρ data. The better discriminating ability of the damping coefficient, compared with the specific modulus, was also previously found in comparing woods from 13 botanical families.[25]

Radial trends of vibrational properties and MFA

Radial variations of E'/ρ and $\tan\delta$ were examined for each species (Fig. 4). Generally, E'/ρ decreased from pith to bark, whereas $\tan\delta$ showed the reverse tendency. For species Cp, each point represents an average value of 15–25 specimens, therefore a continuous decrease of E'/ρ along the radius is convincing. Regarding $\tan\delta$, a distinguishable two-step variation is evident, matching with the separation between heartwood and sapwood. For Sg, although only three average points were obtained, the patterns of E'/ρ and $\tan\delta$ were similar to those of Cp.

These radial patterns were applicable to Sa as well and, furthermore, they provide a reference for classifying Sa specimens, which cannot, as previously mentioned, be grouped by visual assessment. Specifically, the value of E'/ρ remains almost constant when the distance from the pith is smaller than about 60 mm and then decreases until the bark is reached. From the $\tan\delta$ data, it seems also that there exist two groups, i.e., a moderate decrease of $\tan\delta$ from pith to about 80 mm, and then an increase. This threshold distance of 60–80 mm is used later to classify Sa specimens into "inner" and "outer" groups, and a hypothesis is made that these two groups are different in their extractive contents.

Radial variation of vibrational properties is actually a reflection of the growth history of the tree. During tree growth, ontogenetic and environmental changes determine the microstructural features (such as fiber length, cell wall thickness, and MFA) as well as its chemical constitution (e.g., molecular configuration, volume fraction of each constituent, and extractives content). The microstructures and chemical constitution then endow the wood with varying properties along the radius.

The radial trend of MFA is shown for all species in Fig. 5. An increasing trend of MFA from pith to bark was found for all three species. It was noted that the MFA of Sa increased slowly when the distance from the pith was less than about 60 mm, and then MFA increased quickly at distances greater than 60 mm. This behavior is similar to that of E'/ρ noted previously (Fig. 4), which confirms the validity of the classification of Sa specimens. From pith to bark, the MFA of Sa rises about 64%, while the MFA of Cp shows a continuously moderate rise of 27% , and the rise in Sg is only about 10%.

In all three species studied, we found that the MFA increased and the specific modulus decreased from the pith toward the bark. This trend is the opposite of that usually observed in temperate trees, in which juvenile wood is generally found to have a larger MFA and a lower specific modulus than mature wood.[8] This opposite trend has already been noted for other species of rainforest trees.[31] A tentative explanation can be given from the viewpoint of tree biomechanics. The usual interpretation of the trend observed in temperate trees is that young trees need a wood with high deformability to allow their stem to bend without breaking in response to wind loads. This high deformability is provided by a large MFA, and results in a low specific modulus. In later stages, when the tree becomes larger, it needs high stiffness to resist its own weight and wind forces.[32,33] This high stiffness is provided by a smaller MFA inducing a higher specific modulus. In contrast, in tropical rainforests, which are in general very dense, i.e., the trees grow close together, the main constraint for the growth of young trees is the poor light availability. Young trees need to achieve sufficient height to receive sunlight. On the other hand, the high density of the forest protects trees from being damaged by external forces such as wind. This condition makes young trees grow preferentially in height, instead of growing in diameter. To prevent buckling caused by the tree's own weight, the young cambium produces trees with a stiff wood (low MFA). In later stages, when the tree is close to reaching the canopy, radial growth becomes quicker and stem stiffness is achieved by a large diameter rather than by a very low MFA.

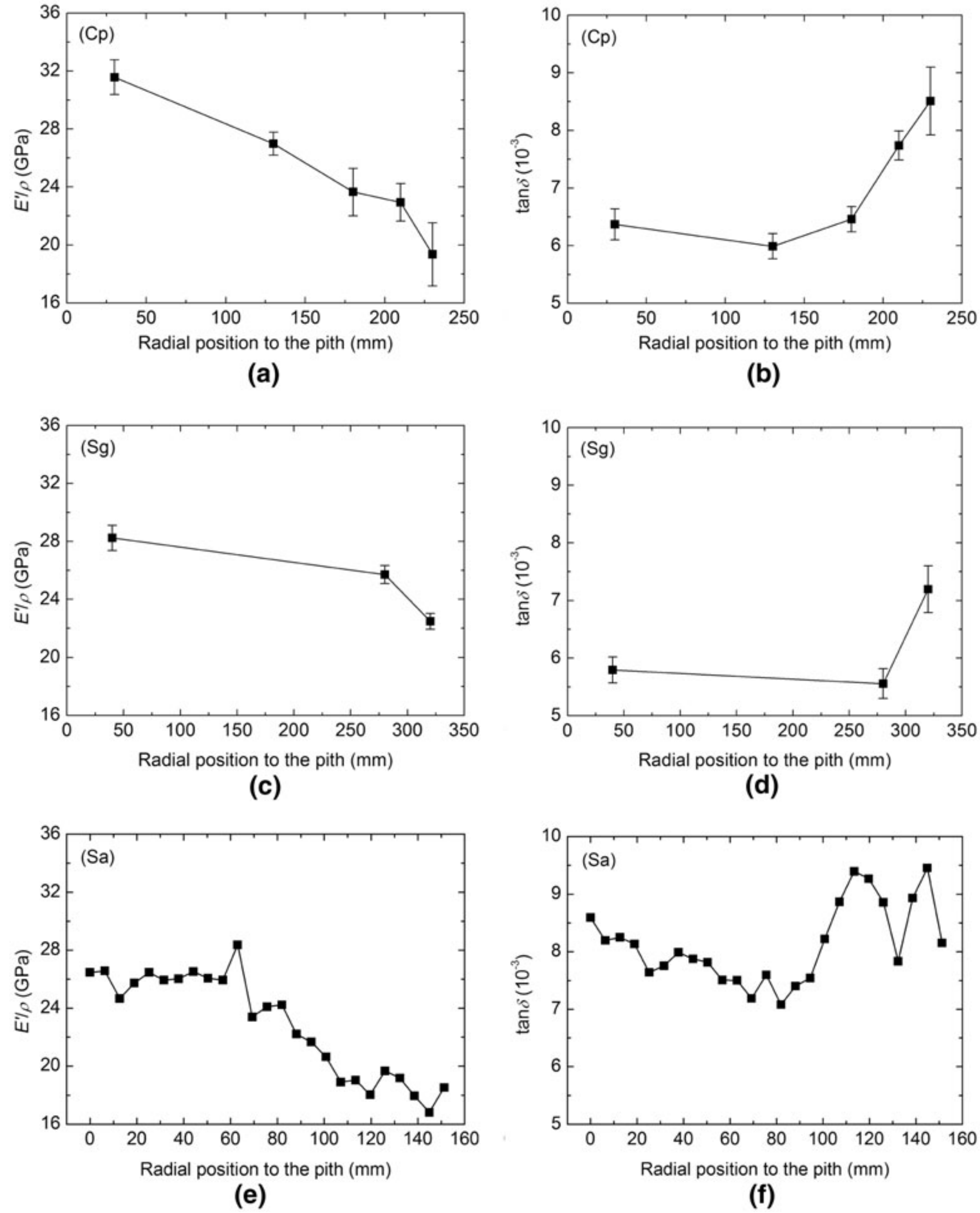


Fig. 4. Radial trends of vibrational properties E'/ρ (**a, c, e**) and $\tan\delta$ (**b, d, f**) for species Cp, Sg, and Sa, respectively. For species Cp and Sg, each *square* represents the mean value of 15–25 specimens and the *error bar* indicates the standard deviation

Relation between vibrational properties, MFA, and heartwood formation

The vibrational properties are examined with respect to MFA in Fig. 6. The specific dynamic modulus shows a good correlation with MFA for each species tested (the coefficient of determination of the linear regressions were R^2 = 0.80, 0.59, and 0.93 for Cp, Sg, and Sa respectively). This relation has repeatedly been observed in other species[9] and can be expected from micromechanical considerations.[34,35] The relation corresponds to the reinforcing effect of cellulose microfibrils: the more they align with the fiber axis, the more efficient this reinforcement is. However, the fact that the regression slopes depend on species cannot be explained from the micromechanical perspective. In fact, the same slope would be expected for woods with similar cellulose contents (which is the case for the trees in this study, see cellulose contents in Table 1) even if they belong to different species. One possible explanation for the different relation between E'/ρ and MFA can be traced back to the measurement of MFA. As previously mentioned, the estimation of MFA using X-Ray diffraction may be submitted to a bias that depends on species. Therefore, the observed differences in slope are more likely due a calibration bias in the MFA estimation (in particular for species Sg and Cp). On the other hand, within each species, the E'/ρ and MFA relation applies equally to different wood types, suggesting that the wood type (which is associated with differences in extractive content) is not a determinant factor of the specific modulus compared to MFA.

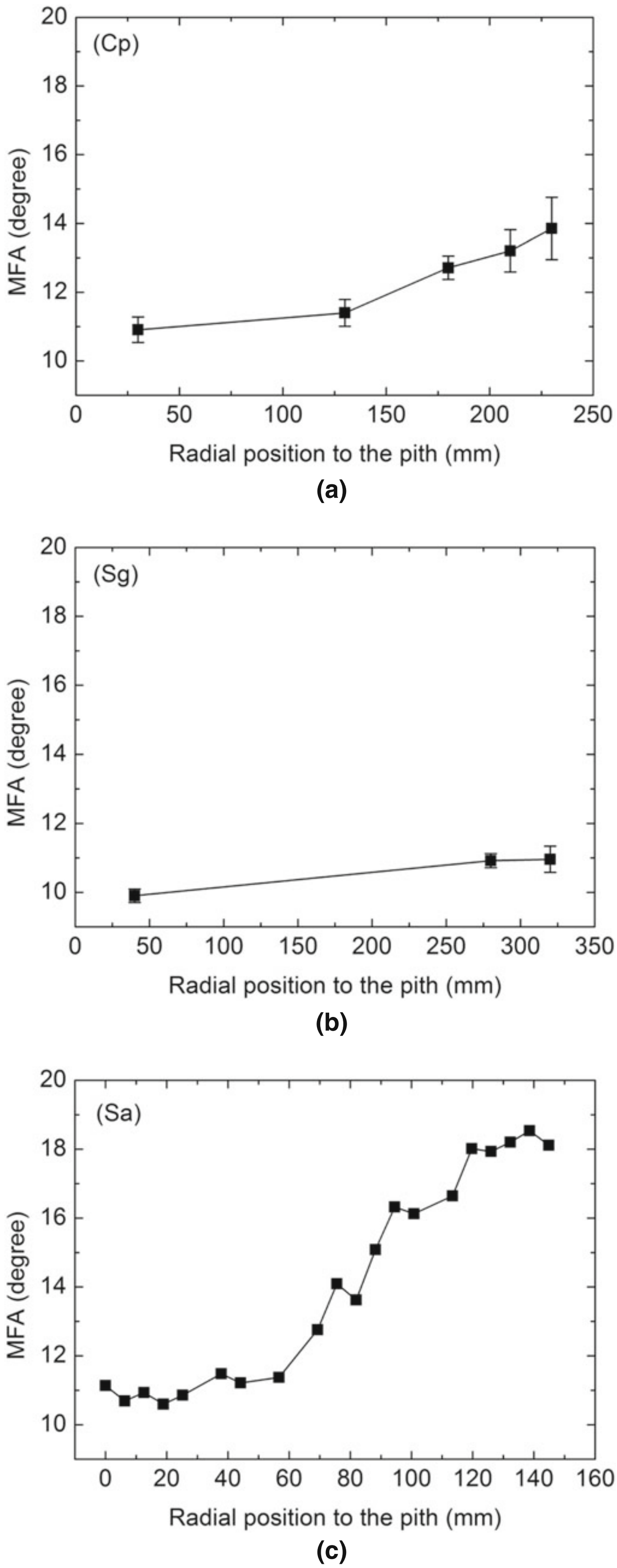


Fig. 5. Radial trends of microfibril angle (*MFA*) for species **a** Cp, **b** Sg, and **c** Sa. For species Cp and Sg, each *square* is the mean value of selected specimens and the *error bar* indicates the standard deviation

From Fig. 6b,d,f, the linear regression shows that the influence of MFA on damping coefficient $\tan\delta$ depends on the species: it was stronger for Cp ($R^2 = 0.63$), weaker for Sg ($R^2 = 0.11$), and not significant for Sa. A significant relation was, however, also found for Sa if only the "outer" group was examined. The relationship between $\tan\delta$ and MFA can also be explained from the theory of micromechanics: the dissipative ability of wood is mainly due to the viscoelastic nature of the amorphous matrix, and the contribution of the matrix to the behavior in the fiber direction is larger when MFA is large, so the damping coefficient is expected to increase along with MFA. However, unlike the specific modulus, the damping coefficient significantly depends on other parameters than the MFA, namely the viscosity of matrix. This is the reason why data scattering is more obvious for $\tan\delta$ than for E'/ρ (Fig. 6). The viscoelasticity of matrix material has its origin in the main polymers and can be modified by the presence of extractives. Inside trees of the same species, it is reasonable to suppose that radial variations in polymer content and configuration are lower than radial variations in extractive content. Therefore, we assumed that extractives were responsible for the variations of viscoelastic properties between our specimens. The influence of extractives on the damping coefficient was also revealed by other researchers[2,3,4,17] by changing the extractive content either through extraction or impregnation.

Relation between specific modulus and damping coefficient

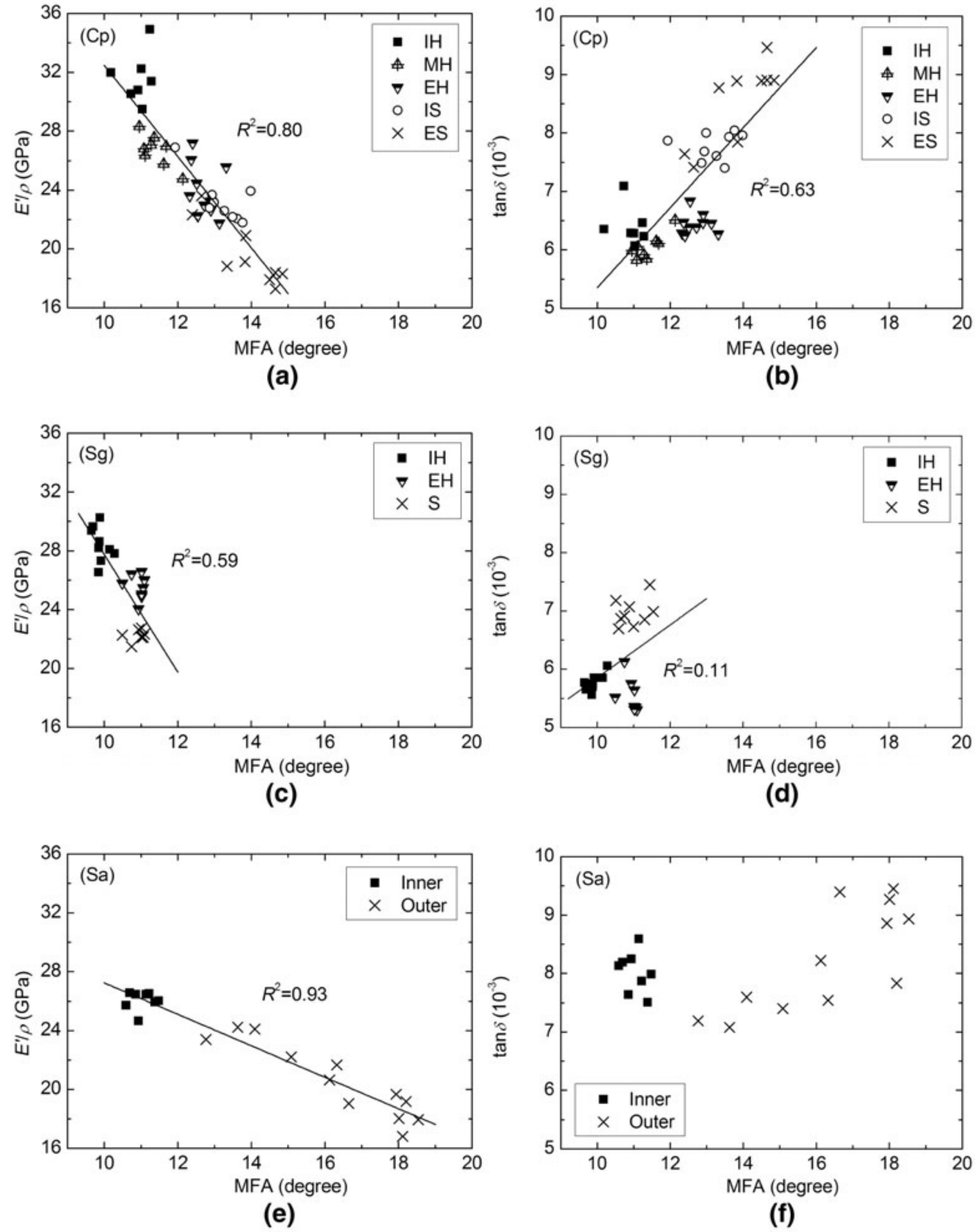
The linear relation between $\log(\tan\delta)$ and $\log(E'/\rho)$ proposed by Ono and Norimoto,[15] based on the study on more than 1000 specimens of 20 softwood species, is addressed here as the "standard relation" and is expressed as:

$$\log(\tan\delta) = -1.23 - 0.68\log\left(\frac{E'}{\rho}\right) \tag{2}$$

For the purpose of comparison, the curve of Eq. 2 is plotted in Fig. 7, together with the values obtained from our species. Linear regression based on all the data together was performed first to check the validity of the "standard relation." The resulting slope was -0.72 ± 0.10, which is in rather good agreement with the standard value (-0.68). For each species, linear regressions were also performed and it was found that the slope was comparable with the standard value when the experimental data cover a relatively wide range of specific dynamic moduli, as in the case of Cp (-0.62 ± 0.11), although different numerical values were obtained for species with a narrower range of E'/ρ variations, as for Sa (-0.33 ± 0.17) and Sg (-0.98 ± 0.21). A conclusion can be drawn here that the "standard relation" describes quite well the relation between $\tan\delta$ and E'/ρ, regardless of the wood species, radial position, and chemical composition.

As shown in the previous section, E'/ρ and MFA are strongly correlated. In other words, the variation of E'/ρ is equivalent to that of MFA and may be even more precise than that shown here, considering the measurement bias of MFA. Therefore, by the $\log(\tan\delta)$ versus $\log(E'/\rho)$ relation,

Fig. 6. Dependence of E'/ρ (**a, c, e**) and $\tan\delta$ (**b, d, f**) on MFA



the damping coefficient can be, to a certain extent, related to wood microstructures. Following this argument, the coefficient in front of E'/ρ in Eq. 2, the slope, can be considered as the influence of MFA on $\tan\delta$. On the other hand, the intercept, which indicates how far the actual $\tan\delta$ deviates from the MFA-predicted contribution, reflects the influence of chemistry, i.e., the extractives in our case (Fig. 8). Evidence for this can be found from the study of Brémaud et al.,[3] who performed extraction on African padauk wood and compared the vibrational properties before and after the extraction. It turned out that only the intercept changed while the slope was identical. Based on this observation, it is reasonable to ascribe intercept changes to changes in extractives content.

Based on this hypothesis, wood specimens can be regrouped according to the difference between experimental $\tan\delta$ values and "standard" values predicted by Eq. 2, assuming that that these new groups should actually reflect the dependence of damping coefficient on the chemistry of matrix. Distances to the "standard relation" (DSR) were previously found to be correlated to extractives content in 25 tropical hardwoods[5] and to be quite good descriptors of species classification.[25] DSR for each group of woods in this study is indicated in Fig. 9.

For Cp, DSR values of middle heartwood and external heartwood are not significantly different and can be grouped together (Fig. 9). This suggests that they have approximately the same "mechanically active" extractives. Based on this

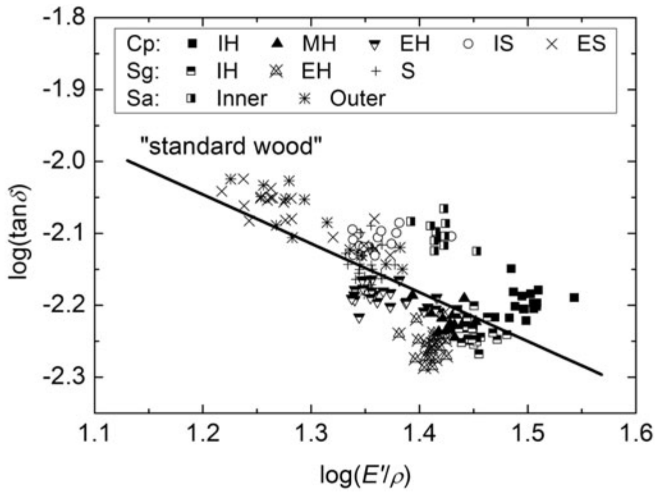


Fig. 7. Log(tan δ) versus log(E'/ρ) diagram for all studied species. The "standard wood" *line* is defined by the "standard relation" (Eq. 2)[15]

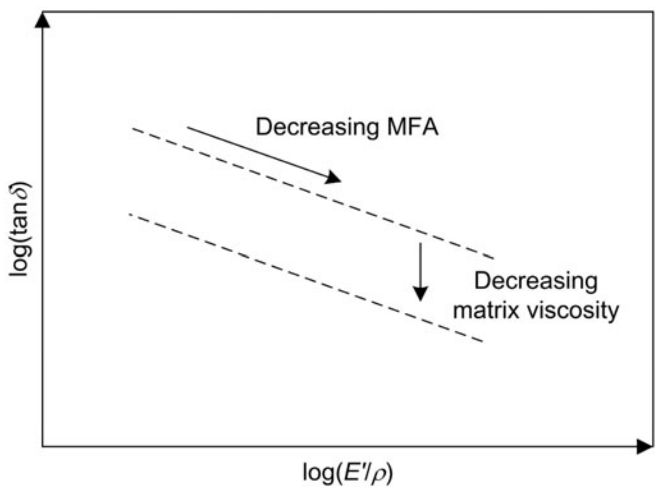


Fig. 8. Principle of the interpretation of log(tan δ) versus log(E'/ρ) diagrams[3,17]

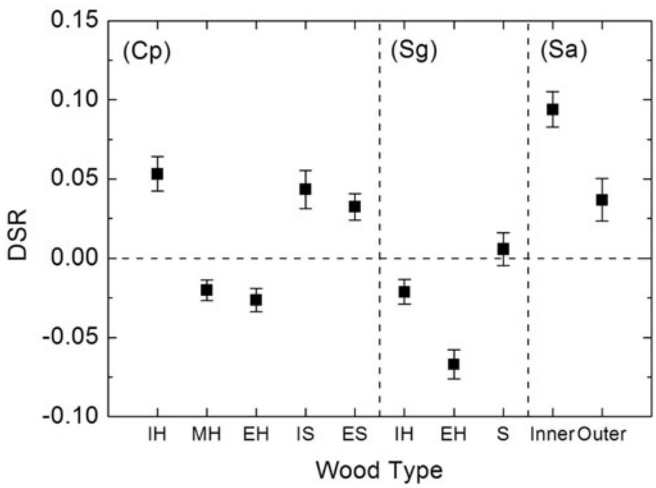


Fig. 9. Averages and 95% confidence intervals of the distances to the "standard relation" (*DSR*) for each group of species Cp, Sg, and Sa

result, linear regression with the slope fixed at –0.68 was performed for new groupings (Fig. 10). The groups MH and EH of Cp are therefore on the same line since their chemical constituents are quite similar, but they are located at different positions on the curve because their MFAs are different. By the same process, inner sapwood, external sapwood, and inner heartwood can be grouped together (Fig. 9). The inner heartwood, surprisingly, falls into the group of sapwoods. This could mean that the oldest extractives in the inner heartwood are no longer mechanically active, as has been shown for biologically active extractives in other species.[36] The other possible explanation is that there is less extractive content in inner heartwood.[37] Since we didn't perform extraction on our specimens, this is still an open question. On the other hand, the polymeric constituents vary along the radial direction, which may also be a cause of damping variation. The same observation can be done for Sg (Fig. 10), except that the ageing of inner heartwood mechanically active extractives is less obvious. For Cp and Sg, mechanically active extractives work in the same way: lowering the damping coefficient. Sa is known to have some biologically active extractives, although in rather small amounts (Table 1), and it seems that there are also some mechanically active extractives; however, these two types of extractives act in opposite ways, i.e., biologically active extractives enhance instead of weaken the damping of the heartwood, as is the case for the extractives of Fagaceae or Sapindaceae.[25]

Conclusions

By carrying out free–free flexural vibration tests and X-ray diffraction measurements, the vibrational properties and their relations to radial location and microfibril angle were investigated for three tropical rainforest trees. Based on the results, the following conclusions can be drawn:

- The radial trends of vibrational properties were typical of rainforest trees, which differ from those obtained from temperate trees. This original remark suggests that the growing conditions have a major impact on patterns of radial variations in wood properties.
- The specific dynamic modulus, E'/ρ, is, to a large extent, dependent on the MFA. However, this relationship differs between species, which, according to our analysis, confirms that MFA measurement by the XRD method needs to be calibrated for many tropical species.
- The damping coefficient, tan δ, is influenced by both MFA and the chemical constituents, the effects of which may be separated in the log(tan δ) versus log(E'/ρ) diagram.
- In all species tested, outer heartwood showed distinct viscoelastic behavior (larger damping for Sa, lower damping for Cp and Sg). These differences in damping behavior could be due either to changes in the constitution of matrix polymer or to changes in extractive content. We assumed that the main factor contributing to the difference between sapwood and heartwood is the extractive content.

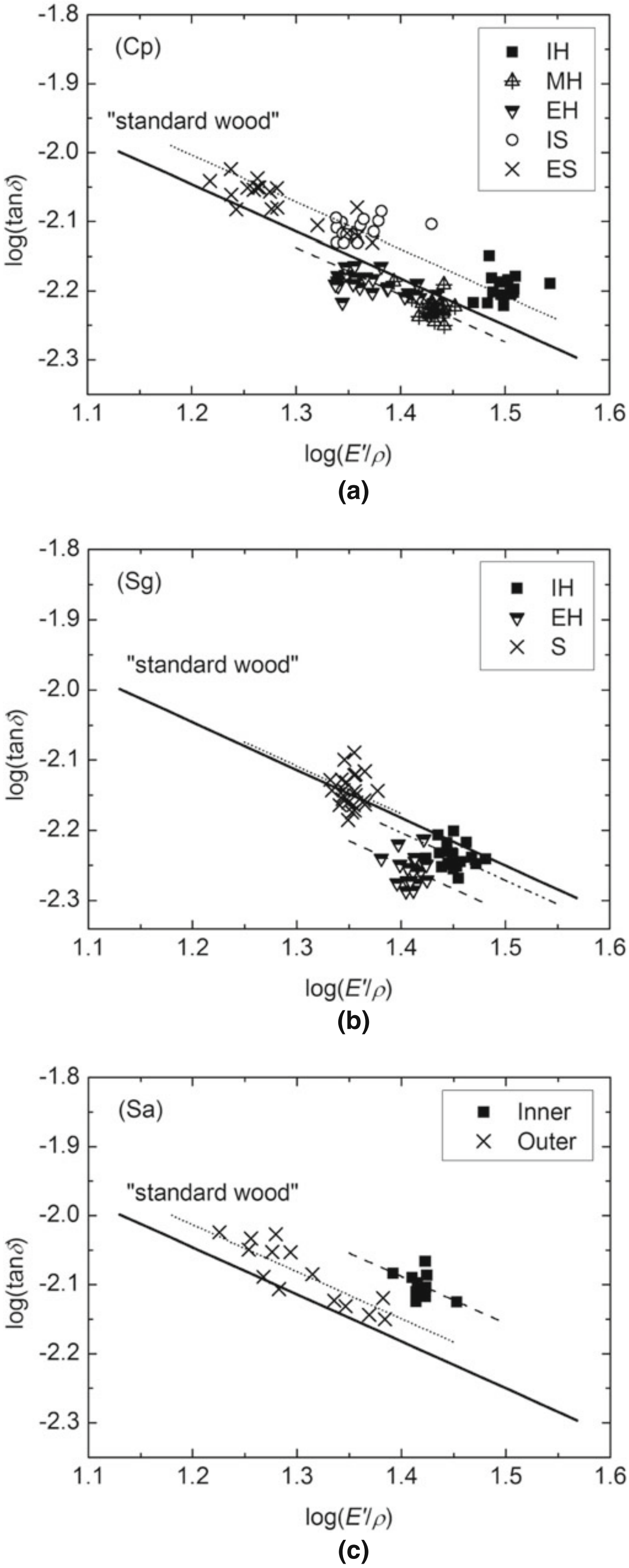


Fig. 10. Linear fitting of log(tan δ) versus log(E'/ρ) with the slope fixed at –0.68 for species Cp (**a**), Sg (**b**), and Sa (**c**) (*lines other than the solid line*) using groupings suggested by similar DSR values in Fig. 9. The *solid line* shows the relation for "standard wood"

– Furthermore, the behavior of inner heartwood differs from that of outer heartwood. This difference was ascribed to the chemical ageing of extractives.
– Mechanical analysis by vibration of radial sequences of specimens is an efficient way to study ontogeny of the xylem through both phenomena of juvenility and duraminization combined with ageing.

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