Effect of extractives on vibrational properties of African Padauk (Pterocarpus soyauxii Taub.)

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Abstract Extractives can affect vibrational properties tanδ (damping coefficient) and E'/ρ (specific Young's modulus) but this is highly dependent on species, compounds, and cellular locations. This paper investigates such effects for African Padauk (*Pterocarpus soyauxii* Taub.), a tropical hardwood with high extractives content and a preferred material for xylophones. 5 groups of 26 heartwood specimens with large, yet comparable, ranges in vibrational properties were extracted in different solvents. Changes in vibrational properties were set against yields of extracts and evaluation of their cellular location. Methanol (ME) reached most of compounds (13%), located about half in lumen and half in cell-wall. Water solubility was extremely low. tanδ and E'/ρ were very strongly related ($R^2 \geq 0.93$), but native wood had abnormally low values of tanδ, while extraction shifted this relation towards higher tanδ values. ME extracted heartwood became in agreement with the average of many species, and close to sapwood. Extractions increased tanδ as much as 60%, irrespective of minute moisture changes or of initial properties. Apparent E'/ρ was barely changed (+2% to -4%) but, after correcting the mass contribution of extracts, was in fact slightly reduced (down to -10% for high E'/ρ), and increasingly so for specimens with low initial values of E'/ρ.

Keywords: Damping coefficient Extractives; Heartwood; Pterocarpus soyauxii; Tropical hardwood; Vibrational properties; Young's modulus

Introduction

Extractives are amongst the most diversified features of woods, both in their nature and their amounts. They can represent only a minute percent in some woods, while the mean for tropical hardwoods is of 7.6% total extracts (4.6% for ethanol-benzene extracts), based on 653 species from the CIRAD database (unpublished), and in some species they can represent as much as one third of wood mass, e.g. 35% total extracts in *Excoecaria parvifolia*, Euphorbiaceae (Gérard 1991). Extractives are known to affect wood colour, biological durability or moisture-related properties (e.g. Wangaard and Granados 1967; Hernandez 2007). On the other hand, their contribution to wood mechanical behaviour is not generally acknowledged. Notwithstanding, extractives can deviate wood properties from well established relations, slightly strengthen wood (Luxford 1931), or hamper creep (Ajuong and Breese 1997).

Particularly pronounced effects of extractives are found in the case of vibrational properties: they can diminish the damping coefficient (tanδ) as much as a 2-fold order in some tropical Leguminosae (*Caesalpinia echinata*, *Dalbergia nigra* and *D. latifolia*) or the softwood *Thuja plicata* (Sugiyama et al. 1994; Matsunaga et al. 1999; Yano et al. 1995; Yano 1994). Though, effects are species-dependant, and completely opposite effects are found for sugar extractives of Reed (*Arundo donax*) (Obataya et al. 1999), or in sapwood of *Thuja plicata*, in contrast with results on heartwood of this same species (Yano 1994). Impregnation of Spruce wood with natural polycyclic molecules or other simple phenolic compounds (Matsunaga et al. 1999; Minato et al. 1997, 2010; Sakai et al. 1999) demonstrated that mechanical effects are highly compound dependant. When extractives are active on vibrational properties, they principally modify tanδ, yet some minor effects are also observed on specific modulus of elasticity (E'/ρ). However, as extractives contribute to specific gravity (ρ), their intrinsic repercussions on E'/ρ are difficult to determine.

Extractives can be assimilated to "natural chemical modifications". In this scope, rheological analyses (Akitsu et al. 1993) indicate that effects would be dependant on the properties of compounds, their interactions with wood polymers, and their location within wood structure. Cellular localisation of extractives may be approached by using sequences of solvents of different polarities and abilities to swell the cell-wall (Morgan and Orsler 1969b). Yet, as this also depends on time and temperature (Morgan and Orsler 1969a; Mantanis et al. 1994, 1995), these indicators should be monitored, for example through shrinkage due to extractives' departure.

The bright red heartwood of African Padauk (*Pterocarpus soyauxii* Taub., Leguminosae-Papilionoideae), has a high hygroscopic stability, and good natural durability due to its extractives (Déon et al. 1980). It contains about 13% extractives, which were abundantly used as a dye-source in the 19th century. At least 13 compounds were identified, mainly of polyphenolic nature (Surowiec et al. 2004; Cardon 2007). This medium-heavy (specific gravity 0.6-0.9) wood is favoured for xylophone making, both in modern western manufacture and in African traditions, as for the slit-drums that were used for long-distance transmission of messages. As very low damping should be of prime importance for xylophones woods (Aramaki et al. 2007), such predominant uses strongly suggest that this species might have peculiar vibrational properties.

This paper aims at clarifying if, and how far, extractives determine the vibrational properties of this selected tropical hardwood. The changes in properties caused by extractions with different solvents are set against calculations of cell-wall and lumen extracts removed, and their purely mass contribution is taken into account. In addition, as interlocked grain is a common feature of this wood, effects of extractives were observed on specimens with varying initial properties.

Material and methods.

Material & preparation of specimens

Wood material of *Pterocarpus soyauxii* Taub. (one tree from Cameroon) was obtained from CIRAD sawmill. Two adjacent flat-sawn planks were selected in medium heartwood for being the most uniform in colour. Vibrational specimens (12×2×150mm R×T×L) were cut parallel to the main axis of the tree (they showed grain angles of 0° to 25° due to interlocked grain). The heartwood sampling contains 158 specimens. A smaller (N=18) sampling of sapwood was added, but its grain angles could not be recorded.

Vibrational properties

Samples were first dried (in order to reach equilibrium in adsorption) for 48h at 60°C. All tests were performed after at least 3 weeks stabilization in controlled conditions at 20±1°C and 65±2%RH. Measurements were made by non-contact forced vibrations of free-free bars. Vibration emission and detection were computer-driven using a specific development in Labview® Software and a laser triangulation displacement sensor (Brémaud 2006). E'/ρ was deduced from the first resonant frequency according to the Euler-Bernouilli equation. Damping coefficient $\tan\delta$ was measured both through the 'quality factor' and through logarithmic decrement of amplitude after stopping the excitation. Frequencies were in the range of 200-400Hz. 3 repetitions were made for each specimen and the mean error was <5%.

Extraction procedure

After vibrational measurements in native state, 6 matched groups of 26 heartwood specimens each were separated: 5 for extractions and 1 reference. Groups were defined so that their ranges in grain angle (0-25°) and in vibrational properties were equivalent between them (mean and variance were not statistically different at the level of 0,001). Sapwood specimens were not subjected to any extraction.

5 groups were extracted in parallel in the following solvents (technical grade): diethyl ether (ET, group B); methylene dichloride (MD, group C); acetone (AC, group D); methanol (ME, group E); hot water (HW, group F). Samples were previously dried and all extractions used Soxhlet apparatus. Temperature in the extraction tube would be of 30-60°C for organic solvents. The time necessary to obtain as complete an extraction as possible varied from 20h to 30h. Organic solvents were then rinsed in ethanol followed by cold distilled water. Specimens were slowly dried by steps to 'air-dry' to avoid checking or collapse and finally oven-dried again and submitted to the same conditioning and measurement steps. The percentage of extractives was calculated both from the differences in oven-dry weight and from the weighting of residuals, with good consistency between the two. The reference group was submitted to all conditioning/drying steps, but to no extraction procedure.

Results and discussion

Yield and location of extractives

The less polar solvents, diethyl-ether (ET) and methylene-dichloride (MD) reached rather small amounts of extracts (2.3 and 3.1% of dry-wood weight, respectively). Higher quantities were removed by acetone (AC, 8.8%) and mostly so by methanol (ME), the most efficient extraction with a yield of 13.4%. Although extractions from solid are expected not to be fully exhaustive, overall yields (Fig. 1) are consistent with those on ground wood from Déon et al. (1980). The very low water-solubility (0.6%) of Padauk, an "insoluble" red dye source (Cardon 2007, Surowiec et al. 2004), is confirmed.

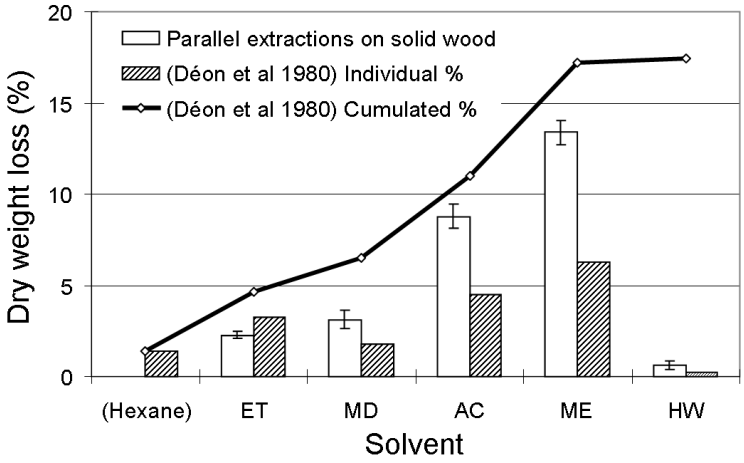

Fig. 1 Extractives removed by the different solvents (percentage of oven-dry weight of wood) compared with results of Déon et al. (1980)

Solubility patterns could give a first idea of the cellular location of removed extractives: low polarity solvents (ET and MD) should attain mostly lumen deposits, while AC, ME and HW can swell the cell wall and reach compounds located inside. Though, our parallel extraction procedure means that lumen compounds are still present in the wood subjected to polar solvents. In addition, the swelling capacity of ET and MD is not strictly zero and the swelling by different solvents depends on the treatment time and conditions (Morgan and Orsler 1969ab, Mantanis et al. 1994, 1995). Thus, the modifications in oven-dry volume could bring useful additional information. The proportion of cell-wall compounds retrieved by a given extraction could be approached by:

$$E_{cw} = \frac{\Delta V_{0ext} \times SG_E \times VR_{ex}}{W_E} \quad (1)$$

Where:

E_{cw}: Proportion of cell-wall extractives from total yield of a treatment;

ΔV_{0ex}: Diminution (in cm^3) of oven-dry volume of wood;

W_E: Weight (g) of removed extractives;

SG_E: Density (in g cm^{-3}) of extractives;

$VR_{ex}=(V_{Ecw}/\ \Delta V_{0ex})$: Ratio between volume of wall-located-extracts removed, and diminution of macroscopic oven-dry volume.

The last two parameters are not known but we could assume, as does Chafe (1987), the mean experimental values of $SG_E \approx 1.4$ and $VR_{ex} \approx 0.9$ obtained by Kuo and Arganbright (1980). Estimated percentage of cell-wall compounds gets smaller when assuming lower

densities of extracts and/or smaller volume ratios (Fig. 2a), but estimations using different sets of plausible values are not statistically different except for Methanol (shown on Fig. 2a). Percentages, calculated with the above mean values, of extracts (dry-wood basis) removed by each solvent from cell-walls and from lumens are shown on Fig. 2b.

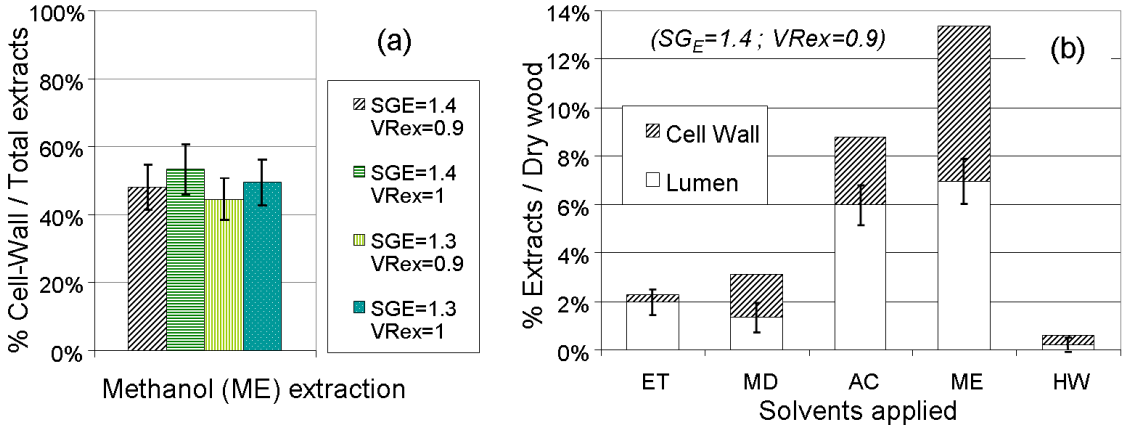

Fig. 2 (a) proportion of cell-wall extractives from total methanol extracts, calculated with different assumptions about specific gravity of extractives SG_E and volume ratio VR_{ex} ; **(b)** estimated percentages of lumen and cell-wall extracts removed by each solvent (oven-dry native wood basis)

As expected, the proportion of cell-wall extracts was very low for ET. But it reached more than 50% for MD which is a rather low-polarity solvent. Acetone retrieved a bit more cell-wall compounds (2.8%), and mostly a good quantity from lumen (6.0%), which is much higher than the sum of those removed by ET and MD. This was also observed with ME and suggests that lumen deposits in Padauk are more soluble in medium-high polarity organic solvents (but not water), than in lower polarity solvents. ME reached about half cell-wall and half lumen compounds.

Specific modulus and damping for native and extracted wood

The relationship between tanδ and E'/ρ was equally strong for native and extracted wood (Fig. 3), with coefficients of determination R^2 of 0.93 to 0.97. All native groups followed a single trend and after extractions still followed a trend of similar shape and range of E'/ρ. Removal of extractives clearly resulted in a vertical shift of the relation towards higher damping coefficients (groups C to F). Group B was little affected, but a slight diminution of damping may be noticed over the lower range of E'/ρ. After the most efficient (ME) extraction, treated wood came close to the "standard" relationship that Ono and Norimoto (1983) obtained on woods with varying microfibril and grain angles. Yet, as this is a statistical relation on several species, Padauk sapwood was the closest reference of similar polymeric composition and microstructure, but with much less extractives. It came close to the standard trend or with a bit higher damping. ME extracted heartwood still remained a bit under the damping of sapwood or of the standard: extraction from solid wood may not be fully exhaustive, as samples retained a shade of the distinctive reddish hue of Padauk. Also, some "extractives" can only be removed by harsher procedures such as alkaline extraction (Lange and Faix 1999, Surowiec et al. 2004).

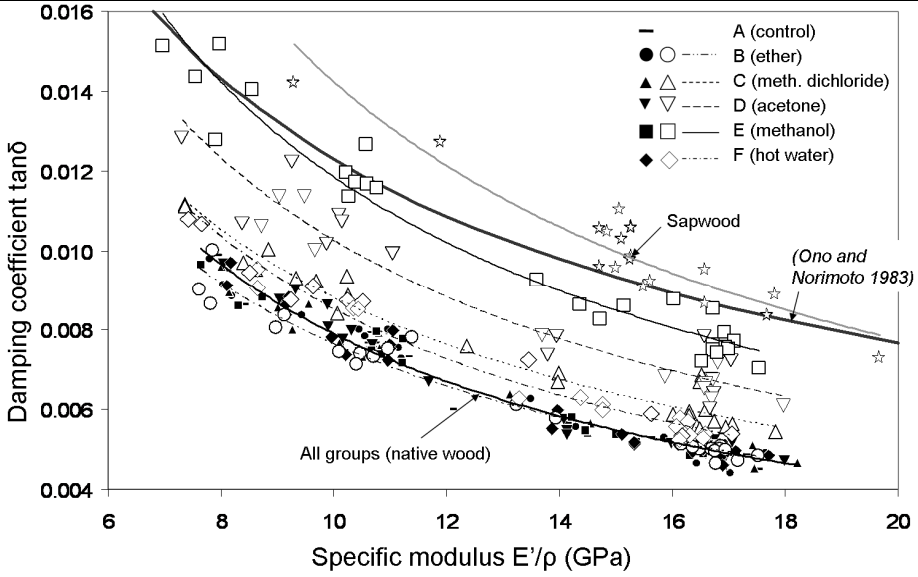

Fig. 3 Damping coefficient and specific Young's modulus for native and extracted woods

Modifications of damping coefficient

The variations in damping coefficient tanδ after extractions are set against the corresponding amounts of extracts on Fig. 4.

Extraction in the most apolar solvent ET induced nearly no change in tanδ: as it mostly removed lumen compounds, this seems to confirm that cell-wall location is a necessary condition for extracts to be able to diminish wood viscosity. Apart from ET, all extractions markedly increased tanδ. The removal of about 1.8% and 2.8% of cell-wall extractives by MD and AC, respectively, induced mean increases in tanδ of 19% and 36%. These quantity/effect ratios are close to those for haematoxylin impregnation of wood (Minato et al. 1997, Matsunaga et al. 1999). ME removed most of both lumen and cell wall compounds and increased tanδ of circa 60%. Damping increased of about 4.7% per 1% total extracts (or 9% per 1% cell-wall) removed by ME on Padauk, this is similar to *Dalbergia nigra* but at least twice more efficient than for *D. latifolia* or *Thuja plicata* (Yano 1994; Yano et al. 1995).

Hot-water induced quite an important change (13%) in tanδ despite its very low yield. Perhaps, hemicelluloses might also have been affected, but the temperature was below 90°C and wood equilibrium moisture content barely changed. Possibly, changes in damping after HW treatment may mostly result from the effects of the small amount of water-soluble extracts. Such extractives – barely coloured – would be of a different chemical nature than those – of a deep brick-red – removed by organic solvents. Padauk water solubility is opposite to that of Pernambuco (*Caesalpinia echinata*), for which extractives strongly diminishing tanδ are mostly water-soluble (Sugiyama et al. 1994), yet damping of both woods is equally very low, which suggests that polarity of extractable compounds is not a key factor concerning their effect on mechanical properties.

Extractives can also affect the equilibrium moisture content (EMC) of wood, which affects tanδ. To observe EMC variations after extraction, it must be corrected for the initial and remaining extractive content (Hernandez 2007a). EMC was little affected by ET, MD, AC and HW extractions (from -0.14 to +0.16% or relative changes of -1.5 to +1.8 %/%) and a bit more by ME (+0.82% absolute, or +9.1%/% relative variation). These variations (at 20°C and 65% RH) were moderate, as extractives mostly affect EMC over the higher range of RH (Wangaard and Granados 1967). According to Obataya et al. (1998, 2001) the changes in EMC after ET, MD, AC and HW extractions would induce no more than 1-2% change in

tanδ, and could reach about 6% for ME. Thus, MD, AC and HW increased tanδ of Padauk wood independently of minor moisture changes, and ME extraction strongly increased tanδ by itself and through a smaller additional effect due to moisture change.

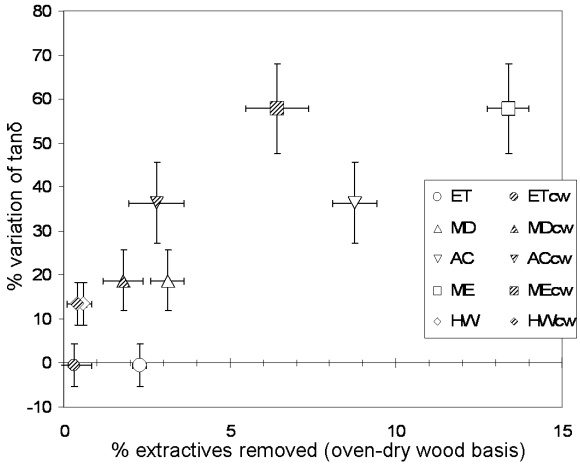

Fig. 4 Variation in damping coefficient plotted against the percentage removed by each solvent: total extracts (open symbols) and estimated cell-wall extracts (hatched symbols). Points are the mean, and error bars the standard deviation, for each group (N=26)

Modifications of Specific Young's Modulus

Specific modulus (E'/ρ) requires to be corrected to take into account the contribution of extractives to specific gravity, and its modifications after extraction:

$$\left(\frac{E'}{\rho}\right)_C = \left(\frac{E'}{\rho}\right)_A \times \frac{\rho_A}{\rho_{X\max}} \quad (2)$$

Where subscripts C and A mean, respectively, corrected and apparent values for native wood or any treatment, and ρ_{Xmax} is specific gravity after the most efficient extraction (ME). All native groups had a specific gravity of 0.76±0.02, and ME extracted wood of 0.70±0.02. Apparent E'/ρ of native wood was under evaluated by 0.7 to 1.8 GPa as compared to corrected values (table 1). Though, the contribution of extractives' mass to apparent E'/ρ had very little influence on the exceptionally low damping of the relationship for native wood (Fig. 5).

Table 1: Ranges in vibrational properties for groups B to F before and after extraction.

Group → Extraction	**Extractives yield (%)**	**E'/ρ corrected (Gpa)** *E'/ρ apparent (Gpa)*		**tanδ (‰)**	
		Native	Extracted	Native	Extracted
B → ET	2.3	8.6 – 19.4 *7.8 - 17.7*	8.2 - 18.9 *7.6 - 17.5*	4.4 - 9.8	4.7 – 10.0
C → MD	3.1	8.8 - 20.0 *8.0 - 18.2*	7.9 - 19.2 *7.4 - 17.8*	4.5 - 9.6	5.4 - 11.2
D → AC	8.8	8.8 - 19.6 *8.1 – 18.0*	7.5 - 18.3 *7.3 – 18.0*	4.6 - 9.6	6.0 - 12.8
E → ME	13.4	8.4 - 18.9 *7.6 - 17.1*	7.0 - 17.5 *7.0 - 17.5*	4.6 - 9.7	7.0 - 15.2
F → HW	0.6	8.9 – 19.4 *8.1 - 17.7*	8.1 - 18.6 *7.4 - 17.1*	4.6 - 9.7	5.3 - 10.8

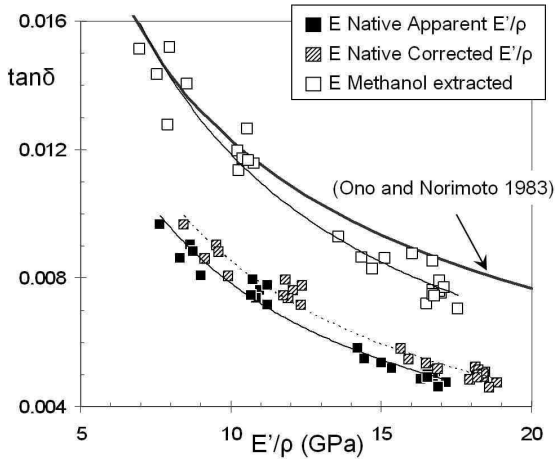

Fig. 5 Relationship between damping and specific modulus: apparent and corrected values for native wood, and properties after exhaustive extraction

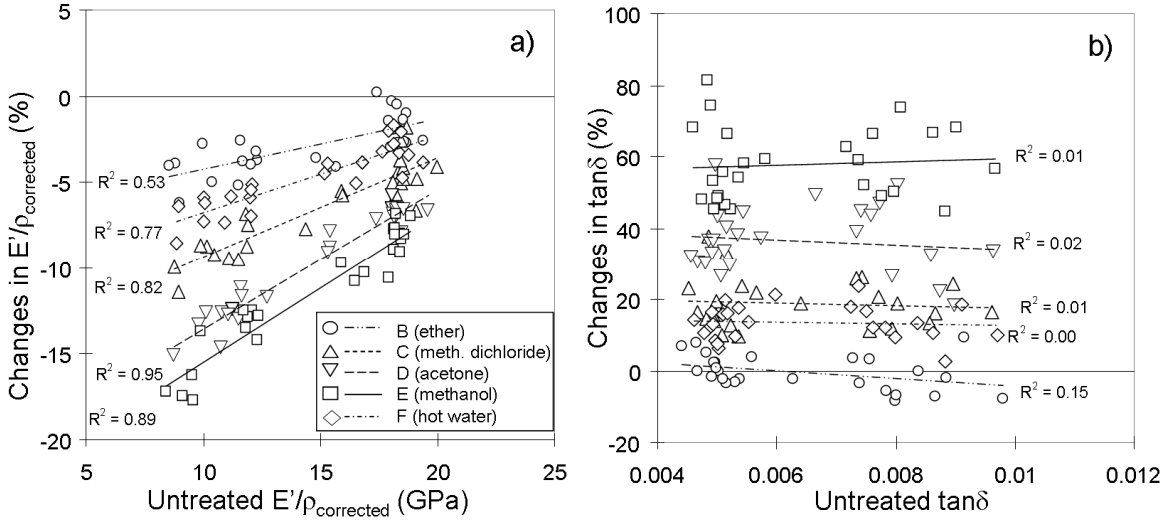

Fig. 6 Variations in vibrational properties after extraction plotted against values for untreated wood; **(a)** changes in specific modulus (corrected for extractives' mass); **(b)** changes in damping coefficient

Corrected specific modulus E'/ρ was decreased after all extractions (table 1). The amplitudes of its relative variations clearly depended on the initial E'/ρ of untreated specimens (Fig. 6a), but were usually much smaller than those in tanδ, which were independent of initial values (Fig. 6b). As the variations in native properties are related to the orientation of wood elements (grain and/or microfibrils) this indicates that extractives effects on tanδ little depended on wood orientation, while their effects on E'/ρ got bigger with angle deviations. An increase of radial E'/ρ is also obtained when impregnating spruce wood with Padauk's extractives (Minato et al. 2007). This suggests that extractable compounds played a role in reinforcing the matrix substance of cell wall, decreasing its viscosity and increasing a bit its rigidity.

In addition, changes in E'/ρ after AC or ME extractions were close, while ME removed twice more cell-wall compounds and resulted in a much higher increase in tanδ. Mechanisms of modifications of these two properties by extractives may be different, and bulking seems of secondary importance.

A few other works report a stiffening of the cell-wall by some extractives, mainly of polyphenolic nature. Hypotheses about their mechanisms of action include the ability to establish strong networks of weak chemical bonds with wood polymers (Ajuong and Breeze 1997), or the rigidity of molecular structures (Bariska and Pizzi 1986), or the addition of both

phenomena (Sakai et al. 1999). In the future, chemical analyses and comparison, over several species and researches, of the molecular features of extractives active on vibrational properties would help to clarify the respective roles of these different parameters.

Conclusion

We investigated the variations in vibrational properties of African Padauk after removal of extractives in different solvents, while taking into account estimations of cellular location and mass contribution of extractives. Results were as follow:

- Apolar diethylether (ET) merely removed lumen-extracts, while methylene-dichloride significantly reached cell-wall. Methanol (ME) removed about as much of cell-wall and of lumen extracts, and most of compounds retrieved by less polar solvents. The very low water-solubility of Padauk was confirmed.
- Damping coefficient (tanδ) of Padauk was exceptionally low. It was very strongly related to specific modulus (E'/ρ), and extractives' removal shifted the relation towards higher tanδ. ME extracted wood became close to the average on many species, and to sapwood.
- tanδ was increased by as much as 60% for 13% extracts (about 6% from cell-wall) removed by ME. Changes in tanδ were independent both of slight changes in moisture content, and of untreated properties.
- E'/ρ was apparently barely modified, but when correcting the mass contribution of extractives it was diminished by extractions: only slightly (< -10%) for specimens with high initial values, but increasingly so for those with lower untreated E'/ρ (i.e. with grain angle).

Padauk extracts were found to be responsible for its exceptionally low damping coefficient, and thus contribute in making this wood well-suited to xylophone uses. They also have a moderate but significant effect on stiffening wood, depending on orientation, which needs to be further analyzed. Our results tend to confirm that cell-wall location is a necessary condition for extractives to affect vibrational properties. It is also suggested that cellular location of extracts should be monitored on a case-by-case basis in studies about extractives' effects.

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