Effet de traitements thermiques modérés et de revêtement sur les propriétés vibratoires des bois d'Epicéa et de Mûrier

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EFFETS DE TRAITEMENTS THERMIQUES MODERES ET DE REVETEMENTS SUR LES PROPRIETES VIBRATOIRES DES BOIS D'EPICEA ET DE MURIER

Soutenance prévue le 4 mai 2016 devant le jury composé de

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To my Family…

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PART A. INTRODUCTION AND PROCEDURES

A.1 General Introduction

Wood and musical instruments

From the earliest times, wood has played a prominent role in human life. Wood has been one of the most important materials for making different objects. People tried to improve the durability and to conserve the wood by various ways as they found this material susceptible to the effects of changes in environmental conditions. The attempts to increase durability of wood refers to several thousand years ago (Navi and Sandberg, 2012). While there is not enough scientific context about the history of using wood from all the diversity of existing species, the historical and archaeological evidence show a long history of its utilization. Wood with its aesthetic, workability and sonority role in various musical instruments, since many decades or centuries ago has been recognized as a special and sought after material in many cultures (Brémaud, 2012). Beside its interesting properties which makes it able to act as vibratory plates of stringed musical instrument, the individual "recipes" and technical trade-offs of each makers raises curiosity to experiment this material – untreated wood and after various applied treatments

When studying musical instruments, the variety of objectives and techniques and the different treatment recipes raises different questions in the mind. When reviewing the literature and past attempt in this field, more unresolved questions come to the mind.

Wood has special mechanical and acoustical properties and this had been traditionally used in the field of musical instruments making. Different cultures use different wood species to make traditional musical instruments (Brémaud, 2012). Although the reasons of choice are mostly multi-criteria, the prominent reasons to choose different wood from different species can also be attributed to some "sonority" features of the selected woods.

Objectives

The first objective of this study is to scrutiny the influence of different treatments on the properties of wood which is used in musical instruments. The types of treatments in current study have consisted of two main categories:

-Thermal treatments, which is to expose wood to different ambient conditions of temperature and relative humidity under controllable conditions.

-Surface treatments, which consist of different varnishes and coating systems used by instrument makers.

For our purposes, two different wood species were studied: Spruce and Morus (White Mulberry), as these two species are used for making European (violin familly) and Iranian (lutes as Tar and Setar) stringed musical instruments.

- **Objective Number 1: investigating the effects of mild thermal treatments as a pre-treatment or accelerated aging on wood for musical instruments.**

Thermal treatments were chosen to apply for both species with two different methods. Some instrument makers sometimes use thermal pre-treatments, and there is currently some interest from research and development studies. From another point of view, thermal treatments might be considered as a kind of accelerated aging. From past research, accelerated aged wood can be, at least partially, similar to naturally aged wood.

- **Objective Number 2: studying the properties of surface treated wood used for musical instruments. As well as, the effect of time of "drying" on the properties after several months following application.**

Different coating systems (Iranian and European), have been chosen to apply for two different species. So, the effect of varnishing can be deduced, following the different steps of the application process, and the immediate effect can be compared to long-term changes in properties.

- **Objective Number 3: comparing two different "wood+coating" systems: European and Iranian.**

By applying different types (solvent-based or siccative oil-based) and different recipes of varnishes (Iranian and European), according to the professional instrument makers, it would be possible to compare them together and investigate the influence of such coating systems.

By our main objective, we can study why instrument makers choose special coating systems and how will be the changes during time.

A.2 General Bibliography

A.2.1 Wood structure

A.2.1.1 Macroscopic structure

Wood is one of the few bio-polymeric renewable materials (Navi et Sandberg, 2012). Wood is also a store for carbon and helps to minimize carbon dioxide in the atmosphere (Navi et Sandberg, 2012). A cross section of a tree is shown in Fig. A.1 and gives a description of wood at a macroscopic scale from outside to centre:

- the dead part of bark (A) and the living part which is thinner (B), bark has the role of protection and conduction (by living part);
- the cambium (C) which by dividing its cells is responsible for the growth of stems;
- sapwood (D) and heartwood (E)
- pith, which is the core at the centre of a tree (F)
- rays, which radiate from bark to pith of tree (G).

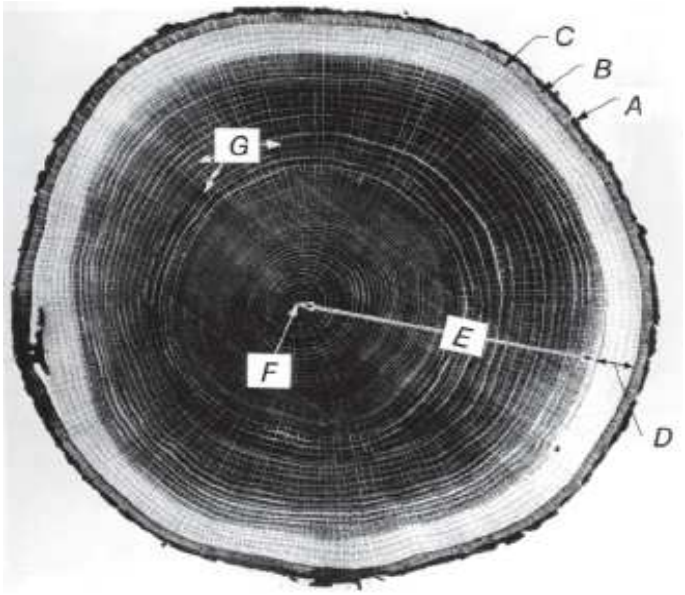


Fig. A.1. Macroscopic view of a transverse section of a Quercus alba trunk (Wiedenhoeft et Miller, 2002)

New wood cells are formed on the inside of the cambium and new bark cells on the outside. Thus, new wood is placed to the outside of old wood and the diameter of the trunk increases (Miller, 1999).

In most species in temperate climates, the difference between woods that is formed early (early wood) in a growing season and that is formed later (late wood) produces annual growth rings.

A.2.1.2 Three principal axes of wood

Wood has three principal directions, which are responsible for its anisotropy and different properties in different directions. A cut perpendicular to the longitudinal direction of stem is

called a transverse or cross section, a cut in the radial plan is called a radial section (or quarter-cut) and a longitudinal cut tangent to the annual rings is called a tangential section (or slab-cut). The Fig. A.2 shows three principal axes of wood with respect to grain direction and growth rings.

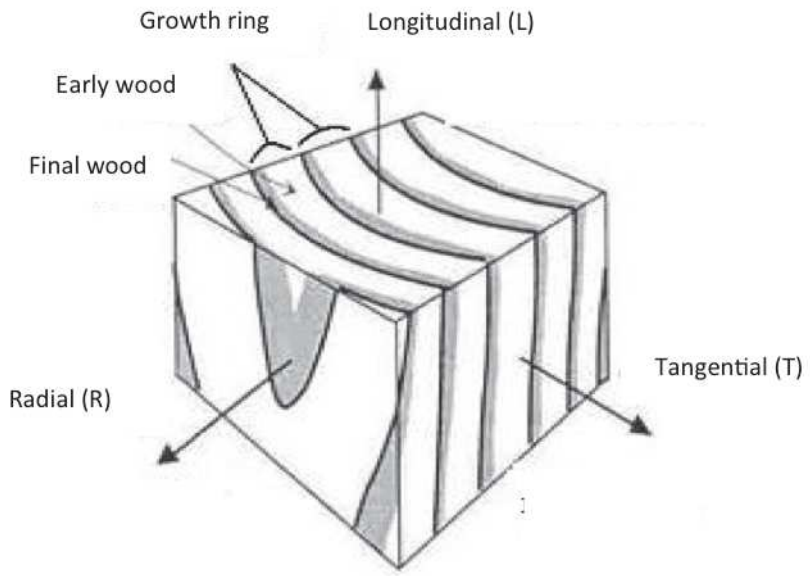


Fig. A.2. Three principal axes of wood

A.2.1.3 Microscopic structure

The primary structural part of wood is the wood cell. In a mature (dead) wood cell in which there is no protoplast, the open portion of the cell is lumen. Each individual wood cell wall is actually a multi-layered structure (Winandy, 1994) and has four distinct cell wall layers (Primary, and secondary: S1, S2, and S3) (Fig. A.3).

Each layer is composed of a combination of three chemical polymers: cellulose, hemicellulose, and lignin. The cell wall has 2 principal layers. The primary cell wall, which is formed during or following the cell division and later modification, the secondary layer, which is formed when cell enlargement is completed (Butterfield and Meylan, 1980).

Between 40% and 50% of cell wall is cellulose, which is organised in microfibrils (MF). The S1 layer is the thinnest layer, being only 0.1 to 0.35 μm thick, and representing just 5% to 10% of the total thickness of the cell wall. The MF angle with regard to the cell axis is 60° to 80°, in S1 layer. This layer is considered as an intermediate between the primary cell wall and the S2 and S3 layers. The thickness of the S2 layer varies between 1 and 10 μm, and is considered for 75% to 85% of the total thickness of the cell wall (Plomion et al. 2001). The inner layer of the secondary cell wall is the S3 layer it is relatively thin, which include only 0.5 to 1.10 μm thick. The microfibril angle (MFA) is 60° to 90° with regard to the cell axis (Plomion et al. 2001). As it is shown in Fig. A.3, each layer has different microfibril angles.

The angle of the cellulose MFs in the S2 layer influences the physical and mechanical properties of the cell and stem wood, because this layer forms the 75% to 85% of the total thickness of cell wall (Plomion et al. 2001). MFA had been recognized as the primary factor affecting axial vibrational properties (Norimoto et al. 1986, Obataya et al. 2000, Brémaud et al. 2010b, 2013).

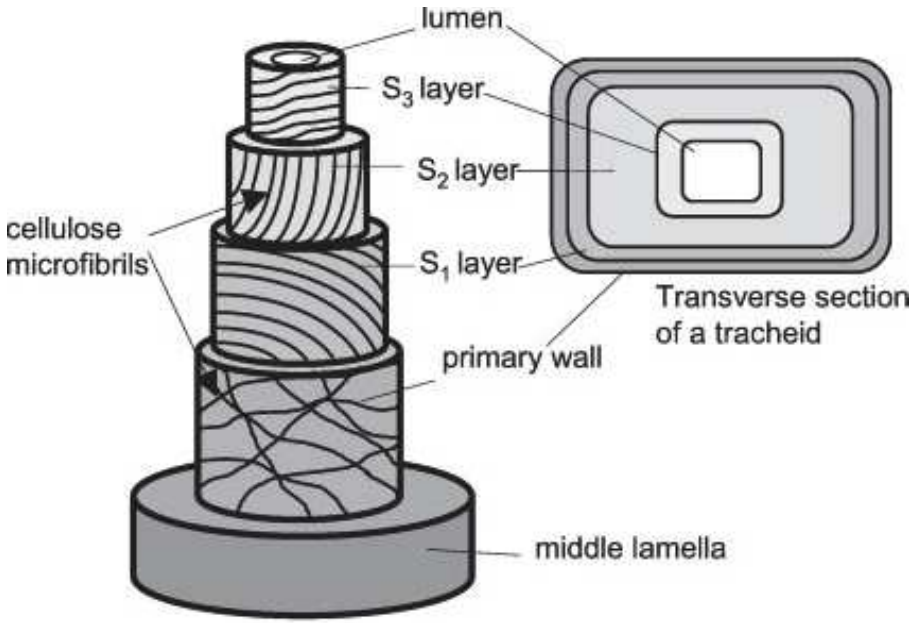


Fig. A.3. Wood cell structure, the layers of the cell wall (Plomion et al. 2001).

The cellulose and hemicellulose are linear polysaccharides (i.e., hydrophilic multiple-sugars), while the lignin is an amorphous phenolic (i.e., a three dimensional hydrophobic adhesive) (Winandy, 1994). The length of wood fibres (cells) varies within a tree and among species. Hardwood fibres average about 1 mm in length; softwood fibres range from 3 to 8 mm in length (Miller, 1999). In addition to fibres, hardwoods have cells of relatively large diameter, which are known as vessels, and do not exist in softwood. These cells form the main conduits in the movement of sap. Both hardwoods and softwoods have cells that are oriented horizontally in the direction from pith toward bark and are called rays. These groups of cells conduct sap and store nutriments across the grain.

A.2.2 Physical properties

A.2.2.1 Wood density

Wood is a hygroscopic material, and its mass and volume change depending on the surrounding relative humidity. Wood density indicates the amount of wood substance present in a unit volume of wood (Zobel and Jett, 2012). It can be affected by some factors like: the cell wall thickness, the cell diameter, the early wood to latewood ratio and the chemical content of the wood (Cave and Walker, 1994).

Density is one of the important properties, which can affect the usage of wood (Brazier and Howell 1979). A significant part of the variation in wood properties between and within species can be attributed to differences in wood density (Schniewind, 1989). Tsehaye et al. (1995) have shown that higher density species tend to have a higher rigidity than lower density species. Density variation between species is due to differences in anatomical structure.

Wood density varies from early wood tissue to latewood tissue within each annual ring. As Haygreen and Bowyer (1996) mentioned, latewood tissue is composed of cells of relatively small radial diameter with a thick wall and a small lumen and therefore, has a higher density than thin walled early wood cells with a larger cell lumen. The specific gravity of wood is defined as the density of wood relative to the density of water (ρ water), which is 1.000 g/cm^3 at 4.4 °; therefore, d is unit less:

$$d = \frac{\frac{m}{V}}{\rho_{water}} \qquad \text{Equation 1}$$

where m and v are the mass and volume of the wood after stabilisation in standard conditions (20°C, 65% RH in the current study), and ρ_{water} is the density of water.

The specific gravity of wood depends on the proportion of cell walls, but the cell-wall density is relatively stable, irrespective of the relative proportions of cellulose, lignin and hemicelluloses. The macroscopic density of wood can be affected by high amounts of extractive components, and by moisture content (MC). Because the moisture content of wood can vary greatly, several specific gravities are defined to facilitate comparisons within and across species, all are based on the oven dry (101 – 105 ° C) mass of the wood.

A.2.2.2 Wood moisture content

Wood is a hygroscopic material. It absorbs moisture and swells in wet or humid conditions and releases moisture and shrinks during dry periods. The amount of water that the wood contains at any time is called moisture content (MC). This value is expressed as a ratio of the weight of water in the wood to the weight of the oven-dry wood. The moisture content of wood can be expressed in two ways:

• The mass of water as a percentage of the total mass of wood plus water. This method is used in the industries. The moisture content on this basis is always less than 100%, and is sometimes described as moisture content on a wet basis.

• The mass of water as percentage of the oven-dry mass of wood. Moisture contents will be greater than 100% if the mass of water exceeds the oven-dry mass of the wood. This occurs in green wood of many lower density species, e.g. the sapwood of pines. This method is sometimes described as moisture content on a dry basis. Many physical and mechanical properties of wood depend upon the moisture content of wood. Moisture content (MC) is usually expressed as a percentage and can be calculated from:

$$MC = \frac{m_{water}}{m_0} \times 100\% \qquad \text{Equation 2}$$

where m_{water} is the mass of water in wood and m_0 is the mass of the oven dry wood.

Water exists in wood as bound water (in the cell wall) or free water (in the cell cavity). Bound water is bonded (secondary or hydrogen bonds) within the wood cell walls, and free water is present in the cell cavities. When wood dries, most free water separates at a faster rate than bound water. Because, there is more accessibility and also the secondary bonding does not exist.

A.2.2.3 Equilibrium moisture content

The moisture content at which the cell walls are still saturated but there is no water in the cell cavities is called the fibre saturation point (FSP) and usually varies around 30% (but sometimes as low as 20%). When wood reaches a balance with the surrounding atmosphere and does not absorb or release moisture, it is said to have reached its equilibrium moisture content (EMC).

$$EMC(\%) = \frac{m-m_0}{m_0} \times 100 \quad \text{Equation 3}$$

where m is the mass of wood at the equilibrium state and m_0 is the oven-dry mass.

A.2.2.4 Shrinkage and swelling

As it has been mentioned, the moisture content of wood changes with the relative humidity of surrounding air. Stamm (1964) stated shrinkage and swelling occur in wood when the moisture content changes. With increasing and decreasing moisture content of wood, swelling and shrinkage take place respectively. They take place below the fibre saturation point (FSP) where all of the water exists only within the cell wall.

Volume change does not take place equal in all directions. The shrinkage is about 5% to 10% in the tangential direction and about 2% to 6% in the radial direction. It is because of the anisotropy of wood, which leads to different properties in different directions. Shrinkage or swelling can be calculated by the following equation:

$$S = \frac{X_1 - X_0}{X_0} \times 100 \quad \text{Equation 4}$$

where S is swelling or shrinkage, X_1 is dimension or volume after swelling or shrinkage and X_0 is dimension or volume before swelling or shrinkage.

A.2.2.5 Weight loss

When applying an hygrothermal treatement, the mass of the specimens might change, reflecting chemical degradation. An indicator is defined to represent this change: weight loss (WL), and is calculated with the following equation:

$$WL(\%) = \frac{m_0^u - m_0^t}{m_0^u} \times 100 \quad \text{Equation 5}$$

where m_0^u and m_0^t are oven-dry mass of untreated and treated wood respectively.

A.2.3 Mechanical properties

A.2.3.1 Elasticity

Elasticity implies that deformations produced by low stress are completely recoverable after loads are removed and it can be described through Hooke's law:

$$\sigma = E\varepsilon \qquad \text{Equation 6}$$

where E is the elastic modulus, σ is the stress and ε is the strain.

A.2.3.2 Anisotropy

Wood may be described as an orthotropic material; that is, it has unique and independent mechanical properties in the directions of three perpendicular axes: longitudinal, radial, and tangential (Bucur, 2006). The longitudinal axis L is parallel to the fibre (grain); the radial axis R is normal to the growth rings (perpendicular to the grain in the radial direction); and the tangential axis T is perpendicular to the grain but tangent to the growth rings (as described in Fig. A.2).

Global mechanical characterization of wood as an elastic anisotropic (orthotropic) material is based on the assumption that its properties can be represented by an equivalent homogeneous anisotropic continuum

In this system of axes, elasticity law is described using nine elastic constants (Bodig and Jayne, 1982):

- three elastic moduli: E_L, E_R and E_T;
- three shear moduli: G_{TL}, G_{LR}, and G_{RT};
- six Poisson ratios (could be reduced to 3): ν_{RT}, ν_{TR}, ν_{TL}, ν_{LT}, ν_{LR}, ν_{RL}.

The constants are related to the compliances in the following matrix (Bucur, 2006):

$$\begin{bmatrix} \epsilon_{xx} \\ \epsilon_{yy} \\ \epsilon_{zz} \\ 2\epsilon_{yz} \\ 2\epsilon_{zx} \\ 2\epsilon_{xy} \end{bmatrix} = \begin{bmatrix} \frac{1}{E_x} & -\frac{\nu_{yx}}{E_y} & -\frac{\nu_{zx}}{E_z} & 0 & 0 & 0 \\ -\frac{\nu_{xy}}{E_x} & \frac{1}{E_y} & -\frac{\nu_{zy}}{E_z} & 0 & 0 & 0 \\ -\frac{\nu_{xz}}{E_x} & -\frac{\nu_{yz}}{E_y} & \frac{1}{E_z} & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{G_{yz}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{G_{zx}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{G_{xy}} \end{bmatrix} \begin{bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{yz} \\ \sigma_{zx} \\ \sigma_{xy} \end{bmatrix}$$ Equation 7

The elastic modulus is much higher in L direction than in R and T: $E_L >> E_R > E_T$ (Guitard and El Amri 1987, Nairn 2007, Katz et al. 2008).

A.2.3.3 Elastic modulus

The speed of sound in a structural material is a function of the modulus of elasticity and density. In wood, the speed of sound also varies with grain direction (Wood Handbook, 2010). So, E can be derived from the following equation:

$$E = V^2\rho$$ Equation 8

where V is the sound velocity and ρ is the density (Kg/m^3). Wegst (2006) has mentioned it is roughly independent of wood species, but varies with grain direction. *V* decreases with an increase in temperature or moisture content. It decreases slightly with increasing frequency and amplitude of vibration (Green et al, 1999). Bucur (2006) stated this equation is idealized for elastic materials shaped as a long slender rod in longitudinal direction.

The frequency (Braune, 1960) is deduced from the relation:

$$f = 2.05\times10^4\times\frac{\sqrt{\frac{d}{E}}}{t}$$ Equation 9

where d is the specific gravity of the wall, t is the thickness of the wall, and E is the Young's modulus of the wall material.

Results from specimens tested by the impedance head method to determine the loss factor, the mechanical impedance, the sound velocity is reported by Craik and Barry (1992).

For a homogeneous and no dispersive medium, the Young's modulus of a bar specimen can be deduced from the following equation:

$$E = 4\rho L^2(f_{L,n}/ n)^2 \qquad \text{Equation 10}$$

where ρ is the density, L is the length of the specimen, and $f_{L,n}$ is the frequency of the longitudinal mode (n =1). For other modes (n =2, 3, etc.) this relation must be corrected with specific coefficients.

As it has generally been considered, suitable wood for making musical instruments is often defined by higher modulus of elasticity (E) per specific gravity (d) and lower internal friction (tanδ) than other wood (Ono et Norimoto, 1983). So, specific Young's modulus is used to describe mechanical property of wood, and is defined as Young's modulus (E) divided by specific gravity (d).

A.2.3.4 Complex modulus

In a viscoelastic system some of the energy stored during loading is recovered by removing the load, and the remainder is wasted in the form of heat. For this kind of material, when applying a sinusoidal vibration at a specified frequency (f), the response would be also sinusoidal in the same frequency but is shifted by an angle of δ (Bardet 2001, Brémaud 2006). The modulus is represented by a complex quantity.

In this term, storage modulus (E'), which is the real part, relates to the elastic behaviour of the material, and describes the stiffness. Loss modulus is the imaginary part (E''), which relates to the material's viscous behaviour. Loss modulus (E'') defines the material ability to dissipate energy. By using Hooke's law for this kind of system, complex modulus (E*) can be defined as:

$$E^* = E' + E''i = \frac{\sigma^*}{\varepsilon^*}$$ Equation 11

Where σ∗ and ε∗ are the complex stress and strain and can be defined as:

$$\sigma^* = \sigma_0 e^{(i\omega t+\delta)} \qquad \varepsilon^* = \varepsilon_0 e^{i\omega t}$$ Equation 12

where ω is the pulsation and δ is the phase angle.

A.2.3.5 Damping

In the theory on free vibrations, when a system is vibrating it would carry on oscillating for ever because the energy put into the system cannot get out of the system. But in reality, the oscillation always fade away with time, because there are some frictions in the system. The graph of Fig. A.4 shows the displacement versus time of a typical damped oscillation.

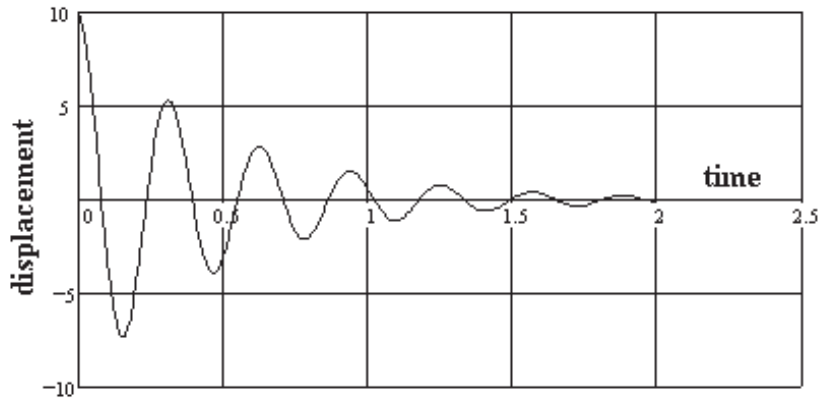


Fig. A.4. Displacement versus time of a typical damped oscillation

Such vibration is called free and damped. Damping is associated with energy dissipation in a material or a system under cyclical loading. Damping is evident in all real systems. Different methods are used to calculate damping in wood.

Several parameters are used to describe the internal friction or the absorption of mechanical energy by solids in the audible frequency range. The most common are:

- The mechanical damping, tan δ, defined as the "logarithmic decrement", that is the logarithm of the ratio of two subsequent amplitudes of free vibrations calculated as 2π tan δ, where δ is the phase angle;
- The quality factor Q, for steady-state forced vibrations, defined by analogy with the theory of electric circuits, as the ratio of the width (Δf) of the resonance curve at half maximum amplitude or at half power level (or at −3 dB) to the resonance frequency f_r.

- Logarithmic decrement

tanδ is used to find the damping ratio of an under damped system in the time domain (Fig. A.5). The logarithmic decrement is the natural log of the amplitudes of any two successive peaks:

$$\delta = \frac{1}{n} log_e \frac{X_1}{X_n} \qquad \text{Equation 13}$$

where δ is the logarithmic decrement and X_1 and X_n are two amplitudes n cycles apart.

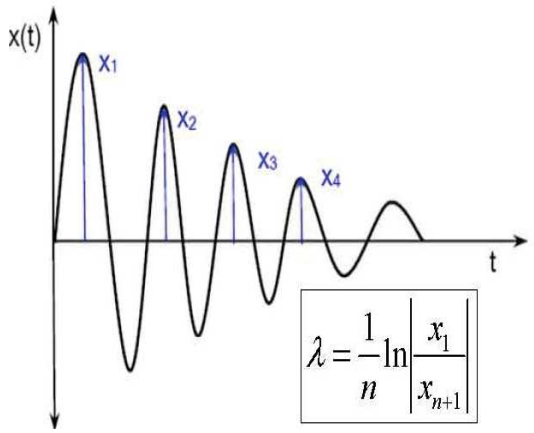


Fig. A.5. Illustration of the loss factor (tanδ) in the time domain

tanδ is dependent on the frequency (Afzal et al. 2003).

- Q factor

The quality factor or Q factor is an indicator of the quality of a resonant system. It is a dimensionless parameter that describes how under-damped an oscillator or resonator is, as well as characterizes a resonator's bandwidth relative to its centre frequency. Higher Q indicates a lower rate of energy loss relative to the stored energy of the resonator. It means that the oscillations die out more slowly. The Q factor is calculated as the resonant frequency (frequency of the peak) f_0 or f_c divided by the bandwidth between the half power point values (Fig.A.6).

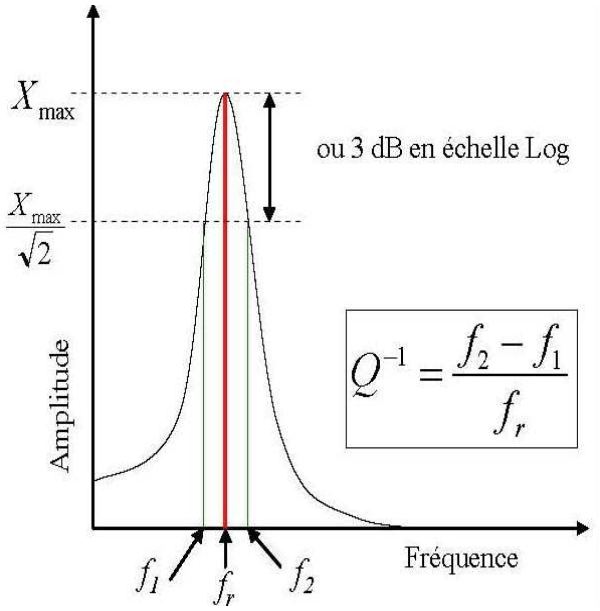


Fig. A.6. Illustration of the loss factor (tanδ) in the frequency domain via bandwidth at the half power (Q or quality factor).

$$Q = \frac{f_c}{f_2 - f_1} = \frac{f_c}{\Delta f} \qquad \text{Equation 14}$$

where ;

Q = Q factor, f_2 = the upper half-power frequency, f_1 = lower half-power frequency, $\Delta f = f_2 - f_1$ and f_c = frequency of the peak.

The Q factor is related to the damping ratio by the following equation:

$$\tan \delta = \frac{1}{Q} \qquad \text{Equation 15}$$

where:

Q = Q factor and tanδ=damping ratio.

A.2.3 Wood and cultural heritage

From the earliest times, wood has played a prominent role in human life. Wood has been one of the most important materials for buildings, weapons and furniture because it is widely available, shows a diversity of interesting properties, and can be relatively easily worked with tools. It became apparent for people that wood is susceptible to the effects of degrading organisms and to weathering. So, the people tried to improve the durability and to conserve the wood by various ways. The attempts to increase the durability were carried out even before working on it by different treatments. The available quality of wood, the condition of use, the culture and the way of life are some affective factors for using from this material. Nowadays, a wooden cultural heritage can be representative of a period of time.

A survived wood product from 5200 years ago is shown in Fig. A.7 which have been preserved in Sweden. It's a ski and probably was bent by some treatment (Navi and Sandberg, 2012).

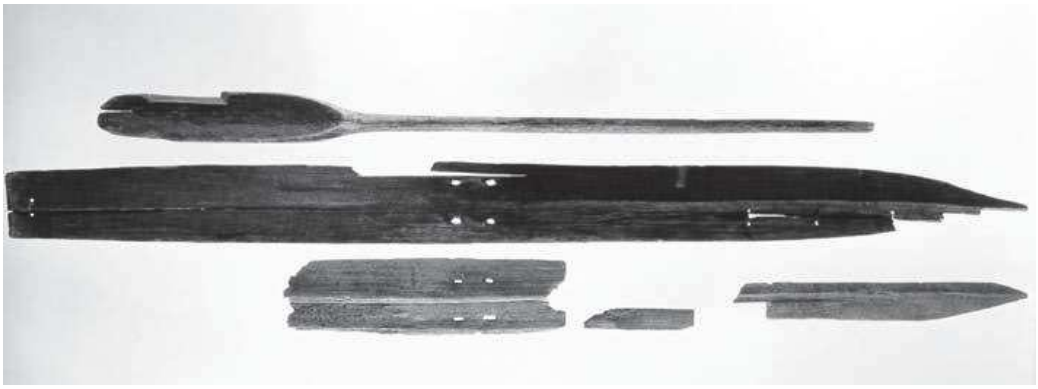


Fig. A.7. 5200-year-old ski made of wood (photo by Sune Jonson, Västerbotten M Museum).

The technic to make this ski is an evidence of using treatments to shape wood from a very long time ago. In some applications wood is dominant as the main material, such as in furniture and decoration. While there is not enough context about the history of using from wood, but the evidence show a long history of its utilisation.

A.2.4 Wooden musical instruments

Acoustical properties of wood and its aesthetic appearance make it the material for musical instruments. Worldwide, several hundreds of different wood species are used for making wind, string, or percussion instruments (Brémaud, 2012).

As wood is an orthotropic material, it has unique and independent mechanical and acoustical properties in the directions of three perpendicular axes, and this is very important concerning the behaviour of plates or shells, as in stringed musical instruments. The acoustic properties of wood between many species are also very different (Brémaud et al, 2012).

In Fig. A.8, the relationships between descriptor factors illustrated for string instruments soundboard, and had been compared to materials used for other vibrating members (strings, violin bows, xylophones) (Wegst, 2006).

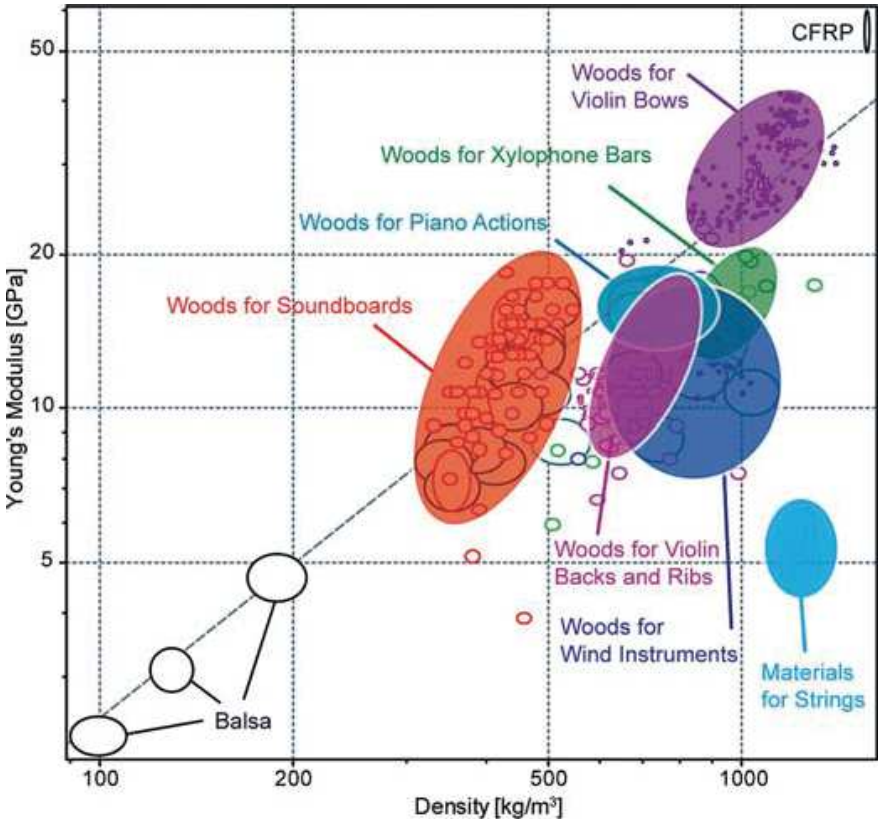


Fig. A.8. A material property chart for woods used for different part of musical instruments, the x-axis is specific gravity (d), y-axis is Young's modulus (E) (Wegst, 2006)

There is a very wide diversity of musical instruments, with different regional distributions (which country/ continent). The different kinds of instruments, but also different parts, usually are associated to different species and type of woods, which is used in their fabrications. The choice of wood can also be different between regions of the world, due to differences in available species, but also to cultural differences (Brémaud, 2012). Musicologists classified ancient and current instruments in organological families (von Hornbostel and Sachs, 1914):

- Idiophones: instruments that produce sounds essentially by vibrating themselves, without using a string (e.g. xylophones);
- Membranophones: instruments that use a stretch membrane for making the sound (e.g. drums);
- Chordophones: instruments that rely on a stretched string (e.g. pianos, violins, guitars). Iranian lutes (Tar and Setar) correspond to this class;

- Aerophones: instruments that use air column for creating sound (e.g. flutes).
- Electrophones: instruments that produce sound by electronic means (e.g. keyboard synthesizers).

The body of a string instruments is usually made by a top plate and back plate which play as soundboard and frame board respectively.

A.2.5 Conclusion

This part included general definitions of wood and some properties of wood, which are related to vibrational properties of wood relevant for making musical instruments. There were two objectives for this part:

- The first objective was basic definitions of wood, which include microstructure and macrostructure of wood. Basic definitions can give a better understanding of its structure and changes in properties.
- According to our purpose to study wood of musical instruments, the second objective was to introduce vibrational properties and different important factors, which can affect vibrational properties of wood and therefore acoustical properties of musical instruments.

According to these objectives, the definitions included properties of wood, which are related to its usage for musical instruments. Presented physical properties (specific gravity, moisture content, swelling/shrinkage and mass change) are also relevant factors in the case of wood of musical instruments.

Mechanical properties, which have been defined in this section, are the most important ones for our purposes. Vibrational properties like damping is an important parameter when studying wood for musical instruments making.

The next session of this chapter consists of the general materials and methods used throughout the different parts of this study.

A.3 Material and methods

A.3.1 Material

A.3.1.1 Studied species and preparation of specimens

For this study, two species, which are major materials for making stringed musical instruments, are used: Spruce (*Picea abies* Karst.) used for European instruments (violin, guitar), and White Mulberry (*Morus alba* L.) used for Iranian instruments (Tar, Setar) (Fig. A.9).

As in Iran Morus is widely used to make musical instruments, Karami et al (2010) investigated the anatomical structure of *Morus alba* and compared it to *Morus nigra* to find if there is a possibility to replace them for making musical instrument. In this study, we will concentrate on *Morus alba*.

Fig. A.9. Iranian musical instruments from left to right: Tar and Setar

Remark: In the document, the English name is used for Spruce, whereas the Latin name "Morus" (without italic style) is used for White Mulberry.

The quality of the specimens preparation is essential when studying vibrational properties (Brémaud 2006, Brémaud et al. 2012). This is even more important in the case of the study of interaction between coatings and wood. The vast majority of all the specimens tested during

this study was prepared and finely machined by Pierre Cabrolier, Post-Doctoral researcher at LMGC, who is gratefully acknowledged for kindly sacrificing so much of his time to help for this study.

- ***Spruce specimens***

For this work, two series of Spruce specimens were used, one for Hygrothermal treatments (HTT), and one for surface treatments (study of violin varnishes).

The first series comes from planks of normal, general supply of spruce, however they were selected by Iris Brémaud in order to be as similar as possible as wood selected according to the criteria for making musical instruments. Therefore the following criteria were visually checked on the planks: narrow and homogeneous growth rings, absence of defects (no knots, no resin pockets, straight grain). Measurement of vibrational properties confirmed that this carefully selected wood had properties similar to specialised supply, however the density is a bit higher. The second series comes from a specialised wood supplier for musical instruments in Jura (France); the planks (from several trees) were selected according to the same criteria, still by Iris Brémaud.

For HTT treatment, 60 samples were cut in two directions: 30 L (longitudinal) specimens ($12\times2\times150$ mm^3, R×T×L) and 30 R (radial) specimens ($120\times2\times12$ mm^3, R×T×L).

For surface treatment, 184 specimens were cut with the same dimensions as previously, to get 92 L specimens and 92 R specimens.

- ***Morus specimens***

Morus specimens were brought from Karaj, a city near Tehran in Iran. All the specimens were from the same tree. A professional musical instrument maker (Mr. Samad Zare) selected the wood and pre-cut the specimens. Then Pierre Cabrolier did the fine machining of the specimens at his personal workshop.

132 specimens were cut in two directions, 66 L (12×2×150 mm^3, R×T×L) and 66 R (120×2×12 mm^3, R×T×L) specimens. They were used for both thermal and surface treatments (Fig. A.10).

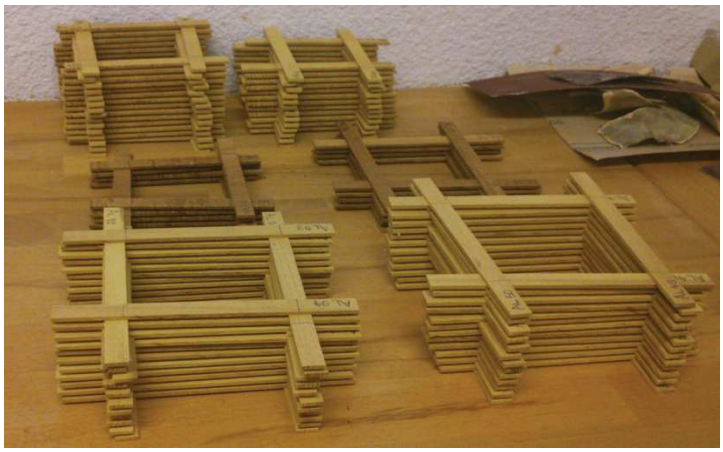


Fig. A.10. Morus samples for TT

A.3.1.2 Specimens conditioning

At first, all specimens are oven dried following a two-steps procedure: 60°C for 2 days, then 103°C for 2-3 hours. Mass and dimensions of anhydrous specimens were measured. Then specimens were stabilised in a climatic room at 20°C and 65% RH, for 3 weeks. This procedure ensure that all properties are measured with a similar hygroscopic history.

After 3 weeks, measurements of mass, dimensions, colour (for thermal treatments) and vibrational properties were done in order to calculate physical and mechanical properties.

A.3.2 Methods

A.3.2.1 Vibrational and physical properties

- ***Vibration test and device***

Vibration tests were conducted on the principle of non-contact forced-released vibrations of free-free beams (Obataya et al. 2000), using the experimental protocol and device and

computer interface described by Brémaud in her thesis (2006) and in Brémaud et al. (2012). This method will be called “Vybris method” in the PhD report.

The apparatus is described in Fig. A.11, a magnetic driver imposes the vibration to the specimen through a thin iron plate glued at the end of the specimen. The laser sensor registers the displacement of the specimen. This signal is analysed with a software developed in LMGC to give dynamic modulus and damping coefficient.

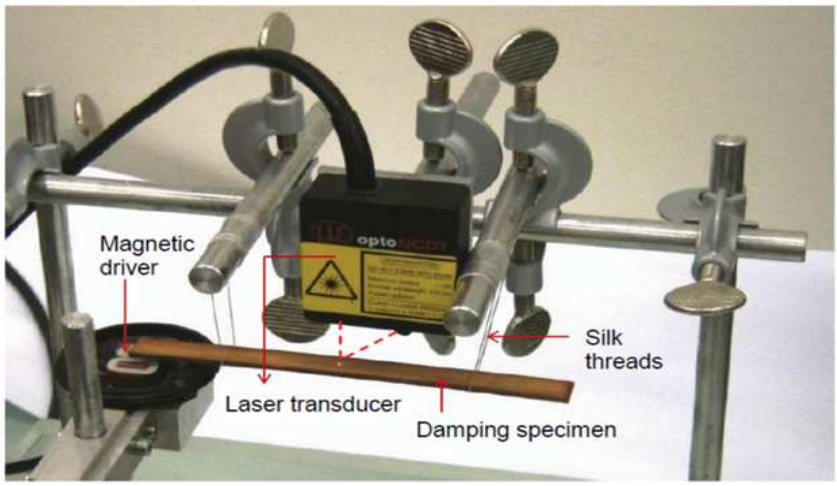


Fig. A.11. Device for measurement of vibrational properties by non-contact forced vibrations of free beams.

Before beginning the test, the position of the vibration nodes of the first bending mode is calculated and marked. Then, an iron plate with 0.05 mm thickness is attached to the end of specimens with super glue. Specimens are hung by two thin threads at the points corresponding to nodal lines of first mode. The laser transducer is placed above, facing the middle of the specimen.

At first, the resonance frequency of specimens is determined by a wide frequency sweep test. This first test can be done by default settings of program, which are 10s frequency sweeps (0.1 Hz resolution) and a scan between 150 and 750 Hz.

From the detected resonance frequency, a second, more precise frequency sweep is conducted. Bandwidth is automatically calculated at half power (-3dB). After this step values of resonance frequency, bandwidth and quality factor Q are registered (as described in Fig. A.6).

Dynamic modulus of elasticity in L or R direction (E_L' or E_R') is calculated from the resonance frequency resonance according to Bernoulli method. According to Bernoulli equation, for a specimen with a rectangular section, defined by h the thickness, the expression of specific modulus for the first mode can be expressed as:

$$\frac{E'_x}{\rho} = 48\pi^2 \frac{L^4 f_1^2}{h^2 P_1} \qquad \text{Equation 16}$$

where :

E_x : E_R or E_L

ρ : specific gravity

L : length of specimen

h : thickness of specimen

f_1 : resonance frequency for the 1st mode of vibration

$P_1 = m_1{}^4$ = and $m_1 = 4{,}730$

The next step is to impose vibrations at the resonance frequency, which has been determined from the last step, to obtain the value of damping factor in L or R direction ($\tan\delta_L$ or $\tan\delta_R$) according to the logarithmic decrement method. After less than 10 seconds, the vibration is stopped manually. If the determined curve is not sufficiently regular, the test can be done again. The results of quality factor (1/Q) and tanδ are theoretically the same: if they differ by a small amount (typically less than 0,0005), the results are considered as reasonable and they can be accepted.

For evaluating experimental error for tanδ, one specimen was tested 10 times and each time it was taken off and on of the supports, all the measuring parameters were kept constant. The error on calculated tanδ (expressed as coefficient of variation = standard deviation divided by the average value) was of 1.3% by bandwidth method (equal for R and L specimens) and of 9.5% and 11.9% (for L and R specimens respectively) by logarithmic decrement method. Therefore, for all the analyses of this study, we used the results from bandwidth method, which are very reliable.

For Spruce specimens the frequency range was 100-400 Hz for R and 350-750 Hz for L specimens. For Morus specimens it was 100-500Hz for both R and L specimens.

For conducting accurate measurements of vibrational properties, all the specimens should be stabilized in climatic room with standard conditions. Vibrational properties are very sensitive about change in humidity (Brémaud and Gril 2015). So, measured specimens should be in a stable condition of humidity and temperature.

- ***Physical properties***

    - Mass and dimensions

Measurements of mass and dimensions are very important because they are related to moisture content variations. Physical and mechanical properties, especially dynamic properties are very sensitive to moisture content, so this data is of primary importance.

    - Mass

The mass was measured by two balances (Fig. A.12), the first balance was used for measuring oven dry weight of specimens (Fig. A.12a). The precision for this balance is 0.001g.

For oven dry measurements, the time between taking specimens from oven and measurement was maximum 4 minutes, which include taking specimens and putting in desiccator to be cold.

Fig. A.12. Balances for (a) oven dried specimens and (b) stabilized specimens measurements

In order to verify that this delay between oven and anhydrous mass measurement doesn't disturb the data, a test was performed. The same measurement was done to see the changes of mass in specimens (one that had been thermally treated and one untreated specimen) after taking them out of the oven until mass measurements. As it can be understand from the Fig. A.13, the mass variation due to adsorbed water from the environment after 10 min is around 0.24% and 0.4% for treated and untreated specimens respectively. According to this figure, for 4 minutes the changes in mass can be negligible as the variation amounts to 0.12% and 0.24% for treated and untreated specimens respectively.

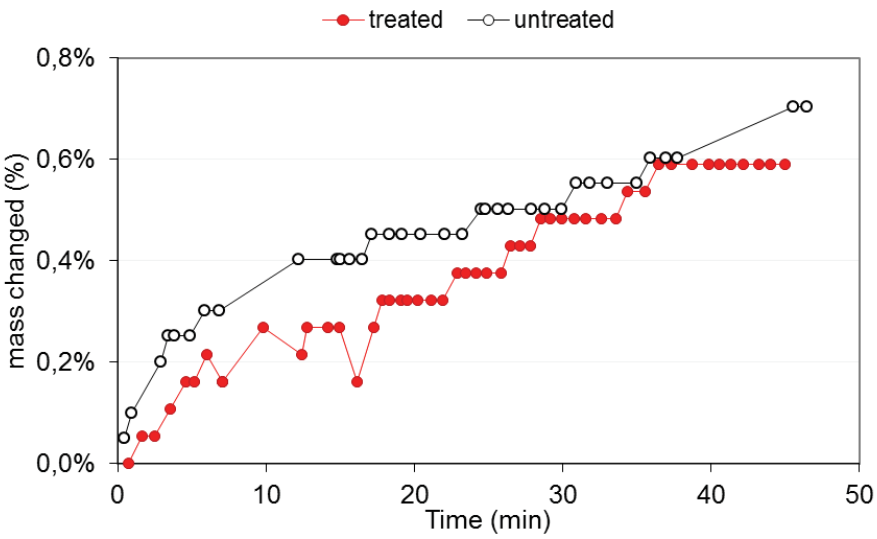


Fig. A.13. The change of mass during around 45 minutes immediately after bring specimens out from oven in ambiant condition

The second balance (Mettler PM100) was used for stabilized specimens with 0.001g precisions (Fig. A.12b). In this case the time between taking specimens and measurements is not important because they were used in climatic room where the specimens are placed.

- Dimensions

As wood is an anisotropic material, changes in dimensions are different in each direction. For measurements of dimensions, Vernier scale was used which is shown in Fig. A.14. The precision of tools is 0.01 mm. For R specimens, it is necessary to make it flat with hand and maintain it so during measurements of length: after oven drying some of the radial specimens may have been temporarily bent so it should be kept flat to obtain the correct value.

Fig. A.14. Vernier for length measurement

- Equilibrium moisture content

Moisture content was calculated based on the oven dried and stabilized masses of the specimens in mentioned conditions.

Equilibrium Moisture Content (EMC) is calculated from equation 3.

- Specific gravity

Specific gravity (d) was calculated on the basis of mass (m) and volume (V) of specimens after stabilization in standard conditions and is calculated by using equation 1.

Measurements of density, mass and dimensions have to be done very carefully as they interfere in the calculation of specific dynamic modulus (E_L'/d and E_R'/d).

- ***Colorimetery***

Colorimetric test is a non-destructive method, which allows outlining the property changes in specimens during treatments. So, it can indicate structural changes or degradation during treatments (Matsuo et al, 2009, 2010, 2011). Two colorimetric devices were used: one during the different steps for HTT of Spruce specimens in Switzerland and the other for thermal and surface treatments specimens in Montpellier

- Apparatus used to monitor HTT in Biel/ Switzerland

For HTT in Switzerland, colour was measured with a spectrophotometer "spectra-guide sphere gloss" from BYK, type 6834 (Fig. A.15), with a diameter of aperture of 10mm. Standard illuminant D65 and standard observer curve of 10° were used. Data were collected in the CIELab system, were L* (lightness) is 0 for black, 100 for white, axis a* ranges from green (-a*) to magenta (+a*) and axis b* ranges from blue (-b*) to yellow (+b*).

Fig. A.15. Colorimetery tool used in Switzerland

Data are also expressed in the more intuitive CIELCh system:

$$C^* = \sqrt{a^{*2} + b^{*2}} \qquad \text{Equation 17}$$

Where, C* is chromaticity (intensity of colour) and:

$$h° = arc\tan\frac{b^*}{a^*} \qquad \text{Equation 18}$$

Where, h° is hue angle.

For treated specimens in Switzerland, the measurements was done three times in three different points for each one and average value of lightness L*, redness a* and yellowness b* calculated after. Intermediate measurements were done at different time intervals during the treatment procedure, to be able to find the kinetics of changes during the treatments.

- Apparatus used to assess thermal and surface treatments in Montpellier/ France

For thermal and surface treatments in Montpellier, the same colorimetric parameters were measured. A spectrophotometer "Datacolor 400TM" was used (Fig. A.16). Standard illuminant D65 and standard observer curve of 10° were used. For each treatment, colorimetric properties were measured on 3 points on each specimens and the value used is the average of these 3 points.

Fig. A.16. Colorimetery tool used in Montpellier

A.3.2.2 Synthetic presentation of all conducted treatments

In table 1, all treatments, which have been done in this study, are shown with detail and conditions. As it has been presented, 6 series of different treatments were studied. As it was mentioned before two different species, Spruce and Morus, were used.

In the present table, as it is shown, one HTT (with 6 groups of specimens for different treatment conditions) was done for Spruce (Series 1). This treatment was done in Switzerland. Also, two different surface coating treatments were applied for two sampling of Spruce specimens (Series 2 and 3):

Series 2 is used for preliminary tests to define the needed time to dry varnish and to select the most appropriate ground layer from two points of view: the amount of varnish uptake on wood, and the acoustical properties. 3 different ground layers followed by 2 different oil-based varnishes (provided by violin maker Nicolas Gilles).

Series 3 is used to evaluate the drying kenetics for 3 different oil-based varnishes (provided by varnish specialist François Perego), for each varnish 3 modalities are applied: 1 thin layer, 1 thick layer, and 2 thin layers. UV (ultra-violet light) treatment was applied between each layer for drying the varnished surface.

Three different treatments were done on Morus specimens, which were brought from Iran.

Series 4 is surface treatment with a common varnish used for musical instrument in Iran: Sandarac (provided by Tar and Setar maker Samad Zare). 3 different modalities were applied; 1 layer, 3 layers and 6 layers (plus control groups for effect of solvent and of ambient conditions). Varnish application conducted in CIRAD laboratory.

Series 5 is another surface treatment, which was performed by a professional instrument maker (Samad Zare) in his own workshop. 3 different varnishes were applied; Sandarac, Polyester and Shellac.

Thermal treatment TT (Series 6) was performed at 150°C with the following protocol: 1.30 hour, 8 hours, 1 day, 3 days and 9 days.

Remark: For Series 2 and 3 (protocols on violin varnishes), the author has participated to the preliminary steps of the protocols, but the subsequent experiments were conducted by Iris Brémaud, Sandrine Bardet, Lydéric Rebecchi and Capucine Carlier.

Table 1. Detail of treatments

Series	Species	Origin of the species	Treatment	Detail of each treatment						Number of specimens
Seies 1	Spruce	France	HTT	Control	130°C + 0% RH	130°C + cyclic RH (0-25%)	150°C + 0% RH	150°C + Constant RH (25%)	150°C + Cyclic RH (0-25%)	30 R, 30 L

Series 2	Spruce	France	Surface treatment	Control	Liquid GL+ varnish silica	Liquid GL + varnish 1	Solid GL + varnish 1	Varnish 1	20 R, 20 L
					Solid GL	Liquid GL + varnish 2	Solid GL + varnish 2	Varnish 2	

Series 3	Spruce	France	Surface treatment	Control	Control UV	GL	GL+ 1 thin VL1	GL+ 1 thick VL1	GL+ 2 VL1	72 R, 72 L
							GL+ 1 thin VL2	GL+ 1 thick VL2	GL+ 2 VL2	
							GL+ 1 thin VL3	GL + 1 thick VL3	GL+ 2 VL3	

Seies 4	Morus	Iran	Surface treatment	Control	Solvent	1 VL	3 VL	6 VL	30 R, 30 L
Seies 5	Morus	Iran	Surface treatment	Polyester		Sandarac		Shellac	18 R, 18 L
Seies 6	Morus	Iran	TT	Control	2.30 H	8 H	1 d	3 d	9 d	36 R, 36 L

Abbreviations: GL for ground layer, VL for varnish layer, d for day and h for hours

A.3.2.3 Sampling based on the initial value

Sampling is a very important and effective step especially about wood because its properties are varying from one position to another. So, depending on the objective of the study, different methods can be used for sampling. In the present study, samplings were done by different methods depending on the conditions of experiment (Table 2)

Table 2. Different sampling methods for different treatments

Treatment	**Spruce**	**Morus**

Thermal treatment	HTT: Based on the position of specimens in each tree, one specimen from each tree in each group (3 trees: A, B and C)	TT: Based on the initial values > By macro
surface treatment	Series 3, varnish drying kinetics: Based on the initial values > By macro	Lab. varnishing (in Montpellier): Based on the initial values > By macro
	Series 2, preliminary test on varnish and ground layerss: Based on initial values, "manual" pairing (groups of 2 specimens only)	Workshop varnishing (in Iran): Based on initial values > By macro Limited number of specimens

According to the present table, 3 different types of sampling were done in this study. In the following, the method which was used for surface and TT and which was based on the initial values is presented.

- ***Initial value***

The initial values from the primary measurements after stabilisation of specimens in standard conditions are shown in the following figures. In Fig. A.17a and b the value of tanδ versus E'/d for R and L direction for all samples are shown. For R direction, the two groups of Spruce specimens are approximately in the same area, except for a series of points, which is separate. To understand this trend, the data were plotted only for Spruce specimens used for HTT and by separating the 3 trees (Fig. A.18). It shows that specimens from trees B and C have different properties than specimens from tree A, this explains the trend in Fig. A.17.

In L direction, Morus specimens have a higher damping coefficient than Spruce and lower specific modulus; specimens of Spruce for HTT and surface treatments are quite homogeneous.

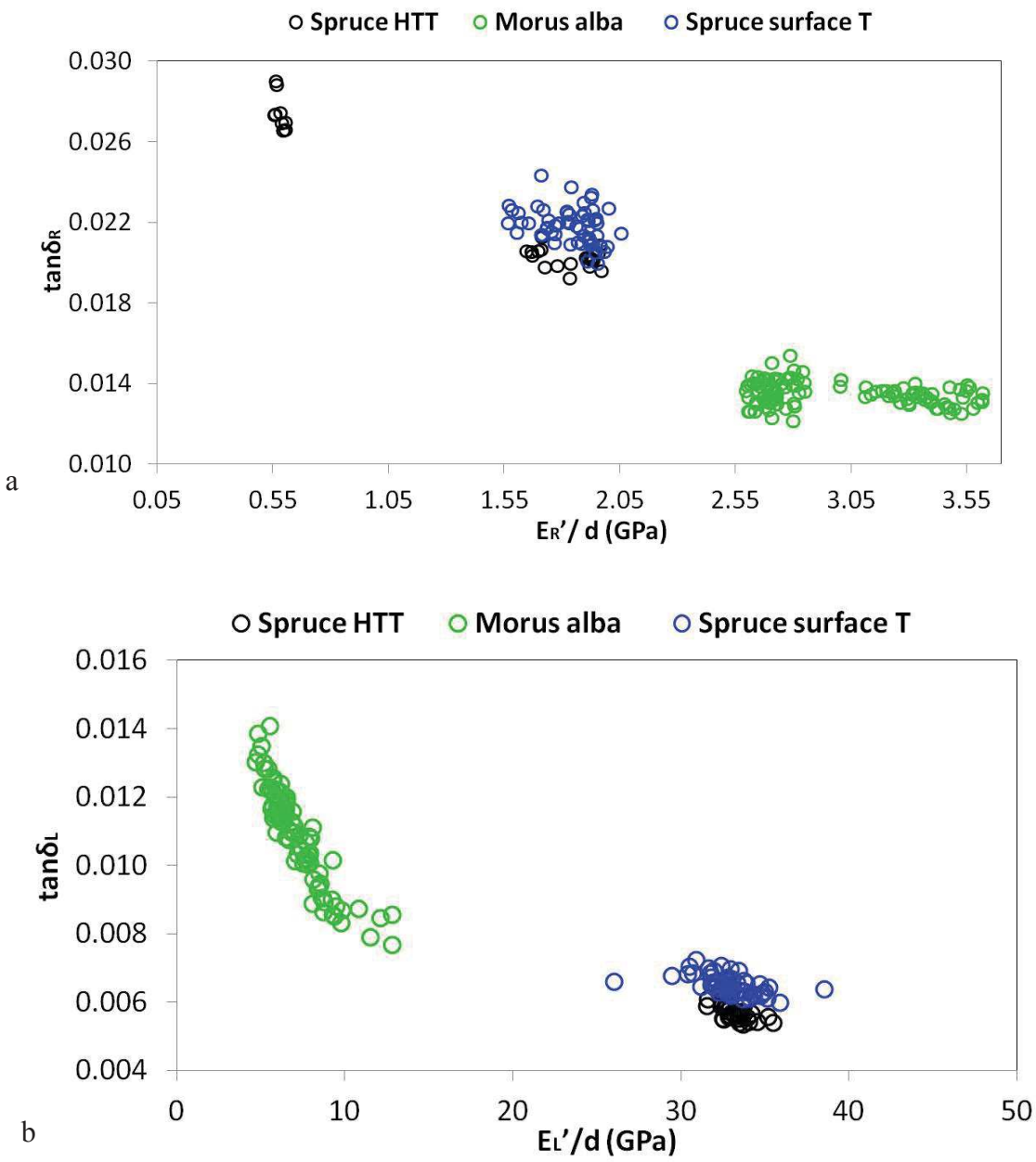


Fig. A.17. tanδ versus E'/ρ for Morus and Spruce specimens in R (a) and L (b) direction

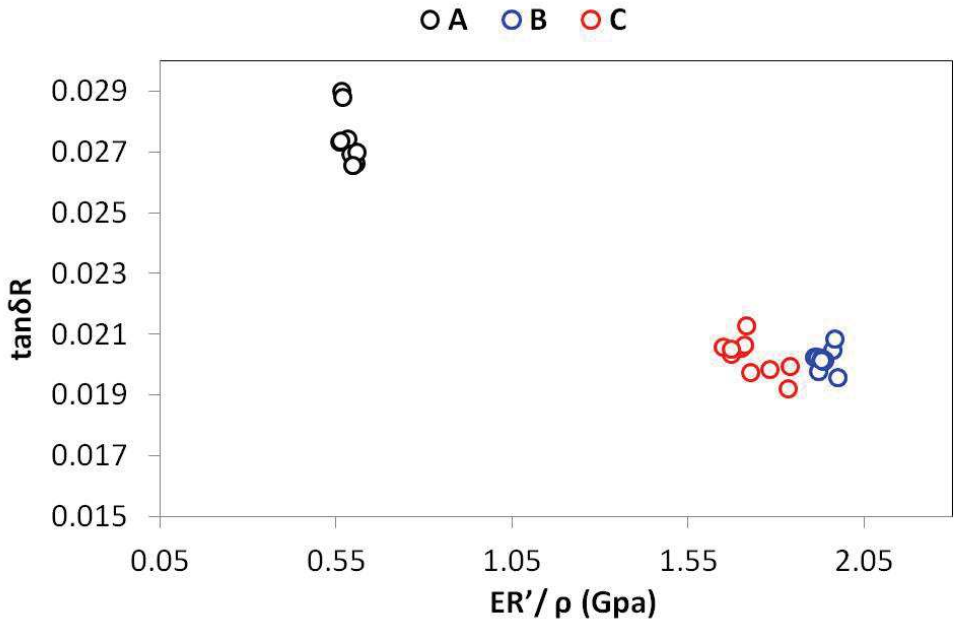


Fig. A.18. tanδ versus E_R'/d of Spruce specimens for HTT treatment in R direction for 3 trees (A, B and C).

In Fig. A.19, the value of tanδ versus E'/d for Morus specimens in R and L direction is shown. Most of the values of specific modulus in L direction are very close to those for R direction. It is a strange result, because as Morus is a ring porous species a big difference is expected between the value for R and L direction. Reduced anisotropy for Morus was already reported (Se Golpayegani et al, 2012), but the anisotropy for the specimens in the current study was interestingly even lower.

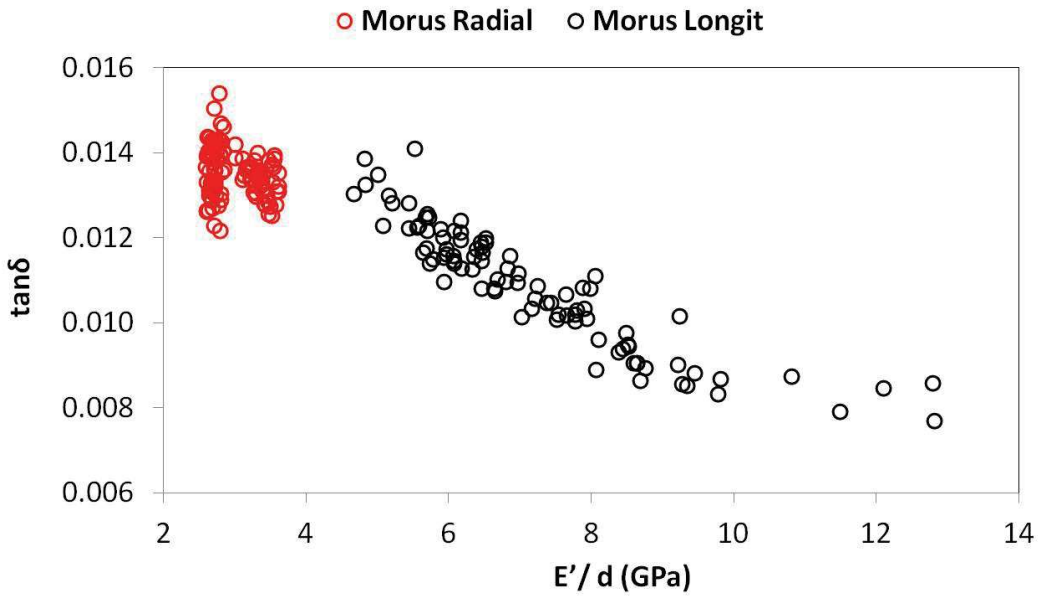


Fig. A.19. tanδ versus E'/d for Morus specimens in R and L direction.

The values of E' versus d for all the specimens have been plotted in Fig. A.20a and b. For the two groups of Spruce, the values are very close and consistent (the average values are 0.67 and 0.86 in R and 15.7 and 15.61 in L for HTT and surface treatment respectively). For Morus specimens the correlation between d and E' is consistent but the value of modulus in both R and L are surprisingly close. It can be suggested that the very low value of modulus in L direction, accompanied by rather high values in R direction, may reflect a very high microfibril angle. This hypothesis would be interesting to verify in the future.

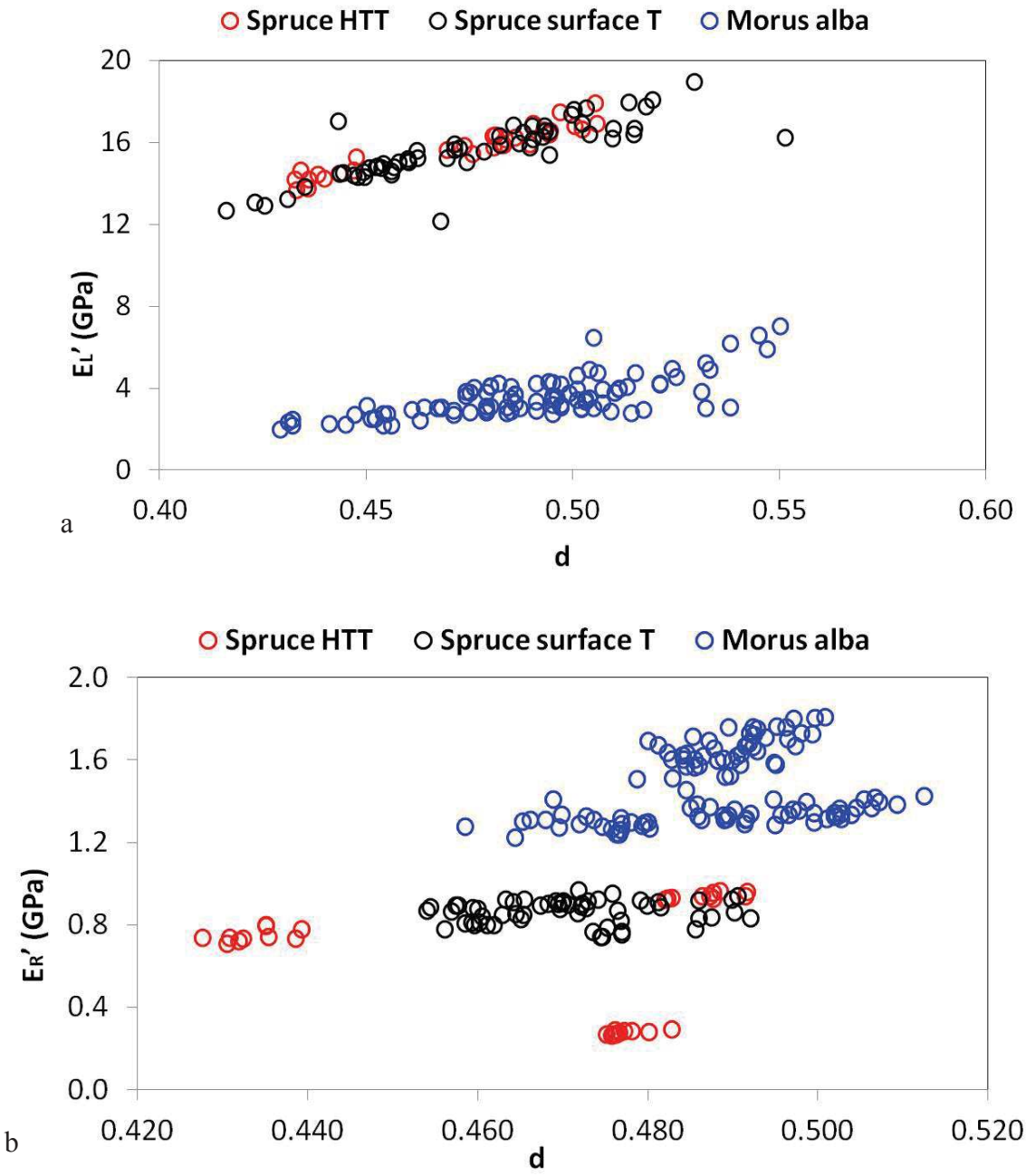


Fig. A.20. E' versus d for Morus and Spruce specimens in L (a) and R (b) direction.

- ***Sampling by Macro in Excel***

As it has been presented in Table 2, for some series of specimens sampling (i.e. distribution into groups to be used for different modalities of each kind of treatment) was done based on initial values. This sampling was done by using a Visual basic Macro in excel, computed by a researcher of the Wood team in LMGC, Tancrède Almeras. After primary measurements,

EMC, d, E'/ d and tanδ were calculated and these initial values were used as descriptors of specimens.

After obtaining these parameters, minimum, maximum, standard deviation and coefficient of variation were calculated. Then, with VB Macro in excel, a scale was defined for each parameter to be able to see the interaction between each parameter and another. So, by the interaction between parameters it will be possible to evidence outlier specimens, which should be removed from the sampling.

After finding outliers and extracting them from the sampling, maximum, minimum, standard deviation and coefficient of variation were re-calculated. Then, all the specimens were re-sorted from the minimum to maximum value of tanδ.

For each group standard deviation and average of all parameters were calculated. Then a coefficient of variation, mass and score were calculated for average value of all the groups.

Then the VB Macro in excel allowed to improve the score. To find the best score, the best match on the graph of tanδ versus E'/d of all the groups should be found. To visualize the importance and influence of sampling by macro, values of EMC against d for Morus (as an example) in L direction were plotted in Fig. A.21 before (a) and after sampling (b).

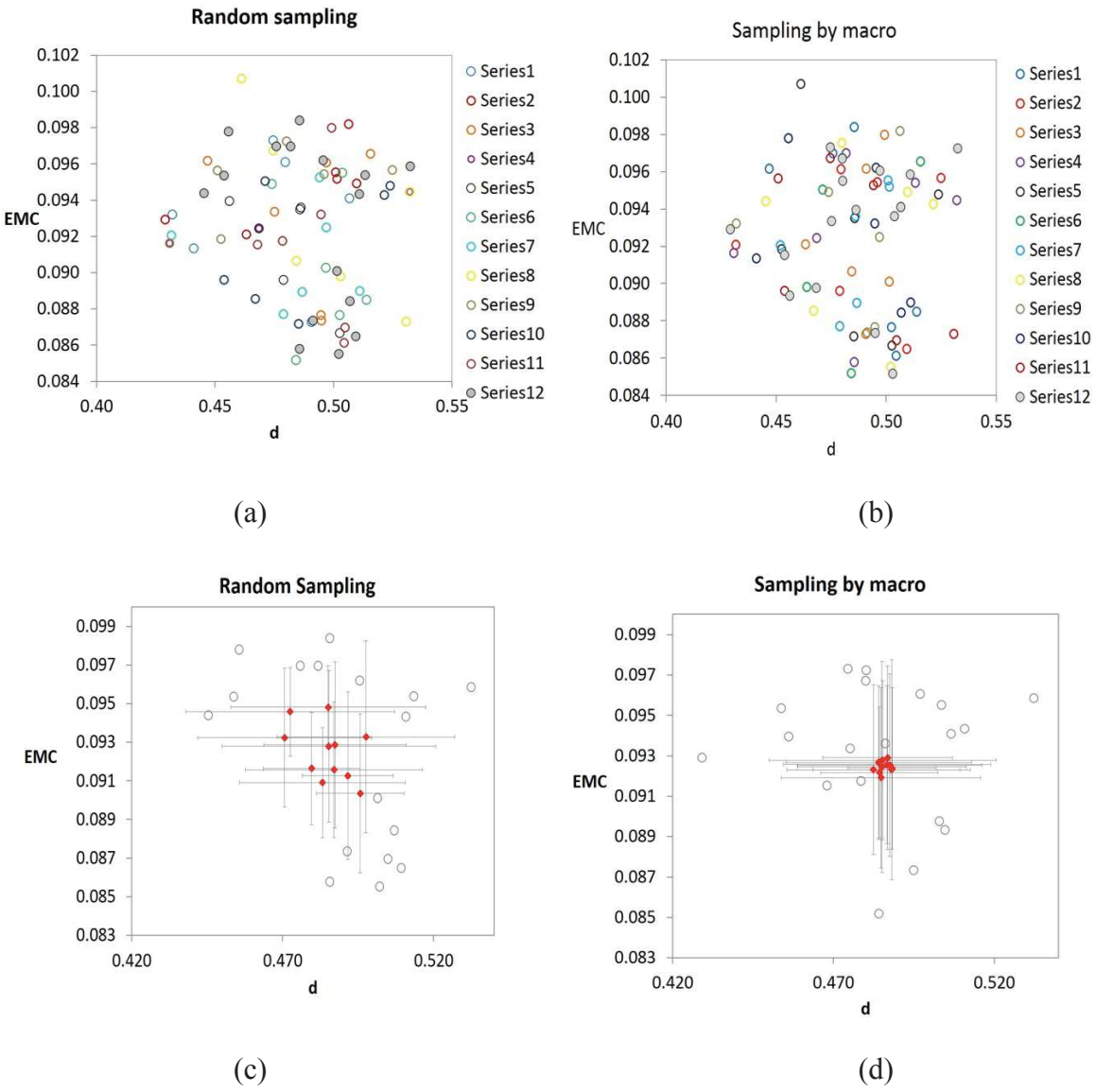


Fig. A.21. Average EMC versus d for Morus tested in L direction before (a and c) and after sampling by macro (b and d). (a) and (b) present one point for one data for each series, (c) and (d) show the average value and standard deviation of the different groups that have been defined.

In Fig. A.21, after sampling, series 12 corresponds to outliers, series 1 to 11 are the final groups.

In Fig.A.21c and d, the average EMC versus average d of all the groups, before sampling (c) and after sampling (d), are shown. As it can be seen, before sampling, the match between groups is clearly weak while after sampling with macro the match between different groups is very good. The best match is achieved by the best score.

As a result, we obtain groups of specimens having the closest average values for all properties and the closest standard deviations.

PART B. THERMAL TREATMENTS

B.1. Bibliography

B.1.1. Definition of HTT and effects on some properties

Hygrothermal treatments (HTT) are processes involving temperature and relative humidity developed to improve and change the properties of wood, especially to increase its dimensional stability. HTT can also be considered as an accelerated or artificial aging.

Effects of mild (below 150°C) and severe treatments (between 150 and 230°C), on different physical properties such as equilibrium moisture content (EMC %), swelling, shrinkage, mass loss and colour properties, and on mechanical properties of some wood species have been studied by various authors.

For a mild treatment at 130°C, resulting decrease in EMC% varies between 5 to 10 % as well as swelling and shrinkage are reduced as a consequence of decrease in EMC (Akyildiz and Ates, 2008). Also, a study of HTT at two temperatures (150°C and 170°C) and various relative humilities from 8 to 100 % on Spruce shows that EMC decreases from 30% whereas the mass loss increases up to a value of 16% (Borrega and Kärenlampi, 2010), the relation between EMC and mass loss is presented in Fig. B.1.

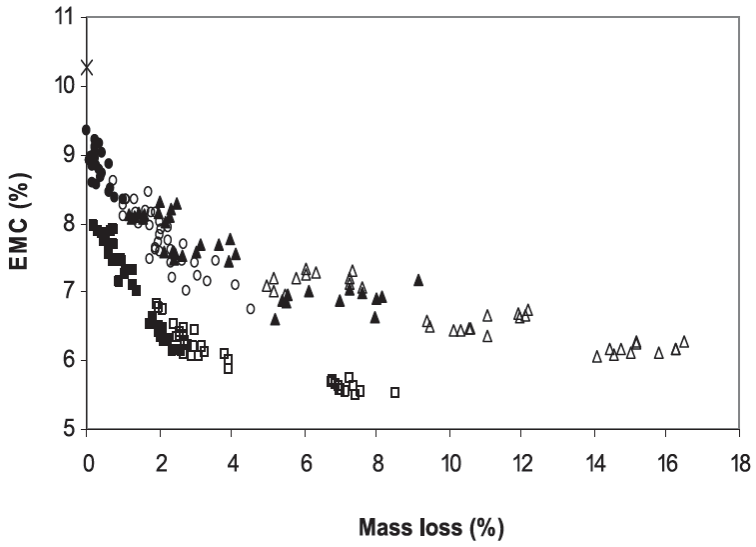


Fig. B.1. Equilibrium moisture content (EMC at 19°C and 65% relative humidity) of heat-treated wood, plotted as a function of the mass loss for each heat-bath treatment. Filled circles, dry conditions at 150°C; open circles, dry conditions at 170°C; filled squares, intermediate relative humidity at 150°C; open squares, intermediate relative humidity at 170°C; filled triangles, water-saturated conditions at 150°C; open triangles, water-saturated conditions at 170°C; marker on the y-axis: mean value for reference wood (from Borrega and Kärenlampi, 2010)

A high thermal treatment (220°C) on *Eucalyptus grandis* was done (Calonego et al. 2012). It was found that, by increasing the temperature, EMC and density decrease by 49.3% and 10.5% respectively and a decrease in the amount of swelling and in modulus of rupture was observed. Another study on *Eucalyptus globulus* Labill, with high temperature treatment (170 and 200°C) indicates that, by increasing the temperature, there is more effect for lower relative humidity. The mass loss, EMC and modulus of elasticity were reduced. For higher relative humidity these properties were even lower. Dimensional stability was improved for both radial and longitudinal directions (Esteves et al, 2007).

According to some authors, the minimum temperature for heat treatment is 100°C (Esteves and Pereira, 2008). But, other authors believe that degradation depends on wood species. For example, a study showed that wood degradation is beginning to happen at 100°C for pine and at 130°C-150°C for oak (Kollmann and Fengel, 1965). A study on softwood and hardwood species from low to high temperature treatment (70°C-200°C) indicates that there is a

decrease in water adsorption after 100°C. By increasing treatment duration there is more decrease in water adsorption (Kollmann and Shneider, 1963). Another results of treatment at 160°C for Spruce reported that equilibrium moisture content of wood decreased from 8.1% to 6.5% and from 18.9% to 15.7% at 30% and 90% relative humidity respectively (Epmeier et al. 2001). Militz (2002) studied the effects on adsorption/desorption curves of heat-treated wood and indicated that there is no significant increase in the adsorption and desorption of treated wood compare to untreated, and there is still an effect of hysteresis.

According to some studies, it can be found that modulus of elasticity increases for mild treatments and decrease for severe ones (Esteves et al, 2007, Esteves and Pereira, 2008). Rusche (1973) showed that heat treatment could lessen the modulus of elasticity for a mass loss between 8 to 10%, in the presence and absence of oxygen, for pine and beech. Vital et al (1983) indicated the similar results for *Eucalyptus saligna* at 105°C-155°C. Mitchell (1988) stated that HTT at 150°C and for different treatment durations and different relative humidities (0%, 12% and green) decrease the MOE with few influence of treatment duration but with a large influence of RH. Indeed, MOE was reduced in a larger scale for green conditions.

Another study by Santos (2000) on *Eucalyptus globulus* showed an increase in modulus of elasticity by heat treatment in 180 °C for three hours. Also, different results for different treatment temperature (180°C-200°C) reported by Esteves et al (2007) concluded that there is a slight increase in the beginning of treatment with a decrease later in the process.

A study on some hardwood and softwood species stated that swelling of treated wood is always lower than untreated wood and it will decrease for sever treatments (Dirol and Guyonnet, 1993).

Mirzaei et al (2012) studied the effects of thermal treatments on the physical and mechanical properties of *Fagus orientalis*. They deduced that shear strength of treated wood was lower than untreated wood whereas density and shrinkage were increased. However the water adsorption was decreased.

Cademartori et al (2012) studied the effects of different temperatures (180, 200, 220 and 240 °C) and different treatment durations on the physical and mechanical properties of *Eucalyptus grandis*. They concluded that modulus of rupture (MOR) and modulus of elasticity (MOE) decreased while weight loss increase by increasing temperature of treatment.

Kol (2010) applied heat treatment for pine and fir at 190°C and 212°C and he deduced that MOR and MOE in bending of thermally modified wood were decreased while shrinkage and swelling were increased by approximately 50%.

Kubojima et al (1998) studied the effects of heat treatment by nitrogen gas or air for Spruce at temperature 120°C-200°C. Young's modulus, shear modulus, and loss tangent were measured by free-free flexural vibration test. Young's modulus and shear modulus increased in the initial stage of treatment and decreased by increasing the temperature. Loss tangent was increased in longitudinal direction whereas for radial direction it increased in the beginning and after 160°C it decreased. Density decreased at higher temperature and longer treatment duration.

Dos Santos (2014) applied heat treatment to improve the properties of low market valued tropical woods. The effect of heat treatment at 160 and 220°C was assessed on colour, weight loss, specific gravity, and equilibrium moisture content (EMC) of cedroarana (*Cedrelinga catenaeformis*) and cedro-marinheiro (*Guarea trichilioides*) woods. They deduced that heat treatment resulted in weight loss, darkening and decrease in EMC while there was no change

in density due to heating. Also, they indicated that heat treatment could improve the properties of low market valued tropical woods.

Rautkari et al (2014) studied the effect of relatively mild hydrothermal treatment (150-180°C) on the mechanical and physical properties of scots pine. They measured static and dynamic MOE, MOR and anti-swelling efficiency. They concluded that lower temperature and less duration of treatment can give better or the same mechanical properties. So, they suggested the temperature can be reduced to 150-180°C rather than 180-220°C which is typically used for thermal treatments. The hypothesis for the decrease in swelling is the decrease of accessible hydroxyl groups.

B.1.2. Chemical origins

The reason for the decrease in water adsorption is a decrease in hydroxyl groups as a chemical change due to treatment (Jämsä and Viitaniemi, 2001). Another reason for dimensional stability can probably be the loss in methyl radicals of some guaiacyl and syringyl units of lignin that results in an increase in phenolic groups. This will increase lignin reactivity and crosslinking. With increasing the crosslinking, the elasticity of molecules is decreased. So there is a decrease in accessibility to hydroxyl groups (Tjeerdsma et al. 1998).

Kubojima et al. (1998) performed a vibrational study on *Picea sitchensis* submitted to heat treatment at 120°C and 160°C. They concluded that the Young's modulus in both radial and longitudinal directions increased for the first two hours and then remained constant. The increase in Young's modulus is followed by an increase in cellulose crystallinity and by a decrease in wood moisture. At the beginning of treatments, the effect on crystallinity is dominant, so there is an increase in Young's modulus. But with increasing the temperature, degradation becomes dominant therefore Young's modulus decreases.

Yildiz (2002) reported a minor increase in density for beech and Spruce (2.25% and 1.73% respectively) for 130°C and 2 hours treatment duration. On another side, a study on Scot pine and Norway Spruce reported by Boonstra et al. (2007) outlined a 10% and 8.5% decrease on density for these two species respectively. He also discussed the mechanical changes after treatment and suggested that the degradation of hemicelluloses and crystallinity of cellulose can be the main factors for loss of the mechanical strength.

Some study on the colour of treated wood indicated that with increasing the time and temperature of treatment the wood become darker (Mitsui et al., 2001, Militz, 2002). Darker tonality of heat-treated wood is attributed to the degradation of hemicellulose or extractives which can change the colour of heat-treated wood (Sehlstedt-Persson, 2003, Mac Donald, 1997). Tjeerdsma et al. (2005) and Mitsui et al. (2001) stated that the formation of oxidation pruducts could be one reason for colour change of heat-treated wood. Patzelt et al (2003) and Bekhta and Niemz (2003) indicated that colour could be a good classification method as it has correlation with weight loss and mechanical properties of wood.

B.1.3. HTT and aging

In the studies of aged wood, hygrothermal treatment (HTT) is considered and applied as a method to imitate artificial aged wood. As studying aged wood is sometimes difficult because of the distractive methods for sampling and experiments, by HTT there is a possibility to study artificial aged wood, which has approximately the same changes in properties with real aged wood. In fact, aging by time and HTT have some common effects on wood properties.

Froidevaux et al (2012) studied the mechanical properties of aged and non-aged wood and showed that there is no significant change in Young's modulus with age. But, there was a decrease in strength for aged wood, which can be caused by a damage of the microstructure of

wood. Another study by Gereke et al (2011) indicated that there is no significant influence of the age of wood on the response to humidity changes. The results demonstrate that even by varnishing on one side of aged wood cold not prevent of its damage in unstable environment conditions.

Esteban et al (2005) studied the effects of cycles of humidity on the dimensional change and moisture content of some hardwood and softwood. They described dimensional and hygroscopic aging as decrease in dimensional changes and moisture changes during several cycles of humidity respectively. They studied the effect of applying several cycles of humidity and called it hygroscopic aging. They indicated that after subjecting to several cycles of humidity the effect of aging is significant.

Noguchi et al (2012) investigated the vibrational properties of new and aged wood (between 100 and 300 years old). They concluded that velocities of sound increased for aged wood and coefficient of damping decreased. These results agree with musicians' hypothesis, which asserts that aged wood has better acoustical properties.

Yokoyama et al (2009) studied 8 historical samples and one modern and did some physical and mechanical tests. They concluded that aged wood is stronger and more rigid than new wood but after moisture content and density correction there was no significant difference between aged and modern wood. Nevertheless, the post linear behaviour was influenced drastically and strength and rupture energy were decreased, especially in R direction.

Matsuo et al. (2011) compared the colour parameters of aged wood from historical building to those of heated wood at temperature ranging from 90°C to 180°C. They found that colour change of aged wood is due to mild oxidation process. In other hand, heat treatment can accelerate the colour change of wood. For a 921 years' old sample at ambient humidity,

colour change was equivalent to heated sample at 180°C and 6.7h, this treatment can be considered as an artificial aging.

Obataya (2007) introduced the experiments that were done by Kohara in 1950. In Kohara's study, the stiffness, strength and the dimensional stability of wood improve slightly by aging, while its rigidity and flexibility degrade significantly. This was attributed to chemical changes in wood structure. Then the effects of heat treatment by steaming or in dry conditions have been described as an aging-like process. The reversibility of physical changes after treatments was described by annealing. Results showed that, for artificial aging, careful moisture conditioning is required. Also, it has been suggested that the reduced anisotropy and improved stiffness of aged wood make it favourable for using in soundboards of musical instruments.

Tomak et al. (2012) applied thermal treatment as an accelerated aging on bamboo and some wood species (scot pine and beech) and compared the physical and mechanical properties. The results showed that the mechanical properties of bamboo decreased drastically by aging whereas for wood species it was less. The dimensional stability of bamboo increased. They concluded the quality of bamboo significantly depend on nodes in culm, which can also affect the aging process.

In 1996, Hunt and Gril studied physical aging of wood. According to their definition, physical aging is time-dependent and corresponds to the approach of a polymer to thermodynamic equilibrium.

B.1.4 Conclusion

As it has been mentioned about the objective of our study, we are going to study thermal treatments on wood, which is used in musical instruments.

- These treatments can be considered as an artificial aging. In this case and as it is resulted from some works (Froidevaux et al, 2012) hydrothermally treated wood looks like an aged wood. In the case of wood for musical instruments, aged wood can give different vibrational properties (Noguchi et al, 2012). By using HTT, some possible effects of aging on wood vibrational properties can be approached by analogy.
- These treatments can be considered as pre-treatments, sometimes used by instrument makers or suggested by applied research to improve some properties of wood. So, it is interesting to study the scientific aspect of this kind of treatment on vibrational properties of wood for musical instruments.

Then, we can understand some influencing parameters of some treatments, see if there is a potential link with interests or practices in instrument making, and how will be the change in acoustical properties of "artificially aged" wood.

B.2. Thermal treatment on Morus

B.2.1. Protocol

As it has been mentioned Morus specimens from Iran were used to study thermal treatments, as this species is one of the main materials for making Iranian musical instruments. They were chosen accordingly to some criteria used in musical instruments. All wood samples were from a single tree, and the same person who chooses the wood pre-cut it in Iran.

Samples were then brought in France, Montpellier and finely machined to final dimensions (by Pierre Cabrolier, Post-Doc at LMGC). Specimens were then oven dried in 60°C for two days and 103°C for 3 hours. After oven drying, mass and dimensions of specimens were measured and they were placed in climatic room in standard conditions (20°C, 65% RH) to be stabilized.

After stabilization of specimens, primary measurements (mass and dimensions) were done. Then the density (*d*), equilibrium moisture content (EMC), shrinkage (S), vibrational properties (1st resonance frequency in bending, specific dynamic modulus *E'/d* and damping coefficient tan δ) and colorimetric parameters CIE*Lab* were measured.

For thermal treatments, 6 groups of specimens were considered (including control groups), as well as 6 repetitions in each group (6L + 6R specimens). Sampling was done by a statistical method presented in chapter A. 2.2.3. It was done based on the initial values of specimens. According to this sampling, specimens in each group have almost the same average and dispersion in terms of initial values of properties.

After sampling, a mild thermal treatment at 150°C and 0% RH was applied for 5 groups with different treatment durations (times distributed on logarithm): 2.5h, 8h, 1, 3 and 9 days (Table 3). This treatment can be considered as a pre-treatment or as basic artificial aging. Treatments

were applied by an oven, which was placed in Cirad, Montpellier. For each step, one group of specimens was taken out out and mass and dimensions were measured. Then the specimens were placed in a climatic room for 3 weeks to be stabilised in standard conditions (65% RH and 20°C). After stabilisation, mass, dimension, vibrational and colorimetric properties were measured (Fig B.2)

Table 3. Treatments with durations

Groups	Treatment duration (Hour)
Group 1	2.5
Group 2	8
Group 3	24
Group 4	72
Group 5	261

Vibrational and colorimetric properties were measured as it has been explained in § A.2. Specific dynamic modulus (*E'/d)* and damping coefficient (tan*δ)* were measured with vibration test. Lightness, intensity of colour and hue angle were measured with colorimetric test.

The kinetics of changes in lightness, specific dynamic modulus (*E'/d)* and damping coefficient (tan*δ)* along the different steps were calculated.

Fig. B.2. Thermally treated Morus specimens

B.2.2. Results and discussion

After thermal treatment, mass, dimensions vibrational and colorimetric properties were measured. Equilibrium moisture content (EMC%), density (d) and weight loss (WL) are calculated from the result of mass and dimension measurements.

Specific modulus of elasticity (E_L'/d and E_R'/d) and damping ($\tan\delta_L$ and $\tan\delta_R$) were characterised in radial and longitudinal directions according to Bernoulli equation (chapter A).

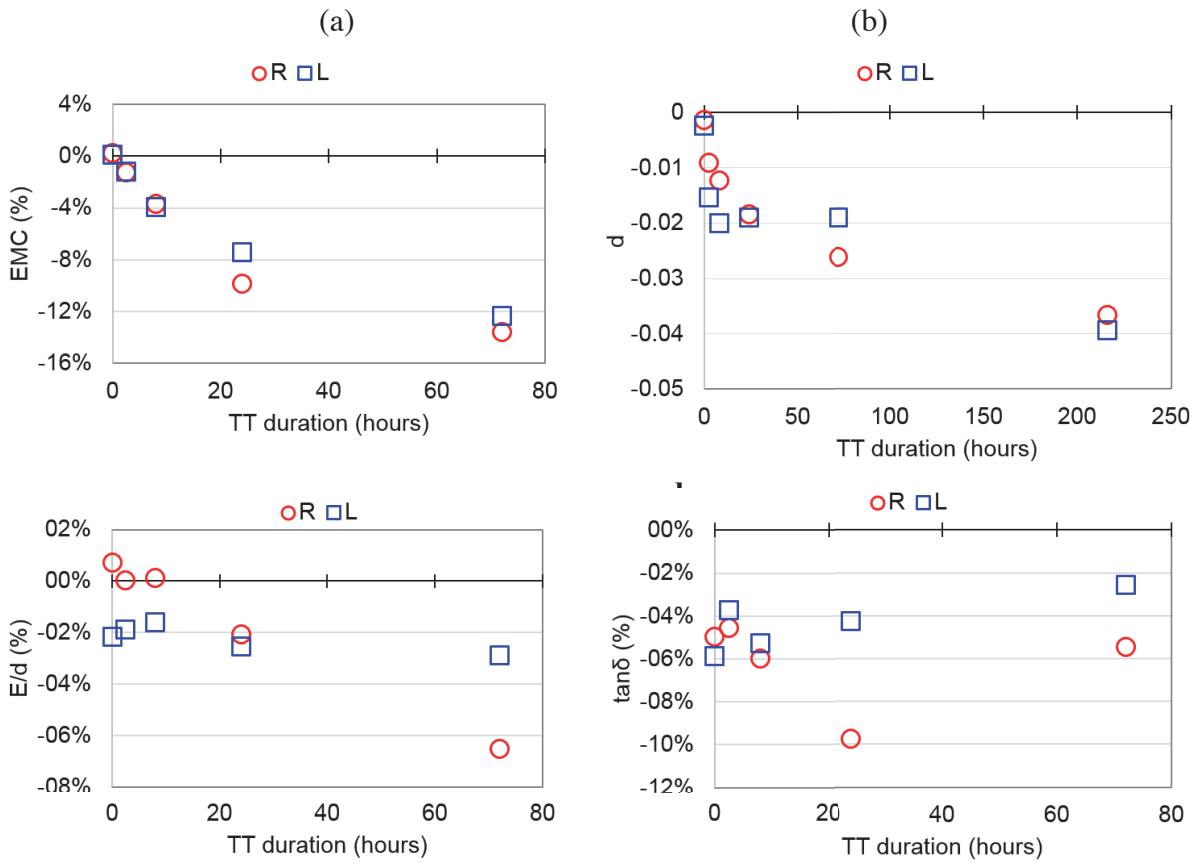
Lightness was measured by colorimetric test, rate of change indicates the intensity of treatment.

The final data that are shown in the graphs are the relative variations in properties according to the following equation:

$$\Delta X_f = (\frac{X_t' - X_u}{X_u}) \times 100 \qquad \text{Equation 19}$$

where ΔX_f is the actual variation due to treatment, X'_t and X_u are the treated value and untreated value for property X, respectively. X'_t is corrected by subtracting the (usually slight) variations measured on the control groups.

Fig. B.3 shows the changes in physical (a, b) and mechanical (c, d) properties after thermal treatments, for R and L directions.



(c) *(d)*

Fig. B.3. The changes in EMC% (a), density (b), E'/d (c) and tanδ (d) during treatment duration.

In Fig. B.3a the variation of EMC (%) after treatment indicates that EMC decreases (similar amplitude for both radial and longitudinal directions, as was expected as EMC is a volume parameter, not directional). It can be expected that the dimensional changes due to changes in moisture content are decreased. Dimensional stability was improved. In Fig. B.3b it can be deduced that increasing treatment duration decreases specific gravity, by as much as 4%, which is the result of changes in mass and lesser so in dimensions.

In Fig. B.3c and d changes in specific modulus of elasticity and damping during treatment are shown. In Fig. B.3c, there is a clear decrease in specific modulus, but moderate (-2%) for L direction, while it is more important and more affected by treatment duration for radial direction. Concerning damping in Fig. B.3d, for both directions, there is first a decrease in

damping and then it is increasing again with duration of treatment. The decrease in the beginning is more obvious for radial direction.

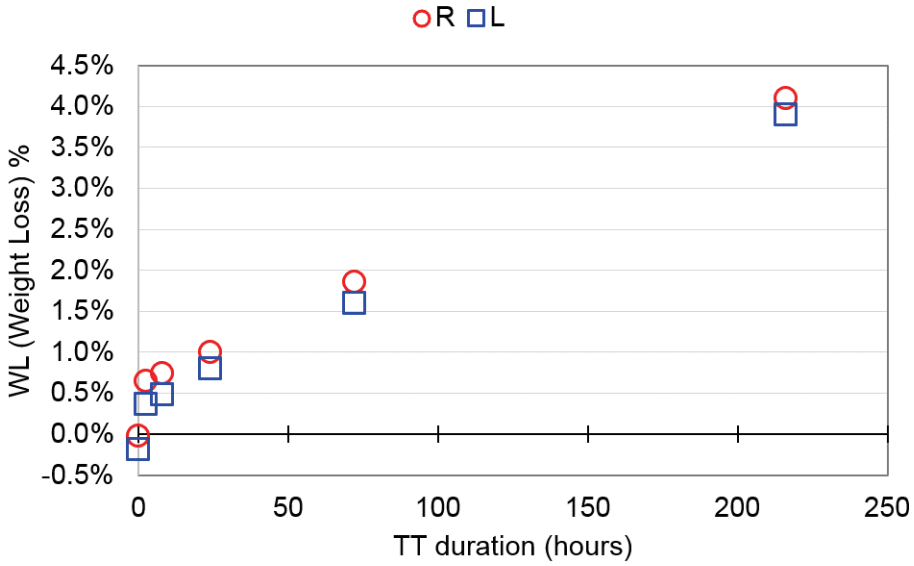
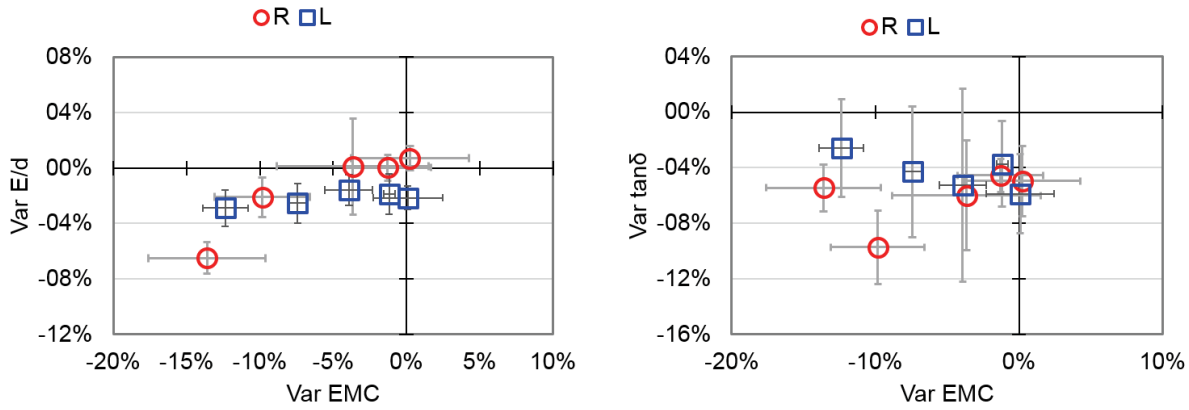


Fig. B.4. Weight loss (WL) during treatment duration

Fig. B.4. shows that weight loss due to treatment is increasing with treatment duration and reaches 4,5%. An increase of weight loss can be the result of some degradation in wood polymers or loss of extractives, which exist in wood composition of Morus and play a role in its vibrational properties (Se Golpayegani et al, 2012).

Because of changes in moisture content of wood it can be deduced that the accessibility to hydroxyl groups has been reduced which can be because of the degradation of hemicelluloses or crystallisation of celluloses. Because of significant weight loss, some moderate degradation is expected.



(a) *(b)*

Fig. B.5. Correlation between variations of E'/d vs EMC (a), and tanδ vs EMC (b).

In Fig. B.5a and b, have been plotted in order to study the existence of a relation between variation of EMC and variation of mechanical properties. Fig. B.5a shows variation of E/d against variation of EMC, and Fig. B.5b shows variation of tan δ against variation of EMC.

In Fig. B.5a, when EMC is decreasing, the specific Young's modulus is decreasing for both radial and longitudinal directions, it enhances a correlation between the variations of these two properties, for both R and L directions.

B.3. Hydrothermal treatment on Spruce

B.3.1. Protocol

Spruce was chosen as it is used for making European musical instruments like violin. The specimens (described in chapter A) have been placed in climatic room in standard conditions (20°C, 65% RH) for 3 weeks to be stabilised. Measurements of mass, dimensions and vibrational properties were done after stabilisation in climatic room. Oven drying was done at 103°C for 5 hours in order to calculate some parameters like EMC%, density and shrinkage. For hydrothermal treatments (HTT), 5 different modalities of treatments were planned, with

also a group of control specimens. As in all this study, the specimens were in both radial and longitudinal directions.

For sampling, there were some specimens from three trees. 6 specimens were needed; 3 R and 3 L. So, 3 specimens were chosen (one from each tree), however in each group a specimen from the same position of each tree were put, so there were 3 specimens in each direction for each group.

After sampling, specimens were exposed to HTT. Two automatic thermo-hydrous reactors (by RINO Sàrl, Blonay, Switzerland) have been used. These HTT were performed together with Miyuki Matsuo from Nagoya University and Sandrine Bardet from LMGC, with the help of Julien Froidevaux and Parviz Navi, at Bern University of Applied Sciences, Department of Architecture, Wood and Civil Engineering, Bienne, Switzerland (Fig. B.6).

Fig. B.6. Reactor for hydrothermal treatment developed by Biel University

The treatments were applied by these protocols: three groups were treated at 150°C with variable or constant RH between 0 and 25%. Another protocol was applied for two other groups at 130°C with varying (between 0-25%) and constant RH (Table 4).

Table 4. Conditions of treatments

Treatments	Relative humidity	Temperature	Duration

A	Constant, 0%	130°C	7 days
B	Varying, 0-25%	130°C	7 days
C	Constant, 0%	150°C	7 days
D	0-25%	150°C	4 days
E	0-25%	150°C	3 days

At 130°C, two different treatments were applied to 2 different groups: at 0% RH (treatment A) and with varying humidity between 0 and 25% RH (treatment B). At 150°C, 3 conditions were applied: constant 0% RH (treatment C), variable humidity by steps or oscillations between 0 and 25% RH (treatments D and E). The whole history of relative humidity was recorded in all cases and can be implemented in modelling.

In Fig. B.7 the evaluation of temperature and relative humidity for each treatment has been shown. For treatments B with 130°C, the cycles were reached very clearly and applied as it was expected. But, as it can be seen for treatments D and E, the cycles, which were expected, could not applied correctly. The cycles were applied with unregularly condition. But as it can be found, treatments D and E can be considered as a cyclic and constant as they are more close to these kinds of treatments.

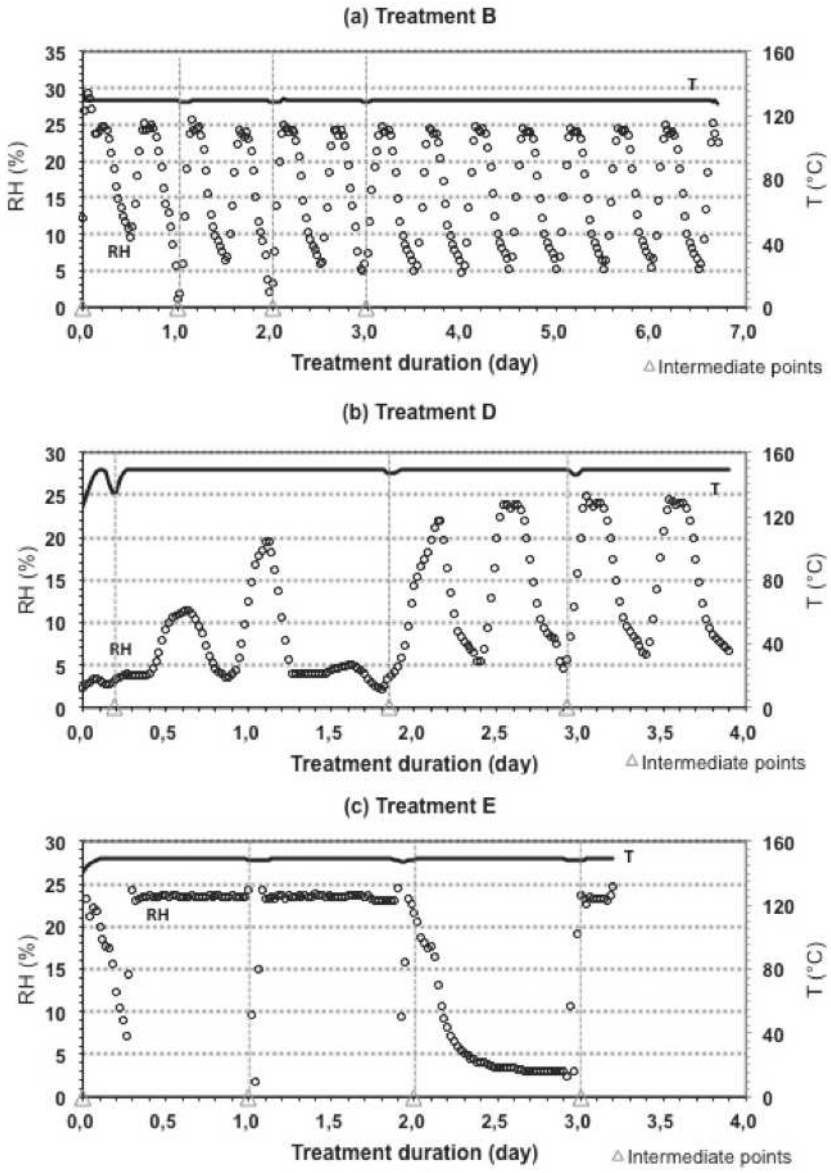


Fig. B.7. Evolution of temperature and relative humidity during tests for treatment: (a) 130°C, treatment B; (b) 150°C, treatment D; (c) 150°C, treatment E. Intermediate points are indicated on the treatment duration axis.

Fig. B.8. is the illustration of experimental procedure: stabilisation, HTT and measurements, step by step. First, treatment stabilisation was done, then, treatment reconditioning was performed to study the recovery of changes resulting from treatments. As it is shown, reconditioning begin after measurements of the HTT treated properties, which are done on specimens stabilised in standard conditions. This step is followed by the "recovery" test, i.e. placing the specimens at a high relative humidity (90%), and then coming back to the same stabilised point in hysteresis as for previous measurements.

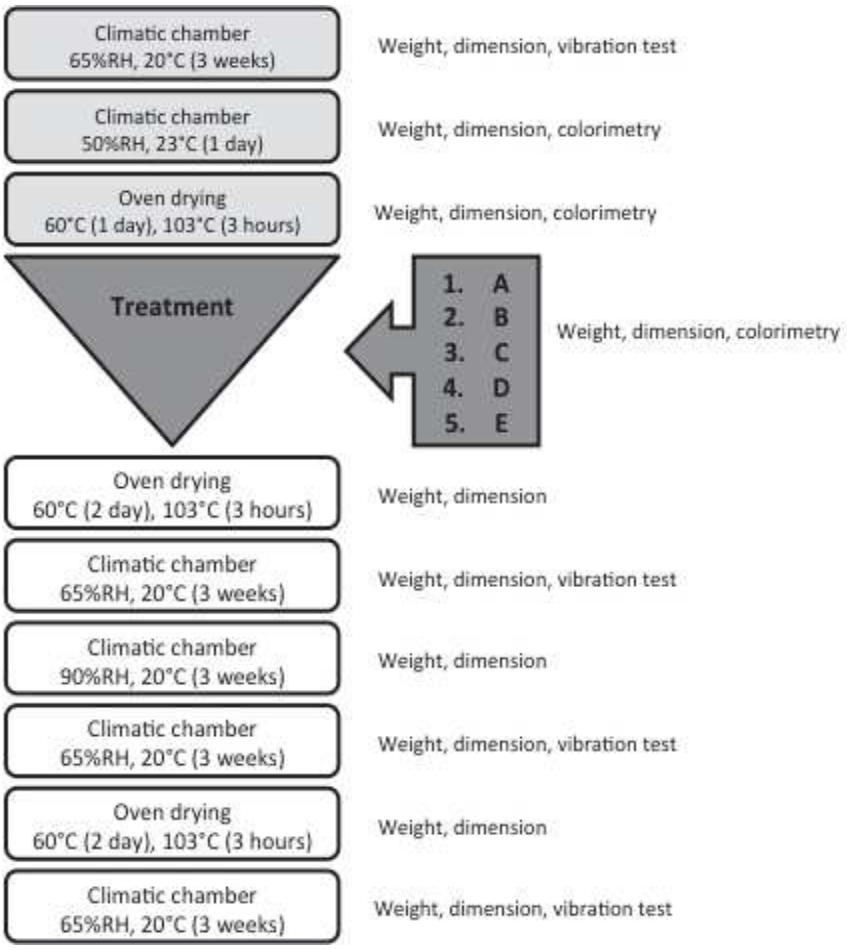


Fig. B.8. Illustration of the process

Fig. B.9. is a photography of treated and untreated specimens. The darkest specimens are for treatments D and E, and the lightest ones are untreated specimens (control). It is clear that, for both radial and longitudinal directions, the colour change is the same and the order of specimens from darker to lighter refers to the stronger condition to the milder condition.

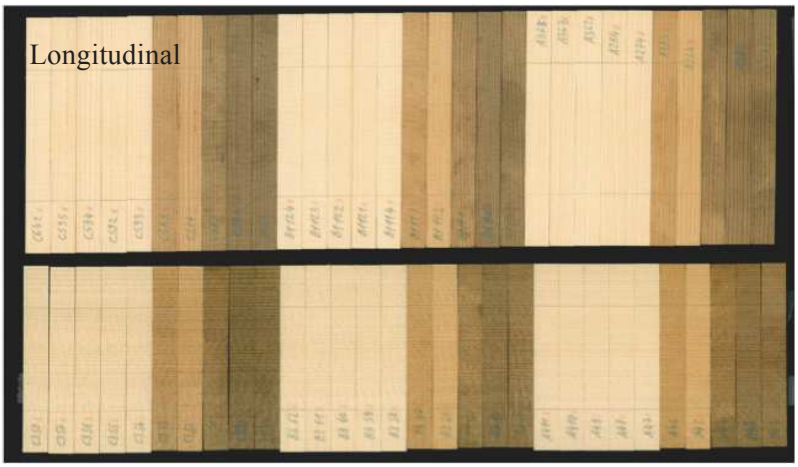


Fig. B.9. The treated and untreated specimens

B.3.2. Results and discussion

Kinetics of modification during treatments

The kinetics of modification was monitored during treatment through oven-dry weight and Colorimetery (Fig. B.10). In the absence of treatment, the colorimetric values measured on air-dry or anhydrous wood are not much different for lightness (-1%), but oven-dry wood appears less saturated (ΔC* = -9%) and more "yellow" (Δh°=+7%). The parameter L* from colorimetric tests is presented in Fig. 8 as a function of treatment duration for the different treatments A to E. The expected decrease with treatment duration is enhanced as well as a clear effect of temperature treatment (after 7 days at 130°C in dried condition, L* is reduced by 20% whereas after 7 days at 150°C in dried condition, L* is reduced by 41%). For the same temperature, the decrease of L* is smaller by treatment at 0% RH than treatment at variable RH. It seems that a variation in humidity induces a larger effect on L*, nevertheless this effect has to be confirmed by further data.

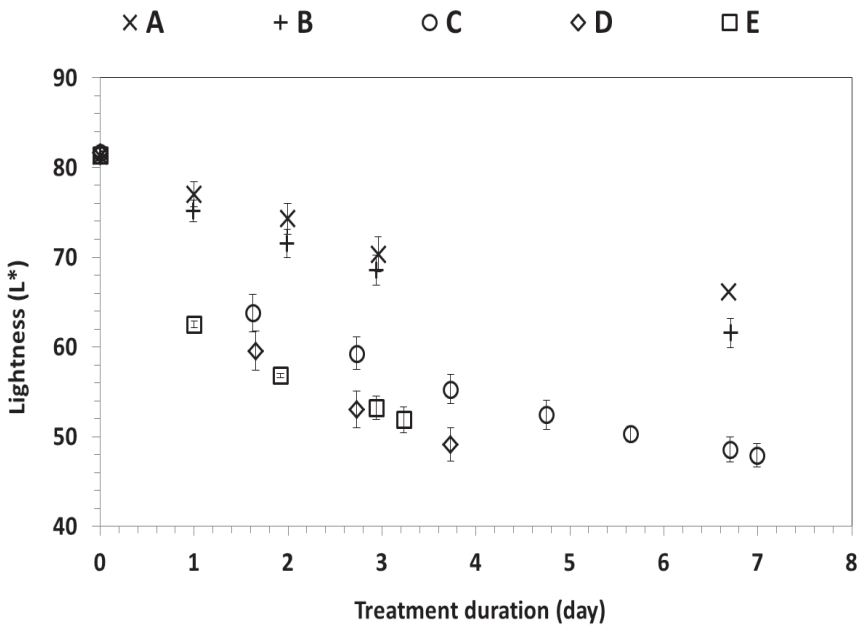


Fig. B.10. Evolution of colour lightness L versus treatment duration of treatment

Two others colour properties, C* and h° were calculated but it appears that they can be deduced from L*, as it is shown if Fig. B.11. Therefore, in the following, only the values of L* will be discussed.

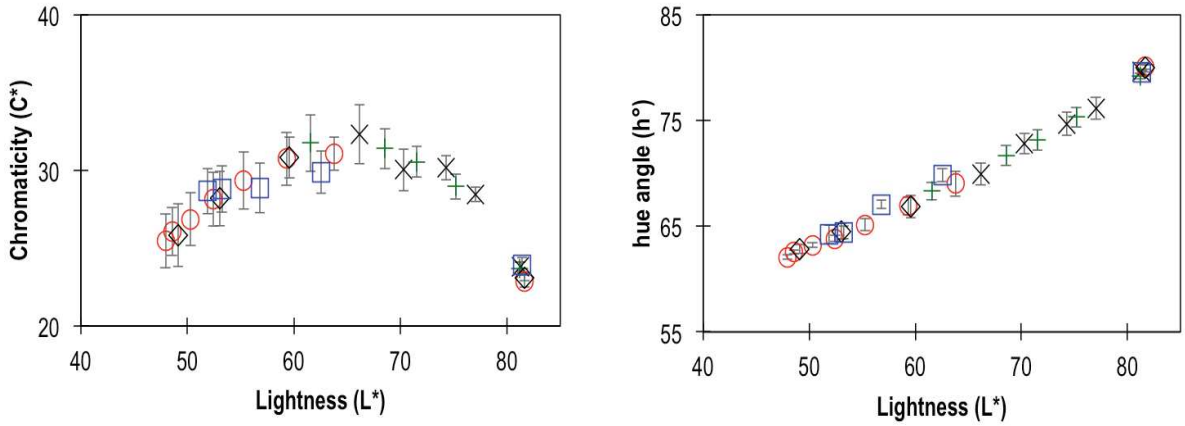


Fig. B.11 Relation between chromaticity (C) and lightness (L*) and between hue angle h° and lightness L*.*

Changes in properties after complete treatment

Average values of physical (specific gravity and equilibrium moisture content), vibrational and colorimetric properties, after complete treatment, are compared in table 5, between control groups and specimens that underwent HTT. Incidentally, for control groups, the effect of drying mainly resulted in a decrease in subsequent EMC (by 11% relative change, that is, from 12.4 to 10.9% absolute values of EMC), with only minor changes in other properties (≤1% for density and module of elasticity, ≤3% for damping). The final values of EMC and d after complete treatment are reduced, compared to control groups, and duration and temperature of HTT influence this effect: a larger decrease is observed for longer duration and higher temperature. HTT is accompanied by a weight loss enhanced by longer duration and higher temperature. Regarding mechanical properties, specific modulus E'_L/d and E'_R/d are increased after HTT whereas $\tan\delta_L$ and $\tan\delta_R$ are reduced. Colorimetric properties are clearly affected as it was shown if Fig. B.11 a and b. L* is reduced as hue angle (the wood becomes more "red"), and c* is increased.

Table 5. Physical, vibrational and colorimetric properties of treated (for treatment A, B, C, D, E) and control specimens

Treatment	D	EMC (%)	E'_L/d (GPa)	E'_R/d (GPa)	$\tan\delta_L$	$\tan\delta_R$	L*	C*	h°	WL (%)
A	0,462	9,0	33,9	1,48	0,0057	0,0179	65,75	32,22	70,24	0,51
B	0,455	8,1	33,7	1,50	0,0053	0,0169	61,57	32,23	68,53	1,35
C	0,449	7,3	31,4	1,45	0,0056	0,0157	48,19	25,80	62,46	4,42
D	0,448	7,2	32,6	1,51	0,0055	0,0153	48,82	26,25	62,85	3,91
E	0,455	7,7	33,7	1,47	0,0054	0,0164	51,99	28,86	64,33	2,52
Control groups	0,465	10,9	32,6	1,42	0,0059	0,0220	81,86	23,91	79,83	- 0,48%

To analyse more precisely these effects, the variations of each property are calculated between treated and untreated specimens, and then these variations are corrected by subtracting the variations for the same property for control specimens, this will be called "relative change".

In Fig. B.12a and b, the relative changes in physical properties are compared between the different treatment modalities. Higher temperature obviously accelerates the processes. Varying humidity conditions also appear to accelerate the process, although, as can be seen in Fig. B.7, the fact that this modality was run for only half the duration of treatment D at 150°C, partly masks the final effects in Fig. B.9a. The decrease in EMC is closely followed by a decrease in radial partial shrinkage (S). When comparing the effects of different modalities of treatment on vibration properties, the same trends can be observed. Properties in radial direction are significantly more affected than those in axial direction, thus leading to a reduction in anisotropy. It was already reported that up to now, most treatments able to reduce

damping involve a reduction in anisotropy (Obataya et al. 2000, Noguchi et al. 2012). There was no obvious effect of treatment in L direction.

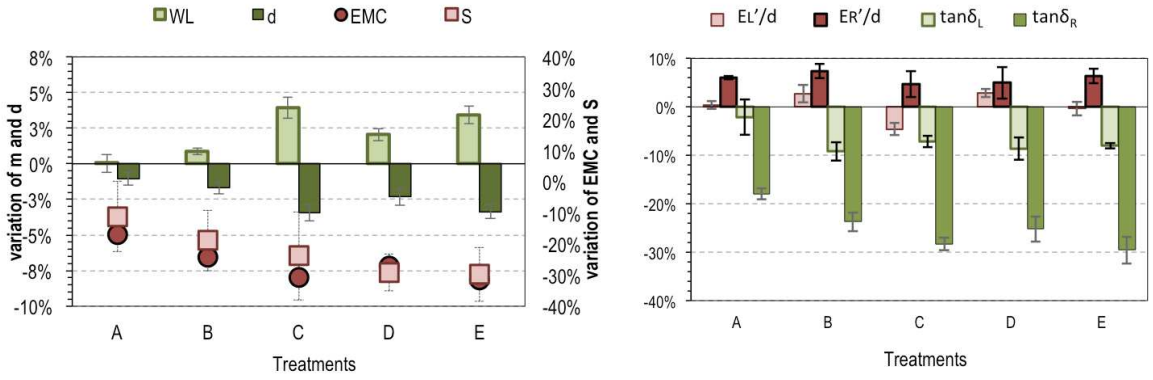


Fig. B.12. Relative changes of properties after completion of the different hygrothermal treatment procedures. (a) Physical properties (WL, d, EMC and S); (b) vibration properties (in axial and radial directions) after completion of the different hygrothermal treatment procedures.

By observing a correlation matrix (not represented here) between relative changes in properties, it can be deduced that WL is a good indicator of changes in EMC and in lightness L* (Fig. B.13), but not so for changes in vibrational properties which are much better correlated with changes in colour (L* and h°, not C* which is a poor indicator in this case). Finally, changes in damping are better correlated with changes in EMC or with L* and h° than with changes in elastic modulus.

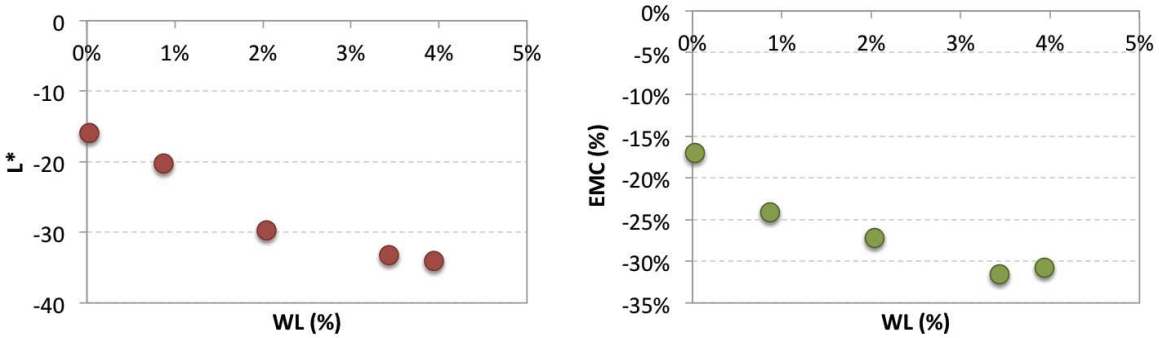


Fig. B.13. Evaluation of changes in colour lightness and in EMC against weight loss at the end of treatments (for treatment A, B, C, D and E)

B.3.2.3. Partial recovery by reconditioning

Fig. B.14 shows the graph of hysteresis from the first point after treatment (1) to the last point after reconditioning (3). Fig B.15a,b, compares the variations in vibrational properties as measured after HTT (point 1 in the hysteresis, Fig. B.14), and after reconditioning at high relative humidity (point 3 in Fig. B.14). For R direction, after reconditioning a clear reduction of the variations in damping and in modulus that were initially measured after HTT, can be seen. It means that some parts of changes due to HTT are reversible. Changes in tanδ have been recovered by up to 7%. The biggest effect has been found for the strongest treatments at 150°C and variable RH (D). For the modulus, a recovery of initially observed variations is obvious for all treatments. In L direction, there was also an observable recovery of the HTT-induced variations of tanδ, except for treatment A. But for modulus, there is no significant effect of reconditioning, as there was no obvious effect of treatment in this direction. Logically, the variations in L direction are smaller than those for R direction, both regarding HTT-induced variations and subsequent recovery effects.

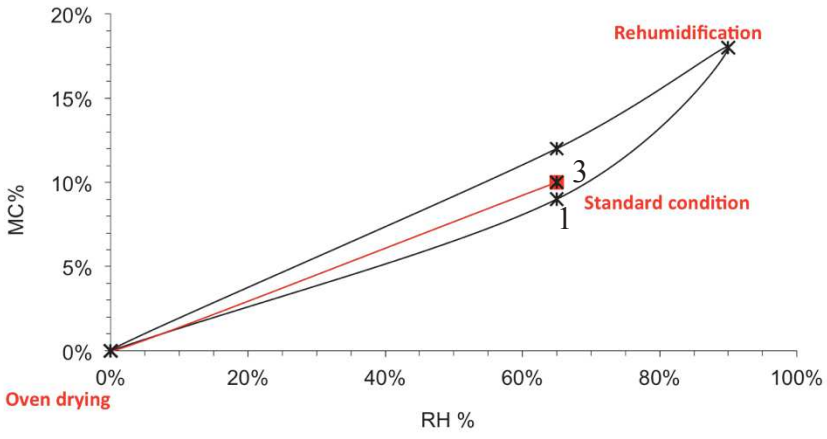


Fig. B.14 visualisation, in the hysteresis loop, of the first point of measurements after HT treatment (1), to the last measurement point after reconditioning (3)

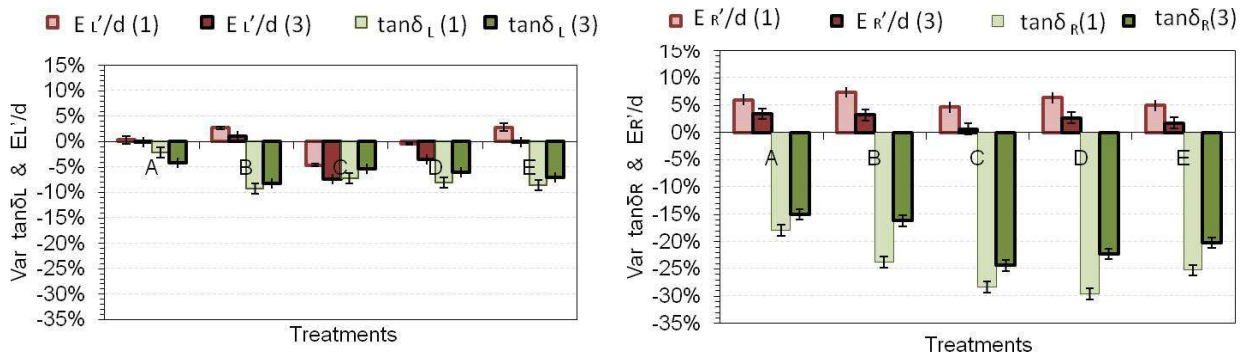
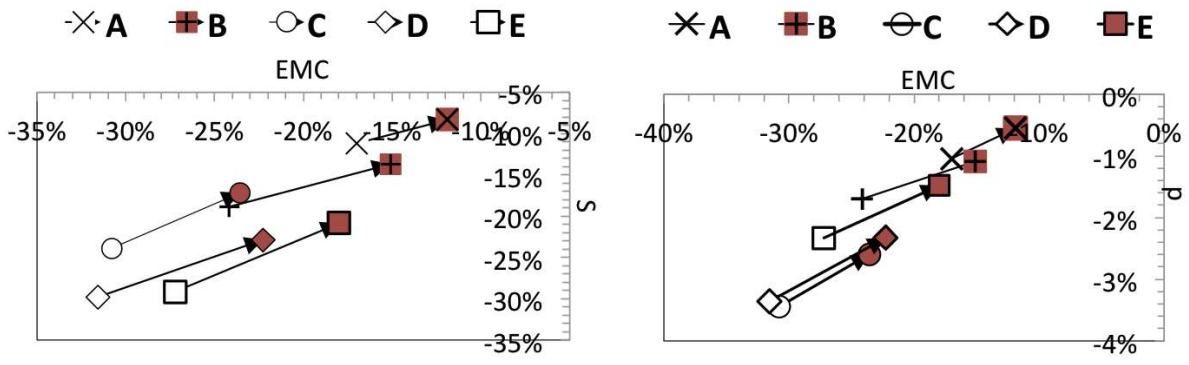


Fig. B.15. Variations of vibration properties measured after HTT (point 1) and then after reconditioning (point 3), for specimens in (a) R direction and (b) L direction.

In Fig. B.16a and b, the variations in partial shrinkage S and in specific gravity d has been shown as a function of variations in EMC, as measured after HTT, then after reconditioning. In Fig.16b, after reconditioning, a clear (but partial) recovery in initially observed changes of EMC is followed by (also partial) recovery of the initially observed diminution of density. It is expected that a decrease in moisture content of wood results in a decrease of specific gravity. In Fig. B.16 a, the same trend can be seen between EMC and partial shrinkage S. With decrease in moisture content, the shrinkage will decrease because the capacity of wood to adsorb and desorb water is decreased. It means that some part of changes after HT treatments is reversible. These changes and subsequent partial recovery can be seen for all the treatments.

For specific modulus also, the effect of HTT is partially recovered or modified by reconditioning. In L direction, for the mildest treatments A and B, initial effects of HTT are completely recovered. For the more severe treatments, L modulus now appears slightly reduced as compared to untreated wood. In R direction, the initially observed increase in E/d becomes smaller (but not null) after reconditioning, which suggest that the initial effect of HTT was in good part due to temporary reduction in EMC.

For damping in R direction, the same recovery trend is very clear, it flows the recovery in EMC in an almost linear way. Whereas in L direction, the recovery effect is small.



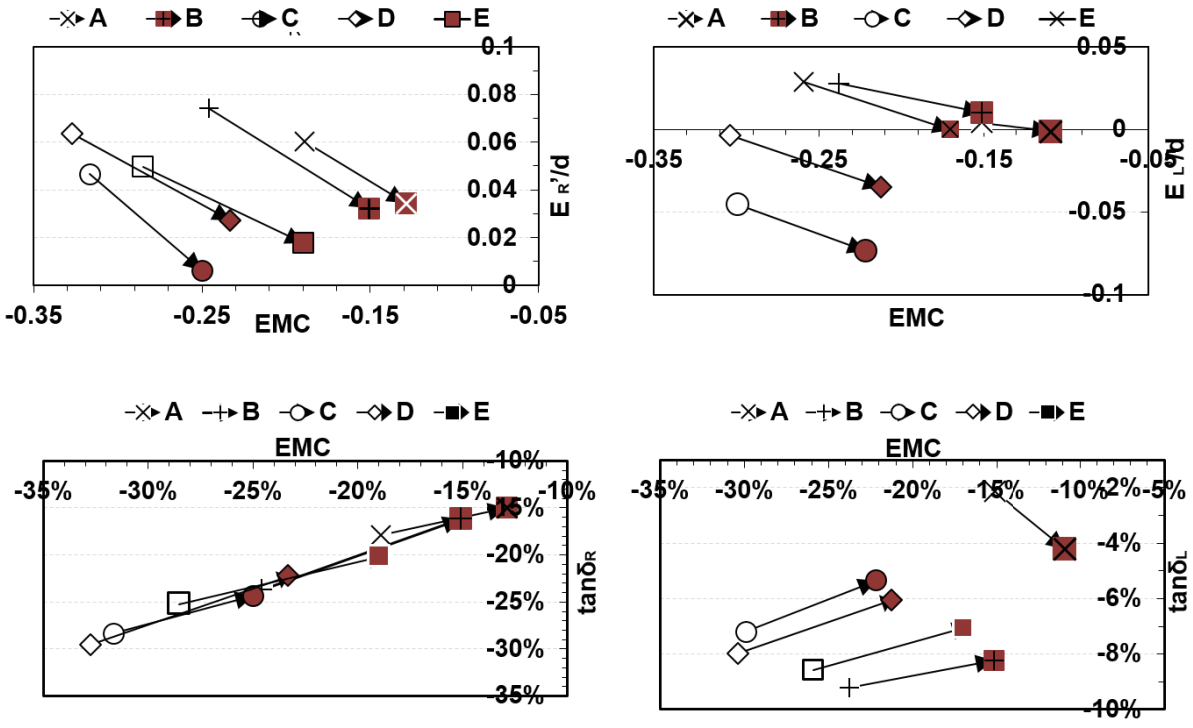


Fig. B.16 Relation between the (relative) variations in EMC after HTT and then after reconditionning, with variations induced by these two steps of treatment on the other studied properties (a) EMC vs S; (b) EMC vs d; (c) EMC vs E'_R/d; (d) EMC vs E'_L/d; (e) EMC vs $tan\delta_R$; (f) EMC vs $tan\delta_L$. Empty symbols correspond to values after treatment; bold symbols correspond to values after reconditioning.

These results show that a part of HTT-induced changes in properties were recovered by reconditioning at high relative humidity. So, by applying more reconditioning (higher humidity and/or longer time and/or cycles?) we could evaluate if all the changes initially induced by HT treatments are reversible or not. Although, of course, the existence of a small but significant weight loss provoked by HTT, as well as the marked change in colorimetry, does indicate some chemical (i.e. irreversible) modifications.

The next section concerns the reconditioning, applied for both Morus and Spruce treated specimens, in the same conditions of high RH (100%) to study reversibility and compare the effects on the 2 species.

B.4. Reconditioning

B.4.1. Protocol

As it has been explained two different treatments were used for two different species, Spruce and Morus. After getting all the results after (hygro-) thermal treatments, two questions have been raised:

- What is the main reason for these changes of properties?

- Are these changes reversible?

For the first question, different chemical and physical factors for these changes should be investigated. The existence of some physical or structural factor may be associated to reversibility in the initially observed changes in properties. To verify the reversibility of changes after treatments, a recovery test needs to be performed. It can be achieved by humidification of specimens at high relative humidity. Such a recovery test has been described in last part, in the case of Spruce specimens.

Here, the recovery was done for thermally treated Morus specimens and a group of treated Spruce (Table 6). As thermal treatment was done at 150°C and 0% of relative humidity for Morus, the equivalent level of treatment was chosen for Spruce to be comparable. So, as it is shown in Table 6, two groups of Spruce specimens treated at 130°C and 150°C and 0% RH were chosen.

Table 6. The groups of specimens tested for reversibility of the effects of mild thermal treatments.

Species	**Temperature of treatment**	**Relative humidity during treatment**	**Treatment duration**	**Re-conditioning**
Morus	150°C	0%	1.30 hours	3 weeks in "saturated" conditions (~100%RH)
			3 hours	
			1 day	
			3 days	
			9 days	
			No treatment – Control	
Spruce	(B) 130°C (C) 150°C	0%	7 days	

Also, 6 Spruce specimens in each direction were selected from control groups (two from each tree). Control groups for both species were kept in climatic room during recovery.

A closed hermetic box was used, which was placed in a cold room. Specimens were placed between water-soaked cotton wool and then placed in this box for 3 weeks. After this step all the specimens were placed in the climatic room for 3 weeks to be stabilized (20°C and 65% RH). After stabilization the physical and mechanical properties were measured, as it has been explained before.

B.4.2. Results and discussion

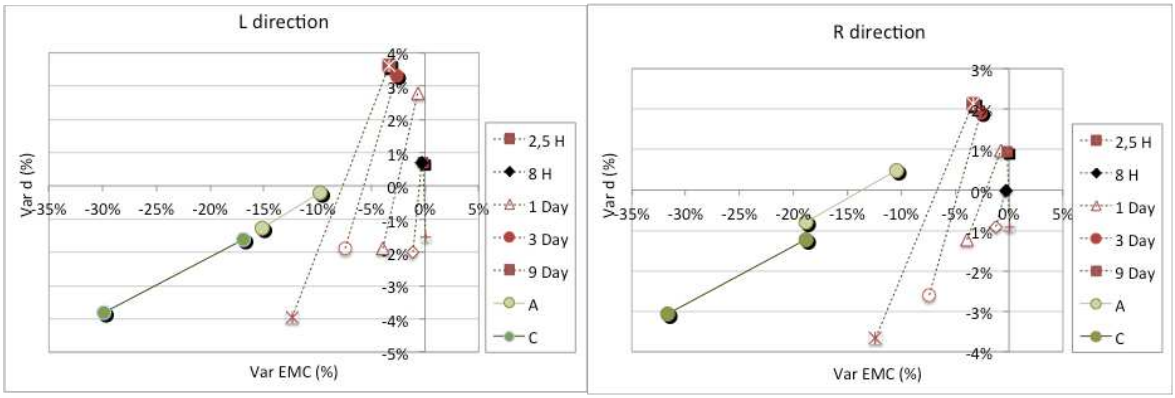
To discover the effect of reconditioning and potential recovery, the results of changes in physical and mechanical properties after treatment and after reconditioning are shown below.

Fig. B.17. shows the relationship between the changes (relative to untreated wood) in EMC and in density, in specific Young's moduli and in damping of Morus (red symbols) and of Spruce (green symbols), after treatment and after recovery. For each specimen the arrow indicates the shift from data after treatment to data after reconditioning/recovery.

In Fig. B.17a, the relation between variations of density versus those of EMC is shown. For Morus specimens, changes were recovered and there is a small final increase in density. But for Spruce specimens, for treatment A, there is a complete recovery while for treatment C the recovery is not complete.

Fig. B.17b shows the variations for the specific Young's modulus. For Morus, for both R and L directions, there is a complete recovery of the initially observed reduction in E/d after TT. In L direction, the specific modulus is now even slightly increased compared to untreated wood, while EMC has returned to the values of native wood. Also for Spruce specimens there is a recovery for both treatments A and C, which is not complete recovery except for the mildest treatment A.

a



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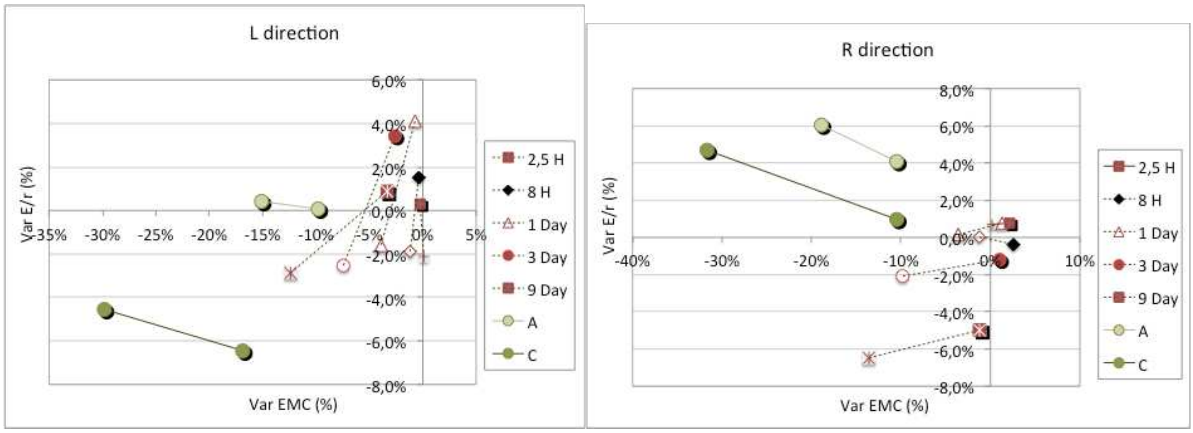


c

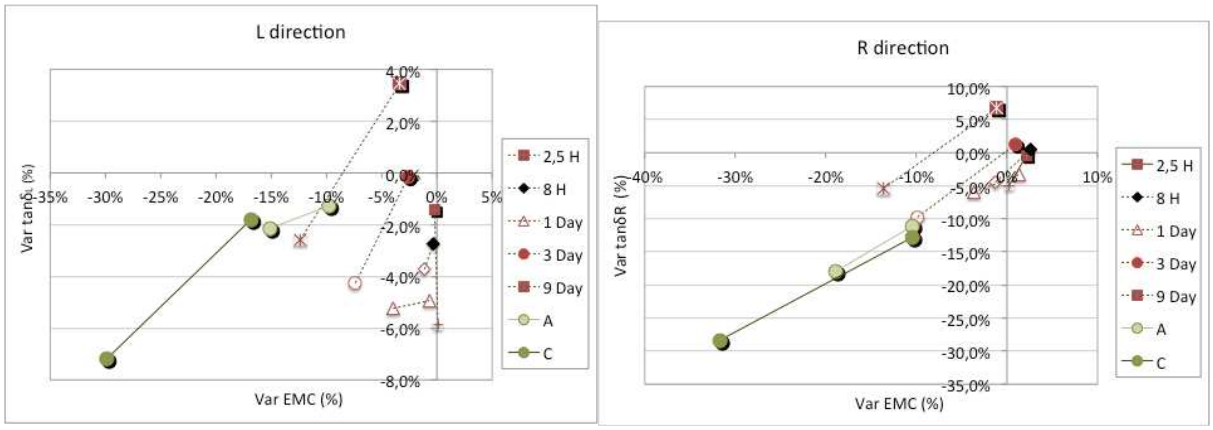


Fig. B.17 Relations between the (relative) variations in EMC and those of others properties, after thermal treatment and then after reconditioning: (a) density (b) specific modulus of elasticity (c) damping. Empty symbols correspond to values after thermal treatment; filled symbols correspond to values after reconditioning.

Fig. B.17c shows the variations in damping. As it can be seen in the graph, for Morus in L direction there is an almost complete recovery in EMC, but significant differences in damping between the groups submitted to different durations of TT remain. For Spruce, the recovery is observed and is well related to recovery in EMC, but it remains partial. For R direction, after

the initial TT-induced decrease there is either a complete recovery or even and increase compared to native wood of Morus, while for Spruce the recovery is clear, and related to recovery in EMC, but still only partial.

So, for R and L directions, for Morus the recovery is more or less complete and can even provoke changes going in the opposite direction as those provoked by the thermal treatment alone. But, for Spruce a large part of the changes have been recovered and for the mildest treatment (A) recovery was closer to be complete than for the higher temperature treatment C, which conditions are the closest to the TT applied to Morus.

B.5. Conclusion

B.5.1. Thermal treatment on Morus

According to the results achieved after thermal treatment for Morus specimens, some physical and mechanical properties changed. Equilibrium moisture content (EMC %), density and specific modulus of elasticity (E'/d) are decreased with treatment duration while for damping (tanδ) there is a decrease in the beginning, which is changed to an increase during end of treatment. The decreases in EMC might have been attributed to the degradation of hemicellulose, which would result in the decrease in accessible OH groups. However, after reconditioning most of the changes have been recovered. So, it can be realised that most part of changes are rather due to structural changes in wood internal structure (or polymers) during thermal treatment. The weigh loss is small but gradually increased along with treatment duration, which can be because either of beginning of depolymerisation of hemicelluloses, or of the loss of extractives that exist in Morus species. The extractives content of Morus may be an explanation for its different reaction both to thermal treatment, and recovery, as compared to Spruce.

B.5.2. Hydrothermal treatment on Spruce

The results of hydrothermal treatment on Spruce specimens show some physical and mechanical changes. Equilibrium moisture content (EMC %), shrinkage and density are decreased, which the largest effect can be seen for treatment C with longer duration and more severe conditions of treatment. The modulus of elasticity (E'/d) is increased in both R and L directions. The increase in L direction is smaller than R. For damping (tanδ) there is a decrease, which is clearly bigger for R direction. The weight loss can be due to the degradation in hemicelluloses. Colour lightness can be a good indicator to monitor changes during treatment. The lightness is decreased by treatment duration and also by degrees of temperature and relative humidity of the treatments. The decrease in lightness is followed by the decrease in weight loss. It can be expected that some parts of changes are the results of chemical changes in wood components.

After the first reconditioning, around half part of changes was recovered. So, it can be deduced that some part of the initially observed changes after HTT were due to structural changes in wood construction. After the second reconditioning, a big part of changes was recovered and for the mildest treatment (A) the recovery was close to complete. But, there are still some parts of properties changes in stronger treatment (C) which can be because of irreversible chemical changes in wood composition.

PART C. SURFACE TREATMENTS: VARNISHES

C.1. Bibliography

The acoustical properties of musical instruments can be affected by different surface treatments. Different surface treatments include different coating systems and preparation/pre-treatment procedures: ground layers, varnishes and (natural or UV) light treatments. In the present study, it is focused on Iranian and European wood+coating systems and they are compared together. Then, the aging of applied coatings is investigated.

C.1.1. Varnish

Quite often, there are some opinions in general public that the varnish plays an important role in the "tone quality" of instruments. However, its application has two original purposes that are different from "sound": beauty and protection (Schleske, 1998). The applied quantity and the (quantitative and qualitative) composition of varnish can affect the acoustical properties. It should be considered which methods and coating systems are used. Nevertheless, some questions are raised: (i) how varnish can affect the acoustical properties? and (ii) how will be the kinetic of changes by passing the time? Another question is which coating system can be more "effective", and are there differences between different traditions of instrument making?

In bibliographic sources prior to 1800, there are very few texts from craftsmen, which describe the materials, tools or processes used for musical instruments varnishes (Echard and Lavédrine, 2008). Mailland in 1859 was probably one of the first to try to rediscover the composition of the Old Italian violin varnish.

A varnish can be defined as one or several layer(s) of an organic film with inorganic particles (Echard and Lavédrine, 2008). Organic compounds are natural products such as drying oils,

essential oils, tree resins and gums, insect resins, dyes, various proteins or polysaccharides. Inorganic compounds (as oxides, hydroxides or carbonates) may be added as pigments, fillers, siccative agents or substrates (Echard and Lavédrine, 2008). Usually, three families of traditional varnishes are distinguished:

- Spirit varnishes: resins dissolved in alcohol;

- Essential oil varnishes: resins dissolved in volatile oils;

- Oil varnishes: resins mixed with siccative oils.

-In addition, modern industrial varnishes increase the types and principles of varnishes.

Michelman (1946) published semi-quantitative analyses of varnishes from a cello made by Francesco Ruggieri in 1691, the ‘‘Prince Gurski’’ cello made by Antonio Stradivari in 1697, a Nicolo Amati cello dated 1682 and from eleven other instruments. Barlow (2008) used some microscopical and chemical techniques for investigating and analysing wood and surfaces of violins.

In 1989 Barlow and Woodhouse studying varnished surfaces of instruments, mostly Italian, dating from between 1650 and 1750 by SEM and EDAX. They stated g round layers certainly exist on some old instruments. In addition, in 2006, Echard was studied varnishes from violin preserved in Musée de la Musique. They were investigated and analyzed by using different methodologies. Also, in 2010 Echard et al investigated the nature of finishes of Antonio Stradivari. They determined the composition of the materials of varnish samples from five instruments made him. In 2002, Simonnent et al studied the effect of violin varnishes on the vibrational properties of wood. The viscoelastic and drying process of varnishes from two different classes were investigated. They also suggested more experiments by improving models and by taking into account several layers of varnishes with different compositions.

Buksnowitz et al (2007) were compared the traditional knowledge and experience of violin makers with science approach for characterizing the properties of resonance wood. Norway spruce (*Picea abies* _L._ Karst.) which is used for the construction of resonance tops of stringed instruments has been investigated in this study. The study showed makers can distinguish the properties of instruments related to visual features, but for mechanical and acoustical properties is not the same. Also, the acoustical properties and external aspects of red Spruce were studied (Di Bella et al, 2002). The average growth ring distance was studied by image analysis thechnic. The results indicated no direct relations between physical and mechanical properties of red Spruce.

C.1.2. Acoustical effects of varnishes

There is not much available literature about the effects of varnishes on the acoustical/vibrational properties of coated wood used for musical instruments. Here it has been tried to collect some important studies.

Like other components in a vibrating system, by adding varnish to a light wooden plate, it can affect stiffness, mass and internal friction (Schelleng, 1968). The application of oils-based varnish leads to a decrease in frequency because of the mass increase. After applying varnish the decrease in frequency is followed by a rise as volatile components evaporate (Schelleng, 1968).

In 1993 Ono investigated coated and uncoated Spruce plates and the Young's modulus and the internal friction were measured for these plates. With varnishing, the sound power level

decreased at low frequencies (<300Hz), and it increased or decreased at high frequencies (>3kHz). Therefore, the acoustical characteristics in the former are improved with varnishing and the sound power level increases at frequencies above about 300Hz. Schleske (1998) studied the effect on acoustical properties of different recipes of violin varnish. He studied the effect of varnishes on the loss factor, velocity of sound and resonance frequency 9 years after applying the treatment. Loss factor and velocity of sound both decreased after 9 years. However, the kinetics of changes were not detailed (3 measurement points in 9 years), and the conditions of ambient humidity were not controlled.

Obataya et al (2001) investigated the possibility of using Urushi as a coating for musical instruments. Urushi is the sap of Japanese lacquer tree, which is used as a coating. Similar coatings from related species are also used in east-Asian countries. Also, they compared Urushi lacquer with "conventional', industrial ones. By coating the most significant change was found in internal friction (Q^{-1}).

Also, Minato et al (1995) investigated the effect of varnish on the vibrational properties of wood during its drying process. They compared natural (shellac) and artificial varnish (cellulose nitrate, CN). The changes in weight, specific young's modulus, and loss factor and resonance frequency of two different varnishes were measured and compared. The results suggested the superiority of natural varnish to artificial one. Hwang et al (2006) investigated the effect of thickness and hardness of coating on the acoustical properties of wood for musical instruments.

C.1.3 Conclusion

Our purpose for this part of study was to find the effect of surface treatments on the vibrational properties of wood used in European and Iranian musical instruments. In fact:

- According to the previous attempts to study the effect of surface treatments like different kinds of varnishes, we are going to study the effects on vibrational properties in both R and L directions of wood and to follow the trends of change along time, with precise kinetics and rigorous monitoring of other possible affecting factors.
- Also, we will compare Iranian and European wood+coating systems and we will discuss their respective effects.

The experiments about Iranian wood+coating systems was fully conducted by the author. The experimental protocol about European (violin making) wood+coating systems started in the same time as beginning the writing of this thesis report. Therefore, the author mostly participated to preliminary tests. Then the research work was essentially conducted by the supervisors of this thesis and by other students at LMGC. However, these “collective” results could be introduced in the present document, to compare European and Iranian wood coating systems.

C.2. Iranian varnishes applied on Morus wood

C.2.1. Protocol

C.2.1.1. Material

- ***Wood species and primary measurements***

As it has been explained, Morus species was used because this species is used for making traditional musical instruments in Iran (Tar, Setar).

The experimental procedure was done in CIRAD, in Montpellier. The specimens were dried in an oven in 60°C for 2 days and then in 103°C for 3 hours. After oven drying, mass and dimensions of specimens were measured. Then specimens were placed in climatic room in standard conditions (65% RH, 20°C) to be stabilized. Stabilization was done for 3 weeks. Then, measurements of mass, dimensions and vibrational properties were done to be able to calculate EMC, density, specific modulus of elasticity and damping.

After primary measurements, Sampling was done by the automated statistical method, which is explained in general material and methods, based on the initial values.

- ***Varnish***

Most string instrument makers in Iran use fa solvent-based varnish with Sandarac rosin as a coating for different musical instruments. Some of them also use shellac or polyester varnishes/coatings. Here, these three different coatings were used and compare.

- Sandarac

Sandarac is a resin obtained from the small tree *Tetraclinis articulata* (Cupressaceae). The resin exudes naturally on the stems of the tree. It is also obtained by making cuts on the bark. In Iran, most of the makers use this resin dissolved with alcohol as a solvent, to apply on the surface of musical instruments.

For preparing Sandarac varnish in the same way as makers do in Iran, ethanol (96%) was used as a solvent. The solution consists of half percent alcohol and half percent Sandarac. Then, they should be mixed to achieve a solution more or less with the same viscosity and colour as honey. This solution has to be applied on the wood by a very soft brush. For applying varnish, depending on the viscosity of solution, it can be applied with between 1 to 6 layers or more.

- Shellac

Shellac is a resin secreted by the female lac bug, on trees in the forests of India and Thailand. It is processed and sold as dry flakes and dissolved in ethanol to make liquid shellac varnish.

- Polyester

Polyester is a category of polymers that contain the ester functional group in their main chain. As a specific material, it most commonly refers to a type called polyethylene terephthalate (PET). Polyesters include naturally occurring chemicals, such as in the cutin of plant cuticles, as well as synthetics through step-growth polymerization such as polybutyrate. Natural polyesters and a few synthetic ones are biodegradable, but most synthetic polyesters are not.

C.2.1.2. Methods

- ***Varnish application in Laboratory conditions (in Montpellier)***

For this treatment Sandarac resin was used, which was bought from Iran (provided my maker of Iranian lutes Samad Zare). It had to be prepared by the recipe of mixing with ethanol, which is explained before. For applying varnish, a soft brush was used.

As it was mentioned, 5 paired groups of wood specimens were defined and used: Control for stability of conditions, Control for the effect of solvent alone, Effect of applying 1-layer, 3-layers and 6-layers of varnish. For one layer after applying varnish, mass and vibration properties were measured after a few minutes of applying. For 3 and 6 layers, depending on the drying process of varnish on the surface the next layer was applied, which normally were

2 layers per day. Because this varnish was alcohol-based there was no need to use UV light exposition to accelerate the drying process, because the main principle is here that of solvent evaporation and deposition of resin material. Kinetics of changes in properties after applying varnish was recorded from the first few hours to after 6 months (Table 7).

Table 7. Gap between measurements for different duration of times from the beginning.

Gap between measurements	Duration of time
Every 30 min	First day
Every 2 hour	Second day
Every day	First week
Every 2 days	Second week
Every week	First month
Every month	Until end

- ***Varnish application in workshop conditions (in Iran)***

The rest of Morus specimens, were chosen for another way of coating application, which was done in Iran. So they were taken in Iran while before they were placed in climatic room and were stabilised in standard condition (20°C and 65% RH). 3 paired groups (defined by the method of statistical sampling explained in Chapter A) of specimens (6L and 6R each) remained from the previous protocols. In Iran, the specimens were kept in ambient, normal unregulated conditions, and after one week they were varnished. For this step, an expert musical instrument maker, Mr Samad Zare (who had selected and provided all the wood material of Morus for this thesis work) has applied 3 different varnishes himself, following his usual procedures. In fact, application by a professional person makes it possible to do comparison between applying by an expert maker and by a non-professional person (such as ourselves as scientists) to see if there is an effect on measurable properties (Fig. C.1).

Varnishes were applied for three groups of Morus specimens with 6 specimens in each direction (L and R) in each group. Polyester, Sandarac (in ethanol solvent) and Shellac (in ethanol solvent) varnishes were applied as three common varnishes used by Iranian

instrument makers. For each one, one layer was applied. The concentration of these three varnishes was the same as varnish used in real musical instrument making. Applying was done by a piece of cotton, which was put in a piece of cloth. After applying varnish the specimens were kept to air dry for several hours. After completely “dry to the touch”, they were taken to Montpellier and were placed in a climatic room for 3 weeks in standard conditions (20°C, 65% RH). After stabilization, mass, dimensions and vibrational properties were measured as explained in chapter A.2.

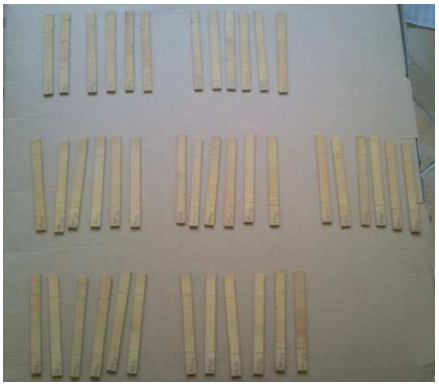


Fig. C.*1 The steps of workshop varnishing procedure for all the specimens varnished by a professional instrument maker in Iran. (a), preparing cotton for varnishing (b), applying Shellac (c), Polyester (d) and Sandarac (e) with a professional instrument maker in Iran.*

C.2.2. Results and discussion on Iranian wood/coating systems

Concerning measurements to discover the effects of different surface treatments, all the results were classified in two series.

C.2.2.1. Laboratory varnishing and monitoring of kinetics (in Montpellier)

The coating treatments conducted on Morus specimens in laboratory conditions (in Montpellier) included 3 main treatment modalities with 1, 3 and 6 layers of Sandarac/ethanol varnish, as well as 2 control modalities (one group to monitor long-term changes in ambient conditions and related changes in properties, 1 group to assess the effects of applying ethanol solvent alone).

Measurements of mass change and vibrational tests were done after applying varnish as explained in section A.2. The results of resonance frequency and damping changes are shown in Fig. C.2 and Fig. C.3.

In Fig. C.2, The changes in damping is shown for both R and L directions and three different treatment modalities. For all the treatments, after applying varnish there is a strong initial increase in damping, followed by a gradual re-decrease over time. For 6 layers of varnish, in the first day after applying the sixth layer, there is a remarkable increase, which is near to 160% and 100% for R and L direction respectively. For 3 layers of varnish, the changes are different. For R direction, there is an increase around 70% in the first day that continues until around 120% in the third day. Then it is followed by a decrease. For L direction, the trend is the same as R, with less increase, by around 40% in the beginning. For 1 layer varnish, the increase in the beginning is close to that for 3 layers, but the trend is different. After the first day of increase, it continues to decrease. After just one month, about half of the initial increase in damping is recovered. After around 11 months, the changes in damping are small, returning to values close (maximum 10% difference) to those of uncoated Morus wood.

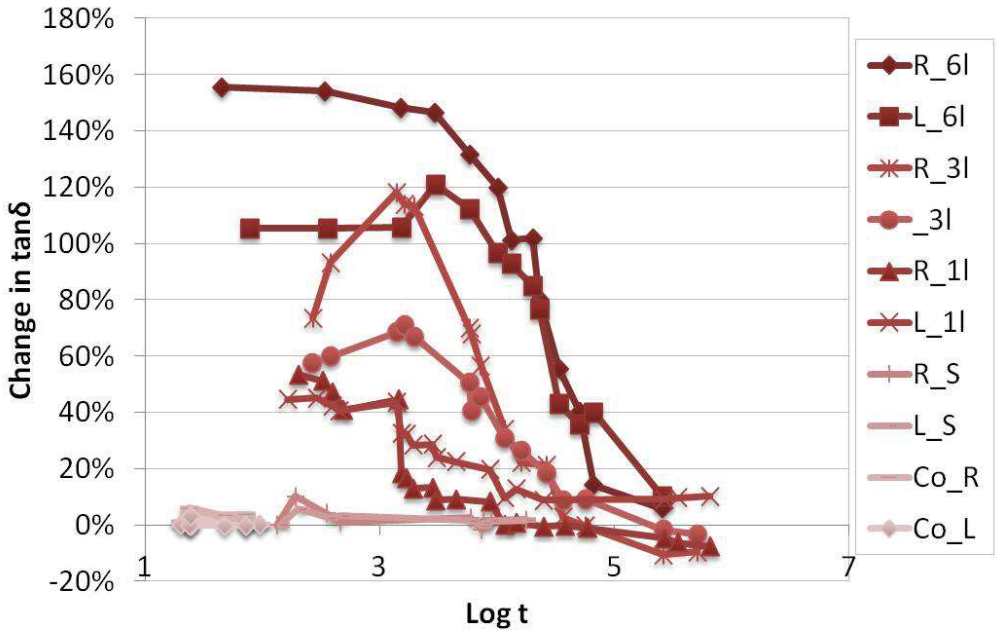


Fig. C.2 .Variation of damping during time for groups with 6layer (6l), 3layer (3l), 1layer (1l), Solvent (S) and Control (Co) for both R and L directions. X-axis = logarithmic scale of time, with minutes as basic time unit.

In Fig. C.3, The change in resonance frequency for all treatments is shown. For 6 layers varnish, the increase in frequency is clearly higher than for the other treatments. In the beginning it is near to 6% for R direction. For 1 layer, there is a small increase in the beginning, which slowly continues during 11 months. For 3 layers varnish, the immediate effects after varnishing are 3% and -1% in the beginning, in R and L direction respectively. Then it continues to increase and after 11 months it reaches 9% and 1% in R and L directions, respectively. In L direction, for all the treatments there is a mild increase, stable after 11 months.

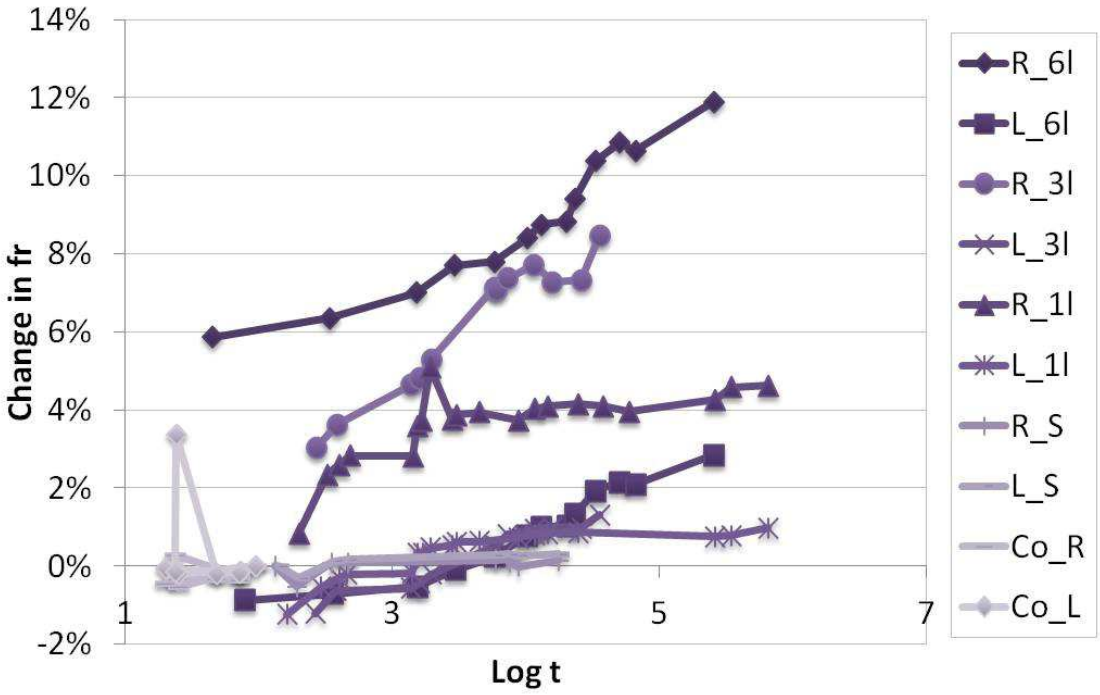


Fig. C.3 Variations of resonance frequency during time after application of solvent-based varnish. The desciprtion is the same as in the previous figure.

So, according to the results, after applying varnish there is a remarkable increase of damping in the beginning. After around 11 months, the values return to close to untreated wood. From the results, it can be seen that after this duration there is no clear effect of varnish on damping. But, for resonance frequency, there is an increase, which is a significant especially in R direction, and during time there is a mild slope of continuing increase, especially for 3 layers and 6 layers. For 1 layer varnish after the small increase in the beginning, the trend is stable during time.

Fig. C.4 shows the mass changes during 11 months after applying the varnish. Immediately after applying varnish the was increased for all the treatments, which is because of the added mass of varnish. Logically, mass change for 6-layer varnish should be the highest, than that for 3 layers or 1 layer. Also, the effect of solvent alone is shown here. In the first minutes of applying solvent, which was ethanol, there was an increase, followed by a decrease as the

solvent evaporates from the surface. For all the treatments, along 11 months, there is only a small further mass decrease. The decrease is due to the evaporation of solvent (and maybe polymerisation of varnish) but the evaporation is a very fast process.

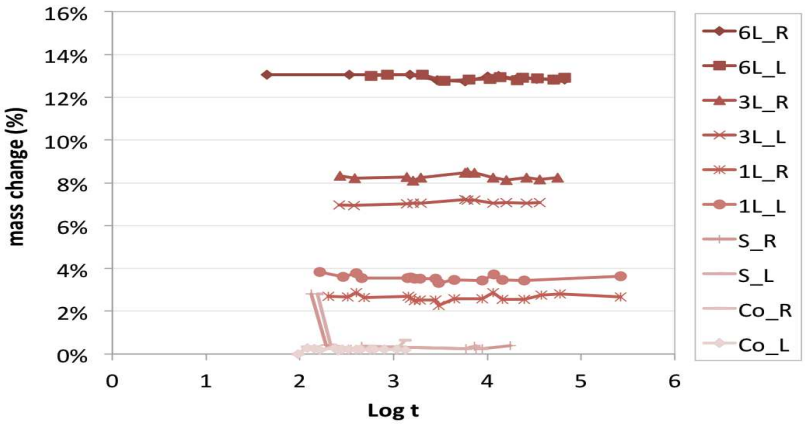


Fig. C.4. Variation of mass during time

In Fig. C.5, damping variations versus mass change are shown. As it is clear, for treatments with more layers of varnish the changes in damping are the biggest. But the time-related changes in damping are not related to mass changes. This suggest that the kinetics of "drying" are little related to the evaporation of solvent, but probably to some changes in the deposited resin.

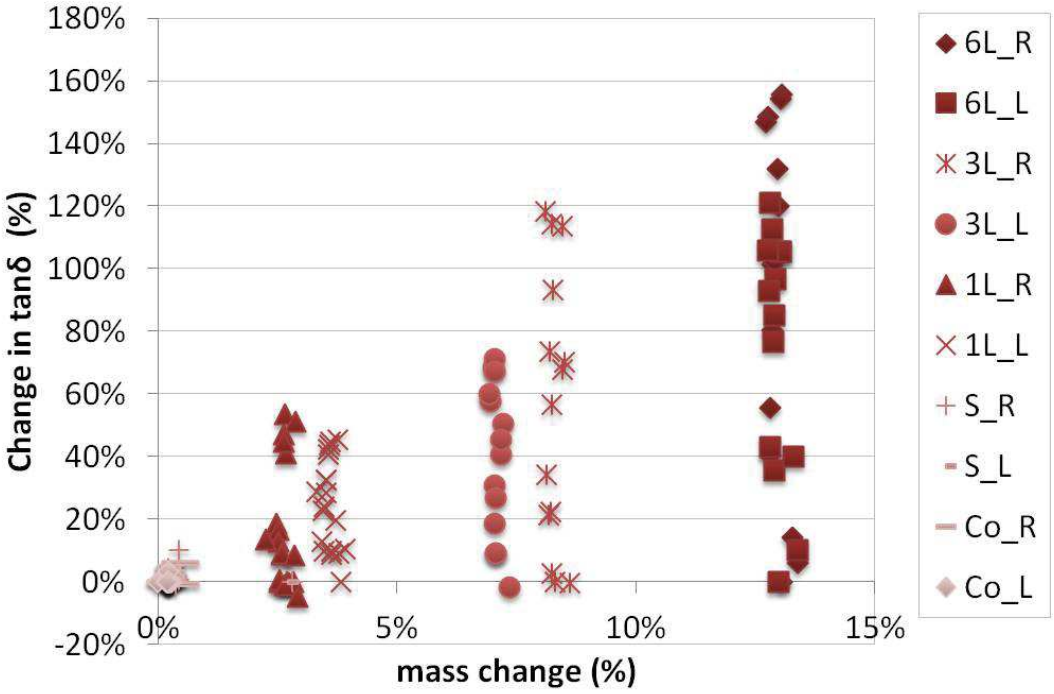


Fig. C.5. Variation of damping versus mass change

C.2.2.2. Workshop varnishing and comparison of three varnishes (in Iran)

Another treatment for Morus was performed in Iran.

The results of change in mass, modulus of elasticity, damping and resonance frequency are shown for three different varnishes and in 2 different durations (2,5 and 7 months after applying varnishes).

Fig. C.6, is the results for R direction. Fig. C.6 a, shows the mass change. After 2.5 months there is increase for all the treatments. For shellac, the mass increase is highest. After 7 months for polyester and shellac the mass decreases a little but for sandarac it increases a little. The application of varnish by a professional instrument maker resulted in thinner layer for sandarac varnish (weight gain of less than 2%) than the previous laboratory test (weight gain of 3-4%).

Fig. C.6 b shows the change in specific modulus of elasticity. For all the varnishes the modulus increases after 2.5 months and then after 7 months it is approximately stable. Fig. C.6 c shows the change in damping. After 2.5 months there is an increase for all the treatments. For Polyester the increase is significant and near to 35%. After 7 months for Polyester there is only a very small re-decrease. For Sandarac there is a small decrease in damping as compared to uncoated wood, and for Shelac there is no change.

In Fig. C.6 d, the changes in resonance frequency is of about 1.5 to 2% for all three varnishes. After 7 months comparing to 2.5 months there is not a big difference.

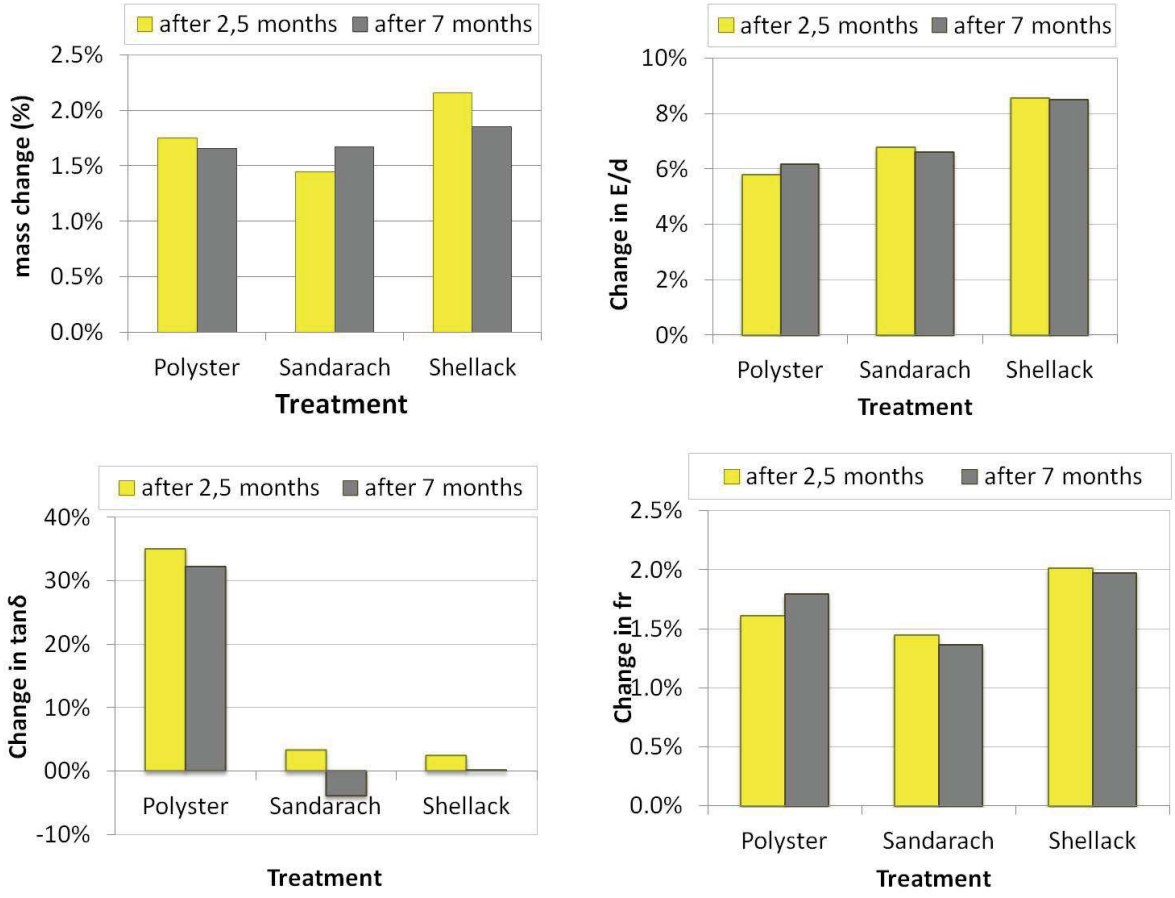


Fig. C.6. Variation of mass (a), Modulus of elasticity (b), damping (c) and resonance frequency (d) in R direction.

Fig. C.7 is the results for L direction. Fig. C.7 a shows the mass change. After 2.5 months there is increase for all the treatments. For shellac, the mass increase is highest, which is the same as R direction. After 7 months the changes are constant.

Fig. C.7 b shows the change in modulus of elasticity. For all the varnishes the modulus increases after 2.5 months and then after 7 months it is approximately stable. The increment for Sandarac is higher and up to 4%. Fig. C.7 c shows the change in damping. After 2.5 months there is an increase for all the treatments, but only very small for the solvent-based sandarac and shellac. For Polyester the increase is significant and close to 20%. After 7 months for Polyester there is some re-decrease but the coated wood remains significally more damping than uncoated. For Sandarac there is a decrease in damping reaches reaches -2.5%. For shellac after 7 months there is no change.

In Fig. C.7 (d), the changes in resonance frequency for Polyester is negative after 2.5 months and 7 months. But for Sandarac and shellac, After 2.5 months there is an increase for Sandarac (up to 1%) and Shellac (0.7%).

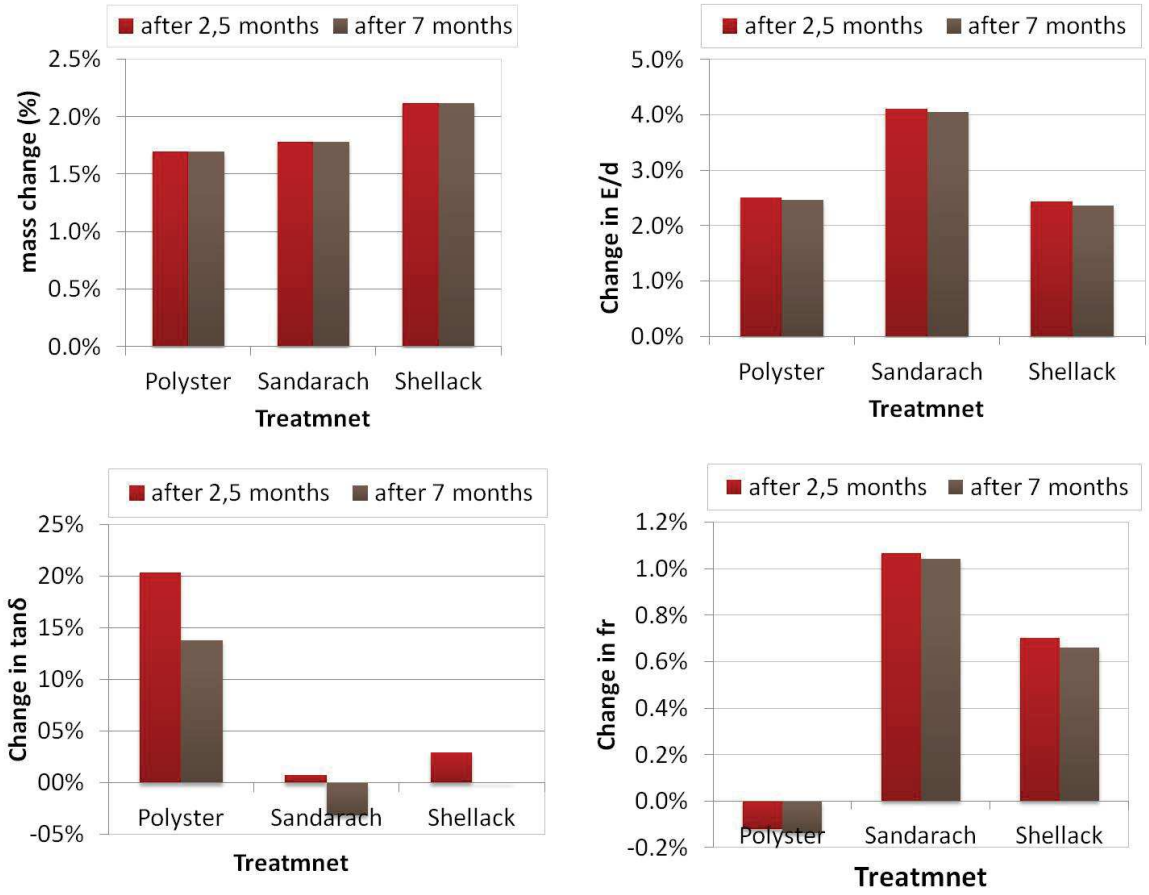


Fig. C.7 Variation of mass (a), Modulus of elasticity (b), damping (c) and resonance frequency (d) in L direction.

C.2.3. Conclusion

Surface treatments of Morus specimens included two different experiments: laboratory treatments and evaluation of kinetics (in Montpellier) and workshop treatments comparing three varnishes (in Iran).

C.2.3.1. Laboratory treatment in Montpellier – kinetics of varnish "drying"

As it has been mentioned, there were 3 different groups of treatments: 1 layer, 3 layers and 6 layers varnish. The results of varnishing for Morus specimens show that ultimately it does not induce an important change of vibrational properties. At first, there was about 160%, 80% and

60% increase in damping for 6 layers, 3 layers and 1 layer respectively. After around 11 months the changes returned to close to 0%.

There was a small increase of resonance frequency for all the treatments. The biggest increase was seen for 6-layer varnish in R direction, around 6% in the first hour after varnishing and 12% after 11 months. After 11 months, there is still an increase in resonance frequency.

To find about the stability of these changes, the kinetics of changes with time should be followed on a longer time, for example to check if there is still a decrease in damping and an increase in frequency. Considering that the solvent evaporation itself is a very fast process, the mechanisms of further changes in vibrational properties through time should be understood.

C.2.3.2. Workshop treatment in Iran – comparison of three varnishes

Treatment in Iran by a professional instrument maker was done by using three different varnishes; Polyester, Sandarac and Shellac.

According to the results, it was found for Sandarac and Shellac that there is not a significant change in damping after around 7 months. For resonance frequency there is a small increase. For Polyester, damping increased after 2.5 months and then in the following it decreased a little but still remained clearly increased as compared to uncoated wood.

The results of two different conditions of varnishing confirm each other. By comparing both results on Morus specimens, it can be understood that traditional varnishing does not have (on the long-term) an effect on the damping for Iranian musical instruments. On the contrary, traditional sandarac varnish has a stiffening effect, both in R and L directions. Considering that native Morus wood has low specific modulus in L direction, in can be thought that the varnish provides some reinforcement. Polyester varnish also has a small stiffening effect, but clearly increases damping, contrary to the two natural (solvent-based) varnishes tested. But to

be sure, the changes should be followed. By following the kinetics it would be possible to see how will be the long-term changes and when they will stay constant.

C.3. European violin varnish applied on Spruce

Remark: as explained before, a comparison between Iranian and European wood+coating systems was originally planned for this thesis. However, the experiments on European (violin making) vanishes only started in the end of this thesis, while starting the writing. Therefore, the author conducted experiments in the beginning (called "preliminary treatment" below) and participated to the first steps of the full protocol (called "main test" below). But most of the experiments of the "main test" as well as the monitoring of kinetics of changes was conducted by supervisors (Iris Brémaud and Sandrine Bardet) with the help of students (Lydéric Rebecchi and Capucine Carlier).

C.3.2. Protocol

Spruce samples were prepared from France and were chosen according to criterion properties of wood used in musical instruments. They were chosen by Iris Brémaud. They were from two planks (soundboards) and were cut by Pierre Cabrolier in Montpellier. The planks were cut in L and R directions with 12×2×150 mm^3, R×T×L and 120×2×12 mm^3, R×T×L dimensions. After cutting and sanding (with very fine grain) the surface of specimens to be very smooth, they were brought to CIRAD. Oven drying in 60°C for 2 days and then in 103°C for 2 hours and then after measurements of mass and dimensions was done. Stabilisation was achieved by placing specimens in conditioning room (20°C, 65% RH) during 3 weeks.

For the main treatment, as it has been explained in chapter A, after primary measurements of properties, the sampling into groups was done (with the Macro developed by Tancrède Alméras) based on the initial values. For the preliminary treatment, sampling was performed with the rest of specimens. So, for the main treatment, in each group there were 6L and 6R and for preliminary test, there were 2L and 2R specimens.

Preliminary treatment – effects of varnishing process

Preliminary test was done to evaluate the process of varnish application, which includes:

- Preparing ground layer
- With or without ground layer
- Applying varnish
- The duration to dry varnished surface

So, preliminary test was performed to find:

- The most appropriate way to prepare and apply ground layer
- The vibrational effect and the uptake of varnish with and without ground layer
- The necessary time to complete drying of varnish

➢ *Ground layer*

For this treatment two usual ground layers employed by violin makers were chosen: Gelatine and varnish including silica. The gelatine can be prepared by two different methods:

- Hot liquid gelatine was prepared by dissolving 1.5 sheet of gelatine in hot water and keep it warm during applying (3% solution).
- Cold solid gelatine was prepared by dissolving 1.5 sheet of gelatine in hot water and leaving it to be dissolute, then keeping it in cold temperature for one night to make it jellified (3% solution).

An expert violin maker, Nicolas Gilles, prepared silica varnish, which contains oil-based varnish and silica.

For gelatine ground layers, there were 6 specimens in each direction and for each kind of application (liquid or jell) with (2L+2R) specimens kept just with ground layer and (4R+4L) others for applying varnish. So, a total of 12L and 12R.

For ground layer with silica varnish, there were 2 in each direction. After applying, they were left for 5 min to allow the penetration of silica. Then, with a piece of cotton the excess of varnish was wiped from the surface.

The liquid gelatine and the silica varnish ground layers were applied with a fine brush onto the specimens' surface and it has been tried to cover all the surface uniformly. After using it with each ground layer, the brush was washed (with hot water for gelatine, with turpentine for silica varnish). The jellified gelatine was applied with a painters' spatula.

- ***Varnish***

Comparing to varnish used for Iranian instruments, in the case of violin-making most of the luthiers use (siccative) oil-based varnishes. Two different oil-based varnishes have been kindly provided by a violin maker, Nicolas Gilles, who prepared the varnishes:

- Rosin / 75% oil of linen. This is a quite classically used varnish.
- "Térébentine de Strasbourg" / oil of walnut. This is more experimental varnish.

As mentioned, 4L+4L specimens for each kind of gelatine were kept for applying varnishes. So, two kinds of varnishes were applied for 2 specimens for each gelatine and each direction.

In addition, there were 2 specimens in each direction just with varnish, without any ground layer. All the surface treatments for preliminary test are summarized in Table 8.

Table 8. Number and classification of specimens for preliminary test

Treatment	Hot liquid gelatine	Cold solid gelatine	Varnish + silica	No ground layer
Térébentine de Strasbourg	2R,2L	2R,2L	0	2R,2L
Rosin ("colophane")	2R,2L	2R,2L	0	2R,2L
No varnish	2R,2L	2R,2L	2R,2L	2R,2L

Applying was performed by a brush, and it was tried to be done uniformly. One layer varnish was applied for all the specimens. After each treatment, measurements of mass and vibrational properties were done. The day after applying varnish, the coated specimens were then exposed to UV light (see below) to accelerate the drying, as is usually done by luthiers. For the first day of treatments (after ground layers, after varnishing, and after UV), measurements were done 2 or 3 times in the day, then gradually with longer steps (days then weeks). All the process of applying varnish and measurements has been done in a climatic room with a standard condition (65% RH and 20°C).

- ***Comparison between ground layers***

After finishing preliminary test, according to the objectives, it had to be decided which kind of ground layer is most appropriate, i.e convenient to apply and effective. So, some comparison between applied ground layers was done to see the effects on mass change and vibrational properties. In Fig. C.8, changes of damping and mass during around 4.5 months were followed. Fig. C.8 a shows the mass changes after applying varnishes. Comparison between R and L direction is just to see if there is differences because of the different cut and penetration of varnishes. Nevertheless they should be approximately the same. According to the results, between two liquid and solid ground layers there is no important difference while for silica, the difference is clear for R direction. Fig. C.8 b shows the value of damping during

4.5 months. The comparison between ground layers shows no important difference between hot liquid and solid gelatine. Comparison between gelatine and silica shows no important change in both R and L directions.

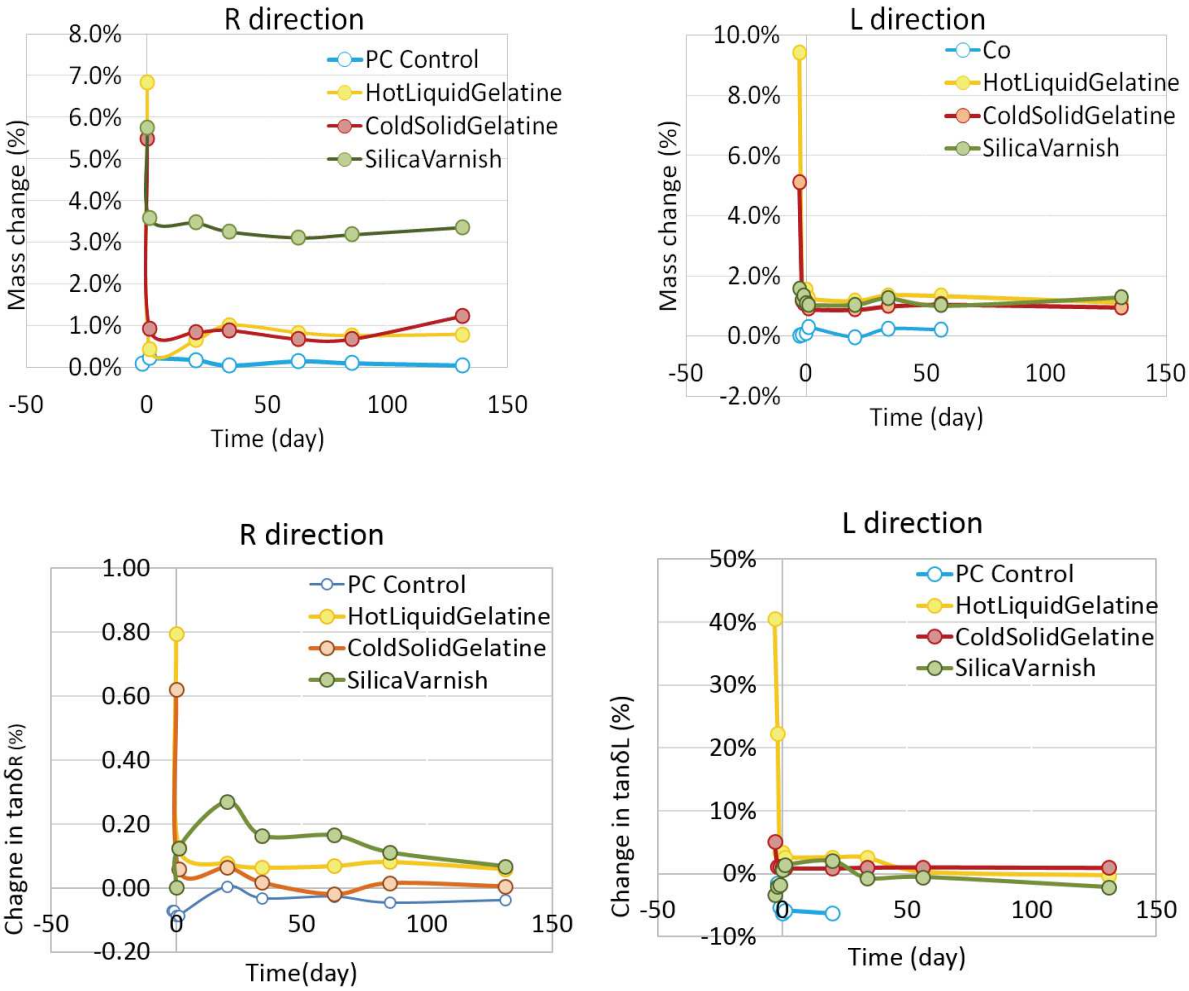


Fig. C.8 Variation of mass (a) and damping (b) in R and L direction, due to different ground layers.

According to the results after preliminary test, it was decided to apply gelatine. Because silica varnish is more difficult to apply exactly the same on all specimens, leading to differences in weight gain. Also, gelatine is more "neutral" and "classical" I order to compare different subsequent varnishes.

Between hot liquid and solid gelatine, the difference was just the way to prepare and apply. Therefore, it was decided to apply hot liquid gelatine, as it was more practical and easy.

Consequently, for the main test that is the next step, it was decided to apply hot liquid gelatine as ground layer for all the specimens.

Main test

- ***Protocol***

As noted above, primary experiments and measurement were done and then sampling for this test was performed based on the initial value. Sampling was done according to the method, which was explained in Chapter A. As here it was decided to have 12 groups in each direction (including 3 control groups: one for effects of conditions, one for effect of ground layer, one for effect of UV) with 9 groups for 3 differents varnishes, described below (Table. 9).

Table 9. Number of specimens and original plan for main test

Treatment	No treatment	Ground layer (GL)	UV	1 layer+UV	2 layer+UV	4 layer+UV
No varnish	6R, 6L	6R, 6L	6R, 6L	-	-	-
Rosin	-			6R, 6L	6R, 6L	6R, 6L
Amber	-			6R, 6L	6R, 6L	6R, 6L
Sandarac	-			6R, 6L	6R, 6L	6R, 6L

But as seen on Table. 10, the actual types of applied treatments is different from the originally planned experiment. Because some of the first varnishes applications gave too thick layers, and also there were not enough time to apply 4th layer.

Table 10. Number of specimens and actually conducted plan for main test

Treatment	No treatment	Ground layer (GL)	UV	1 thin layer +UV	1 thick layer +UV	2 thin layers +UV
No	6R, 6L	6R, 6L	6R, 6L(G3)	-		-

varnish	(G1)	(G2)				
Rosin	-				18L, 18R (G4,5,6)	
Amber	-			12R, 12L (G8,9)	6R, 6L (G7)	6R, 6L (G9)
Sandarac	-			12R, 12L (G11,12)	6R, 6L (G10)	6R, 6L(G12)

- ***Ground layer***

Ground layer was applied by a brush. After applying ground layer and measurement of mass and vibrational properties, varnishes were applied. The gap between applying ground layer and varnish was at least one day to let it dry, although by preliminary test it was found that in some hours they can be completely dry.

- ***Varnish***

The treatments were done with three kinds of varnish. These varnishes are siccative oil-based varnish and were provided and prepared by an expert researcher, Francois Perego (Table. 10). The siccative oil was linseed oil for all of them, with a comparable ratio of oil to resin, and they differed by the nature of the resin:

-Varnish 1: rosin (“colophane”)

-Varnish 2: amber

-Varnish 3: sandarac

The protocol for applying varnishes was as follow:

For the one-layer varnishes, after applying varnish (with a silicon painters spatula), they were left to rest for 24 hours and then placed in UV box for 24 hours to accelerate the drying. Then measurements of mass and vibrational properties were done for the first day after UV treatment. These measurements were then done continuously every day and then every week to monitor the kinetics of drying. For more than one layer varnish, after applying one layer, sufficient time was left so that the fisrt layer was well “dry to the touch”. All the steps of

treatment, measurements and monitoring were done in the climate room (20°C and 65%RH). The illustration of all the steps is shown in Fig. C.9.

- ***Drying of varnish***

Ultra-violet light is the effective technique for drying the varnishes; the sun is an excellent source for ultra-violet radiation for that purpose; but dependence on the sun limits drying operations and consequently varnishing only to times of favourable weather (Michelman, 1946). As drying of linseed oil varnishes takes a long time, UV is used to accelerate the drying process. Makers use different methods for this step. Here, the drying process was performed according to the advices of a professional instrument maker, Nicolas Gilles. So, drying was done as it has been explained above. UV box was made by Pierre Cabrolier in Montpellier (Fig. C.9).

Fig. C.9 UV box, made by Pierre Cabrollier

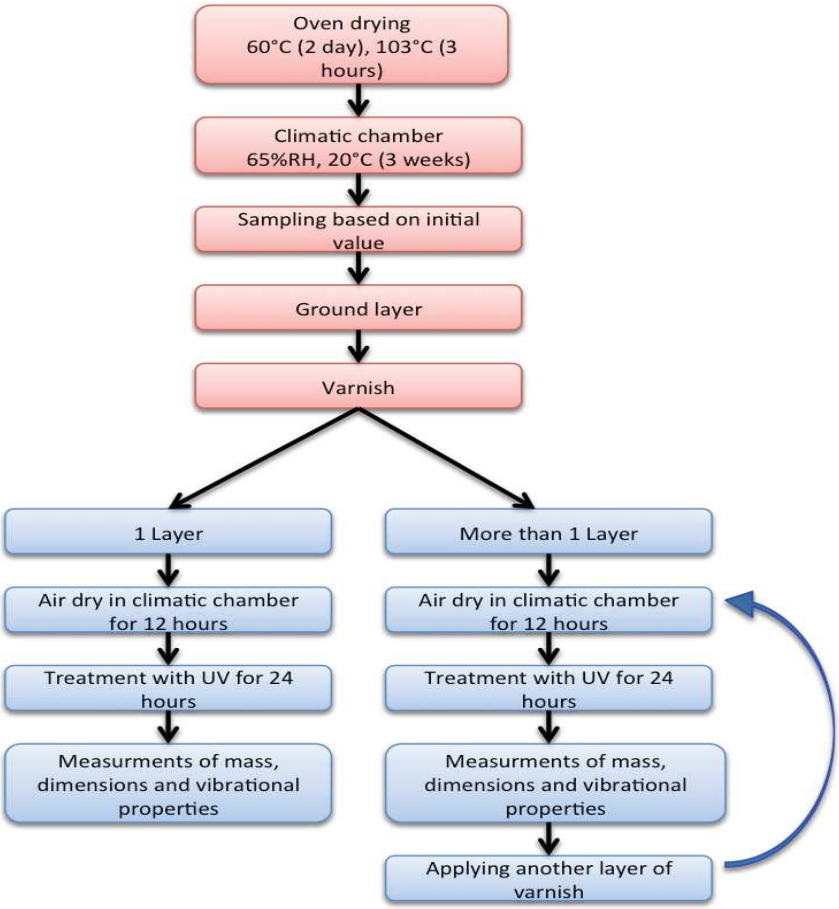


Fig. C.10 Illustration of the process of varnishing for Spruce specimens

C.3.3 Results

Surface treatment for Spruce specimens was done in two steps:

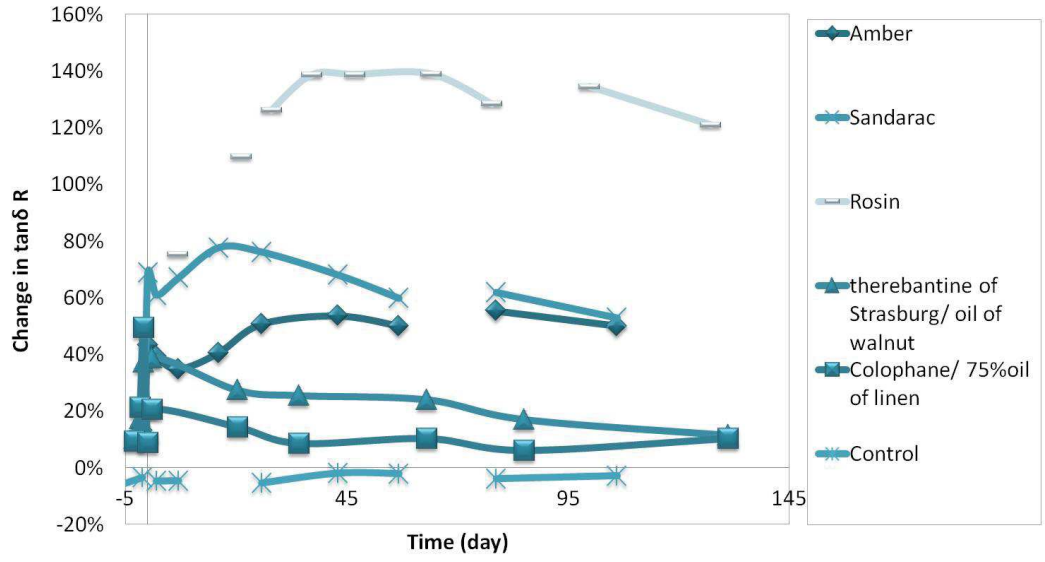
- Preliminary test, which was performed to achieve basic information. According to the basic information it will be possible to find how the process of varnishing, drying and changes in properties is.
- Main test, which was performed according to the basic information from preliminary test.

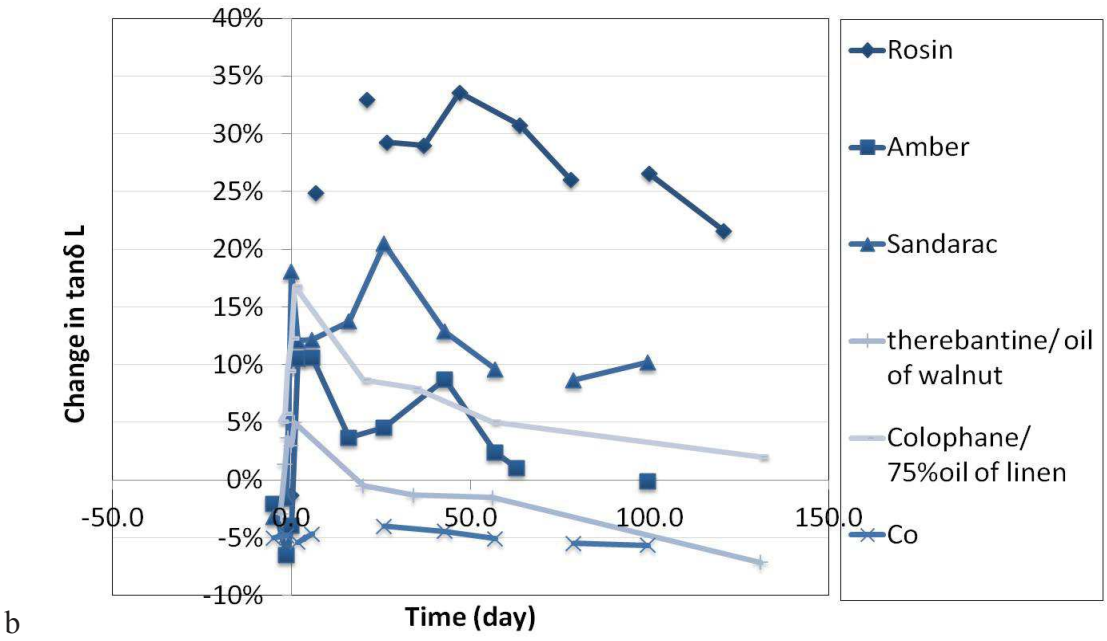
According to table 8 and 10, 2 kinds ("Térébentine de Strasbourg" and rosin) then 3 kinds (Rosin, Amber and Sandarac) of siccative oil-based varnishes were used for preliminary and main test respectively.

The kinetic of changes in damping and mass during some month after applying varnish are shown here. Also, the relation between mass and damping is shown.

Fig. C.11 shows the kinetics of changes of damping in R (a) and L (b) directions during 4 months. The changes are related to the groups with 1 layer varnish of main and preliminary test. In Fig. C.11a, for all the varnishes there is a strong increase in the beginning, which is followed by a very gradual decrease afterwards. This decrement is continued during several monthes. In both radial and longitudinal direction, one Rosin varnish has the biggest change in damping, but this is because the applied layer was too thick. Sandarac and Amber have the same trend. Two varnishes of preliminary test, Térébentine of Strasbourg and Colophane, have the smallest effect. These varnish were also more diluted (by turpentine as solvent).

a





b

Fig. C.11 Variation of damping in R (a) and L (b) direction during time after application of a single layer of 5 different oil varnishes.

Fig. C.12. show the change of mass for 1-layer treatments. There is an increase in the beginning for all the treatments, which is because of the added mass of varnish. Then it became stable for all the treatments. In R direction (a), the biggest change is for Rosin which had been applied too thick (11%) and the smallest change is for two varnishes of preliminary test (between 3 and 4 %).

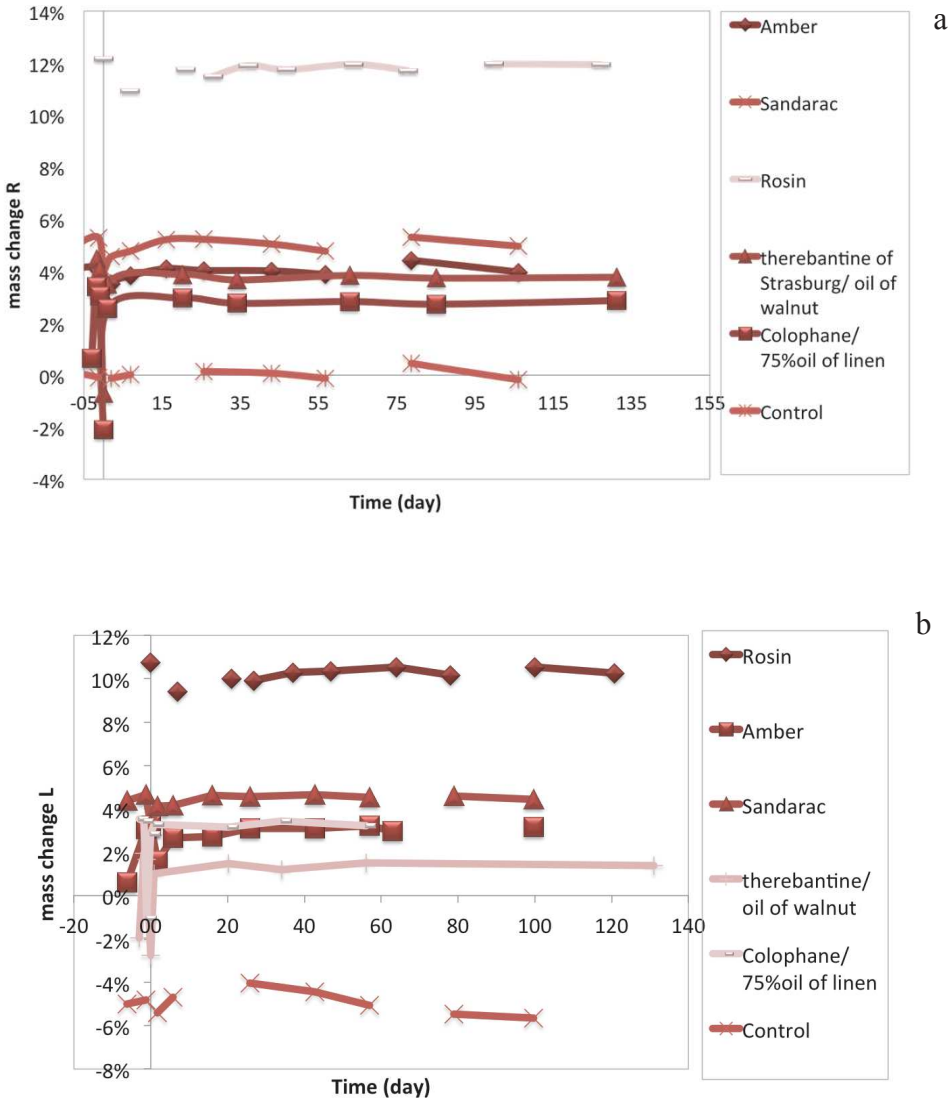
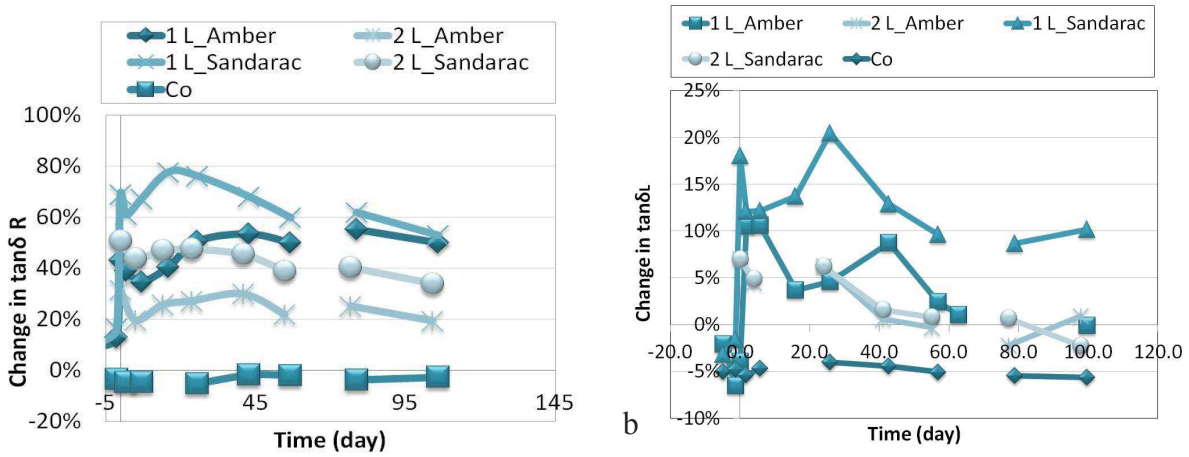


Fig. C.12 Variation of mass in R (a) and L (b) direction.

In Fig. C.11b, for L direction, the trend of changes in damping, according to time and according to different varnishes, is the comparable to trends in R direction. But the amplitude of changes in damping are much lass in L direction, approximately 4 times smaller.

Fig. C.13 compares 1 layer and 2 layers treatments and 2 different varnishes. Fig a and b are the changes in damping and c and d are the changes in mass.

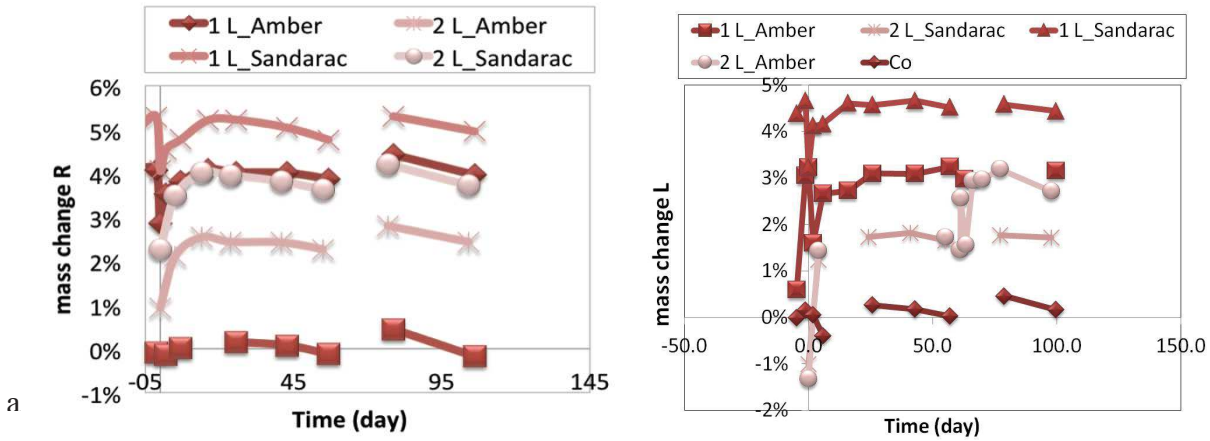
In Fig. C.13a and b, 1 layer varnish of Amber and Sandarac have the biggest change in damping in the beginning. For 2 layers of varnish the additional changes are smaller. For all the treatments, after less than one month the trends are a re-decrease.



a b

Fig. C.13 Variation of damping in R (a) and L (b) direction due to the application or 1 or 2 layers.

In Fig. C.14a and b, the biggest changes in mass are for 1 layer varnishes. 2 layers treatments exhibit smaller additional changes. For all the treatments after less than 1-month the mass decreases a little and they tend to become constant.



a

Fig. C.14 Compare between change of mass in R (a) and L (b) direction for 1 layer and 2 layer treatments.

Fig. C.15a,b,c and d, are the relation of mass variation and damping for L (a and c) and R (b and d) directions. For R direction (b and d), the linear relation is clear. For L direction the relation for 1 layer varnish (c) it is linear while for 2 layers (a) it is less linear.

a

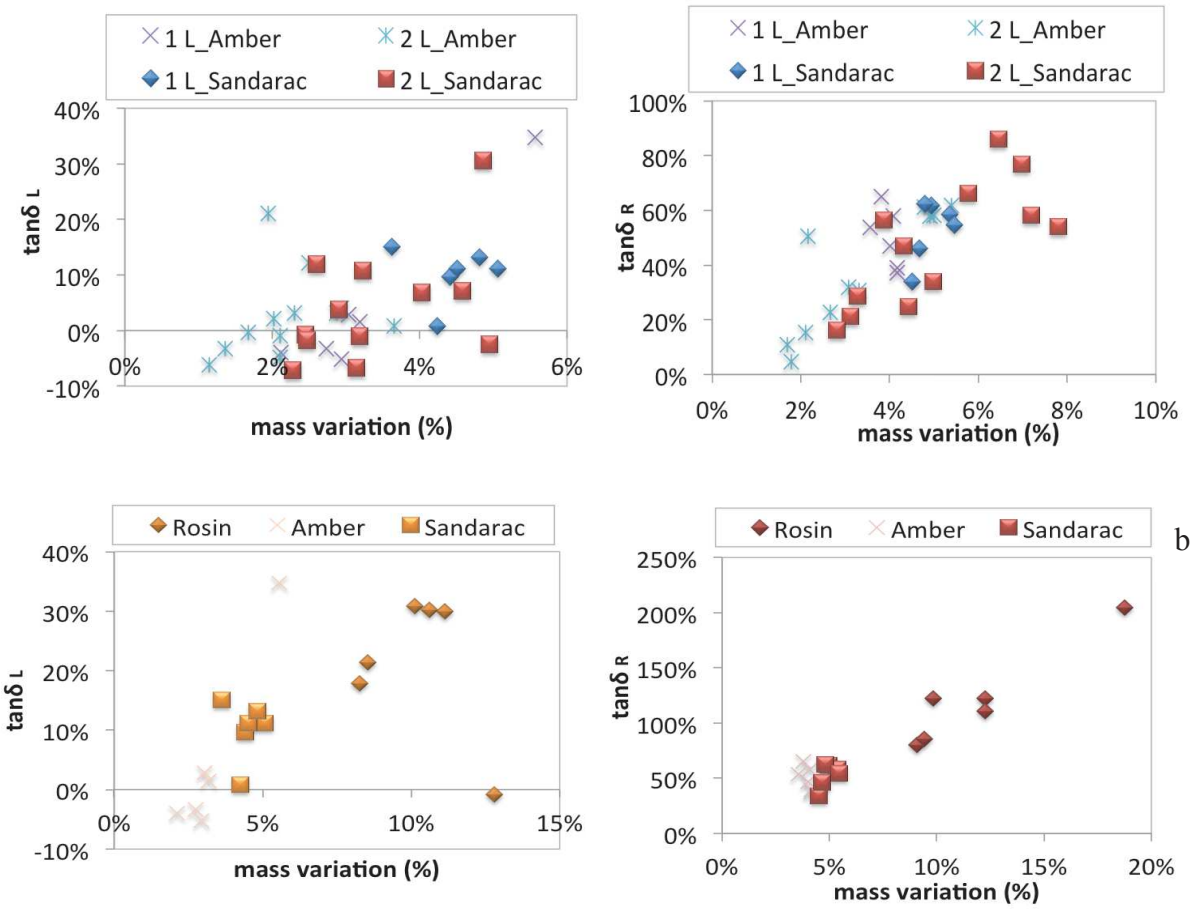


Fig. C.15 Variation of mass versus damping variation in R (a) and L (b) direction, for 1 layer and 2 layers treatments.

C.3.3. Conclusion

The results of oil-based varnishing for Spruce specimens showed some interesting changes in damping with time. The represented results here are a part of a wider study, which can be completed during the coming years. So, we just focused on the results achieved in the first year because there is a limitation in the time to finish this thesis.

According to the primary results, for 1-layer varnishes, for all varnishes, there is an increase in damping in the first weeks of varnishing. Then it starts decreasing. For some of them like Amber, Rosin and Sandarac (main test) it is still decreasing and for some others like Therebantine and Colophane (primary test) it becomes stable.

For all the treatments, there is an increase in the mass, which is because of the added varnish, and then it decreases a little and becomes stable. It can be due to the polymerisation of the varnish.

The kinetics of changes wer clearly much slower for the oil-based varnishes than in the previous experiments about Iranian solvent-based varnishes.

The kinetics of changes can be followed after this study. As there was not enough time to follow the procedure of drying varnish until all the variation became stable, it can be the next step to see the results better and to be able to discuss the results more in-depth.

D. CONCLUSIONS AND PERSPECTIVES

D.1 Return to the study

Our study was based on two main objectives. We focused on the wood which is used for making European and Iranian musical instruments.

The first objective was to investigate the mechanical and physical effects of pre-treatments on the wood which is used for making musical instruments. The effect of mild thermal treatments on the different properties of wood was investigated. Thermal treatment can be considered as an accelerated aging also. According to the literature, accelerated aged wood can have some similar changes in properties as aged wood. So, it can concluded that the effect of accelerated aging on the properties of wood would be achieved and can be discussed in relation to musical instruments.

Thermal treatments in this case can be also considered as pre-treatments that might be employed by instrument makers. Therefore, we could find what will be the resulting changes in physical and especially acoustical properties.

The second objective was to recognize the effect of surface treatments on the physical and mechanical properties of wood used in musical instruments. As we decided to work on both European and Iranian wood used for making musical instruments, European and Iranian common coating recipes were selected for surface treatment. Furthermore, the effect of time on the properties of varnished wood was remarkable.

We studied two species Spruce and Morus which are used for making European and Iranian traditional musical instruments respectively. Spruce samples were from preselected quality as for instrument making. Morus sample from Iran was selected by a musical instrument maker.

Both species were chosen according to criteria properties of wood for musical instruments that should be with fine structure and absence of any defects in wood.

HTT (hydrothermal treatment) and TT (thermal treatment) were performed on Spruce in collaboration with Bienne University of Applied Sciences, Switzerland, and on Morus in CIRAD Montpellier.

After both TT and HTT, a reconditioning was applied to see the reversibility of changes. Physical properties like mass change, shrinkage and swelling, density and EMC were calculated by measuring mass and dimension of specimens before and after treatments. In the case of musical instruments, damping and specific modulus of elasticity are important properties. So they were obtained by vibrational test (non-contact forced vibration test on free-free beams).

The interaction between moisture content of wood and its vibrational properties has decisive role on qualification of acoustical properties of an instrument. Hence, after obtaining all the result we tried to find the relation between different properties.

For the surface treatments, different European and Iranian recipes was applied with the help of experts and professional makers in France and Iran. Surface treatments were performed on Morus and Spruce specimens, then physical and mechanical properties were observed and the change in properties were monitored along time of drying.

By varnishing, change in acoustical properties would be achieved. For European varnishes which are oil based, after polymerisation of varnish the effect of varnish on acoustical properties can be clear. So, after applying varnish, properties were measured at first every day, then every week and after every month. This monitoring is still continued. The obtained result is just some month after varnishing. So, because of limited time in this study, the result of this part is just a part of this project.

D.2 Conclusion according to our objectives

As our study was formed based on two defined objective, here we discuss the obtained result and its correlation to our objectives separately.

By the first objective we tried to do some TT and HTT experiments on two main species for making musical instruments in Europe and Iran.

By TT on Morus specimens EMC decreased and damping decreased a little in the beginning of the treatment process and then there was a small increase again. But totally, there is still a small decrease in damping.

By HTT, the highest change in properties are obtained for strongest condition of treatment. Decrease in lightness was obtained while this decrease is enhanced by treatment duration and treatment temperature. There was an increase in specific modulus of elasticity for all the treatments with a significant decrease in damping which was higher for radial direction.

It could be concluded that by both TT and HTT there was an improvement in acoustical properties of wood based on decrease in damping and also these treatments could improve dimensional stability.

Hence, recovery was performed to be sure about the reversibility of changes after treatments and to know if there is chemical or physical changes in wood construction. For both TT and HTT, after recovery some parts of changes was reversible, but not all. It may be speculated that by doing another recovery maybe additional part of the changes could be recovered, or on the contrary it would highlight the amount of irreversible part. It can be suggested for future work.

So, according to our first objective we find these treatments useful which can improve some physical and acoustical properties of wood, but the reversible part of chanes must be taken into account. By future work and more recovery on this kind of treatments we can confirm the obtained results.

The second part of the work showed also interesting results. Applying different coatings for two different species was an interesting experience: at the beginning it seemed very easy but in fact it was not. It requires much precision and experience. During our experiment on Morus specimens by Iranian varnish, the difference between professional and not professional application was compared. Also, different number of layers has been applied to see the effect of different coating systems by different makers.

By Iranian varnishes (alcohol based), for both applying by a person and by a professional one, for damping in the first hours and weeks there is an increase while after it decreases again and after around 7 months the damping of coated wood became close to before varnishing. So, it can be concluded that by applying such type of varnish there is little final effect on damping, but a small stiffening effect is observed. As the Morus wood has naturally quite low specific modulus of elasticity and quite high damping, the application of such type of varnish on such type of wood appears a logical wood+coating system which is traditionally used by Iranian musical instruments makers.

For Spruce and for different types of oil-based varnishes, we also found an increase in damping and mass changes at first. After some months and with polymerisation of varnishes, damping was slowly decreased again but the trend was much more slow than with the solvent-based varnishes. For some of the applied varnishes like Amber, Rosin and Sandarac (main test) it is still decreasing and for some other like Therebantine and Colophane (primary test) it starts to become stable after some months. Because this part of study needs more time to

obtain clear effect of varnishes, and we did not have enough time, the results obtained up to now cannot be the final result and they need more time and experiments to be concluded perfectly.

D.3 Perspectives

By this study we could find answer for some part of our questions based on objectives. Decrease in damping and moisture content which were resulted from the (hygro-)thermal treatments could be considered as an improvement in wood properties of musical instruments.

In the case of TT, more applied recovery can give clearer results. For surface treatments, because of the limited time we could get just some months of the changes after treatments while, it needs more time to follow the changes and find the effects.

For surface treatment, between Iranian recipes we chose some commonly used ones. The problem is the lack in literature about Iranian varnishes. This study provided data with clear results. According to the obtained results, future works on Iranian musical instruments and surface coatings can represent some new aspect of study in this field.

As by applying reconditioning the reversibility of changes induced by thermal treatments could be investigated so it would be interesting to apply more recovery cycles. Here, with one time recovery, a part of changes in both TT and HTT were recovered. It can be attributed to reversible structural changes. But if the reversibility cannot be achieved with more recovery cycles, it would suggest the occurrence of irreversible chemical changes in wood structure, such as degradation. Therefore, by chemical analysis the chemical component of treated samples can be investigated. This kind of analysis can be considered as a complement to discuss the results with more deep basis.

Our result on TT treatment can be considered as an accelerated aging. Study on aged wood and modelling on accelerated aged wood by obtained result can give us a different view. Considering TT as accelerated aged wood can open a new view to survey if aged wood in musical instruments can affect their acoustical properties.

This study helps to answer to most parts of our objectives. In the case of European musical instruments made by Spruce (violin), surface, oil-based coating is able to clearly modify the acoustical properties, usually by increasing the damping. However, this part of experiments needs more time to obtain final results. But about wood coating used for Iranian musical instruments, the obtained results are clear and show that using some traditional solvent-based varnish can significantly stiffen wood without much change in damping.

TT and HTT treatments which can be considered as pre-treatment could reduce damping for both Spruce and Morus specimens. Therefore, by pre-treatments we could find improvement in acoustical properties of wood which can be considered also as accelerated aging on wood. But it would be interesting to check other kinds of complementary properties on such treated wood, to check if there is no fragility of no difficulty for woodworking.

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Effet de traitements thermiques modérés et de revêtement sur les propriétés vibratoires des bois d'Epicéa et de Mûrier

Le bois est couramment utilisé pour la fabrication d'instruments de musique. Les procédés employés consistent souvent en traitements modifiant le matériau en volume ou en surface. Ce travail s'est focalisé sur deux espèces employées dans les instruments à cordes et représentatives de différentes cultures: L'épicéa (Picea abies Karst.) utilisé en Europe et le Mûrier blanc (Morus alba L.) utilisé en Iran. Pour chacune l'effet d'un traitement thermique modéré (<150°C) et d'un revêtement par vernis sur plusieurs propriétés physiques et mécaniques ont été étudiés. Les principaux résultats sont les suivants. A la différence de l'Epicéa, le Mûrier présente un très faible degré d'anisotropie mécanique. Chez les deux espèces, le traitement thermique entraîne une forte chute dde l'amortissement, particulièrement dans la direction radiale pour l'Epicéa, ainsi que du taux d'humidité d'équilibre, sans dégradation marquée comme indiqué par la faible perte de masse. Toutefois, après reconditionnement à haute humidité, une part importante de la modification est récupérée. L'application d'un vernis à base solvent sur le Mûrier entraîne une rigidification continue, tandis que la forte augmentation de l'amortissement observée après l'application est suivie par un retour au bout d'environ 2 mois aux niveau du bois non traité. Pour l'Epicéa, des vernis à base d'huile siccative ont été appliqués et divers paramètres du procédé ont été testé. Dans ce cas la cintétique de stabilisation des propriétés est très lente et des variations notables continuaient à être observée au bout de 5 mois.

Effet of mild thermal treatment and of coating on the vibrational properties of Spruce and Mulberry wood

Wood is commonly used for making musical instruments. During the process it is often subjected to treatments, that either modify its volume or its surface properties.

Two species used for for string instruments were studied, representative of different cultures: Spruce (Picea abies Karst.) used in Europe and White Mulberry (Morus alba L.) used in Iran. For each of them the effect of thermal treatment at moderate temperature (<150°C) and of coating on various physical and mechanical properties was studied. The main results are as follows. In contrast to Spruce, Morus has a very low degree of mechanical anisotropy. For both species, thermal treatment induces a strong decrease in damping, especially in R direction for Spruce, and equilibrium moisture content, without marked degradation as indicated by the very small weight loss. However, after reconditioning at high humidity, a significant part of the changes is recovered. The application of a solvent-based varnish on Morus induces a continuous stiffening, while a very strong increase in damping after application is followed, after about 2 months, by a return to values close to those of untreated wood. For Spruce, siccative oil based varnish was applied and several parameters of the process were tested. In this case, the kinetics of property stabilisation are very slow and significant changes were still observable after 5 months.