WORKSHOP

Enhanced production of edibles from forests and orchards

November 12th & 13th, 2019Tunis (Tunisia)

Effect on the durability, chemical modifications and mechanical properties of the heat treatment on some Tunisian softwood species

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Introduction

Issue of biological activity

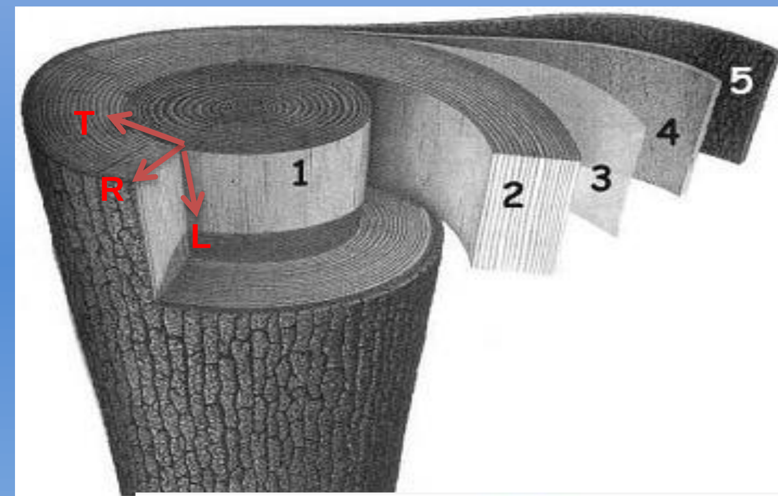
- Heterogeneous
- Anisotropic
- Hygroscopiqic
- Porous
- Permeable

Function

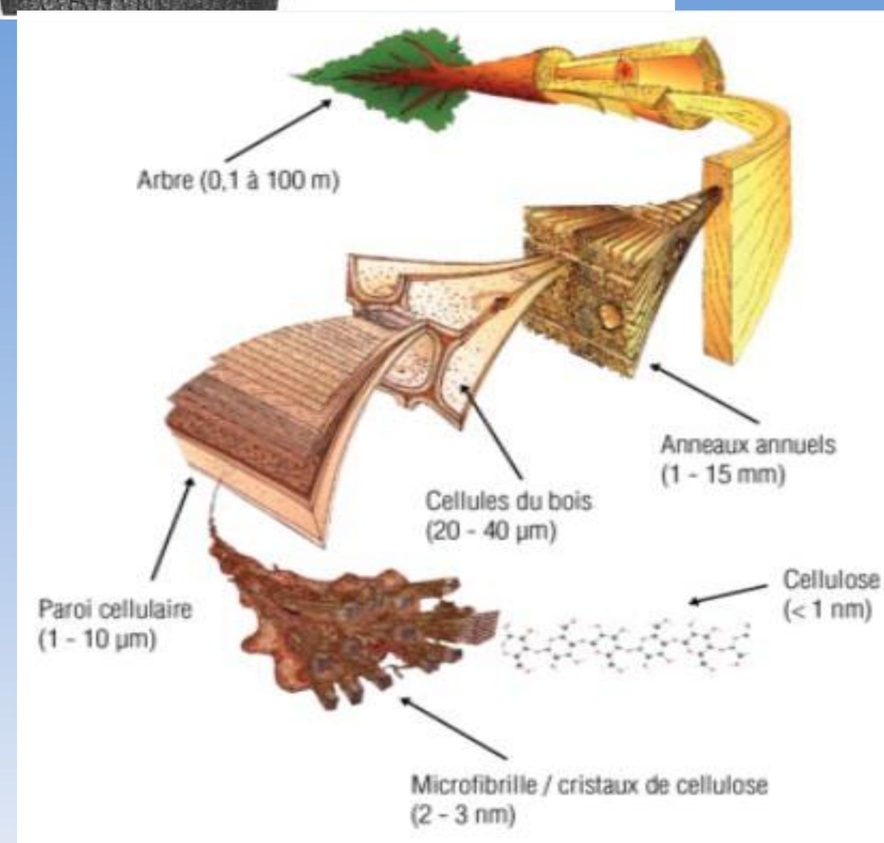
- Conduction
- Storage
- Mechanical support

Chemical composition

- Cellulose (45 ± 2%)
- Hemicellulose (30± 5%)
- Lignin (25 ± 3%)



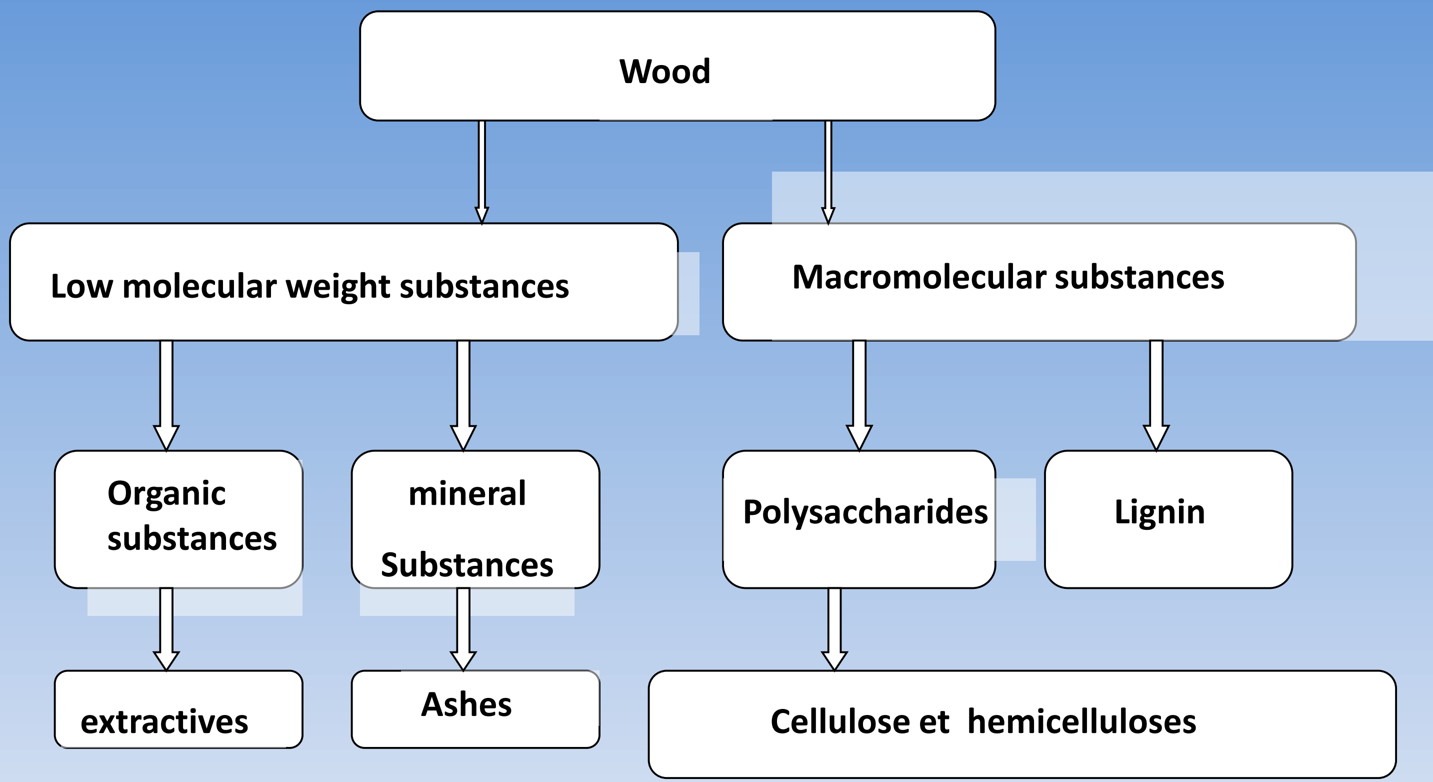
1.Heartwood
2. Sapwood
3.Cambium
4. Phloem
5. Bark



Introduction

The Wood Materials: Chemical Composition

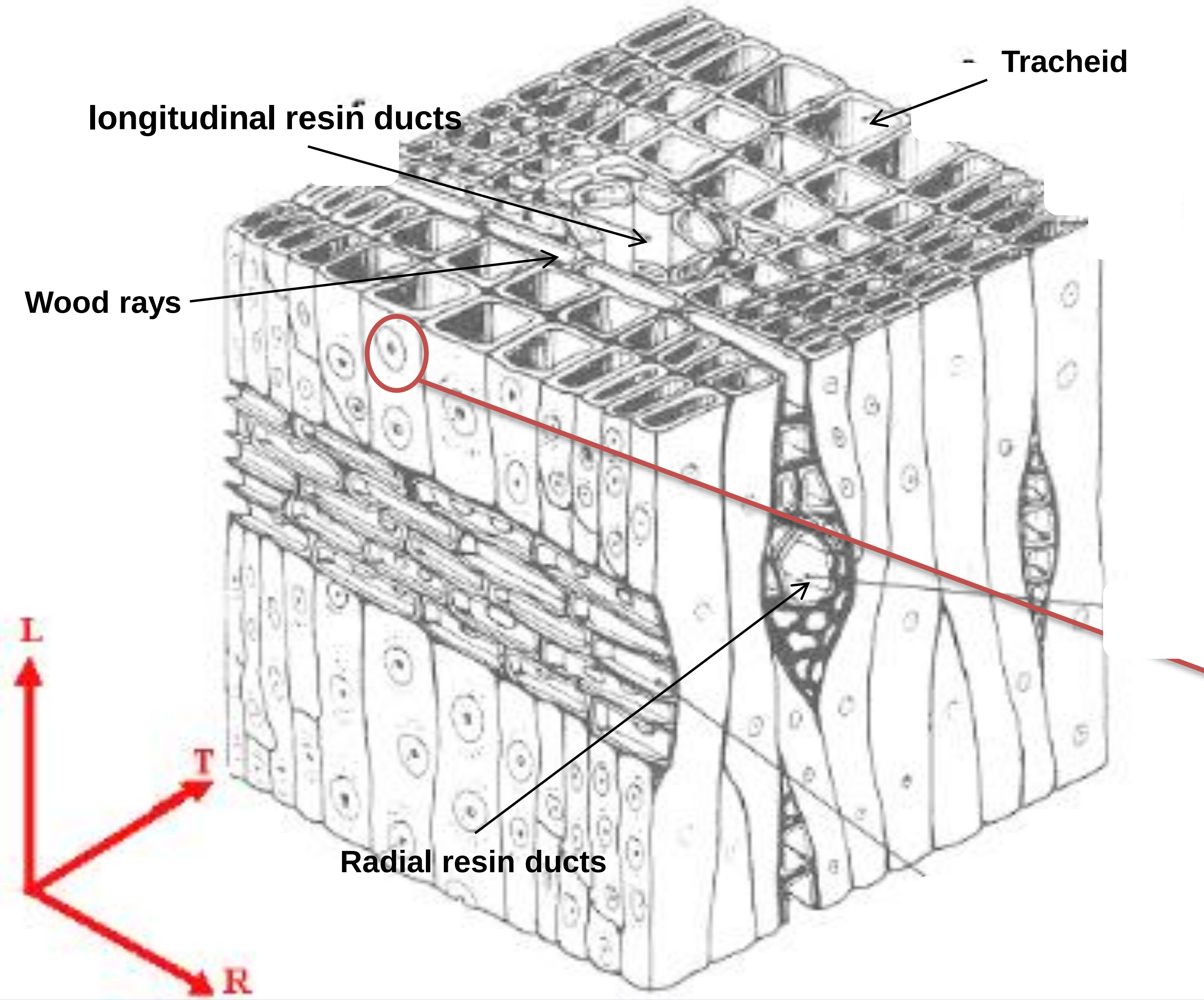
The elemental chemical composition of the organic material of the wood varies little from one species to another. On average it is distributed as a percentage of dry weight, as follows:
Carbon: 50%, Oxygen: 43%, Hydrogen: 6%, Nitrogen: 1%, Ashes (silica, phosphates, potassium, calcium) <1%.



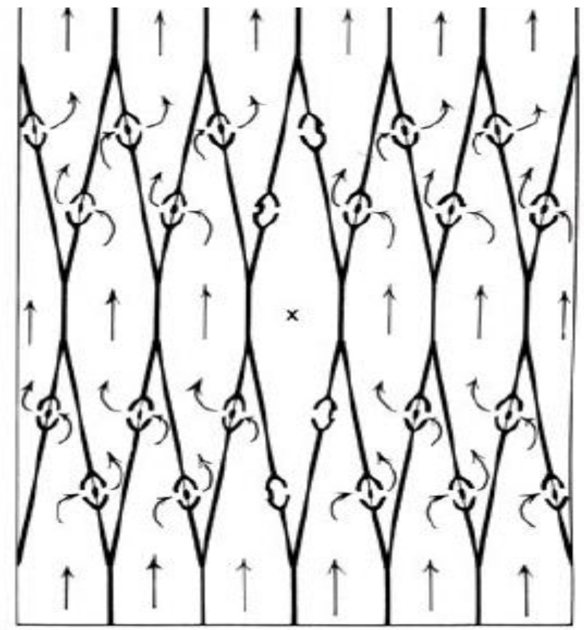
Schematic representation of the chemical constituents of wood(Haluk 1994)

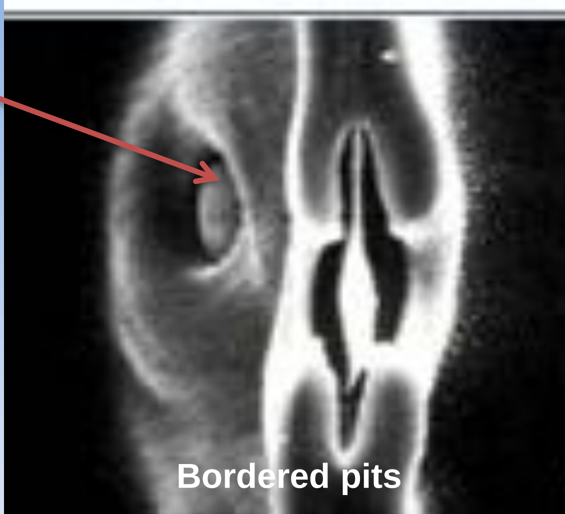
Introduction

Where does water flow in the wood?

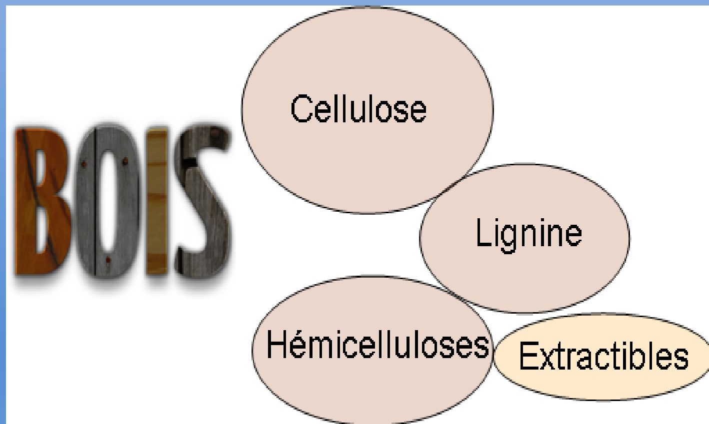


Model of Sap Flow in Softwoods (Zimmermann, 1983





Introduction



Its implementation leads to its exposure to abiotic and biotic degradation agents

- **durability: variable according to species**

Need to protect the wood material (little or no durable species)

Brown rot

White rot

Impregnability- Autoclaving

Process for impregnating dry wood with preservatives

Introduction
Wood preservation

Preservatives based on the use of biocides

3 big families :

- Tars and oils
- Organic products: - PCP (pentachlorophénol) (organochloré)
  - Triazoles (Acaconazole, propiconazole, tébuconazole, cyproconazole…)
  - Carbamates (IPBC...)
  - Ammonium quaternaires (DDAC...)
- Mineral productx: - Multisel formulations (CCA, CCB…)
  - Boron derivatives (boric acid, borax ...)

Problème de toxicité pour certains produits

Directive biocide (98/8/CE)

- Increasing environmental pressures
- New opportunities for the development of more environmentally friendly wood preservation methods

Research of new preservation processes for the wood material

- **Chemical modification**
- **New wood-based materials**
- **Development of biocides respectful of the environment**
- **thermal modification**

Heat treatment of wood

Huile

N_2

H_2O

heat treated wood

Native wood

180°C<T<250°C 20h<t<60h

«Thermowood» Process

«OHT» Process

«Plato» Process

✓ **Good fit with new emerging market trends based on eco-products and eco-design**

Physicochemical modifications of heat-treated wood

Chemical modification of wood polymers by heat treatment

- **Modified lignin polymer network**
- **Ratio between amorphous and crystalline cellulose is changed**
- **Hemicelluloses strongly degraded**

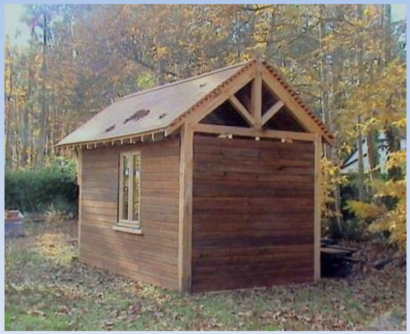
Advantage

*Duarability
* Dimensional stability
*hydrophobicity

Disadvantages

*Mechanical properties
*Reproducibility
*Process control

Use in Hazard class 3, where the wood is not in direct contact with the ground



Goals

1 - Improvement of the durability of the wood of some local species

2- Development of chemical markers for the prediction of ML

- **Study the relationship between the ratio O/C et ML et WL%.**
- **Evaluate the effect of treatment parameters on the mechanical properties of wood.**

MATERIALS AND METHODS

-Hard wood: *Zeen Oak*,

-Soft wood: *Aleppo pine, Maritime pine and Radiata pine.*

- Wood samples have been treated for three different mass losses: 8, 10 and 12%, at 200-230°C.

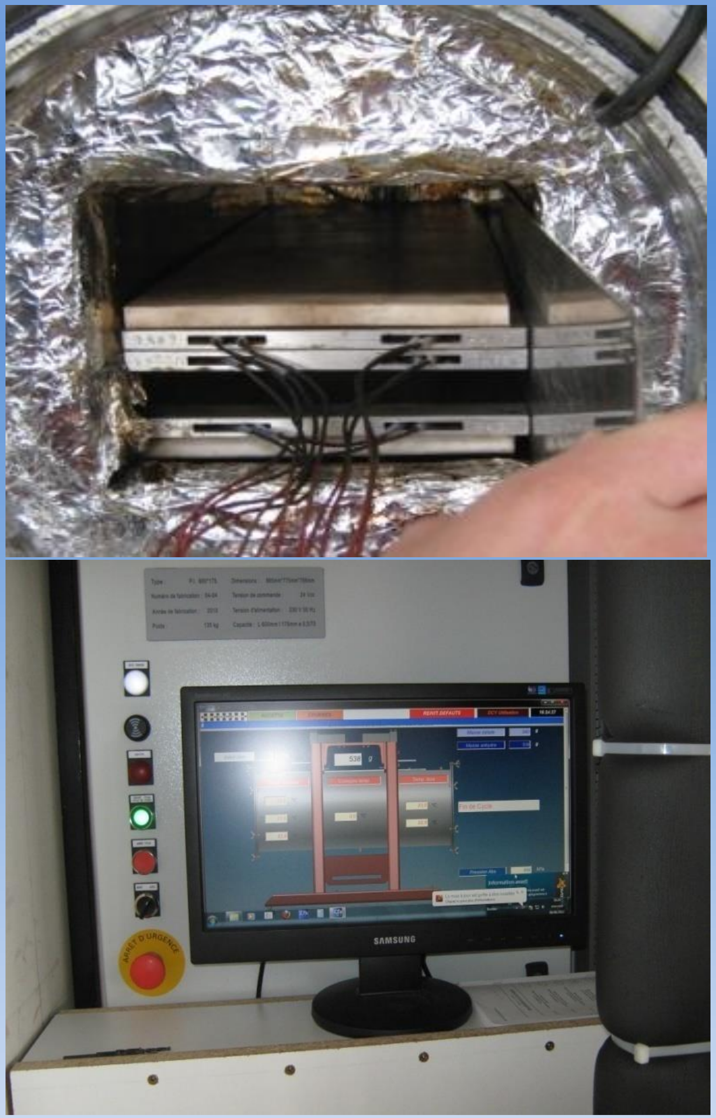
- For each treatment and each species, durability test, O/C ratio, bending and Brinell hardness have been realised.

> Heat treatment:

- Under vacuum atmosphere;
- by conduction between two metallic plates;
- Recording of dynamic weight loss;

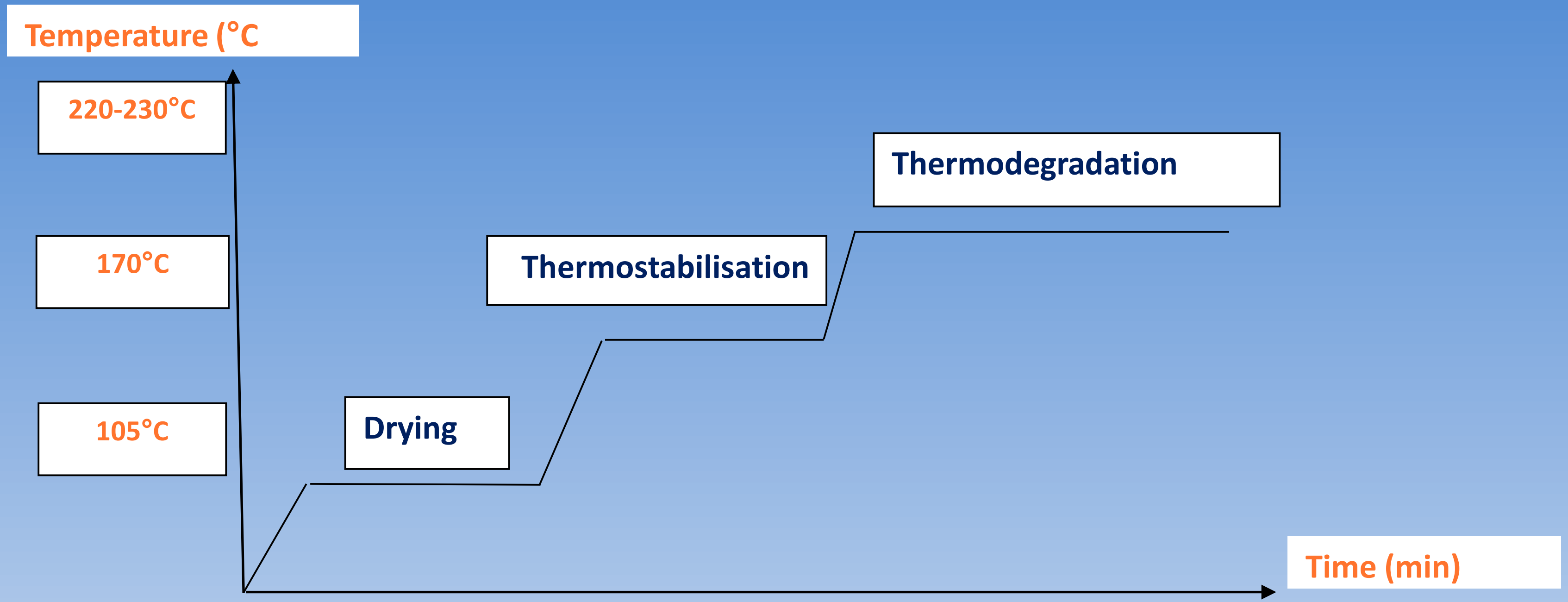
- Samples: 25*110*250 mm (x 2)

- Temperatures of treatment are included between 190°C and 220°C for different mass losses: 8, 10 and 12 %



Heat treatment process

MATERIALS AND METHODS



Temperature evolution to achieve thermal treatment.

MATERIALS AND METHODS

- > **O/C ratio:**

• Wood was grounded to fine sawdust and passed through different sieves to obtain a powder of granulometry comprised between 0.2 and 0.5 mm.

• Sawdust was: - conditioned at 103°C for 24 h
- stored in closed bottle before analysis.

• Elemental analyses were performed on a Thermofinnigam Flash EA1112 micro-analyzer.

MATERIALS AND METHODS

- > Decay resistance:

The decay resistance was tested on four different fungies:
Coriolus versicolor (CV), *Gloeophyllum trabeum* (GT), *Coniophora puteana* (CP) and *Poria placenta* (PP)

WL (%) = 100 x (m1 – m2)/ m1

- > Mecchanical properties:

=> Three point bending (MOE, MOR) and Brinell hardness were carried out for untreated samples and heat-treated samples, and the results were compared.

=> **INSTRON 4467** Universal Mechanical Test Machine was used for the measurements.

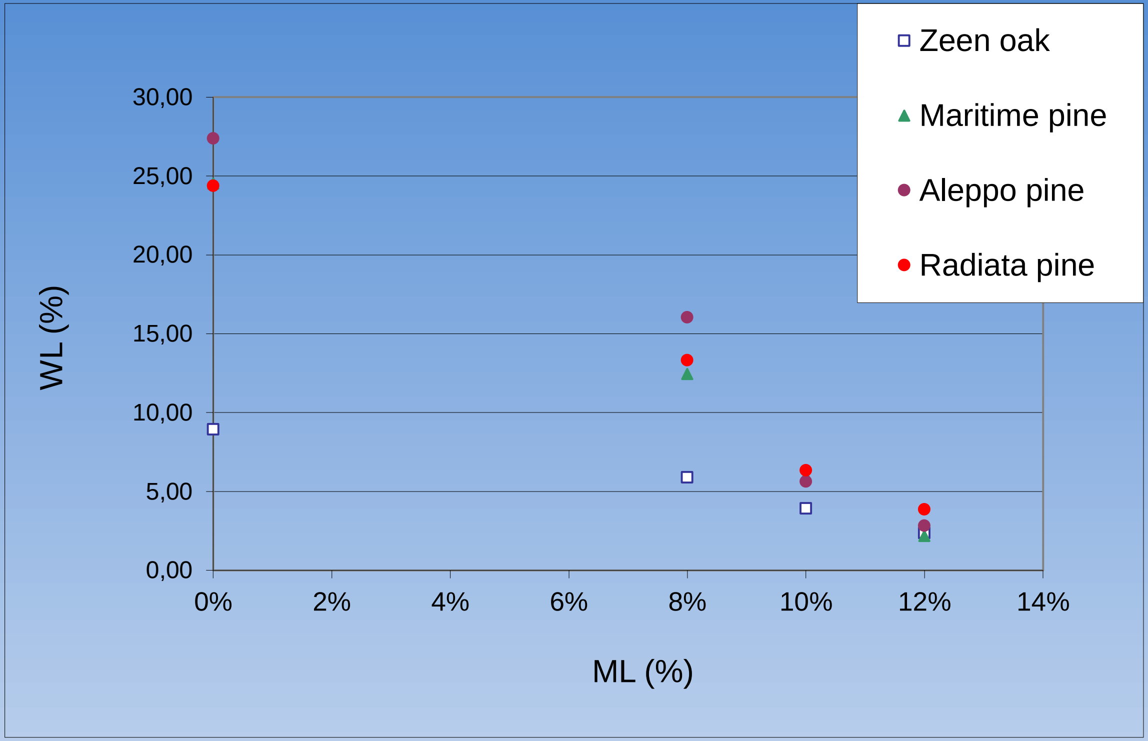
=> Three point static bending tests were carried out according to the **EN 408 (2003)** standard

=> Brinell hardness tests were performed in accordance to **EN 408 (2003)** standard

RESULTS

> Correlation between ML and WL:

<u>Fungies:</u> Coriolus versicolor **(CV),** Gloeophyllum trabeum **(GT),** Coniophora puteana **(CP),** Poria placenta **(PP).**
<u>Conditions:</u> at 22°C and 70% HR for 16 weeks.



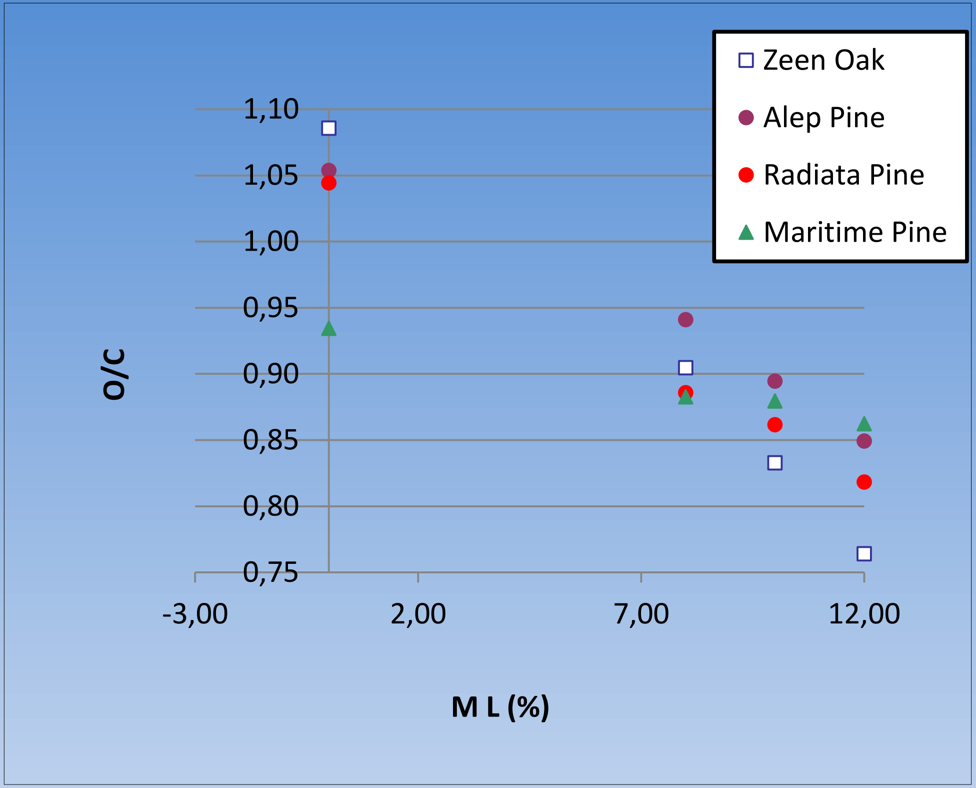
Correlation between mass loss due to thermo-degradation and weight loss after exposure to fungus (Poria placenta)

- **For all wood species, the weight losses (WL) due to fungal attack are correlated with mass losses resulting from thermo-degradation reactions (R2 > 0,92).**
- **The durability is almost complete when the Mass loss reached 12%.**

RESULTS

> Correlation between ML and O/C

<u>O/C:</u> Elemental analyses were performed on a Thermofinnigam Flash EA1112 micro-analyser.
Granulometry of dried sawdust of wood: 0.2 to 0.5 mm



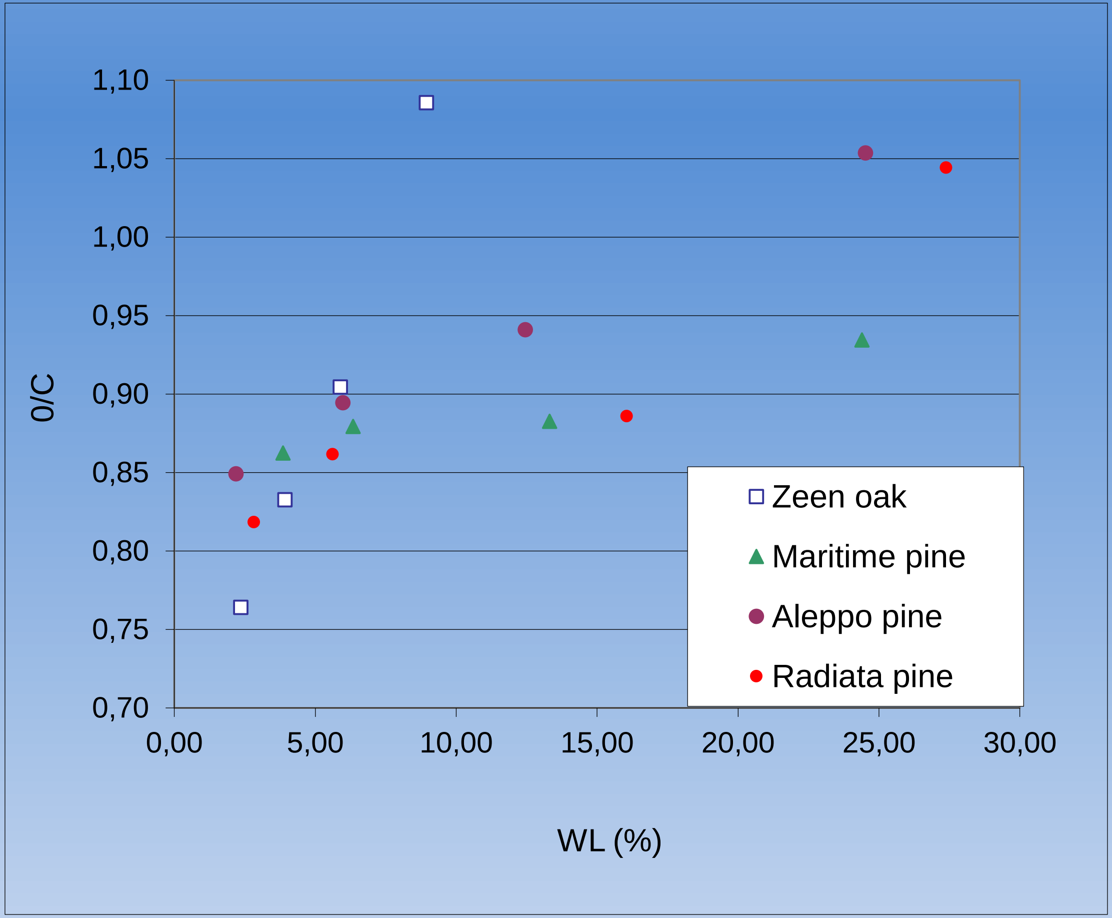
Correlation between mass loss due to thermo-degradation reactions and O/C ratio

- **O/C ratio decreases as the mass loss due to thermo-degradation reactions increases for all wood species.**
- **For each wood species, ML is strongly correlated to O/C ratio ($R2 > 0,9$).**

➡ O/C ratio could be a valuable marker of heat treatment intensity.

RESULTS

> Correlation between ML and O/C:



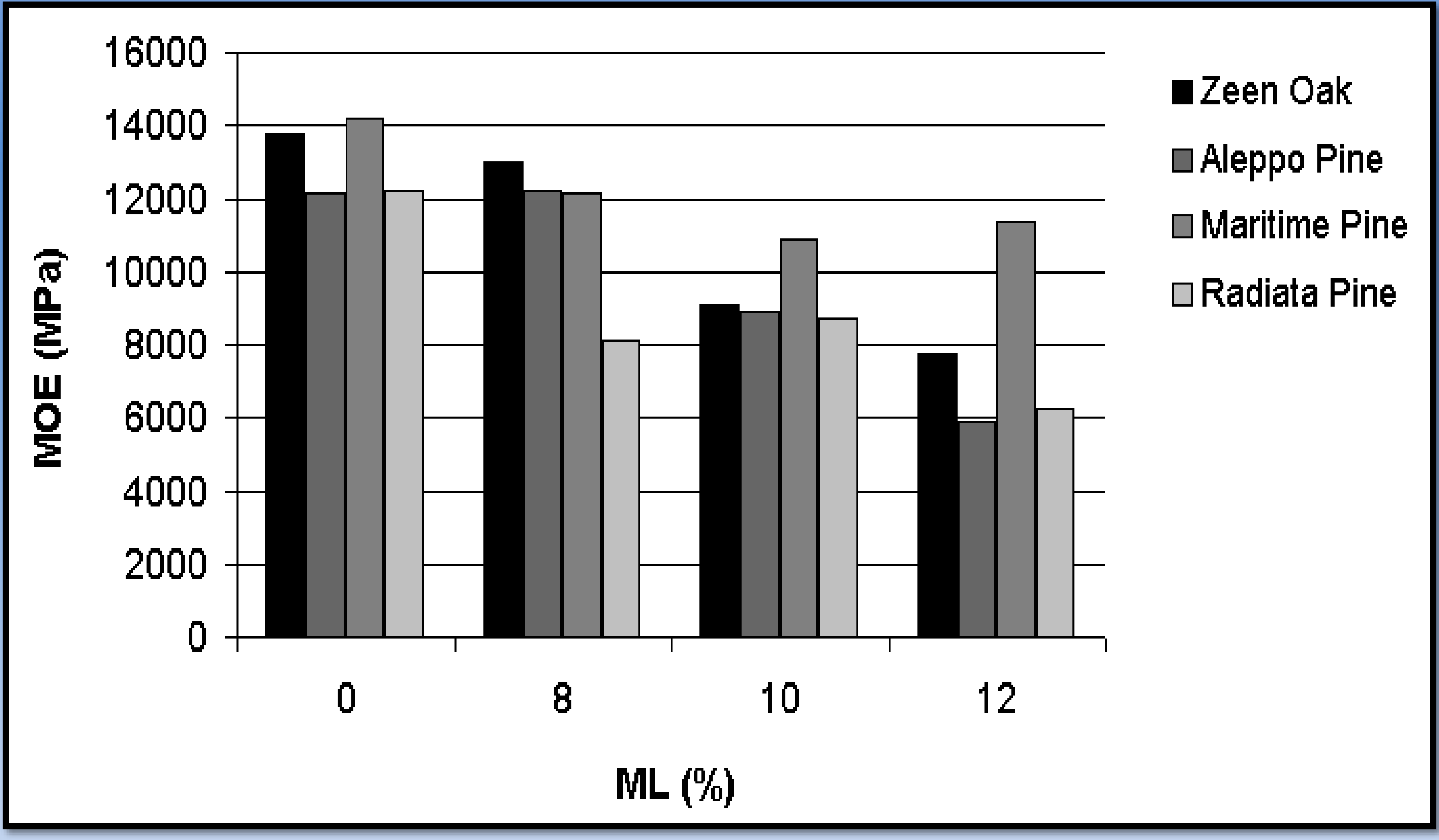
Correlation between O/C ratio and Weight loss after exposure to fungus (Poria placenta)

• O/C ratio is strongly correlated with weight loss due to fungal degradation (R2 > 0,90).

O/C ratio is a marker of the durability of 4 wood species.

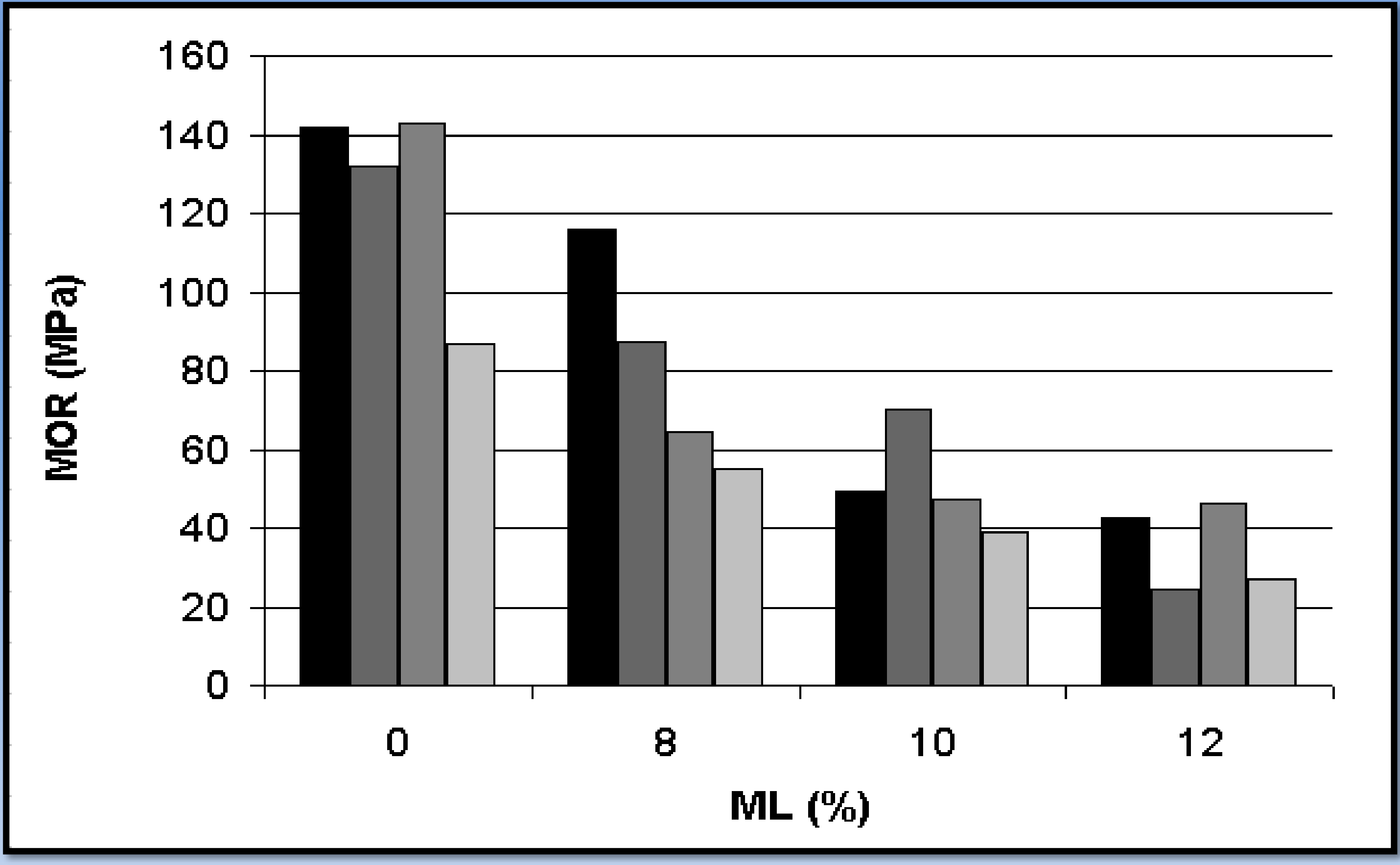
RESULTS

- Mecchanical properties:



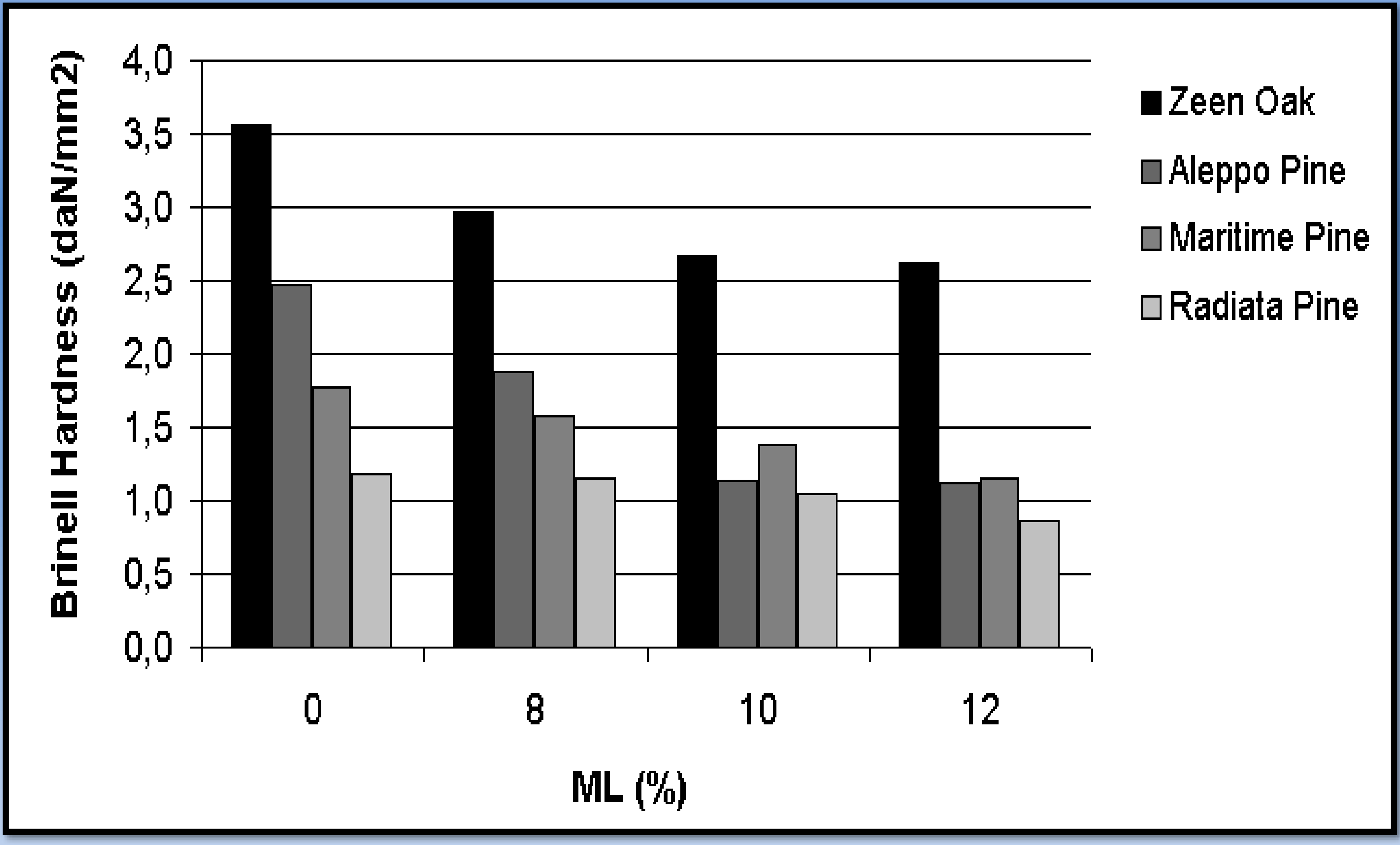
MOE evolution according to mass loses issued from thermal treatment for different Tunisian wood species.

RESULTS



MOR bending evolution according to mass loses issued from thermal treatment for different Tunisian wood species.

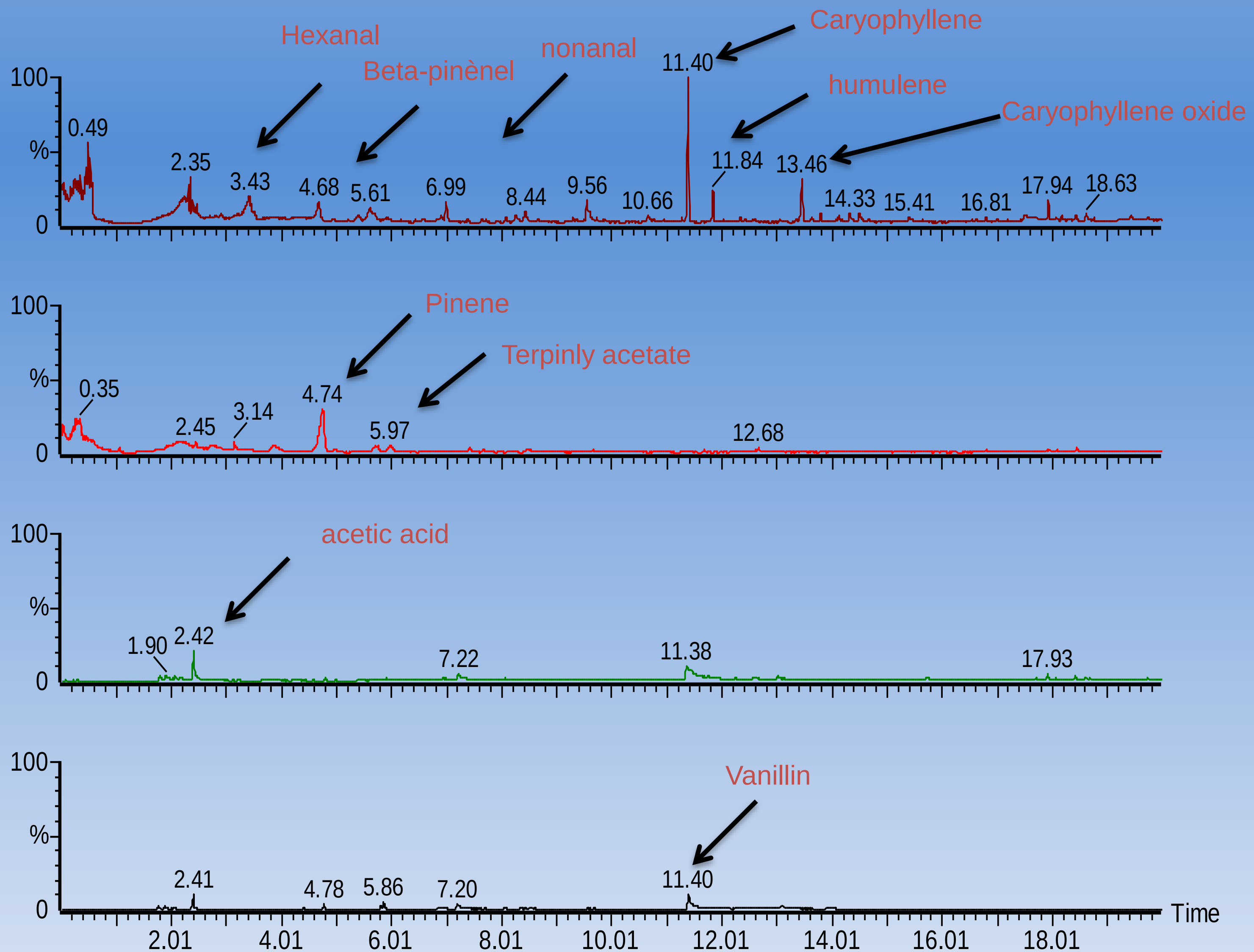
RESULTS



Hardness evolution according to mass loses issued from thermal treatment for different Tunisian wood species.

RESULTS

TD-GC-MS chromatograms according to heat treated Aleppo pine.



CONCLUSIONS

- **Results show that mass loss is an important parameter for the heat treatment control.**
- **Wood species plays an important role on the kinetic of mass loss. This influence can’t be explained by the thermal properties of different species.**
- **Correlations between weight losses recorded after fungal exposure and elemental composition indicated that carbon content and O/C ratio could be used to predict wood durability conferred by heat treatment.**
- **. MOE decreased insignificantly for all wood species samples having weight loss of less than 10% above which the decrease is more significant.**
- **MOR decreases dramatically starting from a mass loss due to thermo degradation of 8 %.**
- **Brinelll hardness is also affected by thermal treatment**

Thanks, for your attention !!!

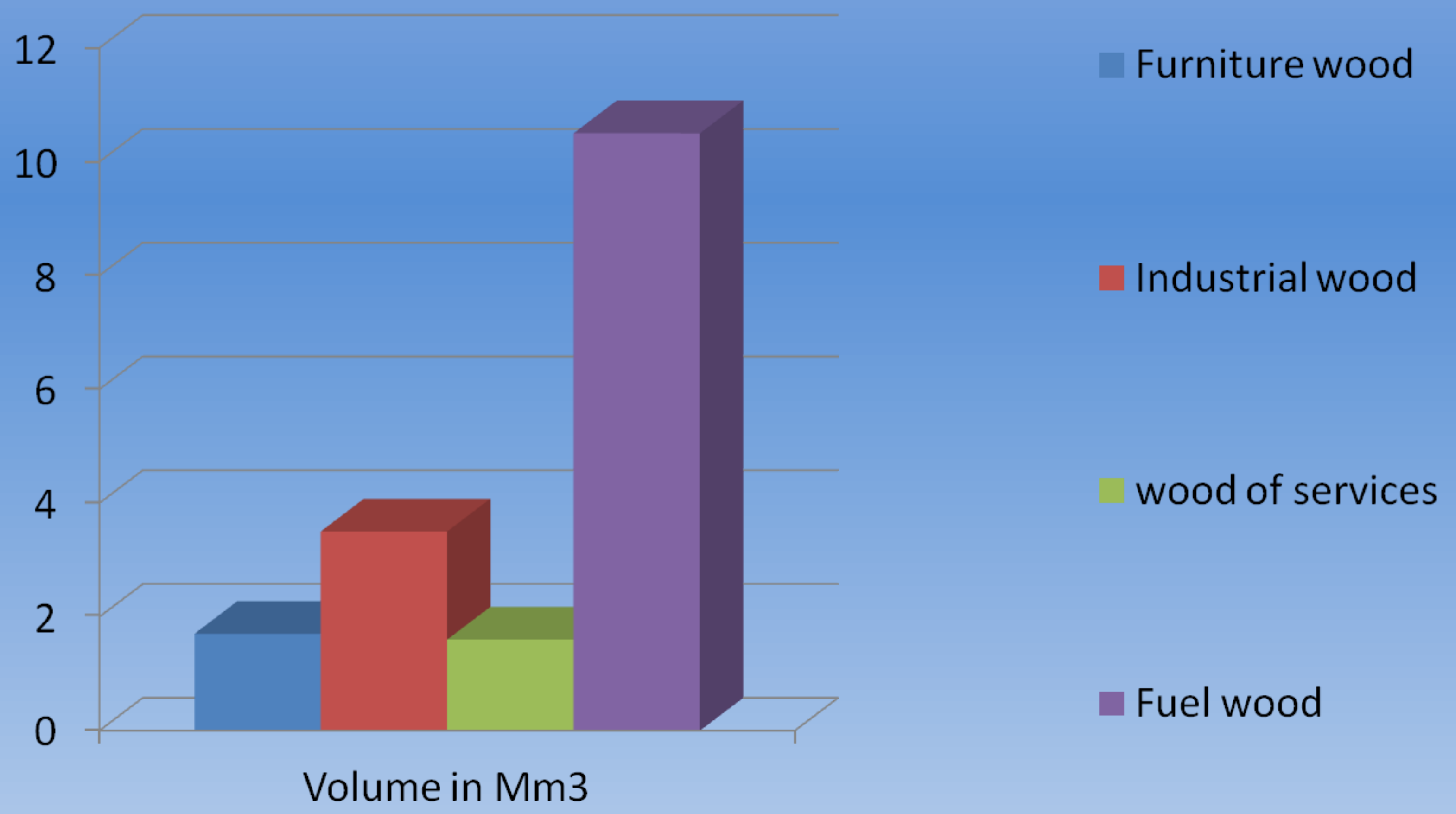
Aleppo pine wood Durability

WL (%) mean values, median values and Durability Class according to decay tests of Aleppo Pine

Fungi	Mean Mass Loss (%) Virulence controls (N=10)	Mass loss (%) – Aleppo Pine samples (N=32)			Classification (TS 15083-1)*
		Mean	Median	Standard deviation	
Gloeophyllum trabeum	48.23	47.39	47.39	4.61	Durability class 5 “Non durable”
Poria placenta	29.80	21.89	22.05	5.54	Durability class 4 “Slightly durable”
Coniophora puteana	38.74	35.22	34.65	4.52	Durability class 5 “Non durable”

Durability classes from TS 15083-1: 1-Very durable; 2 - Durable; 3- Moderately durable; 4 - Slightly durable; 5 - Non durable.

Introduction

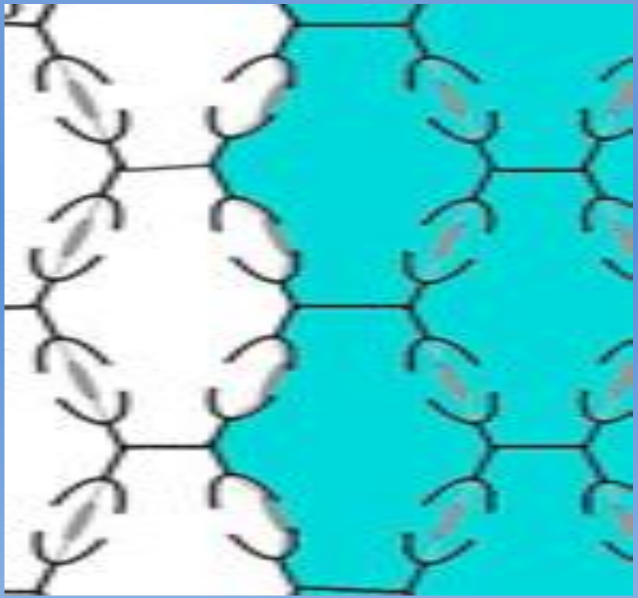


Distribution of the national timber production

Introduction

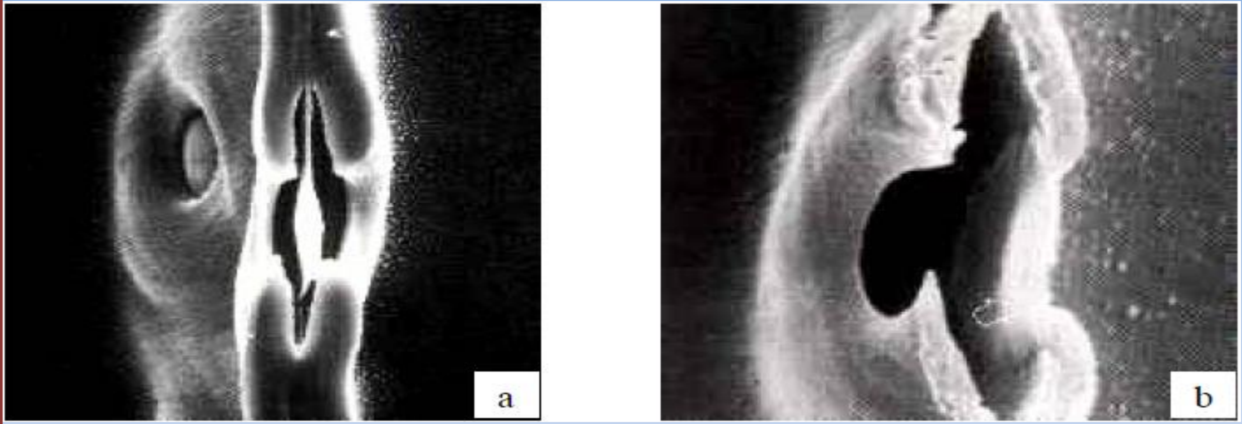
Closing and opening pits

- Consequence of an embolism following an injury, freezing ...
- Embolism: Appearance of air bubbles in conductive cells



Heartwood formation

- Chemical modifications (deposit of phenolic substances, disappearance of starch)
- Physical modification (blocked pit)



Bordered pit not aspirated (a) aspirated (b)

Improvement of Aleppo pine heartwood impregnability to water

Aleppo pine wood has two characteristics unfavorable to its peeling

- ➢ Heartwood dry in the green state(30-40%)
- ➢ Hertwood non-impregnatable

Dry wood + wood heterogeneity

Frequent wedging

Rapid blunting of cutting tools

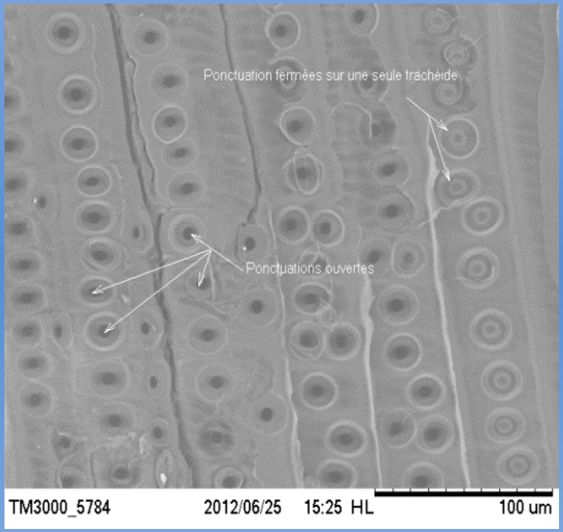
Bad quality + loss of material

Improvement of Aleppo pine heartwood impregnability to water

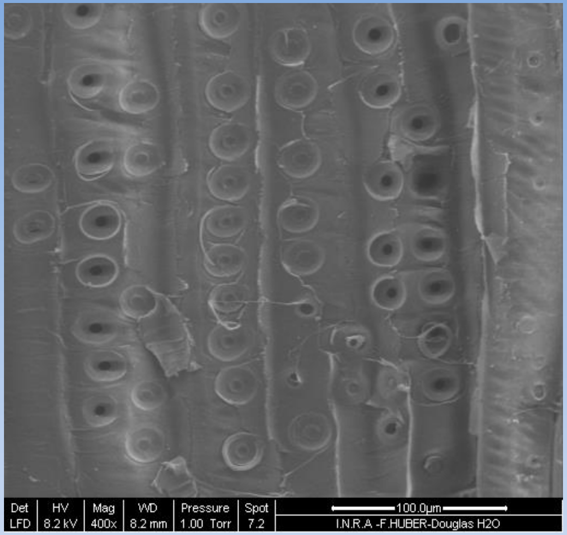
Differences from an anatomical point of view Heartwood-Sapwood

Environmental scanning electron microscope (SEM)
TM 300

Preparation of sample surfaces



Sapwood



Heratwood

In the green state, there are more open pits in the sapwood than in the heartwood.

Improvement of Aleppo pine heartwood impregnability to water

(1) et (2) Thermal drying in a ventilated dryer (3) Immersion of logs in water tanks

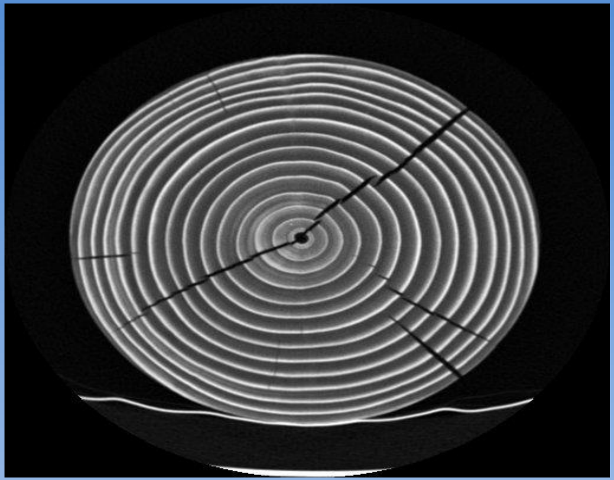
Water uptake

For all logs a weight gain is taken every 24 hours with a brand OHAUS balance. It is a scale with strain gauge sensor, has a measuring capacity of 250 kg and with a precision of 10 g

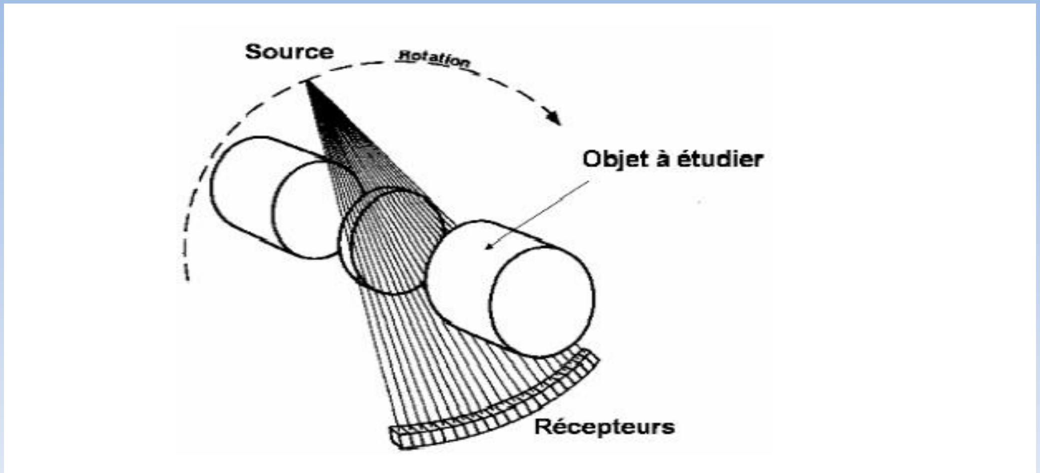
Improvement of Aleppo pine heartwood impregnability to water

Moisture profile

X-ray scanner: Such a mapping would make it possible to follow the distribution of the water absorbed along the treated samples



X-ray medical scanner, type GE BrightSpeed Excel

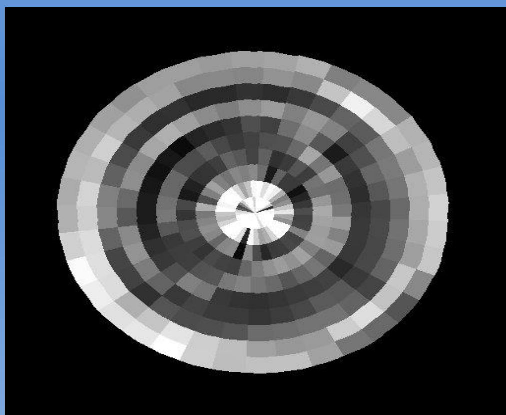


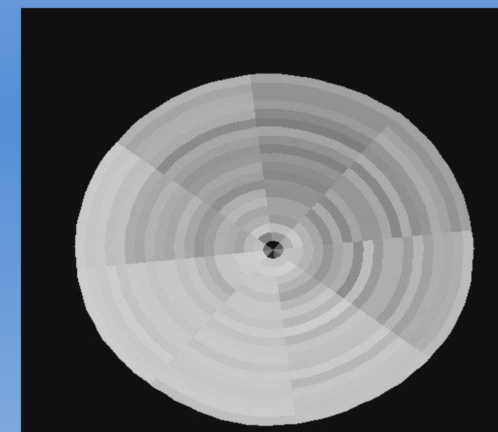
X-ray source rotating around the log placed on a moving table and a bar of receivers that measure the attenuation of X-rays after passing through the object

Improvement of Aleppo pine heartwood impregnability to water

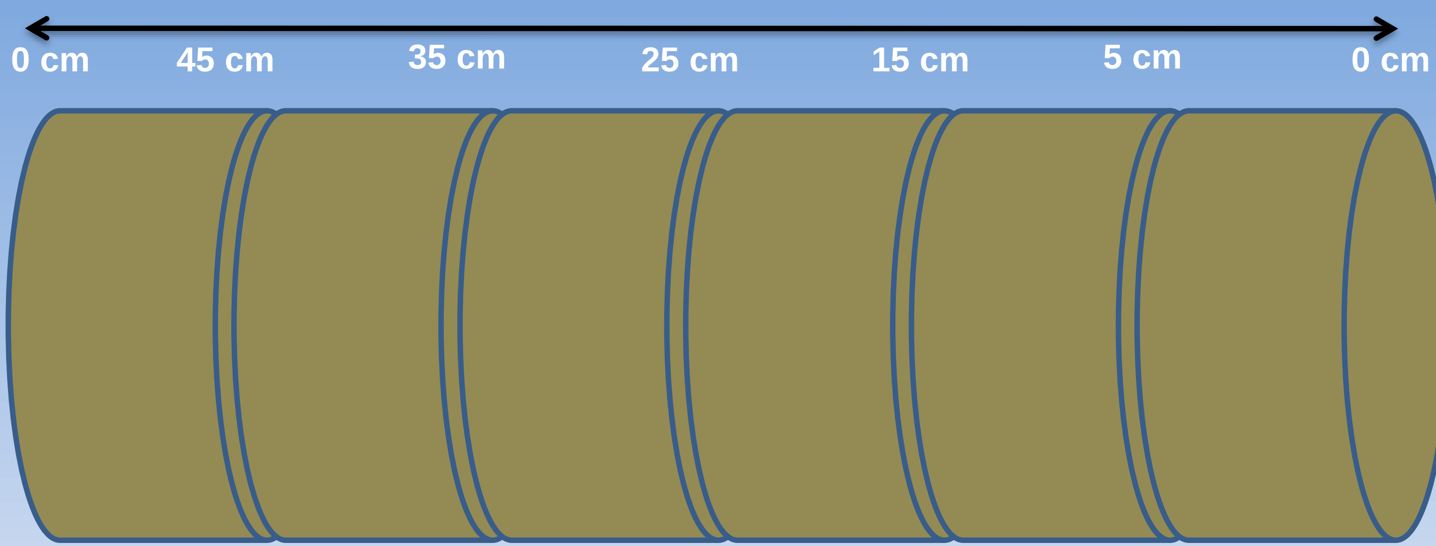
Cutting logs in virtual zones

- **Cutting into 36 rays or sectors of 10 ° × 10 rings - width 1 cm,**
- **Cutting 8 × 20 rays "rings - width 0.5 cm".**





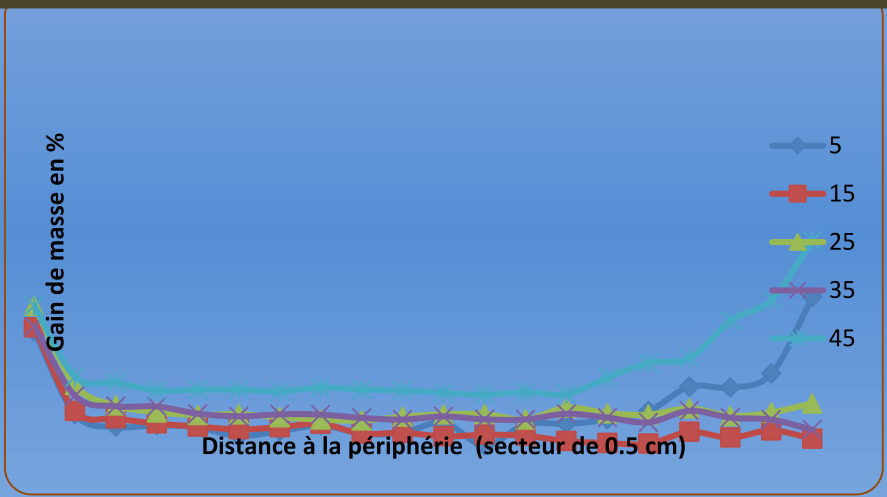
50 cm long



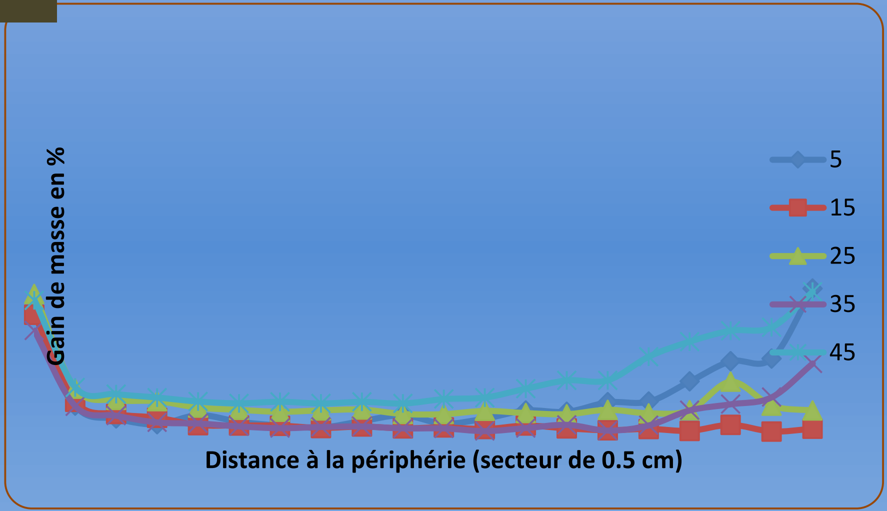
Log cutting into 5 virtual levels

Improvement of Aleppo pine heartwood impregnability to water

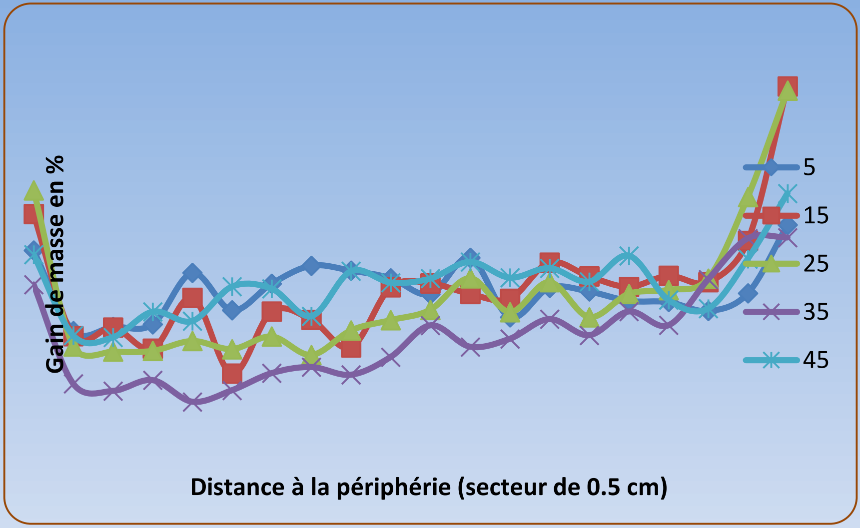
Découpage en 8 rayons × 20 " anneaux de 0.5 cm



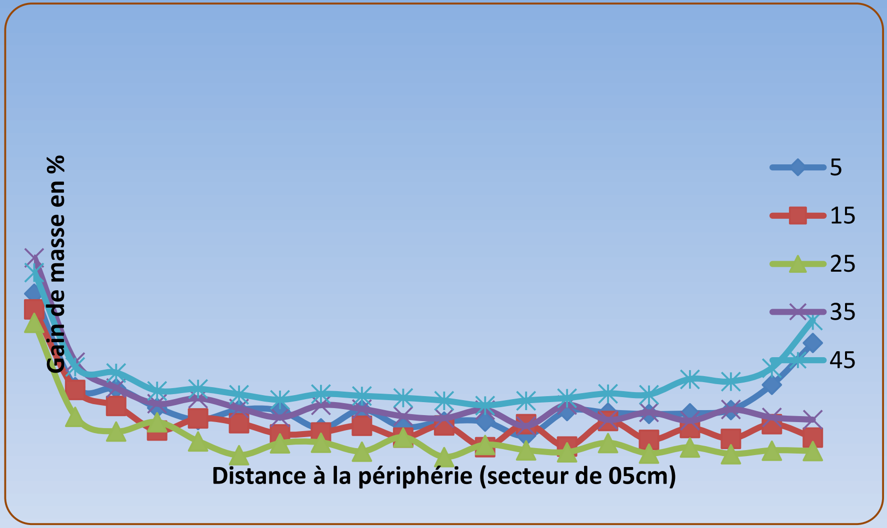
Radial distribution of water adsorbed by the control log (T)



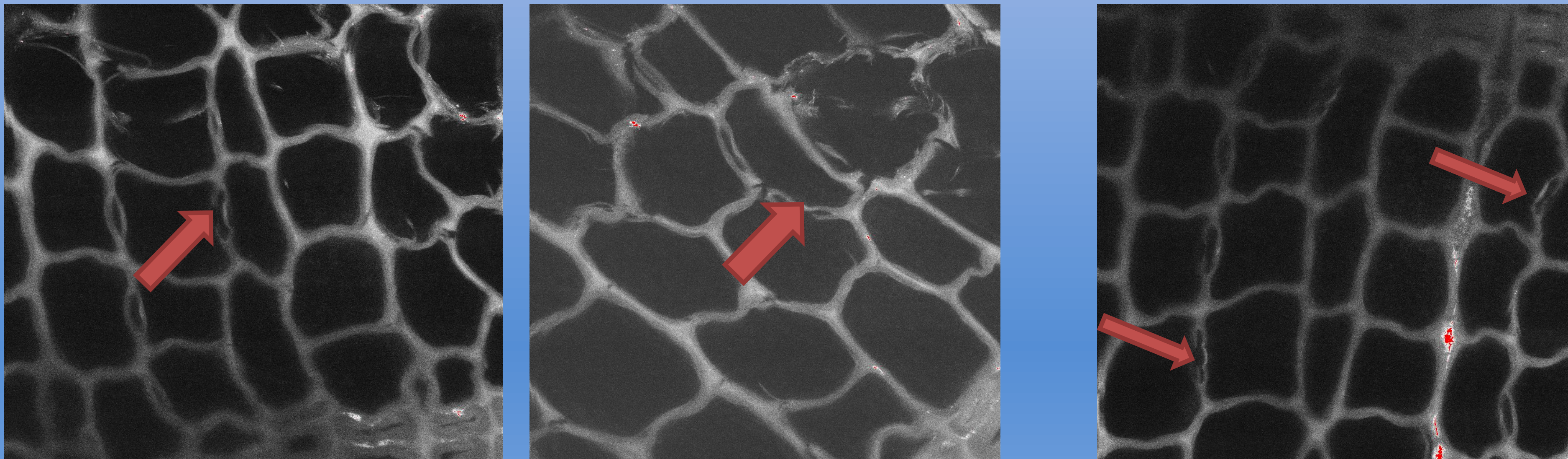
Radial distribution of water adsorbed by marinated logs in standing water (M)



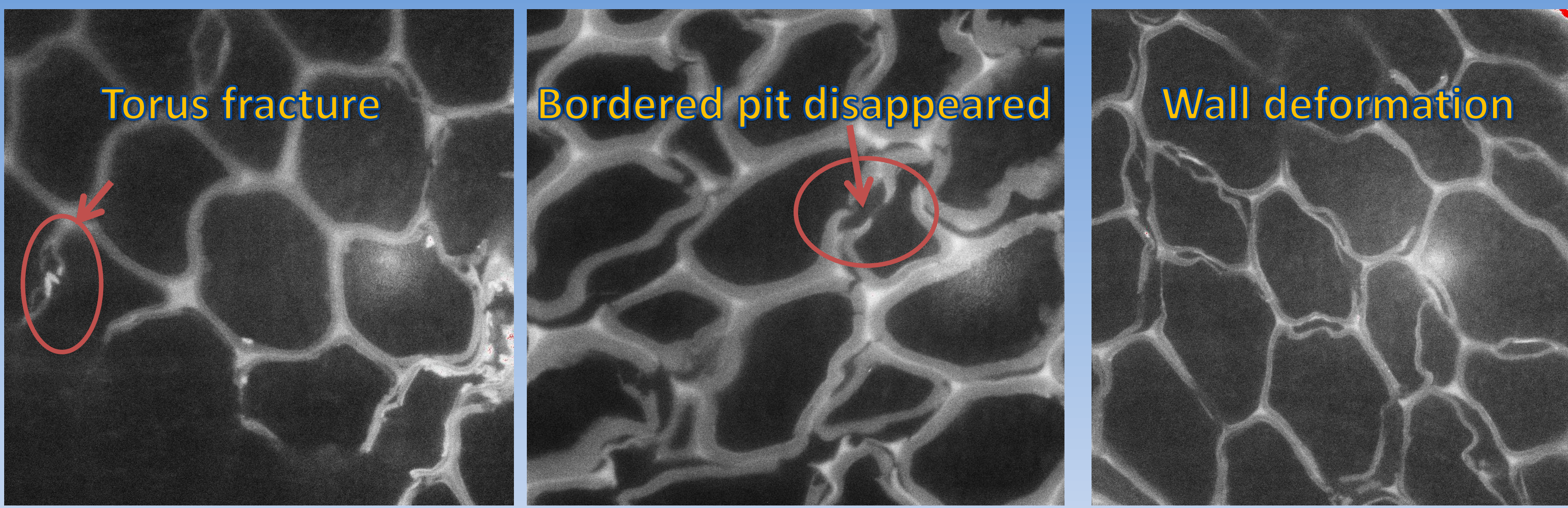
Radial distribution of water adsorbed by the thermally dried log (ST)



Radial distribution of water adsorbed by naturally dried log (SN)



Duramen témoin vert



Heartwood dried at 103°C