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PORE SIZE DIAMETER, SHRINKAGE AND SPECIFIC GRAVITY EVOLUTION DURING THE HEAT TREATMENT OF WOOD

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ABSTRACT

The heat treatment of wood by mild pyrolysis permit to produce a new material called torrefied or retified wood. Heat treated wood possesses new properties like improved decay resistance, higher dimensional stability, hydrophobic character, while strength properties are considerably reduced. The aim of this work was to study the anatomical structure evolution during heat treatment to explain the decreasing of the mechanical properties. The heat treatment has been carried out for a very large temperature range between 180 to 345°C under inert atmosphere on tow European wood species (pine and beech). The anatomical structure has been investigated by Scanning Electron Microscopy before and after heat treatment. On wood block for different treatment temperature mass, volume and shrinkage has been measured. From these results the apparent specific gravity was calculated and the porosity variation estimated. The results of the observations show that they are no evolution of the macroscopic anatomical structure, the vessels and tracheïds diameters remain constant. The volume evolution is probably caused by the diminution of the cellular wall thickness. By combining specific gravity evolution and the microscopic observation it's possible to conclude that the wood porosity increase on a scale lower than the micrometer (meso or nano porosity).

Key words: heat treatment, pore size diameter, porosity, shrinkage, specific gravity, wood

INTRODUCTION

Pine and Beech wood are some important European soft wood species widely used in wood industry. However, their natural durability is too low for hazard class III justifying the recourse to biocides for outdoor applications. Conventional wood preservation systems involve generally impregnation of broadly active biocides into the wood (Barnes and Murphy 1995). However, these techniques are under increasing scrutiny with respect to their environmental impacts, which may limit their scope of applications in the future (Suttie 1997). Furthermore, the increasing environmental pressures appeared since these last years in many European countries led to an important change in the field of wood preservation particularly in regard to the biocides toxicity leading to the development of non biocidal alternatives. Among these alternatives, heat treatment of wood has been intensively investigated to increase its durability (Patzelt 2002, Militz 2002). The obtained product called thermally modified, torrefied or retified wood and can be obtained by mild pyrolysis in a temperature range between 180 to 240°C under inert atmosphere. Such heat-treated wood dramatically reduces its hygroscopicity, its wettability (Hakkou et al. 2005), and improves its dimensional stability (Mouras et al. 2002) and durability (Hakkou et al. 2006). But the main drawback of the heat treated wood is the mechanical brittleness (Santos 2000, Mouras et al. 2002, Unsal and Ayrilmis 2005, Yildiz et al. 2006). For these reasons the heat treated wood is not recommended for use in load-bearing constructions. The aim of this study was to determine how heat treatment modified the physical wood structure: anatomy, shrinkage, specific gravity and porosity. For this purpose, the anatomical structure has been investigated by Scanning Electron Microscopy before and after heat treatment. On wood block for different treatment temperature (constant treatment time, 8 h) the mass, volume and shrinkage (radial, tangential and axial direction) has been measured. From these results the apparent specific gravity was calculated and the porosity estimated. The heat treatment has been carried out in a very large temperature range between 180 to 345 °C, to accentuate the phenomena observed, under inert atmosphere on tow European wood species (pine and beech).

1. MATERIALS AND METHODS

Beech (*Fagus sylvatica*) and Pine (*Pinus sylvestris*) heartwood was used throughout this study. Wood blocks were oven dried at 103°C until stabilization of their mass (approximately 48 hours) before determination of their anhydrous weights (m_0).

1.1. Heat-treatment

Heat-treatment was performed on wood blocks (25 x 15 x 30 mm in tangential, radial and axial directions) in a reactor placed in an oven for different temperatures and times under a nitrogen atmosphere. The oven temperature was increased from ambient to the operating temperature 180 – 260 °C. We have voluntary used higher treatment temperature to increase the tendency that we want to study.

1.2. Pore size diameter

The pore size diameter has been investigated by Scanning Electron Microscopy before and after heat treatment. Before the experiment the investigated sample surface is refreshed by microtomic cut. The observation plan is the transversal face (tangential x radial) that can permit to observe the tracheas (pine) and the vessels (beech).

1.3. Mass, volume, specific gravity and shrinkage measurement and variation

Before (indice 0) and after (indice 1) the heat treatment, on anhydrous samples the following parameters have been measured: mass and 3 dimensions. The nominations of all the different measurements are presented in the table 1.

Table 1: Mass and dimensions nominations

Masse		Dimensions					
		In radial direction		In tangential direction		In axial direction	
Initial	Final	Initial	Final	Initial	Final	Initial	Final
m_0	m_1	H_0	H_1	L_0	L_1	P_0	P_1

From these parameters the volume ($V = H \times L \times P$) and the apparent specific gravity ($\rho = \frac{m}{V}$) have been calculated. For better understanding the influence of temperature we also calculated the percentage mass evolution (Δm), the percentage volume evolution (ΔV), the percentage specific gravity evolution (Δρ), and the different shrinkages (radial R_r, tangential R_t and axial R_a) according to the formulas:

$$\Delta m = \frac{m_0 - m_1}{m_0} 100 \quad \textbf{(1)}$$

$$\Delta V = \frac{V_0 - V_1}{V_0} 100 \quad \textbf{(2)}$$

$$\Delta \rho = \frac{\rho_0 - \rho_1}{\rho_0} 100 \quad \textbf{(3)}$$

$$R_r = \frac{H_0 - H_1}{H_0} \quad R_t = \frac{L_0 - L_1}{L_0}$$

$$R_a = \frac{P_0 - P_1}{P_0} \tag{4}$$

2. RESULTS AND DISCUSSION

2.1. Vessels and tracheas size diameter from mic-roscopic observations

The anatomical observations are presented on the figure 1 and 2 for the beech wood before and after heat treatment at 260 °C during 8 hours. On the figure 3 and 4 we presented the observations for the pine wood. The determination of the tracheas size diameter is easily from visual observation.

The diameter size corresponds to the maximum horizontal value measured on the picture (table 2).

Table 2: Maximum and horizontal size diameter for the macro vessel of beech wood and trachea of pine wood from the SEM observations, D (μm)

Specimen Length [m]	Untreated	Heat treated (260 °C, 8h)	Variation of pore size diameter (%)
Beech, average of the maximum and horizontal size diameter for the macro vessel, D (μm)	38,1	37,6	-1,3
Pine, average of the maximum and horizontalsize diameter for the trachea, D (μm)	2,3	2,1	-0,9

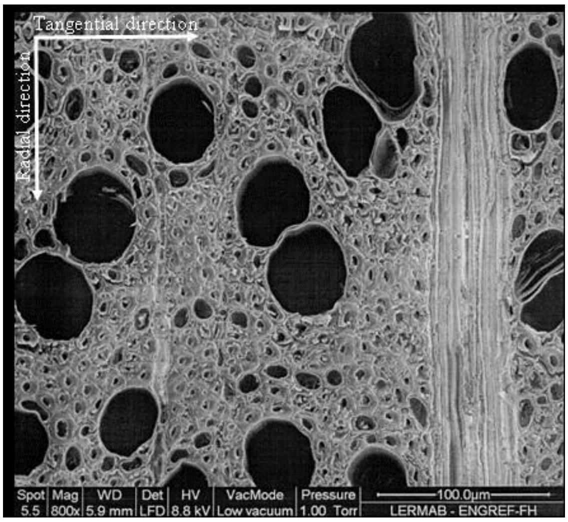

Figure 1: Transversal plan observation for beech wood

In the case of the pine the tracheas have the same structure, so it's easy to determine the average size diameter. For the beech, the anatomical structure is very different and composed by a great dispersion of the vessel diameter. However, the vessels can be classified in two different classes: the macro vessels and the micro vessels. The SEM observations permit a good observation of the macro vessel, that's the raison why the size diameters have been determined only by the measurement of the horizontal diameter of the macro vessel of the beech. In the reason of the non-symmetrical shape of the vessel a special procedure was developed for the beech wood; indeed the two pictures (figure 1 and 2) are twin: the observations were carried out exactly at the same location on the same sample. In these conditions we can easily observe the same vessels before and after heat treatment. In the table 2 the value of the average of the maximum horizontal vessels size diameter, of the 11 mains macro vessels showed on the figure 1 and 2, are presented.

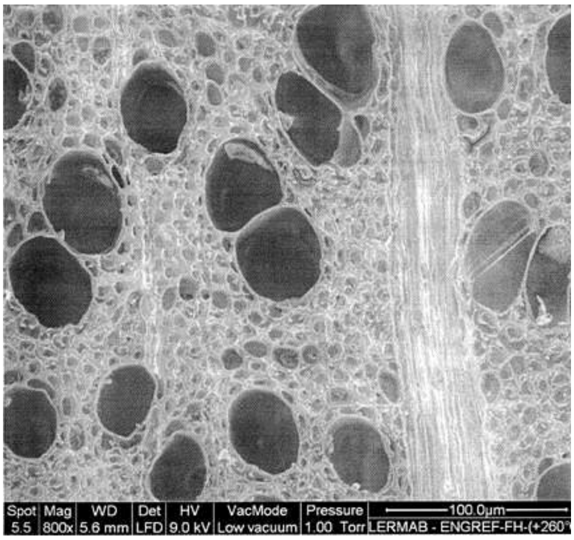

Figure 2: Transversal plan observation for beech wood after heat treatment (260 °C, 8h)

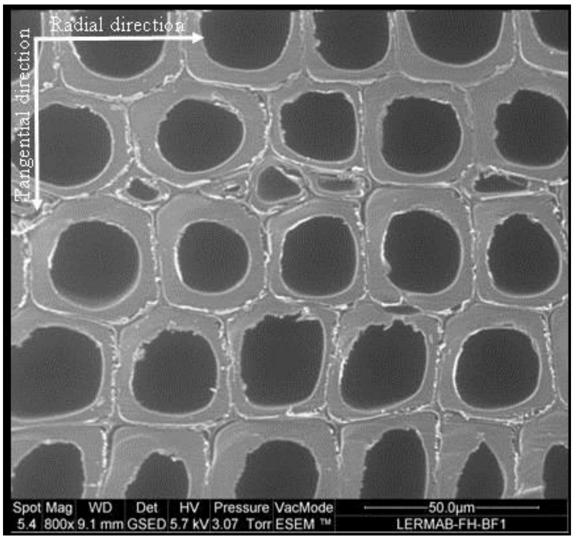

Figure 3: Transversal plan observation for pine wood

A general observation permit to conclude that there is no important anatomical modification. The results presented in the table 2 showed a slightly diminution of the average size diameter for the pine and for the beech. This diminution is equal to -1,3 % for the beech and -0,9% for the pine. So we can conclude that during the heat treatment, at 260 °C for 8 hours, there is not real modification of the tracheas and vessels size. These observations are in good agreement with the results obtain by Pfriem et al. (2009), that obtained a slightly increasing of pore diameter for the maple determined from porosimetry by mercury intrusion. Moreover, for beech wood only, the tangential and radial shrinkage are easily measurable from the figure 1 and 2 (same sample), the results are: Rt = 12 % and Rr = 8 %. We can conclude that the macro pore size diameter (vessels and tracheas) are not affected by the heat treatment. The main modification is an important shrinkage that involves a decreasing of the volume sample and equally a thinning of cell walls.

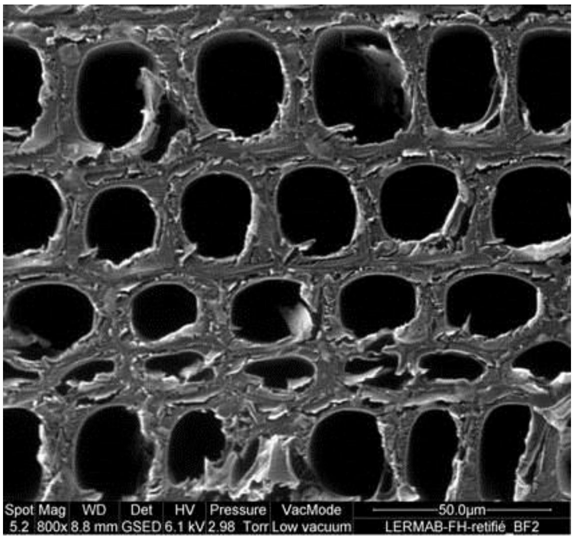

Figure 4: Transversal plan observation for pine wood after heat treatment (260 °C, 8h)

2.2. Mass, volume, specific gravity and shrinkage evolution, macroscopic observations

The figure 5 and 6 show the evolution of mass, volume, apparent specific gravity and shrinkage as a function of temperature treatment; respectively for beech and pine. Concerning the beech the values of the tangential and radial shrinkage increase with the temperature. For 260 °C these values are R_t = 12 % and R_r = 6 %. They confirm the measurements obtained from the microscopic observations (R_t = 12 % and R_t = 8 %). For 345 °C these values are very important

R_t = 36 % and R_r = 21 %. Along the axial direction the shrinkage is close to zero below 280 °C, and lowers than 8 % at 345 °C. We can conclude that the volume diminution (ΔV) is mainly generated in the tangential and radial direction, probably by thinning down of the cellular walls.

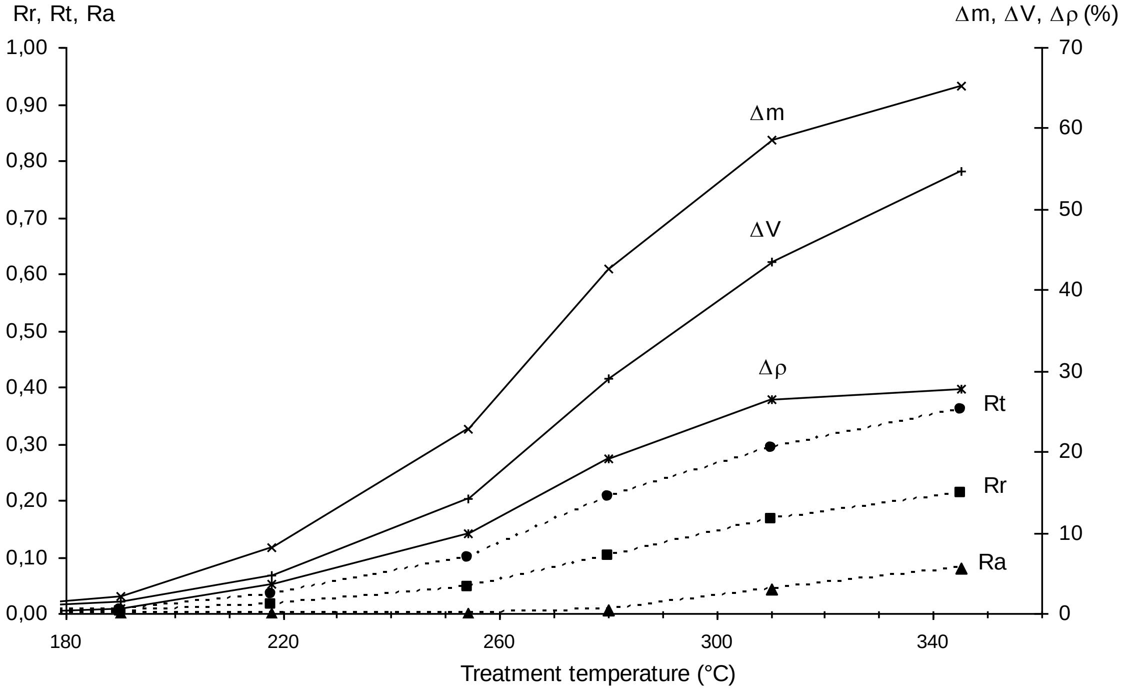

Figure 5: Evolution of mass, volume, specific gravity and shrinkage as a function of temperature treatment for beech wood

The evolution curve Δm = fn(T) and ΔV = fn(T) are very similar, however the volume evolution is lower than the mass evolution. We can note that the parallel evolution of the tow curves involved a stabilisation of the decreasing specific gravity for the high temperatures. In the temperature range that interest us (240 °C) the specific gravity decrease considerably, from this measurement it's possible to predict that the porosity of beech wood increase. This prediction is in good agreement with Pfriem *et al.* (2009). From the SEM observations, that showed clearly that the vessels diameters are not affected, we can conclude that the increasing of the porosity is probably located in the cellular walls.

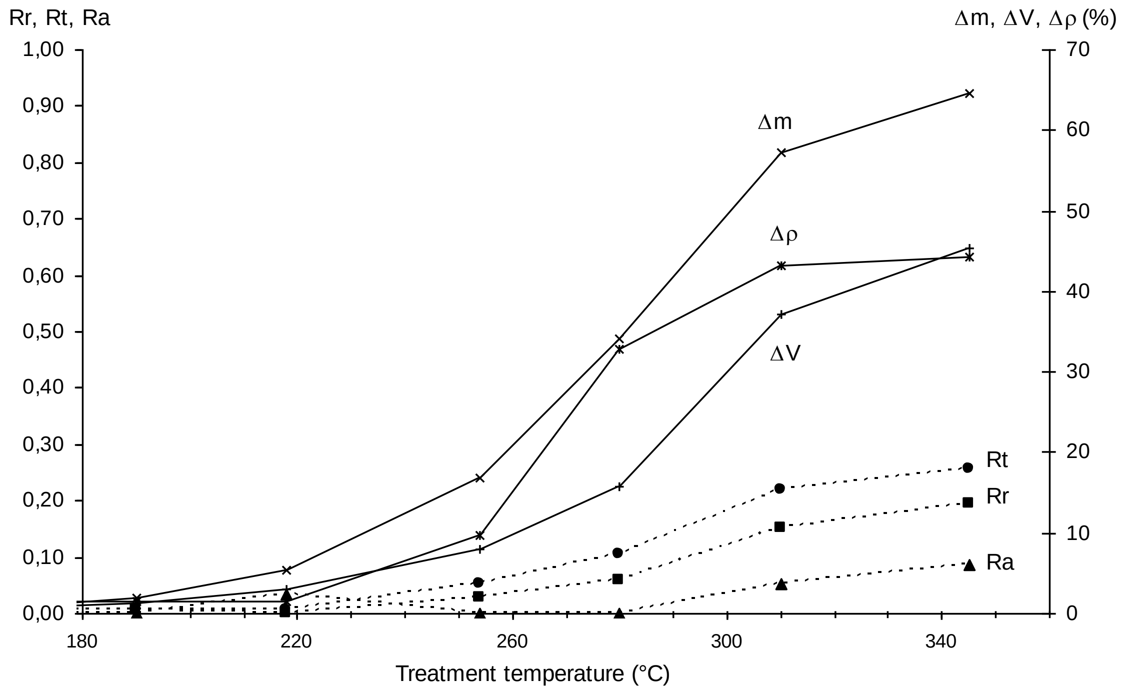

Figure 6: Evolution of mass, volume, specific gravity and shrinkage as a function of temperature treatment for pine wood

For the pine all the observations and conclusions are similar. The values of the tangential and radial shrinkage increase with the temperature also. For 260 °C these values are R_t = 6,5 % and R_r = 3,8 % and R_a = 0,025 %. For 345 °C these values are very important R_t = 25,8 % and R_r = 19,4 % and R_a = 8,6 %. These results are very close to those obtain on the beech wood. So we can conclude that the volume diminution is generated in the tangential and radial direction. As for the beech the evolution curve $\Delta m = fn(T)$ and $\Delta V = fn(T)$ are almost parallel, that involve a stabilisation of the decreasing specific gravity for the high temperatures. For 240 °C the decreasing of the specific gravity is important, these measurements permitted to estimate an important increasing of the porosity. As the SEM observation showed that the trachea diameter remains constant, we can conclude that the decreasing of the porosity is located in the cell wall.

3. CONCLUSION

The heat treatment of wood by soft pyrolysis does not involve significant anatomical modification. During the heat treatment the pore size diameters (vessels and tracheas) can be considered as constant. The main modification is an important shrinkage that involves a decreasing of the volume sample and equally a thinning of cell walls. On the other hand the measurements of mass and volume evolution clearly highlighted a change of the specific gravity of heat treated wood. Consequently we can estimated that the porosity of wood increases during the treatment. This new porosity is probably localized in the cell walls that thinning. This observation can initially explain the decreasing of the mechanical properties of wood after heat treatment.

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