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EUROPEAN OAK'S GROWTH RINGS PROPERTIES: DENSITY DISTRIBUTION AND THERMAL BEHAVIOR ANALYSIS OF EARLY – AND LATEWOOD

Joël Hamada[1], Anélie Pétrissans[1*], Frédéric Mothe[2], Philippe Gérardin[1], Mathieu Pétrissans[1]

[1]Laboratoire d'Etudes et de Recherche sur le Matériau Bois, Université de Lorraine, FST BP 70239, 54506 Vandœuvre-lès-Nancy, France

[2]Laboratoire d'Etude des Ressources Forêt Bois, UMR INRA-AgroParisTech 1092, INRA Centre de Nancy Lorraine, Route d'Amance, 54280 Champenoux, France

[*]e-mail: anelie.petrissans@univ-lorraine.fr

ABSTRACT

Last decades wood is promoted as building material. Unprotected wood exposed to outdoor conditions undergoes a variety of degradation induced essentially by fungi attacks. Heat treatment of wood by mild pyrolysis (180°C<T<240°C under inert atmosphere) is a preservation process with a weak environmental impact, and therefore, is viewed as an interesting alternative to the chemical impregnation methods. Nowadays, the main difficulties of industrial processes are to obtain final products with a constant quality (durability, dimensional stability, mechanical properties and colour). These difficulties may be due to the heat transfer or the inter-specific or intra-specific wood heterogeneity. The aim of this study is to better understand the effect of the intrinsic wood properties: density according to the position in wood, and especially early wood versus latewood, on the thermo-degradation process. Heat treatment using thermo-gravimetric analysis (TGA) was performed on small samples of the European oak (*Quercus petraea Liebl.*), where the earlywood and the latewood constituting the annual rings were studied separately. The relationship between the radial variation of wood density components assessed by micro-densitometer and their thermo-degradation sensibility was investigated. The results show that globally, earlywood and latewood behaved differently under thermal conditions, for the two studied trees; earlywood tended to be more sensitive to thermo-degradation than latewood.

Key words: heat treatment, intra rings, micro-tomography, oak, TGA.

INTRODUCTION

Nowadays, the use of wood as building material is subjected to growing interest due to its intrinsic properties. Exposed to outdoor conditions, unprotected wood undergoes degradations due to biotical and abiotical agents. In the context of sustainable development and the increasing environmental pressure, chemical products are nowadays contested because of their toxicity and the wood products' service lifecycle management. Non biocide methods like thermo-modification appear as an attractive alternative to classical preservation methods. Wood heat treatment by mild pyrolysis improves the biological durability (Kamdem *et al.* 2002, Hakkou *et al.* 2006, Guller, 2012) and its dimensional stability (Mouras *et al.* 2002) of the material. The process remains environmentally friendly. The influence of treatment conditions on chemical composition and conferred properties of heat-treated wood has been intensively investigated. A quality control marker, based on mass loss, has been proposed to find the level of thermodegradation required to reach full decay durability (Nguila *et al.* 2009). Nowadays, the main difficulties of industrial processes are to obtain final products with constant quality (durability, dimensional stability, colour). These difficulties may be due either to the processes their self or to the inter-specific or intra-specific wood heterogeneity. It has been shown in a previous study that mass losses due to thermodegradation reactions are strongly influenced by the nature of the wood species and consequently on some wood initial properties like its chemical composition and density (Chaouch *et al* 2010, Candelier *et al.* 2011). Hardwoods were generally more sensitive to thermodegradation than softwoods, while woods with higher densities seem more sensitive to temperature than lower densities woods (Chaouch *et al* 2010). Wood properties can vary within a species and even inside the same tree. Wood quality is reported to be deeply influenced by rings age, radial growth and ecological factors, the soil quality, forest management, age and genetics (Bouriand 2004, Zhang *et al.* 1993, Knapic *et al.* 2007, Bergès *et al.* 2008). The quality of ring porous wood is influenced by rings density (Polge *et al.* 1973), differences between earlywood and latewood properties contribute to the timber stability (Kretschmann and Cramer, 2007). Kibblewhite *et al.* (2010) have demonstrated that lignin and carbohydrate content of earlywood and latewood vary between normal and compression wood. Shchupakivskyy *et al.* (2014) using a High-Frequency Densitometry have observed after heat treatment of oak that the earlywood's density was significantly more reduced than the density of latewood. Initial intrinsic properties of wood such as density or chemical composition appear as important parameters which may influence the thermodegradation.

The effect of the initial intrinsic wood density directly connected to wood's natural variability on the level of thermodegradation recorded for similar curing conditions and consequently on conferred properties to the final end products has been studied. Such data may be of valuable interest for the improvement of industrial scale treatments performed generally on a single wood species. Better understanding of the effect of initial wood quality on the industrial heat treatment process may result in a better utilization of the resource improving the attractiveness and competitiveness of the wood forestry sector. In a previous study, the density of oak wood boards before and after heat treatment was measured using the X-ray scanning tomography. This non-destructive technique gave the possibility to obtain cartography of density distribution over relatively small zones regrouping several rings. The loss of density was analyzed after heat treatment. However, the obtained observation did not show a clear relation between the initial density and thermodegradation mass loss. The image meshing did not allow considering the effects of earlywood and latewood on the level of thermo-degradation recorded for similar treatment conditions.

This study uses a microdensitometer device in order to characterize the effect of the local earlywood and latewood density on the thermodegradation level. Thermo gravimetric analyses were performed on ground to fine sawdust samples of earlywood and latewood. The thermal sensitivity of each component is related to the intrinsic composition.

1. MATERIALS AND METHODS

1.1. SAMPLING

Wood samples were obtained from radii of two trees of the European Oak (*Quercus petraea Liebl.*). Trees are issued from distinct French stands presenting different density variation, mean rings' width, crown base height, and silvicultural history (Guilley *et al.* 2004). Description is given in Table 1.

Table 1: Classification of wood origins, age, initial density and rings characterization

Sample	Region	Forest management	Soil quality	Age (y)	Average rings size (mm)	Average ring density (kg/m³)	Average earlywood density (kg/m³)	Average latewood density (kg/m³)
2390	Alsace	Even-aged	Bad	171	2.19	744.10	633.27	802.98
2547	Lorraine	Coppice	Medium	147	3.12	700.66	582.94	768.62

Strips of radii were obtained using a saw milling. After X-ray microdensitometer analysis of rings' width and density, the separation of the earlywood and latewood is performed only from some large rings. A microtome knife and hammer were used; each part of earlywood and latewood was then ground to fine sawdust 0.160 mm using a cutting mill Retsch SM 100.

1.2. DENSITY DISTRIBUTION

Radial profiles of wood density were determined. X-ray radiographies of the samples were obtained using a microfocus X-ray source (Hamammatsu L9181-02 130kV) and a digital X-ray detector (Varian PaxScan 4030R). Radial profiles of wood density of the samples were computed from the radiographies using the Cerd software suite (Mothe *et al*. 1998 a, b).

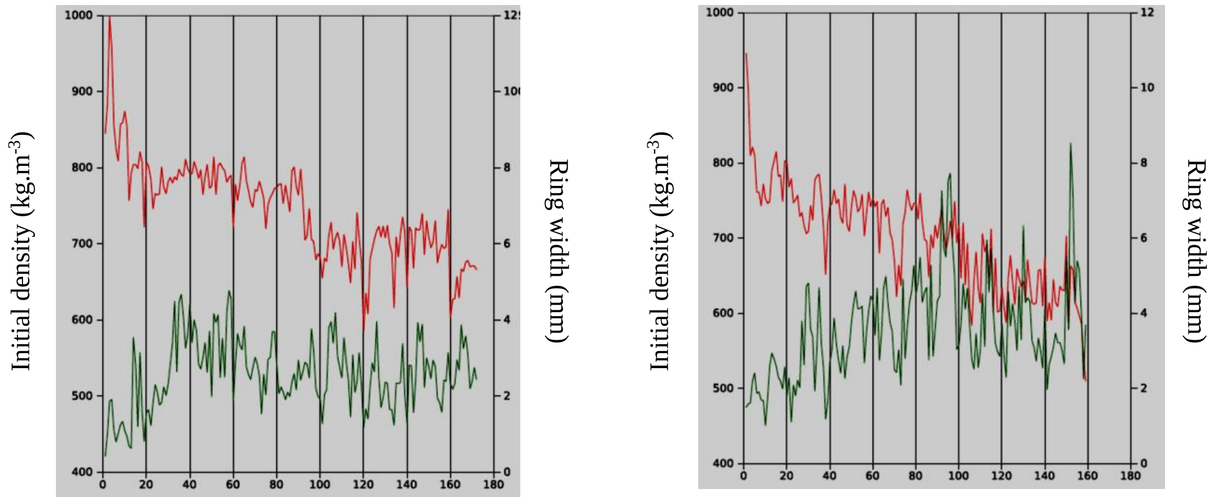
1.3. THERMO-GRAVIMETRIC ANALYSES

Earlywood and latewood behaviour of each tree are examined separately by thermo-gravimetric analyses. Heat treatment was performed on samples amounts of 10 mg, under nitrogen using a thermo-gravimetric Mettler Stare system (Mettler Toledo TGA/DSC). After a drying stage at 103 °C, experiments were performed at 220 °C for 2 hours. The heating rate and gas flow were 10° $K.mn^{-1}$ and 50 $ml.mn^{-1}$, respectively. The earlywood and latewood thermodegradation was analysed individually over 17 rings for the tree 2547 and over 15 rings for the tree 2390.

2. RESULTS AND DISCUSSION

2.1. DENSITY PROFILS

Radial wood density profiles of the trees 2390 and 2547 were studied using an X-ray microdensitometer (Figures 1 a-b). It was found that the average density (upper line) and ring's width (lower line) varied irregularly, without a clear correlation between width and density. For both trees, wood density tends to decrease linearly with ring age whereas ring width is roughly constant after an initial increasing. Numerous studies in the literature give information about the radial oak wood density distribution, prediction models were proposed to describe density distribution in earlywood and latewood, across the ring's age, integrating growth conditions, climate, region and forest management (Guilley *et al.* 1999 a, b; Le Moguédec *et al.* 2002 ; Guilley et Nepveu 2003, Knapic *et al.* 2007). The density profiles obtained using the X-ray microdensitometer are in agreement with the observations reported in the literature. Actually, several studies have been devoted to the radial profiles of wood density, numerous of them give information about the oak wood (Degron et Nepveu 1996, Guilley *et al.* 1999 a, b; Le Moguédec *et al.* 2002; Guilley et Nepveu 2003, Le Moguédec et Nepveu 2004, Guilley et al., 2004b). These studies show a clear difference between the densities of the earlywood and the latewood in the annual ring.



a) Ring age (years) b) Ring age (years)

Figure 1: Variation of mean ring density (upper line) and ring width (lower line) with age from the pith for trees 2390 (a) and 2547 (b).

2.2. DENSITY PROFILS

The earlywood and latewood thermal sensitivity was determined using the thermo-gravimetric analysis. For each tree, heartwood and sapwood were analysed separately. Recorded values of mass loss, density and width of earlywood and latewood are given in Tables 2-3. A global observation of the mass losses shows a clear difference in the behaviour of the earlywood and latewood compartments. For both studied trees, earlywood characterised by a lower density is more degraded than the higher density latewood samples, for similar treatment conditions. Results show a good agreement with the study reported by Shchupakivskyy *et al.* (2014). Authors studied the local density changes of oak wood after thermal modification using High-Frequency Densitometry. They found that density of earlywood was significantly more reduced after thermal treatment than the density of latewood.

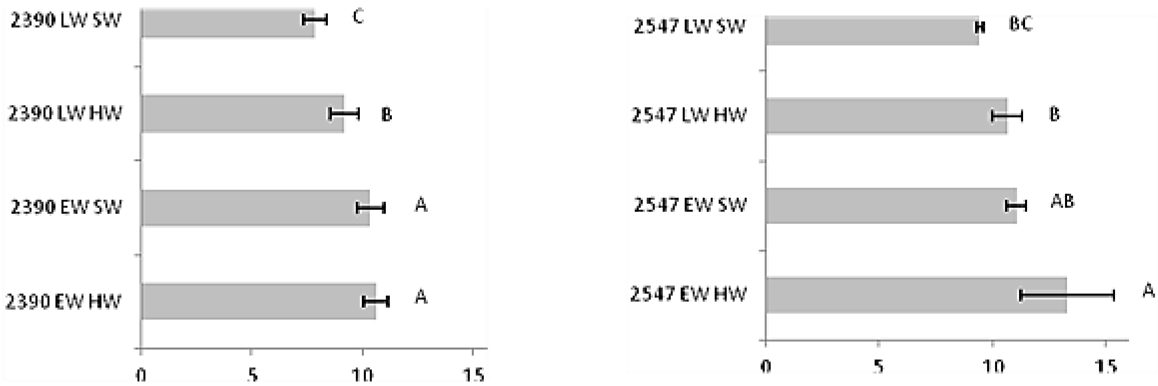
Table 2: Intra-ring study, averaged mass loss, density and width of the earlywood and latewood, tree 2547

	Tree 2547						
			Earlywood			Latewood	
	Ring density ($kg.m^{-3}$)	ML %	EW's density ($kg.m^{-3}$)	EW's width (mm)	LW %	LW's density ($kg.m^{-3}$)	LW's width (mm)
Heartwood	756±35	13.31±2.07	616±39	0.89±0.29	10.64±0.67	818±25	2.05±0.77
Sapwood	727±43	11,04±0,45	608±41	1,43±0,09	9,41±0,38	816±45	1,99±0,56

Table 3: Intra-ring study, mass loss, density and width of the earlywood and latewood, tree 2390

	Tree 2390						
			Earlywood			Latewood	
	Ring density ($kg.m^{-3}$)	ML %	EW's density ($kg.m^{-3}$)	EW's width (mm)	LW %	LW's density ($kg.m^{-3}$)	LW's width (mm)
Heartwood	747±54	10.59±0.57	605±45	0.92±0.15	9.16±0.64	821±59	2.11±0.65
Sapwood	709	10,35±0,59	610	0,8	7,84±0,54	750	1,98

A comparison of thermal behaviours of earlywood and latewood withdrawn from heartwood and these issued from sapwood shows important differences. For both compartments, samples coming from heartwood parts are characterised by higher averaged density and are more affected by the thermodegradation. Earlywood from sapwood seems less sensitive to thermo-degradation than earlywood from heartwood. Likewise latewood from sapwood is less degraded than latewood from heartwood. This observation is in agreement with the results of Bertaud and Holmbom (2014) obtained for another species, the Norway spruce (*Picea Abies L.*).



a) b)

Figure 2: Mass Loss distribution of earlywood (EW) and latewood (LW) from heartwood (HW) and sapwood (SW) parts for tree 2390 (a) and 2547 (b) at 220°C. Error bars represent standard deviations. Systems not connected by the same letter are largely different, at the 5% level.

In order to better quantify the thermodegradation variations for the different wood tissues, a statistical analysis of data, based on the single-step multiple comparison Tukey–Kramer's method was performed using the JMP program (SAS Institute Inc., Cary, NC, USA). Each tree is studied separately, results are shown on Figure 2. The Tukey–Kramer's method allowed the classification of thermodegradation data into categories from A to C. Systems not connected with the same letter are largely different, at the 5% level. Statistical analysis clearly showed that thermal behaviour of earlywood is significantly different from the latewood degradation; results are not connected by the same letter. The individual comparison of each compartment confirmed also the variation between heartwood and sapwood thermodegradation.

It should be noticed that results differ for the two trees. For both compartments, samples issued from the tree 2547 are more degraded than samples withdrawn from the tree 2390. The tree effect previously developed in the literature to describe density variations (Guilley *et al.* 1999 a, Castaño-Santamaría and Bravo 2012) is also observed for the thermodegradation sensitivity.

Attention should be given to the choice to grind wood samples to a particle size of 0.160 mm corresponding to the order of magnitude of the wood vessels. Heat transfer is not influenced by the anatomical structure, only the chemical composition could explain the differences of the measured mass losses.

3. CONCLUSION

The intra-ring study allowed evaluating the behavior of earlywood versus latewood and heartwood versus sapwood during the heat treatment process. It was observed that, earlywood, which has lower density, tended to be more sensitive to thermo-degradation. Heartwood characterized with higher density is more degraded than sapwood. Since thermo-degradation of wood is a combination of two phenomena namely heat transfer and chemical reactions, it can be concluded that the apparent difference between the two compartments could mainly be explained from the chemical point of view. The accuracy of densitometer device was of great importance to view the intra-ring variation of density. The study will be extended by analysis of the chemical composition of the earlywood and latewood compartments; expecting that future results to confirm these initial observations.

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