INFLUENCE OF THE NATURAL WOOD VARIABILITY ON THE HEAT TREATMENT PROCESS: EFFECTS OF THE WOOD INITIAL DENSITY ON THE FINAL QUALITY OF THE HEAT TREATED PRODUCT

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ABSTRACT

This study aims at making a link between the investigations of the wood industry on the control of the quality in forest (production of wood adapted to the industrial requirements) and the studies on the wood heat treatment to improve the wood durability and dimensional stability. Heat treatment was carried out under nitrogen at 220 °C. A set of 14 samples (250x110x25 mm) from 7 European oak (Quercus petraea) trees of various French origins were scanned at dry state by X-ray tomography before and after heat treatment for analyzing the effects of treatment on the variation of wood density within and between the samples. The moisture uptake test also was performed and the equilibrium moisture content rates were calculated. The results showed to the naked eye the evident dark color of samples with differences between samples treated at 230 °C and 220 °C; no color differences within samples of heartwood, but differences between heartwood and sapwood. The density values of all samples decreased after the treatment. Mass loss (ML) ranged from 11,27 % to 13,28 % and was major when density was initially high. The treatment had no significant modification on the homogeneity of the density, but the near portion of sapwood was affected. The effects of wood density on the results of heat treatment were the same within samples of same tree but when comparing different trees the trend was highly variable. In conclusion, studying the effects of wood initial density variability on the heat treatment process needs further analysis such as chemical and biological analysis along with X-ray tomography and needs to be carried out on several species.

Key words: characterisation, control, heat treatment, CT scanning, variability, wood density

INTRODUCTION

In the context of sustainable development there are more requirements for friendly environment products. In the field of wood preservation, the European Biocidal Directive 98/8/EC on the placing on the market of biocidal products recommends limit the use of conventional chemical products based of hydro dispersible and hydro soluble compounds such as CCA (Copper Chrome Arsenic) and CCB (Copper Chrome Boron). Because of leaching and management of wood products end of life cycle, these chemical products are contested. Therefore, new formulations for wood preservation are needed. Heat treatment seems to be an alternative solution. But there are still concerns about the quality of final wood heat treated products at the industrial level. Indeed, since the invention of this method, several processes have emerged in Europe and throughout the world, but all are still struggling to consistently reproducible thermal treated wood in terms of colour, durability, dimensional stability and mechanical strength. Heat treatment is a preservation process of wood consisting of heating the wood up to 260 °C under oxygen deficient atmosphere, immersion in a bath of hot vegetable oil, water vapour under vacuum or in an aqueous phase. It was worked out by the *Ecole des Mines de Saint-Etienne* under the name of Bois rétifié®. Now there are many industrial marks throughout the world (France, Germany, Finland, Netherland, Switzerland, Romania, Canada and others). The aim of all these different processes is the same, i.e using only high temperature to inducing a series of chemical reactions in the wood which eventually modify the structure of the main constituents of wood. Lignin network polymer is modified (Zaman et al. 2000, Nguila et al. 2006, Nguila et al 2007), the ratio between amorphous cellulose and crystalline cellulose is changed (Fengel et Wegener, 1989, Yildiz et al 2006), hemicelluloses are degraded (Gérardin et al. 2007, Nuopponen et al. 2004) and chars precursor products appear (Nguila et al. 2007). These changes give the wood new properties such as durability, dimensional stability and colour, but also can alter others such as mechanical strength and stiffness. Heat treatment is generally applied to those species that are not durable, in order to improve their quality, so that they can be used for application requiring more durability and stability performance, also aesthetic quality. The fig. 1 shows the different temperatures under which wood can be modified, and the fig. 2 shows the effects of chemical modifications taking place at the molecular and macromolecular of wood during the heat treatment.

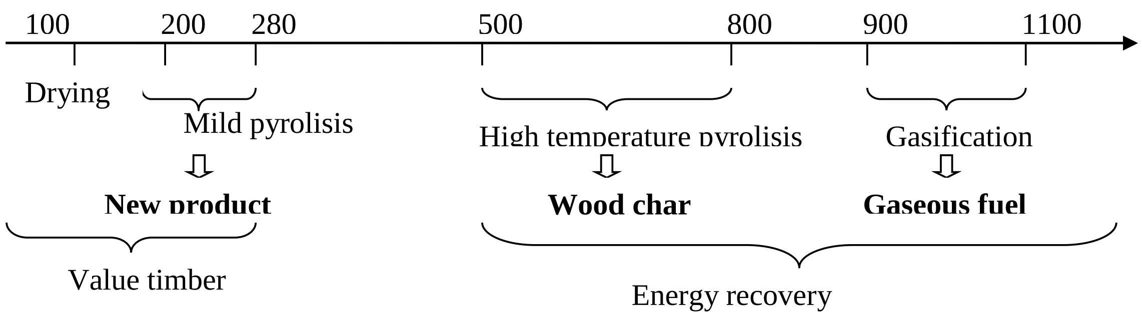

Figure 1: Different temperatures for thermal treatment of wood

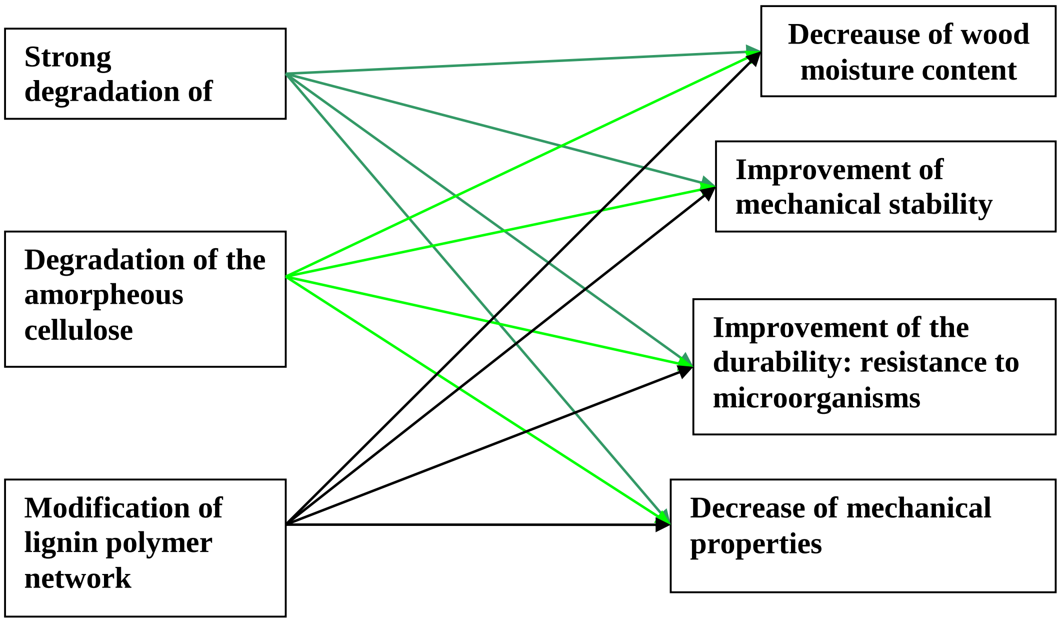

Figure 2: Diagram showing the link between the chemical modifications taking place at molecular and macromolecular level inside the wood during heat treatment and their effects on wood macroscopic properties

Thermal wood treated is generally used for interior carpentry (floors, walls, doors, furniture) and external joinery (cladding, flooring terrace around pool, shutters, frames). Many studies recently showed there is a direct link between ML induced by the heat treatment degradations and the conferred properties (Hakkou et al. 2006, Welzbacher et al. 2007). Subsequent studies conducted in laboratory LERMaB on different European species allowed evidence the influence of the operation conditions such as the processing temperature, the duration of the treatment, and the rise in temperature (Chaouch *et al*. 2009, Chaouch *et al*. 2010). On the other hand high density enhances strong ML (Chaouch, 2011). Therefore it seems important to follow the ML during wood heat treatment in order to develop a quality control of the final product. We hypothesized that initial density variation has effects on the quality of thermal wood treated.

The focus of our study was to estimate ML rates and density variation within samples of the same tree and between samples of different trees, and assess the effect of samples diversity on the properties of the final product using the CT scanning.

1. MATERIEL AND METHODS

1.1. SAMPLES

Samples of 250x110x25mm were taken from the radii of the European oak (*Quercus petraea*) as showed in fig.3: 7 trees from different French origins were firstly chosen depending on the type of the stand, and density diversity. The goal was to get 2 pieces from the same tree: 1piece close to the pith and 1 piece close to the sapwood.

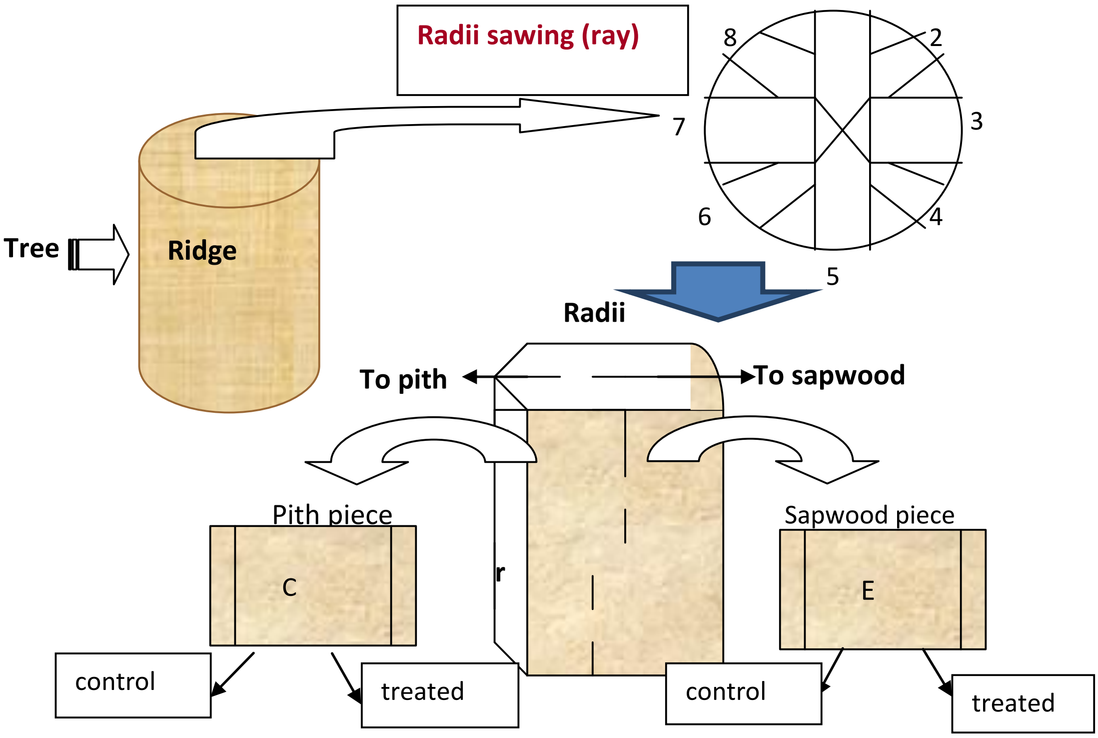

Figure 3: Diagram showing the sampling within the tree

1.2. HEAT TREATEMENT PROCESS

The treatment took place in a pilot furnace, a kind of thermo balance device which allowed us to:

- -carry out heat treatment of pieces of wood of 250x110x25 mm at least by conduction under nitrogen;
- -record ML during the drying and treatment phase (accuracy 0,2 g);
- -record the temperature at the core and the surface of the piece (accuracy 0,1 °) (fig. 4);
- -set the temperature rise of the process in a variable way from 0,1 °C to 0,5 °C per min

Each piece of wood was confined between two metal plates heated and the assembly was placed in a thermally isolated box. The maximum temperature of treatment was 230 °C and the minimum was 210 °C. Each set of treatment lasted 2 h 23min.

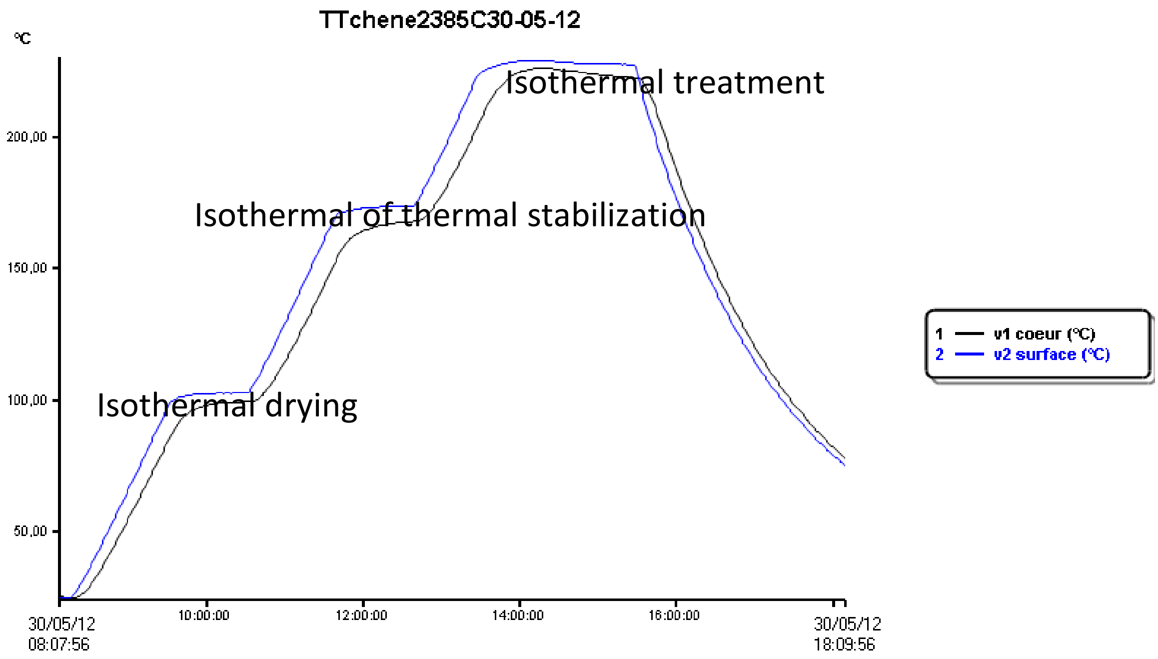

Figure 4: Curve representing the shape of the temperature at the core and at the surface of the piece during the treatment

1.3. TC SCANNING

The characterization of the pieces density was performed by means of TC scanning, which allowed us to get density values before and after the treatment. The TC scanner uses the lights beams that are mitigated by the wood according to its atomic composition for reproducing the volume of the entire piece as JPEG image. The image was then digitally processed, and according to pixels number and piece volume, the density value was calculated. For each piece, the image was divided into 1000 identical cards in the direction of the length, width and thickness. The average of all cards densities gave the piece density. To assess the variation in density within the single piece the coefficient of variation (cv) was calculated.

2. RESULTS

2.1. COLOR

All pieces are clearly dark after treatment: Pieces treated at 230 °C are darker than pieces treated at 220 °C and pieces treated at 220 °C are darker than pieces treated at 210 °C. The three colors seem close to those obtained by the company BDT (Bois Durable de Bourgogne) at the same range of temperature for the European oak. There is very little difference in color within pieces completely homogeneous, and pieces containing sapwood and heartwood have two colors. Since sapwood is naturally clearer than heartwood, it is normal that its color remains clearer than heartwood's. There is no obvious difference of color at the naked eye between pieces close to the pith and those close to the sapwood. Measurement by colorimetry might help distinguish any difference.

2.2. MASS LOSS AND DENSITY

ML ranges between 11,27 % and 13,28 % (tabl. 1). These values match those obtained by the company BDB (Bois Durable de Bourgogne), and are considered as the average needed to guarantee good performance for the European oak. Within each tree piece with high initial density, corresponding to that close to the pith, has major

ML except the tree n° 2482 (tabl. 1). These results correspond to those obtained by Chaouch (Chaouch 2011). But these results are more variable between samples (between trees). The heat treatment doesn't affect the homogeneity of the density heavily as the gap between values of the coefficient of variation (cv) prior and after the treatment is low (tab.1).

Table 1: Results of the densities values, ML (mass loss) values and coefficient of variation (cv) for the densities; the first column contains the samples where the first number refers to the tree code, the second to the ridge, the third to radii, and the letter C stands for C*œur* (heart) while E stands for *Ecorce* (bark)

N° tree and type wood	Density Prior kg/m³	Density After kg/m³	Standard deviation Prior kg/m3 k	Standard deviation After kg/m3	ML (%)	CV Prior (%)	CV After (%)
2385-1-3-C	**655,97**	**611,38**	**67,7**	**64,15**	**12,53**	5,28	5,59
2385-2-6-E	558,2	515,85	57,1	54,04	11,83	**6,19**	**6,38**
2388-2-6-C	**703,41**	**662,93**	**64,64**	**62,05**	**12,24**	5,15	5,62
2388-2-2-E	673,85	630,6	83,63	79,73	11,27	**7,17**	**7,57**
2487-2-2-C	**645,5**	**602,19**	**93,07**	**88,32**	**11,72**	4,22	4,44
2487-2-6-E	581,5	535,75	89,62	84,5	11,57	**6,54**	**6,79**
2492-4-2-C	**751,83**	**697,63**	**97,34**	**92,58**	**13,28**	6,38	6,55
2492-2-2-E	698,64	650,86	89,6	86,91	12,29	**8,95**	**9,18**
2371-2-2-C	**682,43**	**634,22**	**37,79**	**36,93**	**12,39**	5,54	5,82
2371-2-2-E	591,12	541,55	58,68	55,13	11,74	**9,93**	**10,18**
2390-1-7-C	**705,31**	**661,25**	**24,54**	**23,81**	**11,63**	3,48	3,6
2390-1-3-E	647,503	600,91	43,45	41,78	11,55	**6,71**	**6,95**
2482-2-6-C	**683,72**	**638,39**	**45,67**	**44,35**	**11,55**	6,68	6,95
2482-2-3-E	595,12	550,02	43,81	42,19	12,66	**7,36**	**7,67**

2.3. MOISTURE EQUILLIBRIUM

The equilibrium moisture content of pieces after treatment was performed according to the principle of the moisture content determination (NF B51-004 standard). The results show that the heat treated wood pieces are less hydrophilic, with the moisture content rates around 7% and 8% (tab.2). These values are higher than those obtained by the company WTT (Wood Treatment Technology) after laboratory testing by CRITT BOIS in Epinal, France (www.abibois.com). Their value of equilibrium moisture content rate for the heat treated aok is 6,1%. The equilibrium moisture content rate tends to be higher for pieces close to the pith except for the tree 2492 (tabl. 2). But these results are variable between samples.

Table 2: Results of the equilibrium moisture content of pieces prior and after treatment, cv stands for coefficient of variation

Treated wood	Density kg/m3	CV %	Equilibrium moisture content %	Density kg/m3	Treated wood	CV %	Equilibrium moisture content %
2385-C	611,38	5,59	**6,97**	515,85	2385-E	6,38	**6,86**
2388-C	662,93	5,62	**7,7**	630,6	2388-E	7,57	**7,3**
2487-C	602,19	4,44	**7,83**	535,75	2487-E	6,79	**5,88**
2492-C	697,63	6,55	**7,53**	650,86	2492-E	9,18	**9,41**
Natural wood					Natural wood		
2385-C	655,97	5,28	**12,03**	558,2	2385-E	6,19	**11,94**
2388-C	703,41	5,15	**11,59**	673,85	2388-E	7,17	**9,02**
2487-C	645,5			581,5	2487-E		
2492-C	751,83	6,38	**12,1**	698,64	2492-E	8,95	**10,3**

3. DISCUSSION

The high values of the equilibrium moisture content after treatment could be explained by the fact that the test was performed by introducing samples of 30 x 20 x 5 mm^3 into a steam room at 82 % RH (relative air humidity) and 35 °C during 28 days. Whereas the WTT value was performed at 65 % RH and 20 °C according to EN NF B51-006 standard. Besides, only eight samples were tested; which cannot allow us to assess the magnitude of the effect of density.

CONLUSION

The studies of the effects of the wood density on the heat treatment need to analyze the homogeneity of the final properties. Our study showed that within tree:

- density declined with the treatment;
- ML was high when the initial density was high;
- the density homogeneity was not heavily affected by heat treatment.

The difference of ML values between pieces from the same tree seems to confirm the hypothesis that initial density would have effect on the properties of final heat treated wood. However, many tests are needed on other species for evaluating the role of the density over the whole heat treatment process and the final product properties. As well as chemical analysis are needed along with the TC scanning.

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