ONLINE ISSN 2069-7430
ISSN-L 1841-4737

PRO LIGNO
www.proligno.ro

Vol. 13 N° 2 2017
pp. 10-20

DECAY RESISTANCE VARIABILITY OF EUROPEAN WOOD SPECIES THERMALLY MODIFIED BY INDUSTRIAL PROCESS

Kévin CANDELIER *
Dr. Eng. – Cirad, UPR 114 BioWooEB
Address: CIRAD-Unité de Recherches BioWooEB, TA B 114/16, Montpellier, France
E-mail: kevin.candelier@cirad.fr; Phone (33) 3 83 68 40 00; Fax (33) 3 83 68 40 01

Luc PIGNOLET
Tech. – Cirad, UPR 114 BioWooEB
Address: CIRAD-Unité de Recherches BioWooEB, TA B 114/16, Montpellier, France
E-mail: luc.pignolet@cirad.fr

Sylvain LOTTE
Tech. – Cirad, UPR 114 BioWooEB
Address: CIRAD-Unité de Recherches BioWooEB, TA B 114/16, Montpellier, France
E-mail: sylvain.lotte@cirad.fr

Alban GUYOT
Tech. – Cirad, UPR 114 BioWooEB
Address: CIRAD-Unité de Recherches BioWooEB, TA B 114/16, Montpellier, France
E-mail: alban.guyot@cirad.fr

Benjamin BOUSSEAU
Eng. – SEFWOOD S.A.
Address: SEFWOOD S.A., 10 rue de Florivoie, 88640 Granges-Sur-Vologne, France
E-mail : benjamin.bousseau@sefwood.com

Etienne CUNY
Sales Manager – SEFWOOD S.A.
Address: SEFCCO S.A., 10 rue de Florivoie, 88640 Granges-Sur-Vologne, France
E-mail: etienne.cuny@sefwood.com

Marie-France THEVENON
Dr. HdR – Cirad, UPR 114 BioWooEB
Address: CIRAD-Unité de Recherches BioWooEB, TA B 114/16, Montpellier, France
E-mail: marie-france.thevenon@cirad.fr

Abstract:
Thermal modification is now considered as a new ecofriendly industrial wood modification process improving mainly the material decay resistance and its dimensional stability. Most industrial thermal treatment processes use convection heat transfer which induces sometimes heterogeneous treatment temperature propagation within the oven and lead to the heterogeneity in treatment efficiency. Thus, it is common that treatment is not completely effective on several stack boards, in a same batch. The aim of this paper was to study the decay resistance variability of various European wood species thermally modified. Thermal modifications were performed around 240°C during 4h, on about 10m^3 of 27x152x2000mm^3 wood planks placed in an industrial oven having a volume of 20m^3, on the following wood species: spruce, ash, beech and poplar. All of the tests concerning the decay resistance were carried out in the laboratory using untreated beech and pine woods as reference materials. An agar block test was used to determine the resistance of thermally modified woods, leached beforehand according to EN 84 standard or not, to brown-rot and white-rot fungi, according to XP CEN/TS 15083-1. A large selection of treated wood samples was tested in order to estimate the variability of treatment efficiency. Thermal treatment increased the biological durability of all leached and un-leached modified wood samples, compared with native wood species. The treatment temperature of 240°C used in this study is sufficient to reach durability classes ‘‘durable’’ or ‘‘very durable’’ for the four wood species. However, the dispersion of weight loss values, due to the fungal attacks was very important and showed a large variability of the durability of wood which has been treated in a single batch. These results showed that there is a substantial need to develop process control and indicator in

* Corresponding author

order to insure that the quality of treated timber is properly evaluated with a view to putting this modified timber on the market under a chain of custody.

***Key words:** European wood species; heat treatment; industrial process; decay resistance; variability.*

INTRODUCTION

Wood thermal modification process is an attractive method to protect wood material against Moisture Content (MC) variations (Pétrissans *et al*. 2013) and fungal attacks (Borrega *et al*. 2009, Candelier *et al.* 2016a). Heat treatment consists in a wood torrefaction performed in a very poor oxygen atmosphere (vacuum, oil, steam pressure etc.) to avoid wood combustion, at temperatures usually ranging from 180°C to 250°C, depending on the industrial process system used, and according to the final targeted modified wood properties (Candelier *et al*. 2016b). Whatever the process used, earlier works have shown that thermal degradation of wood depends on heat treatment intensity, being directly related to treatment temperature and duration, and conditioning therefore the final properties of heat treated wood (Rep *et al*. 2004, Welzbacher *et al*. 2007). Heat treatment modifies the chemical composition of wood cell wall polymers which results in a wood Mass Loss (ML %). ML is mainly attributed to the degradation of the hemicelluloses, which are the most thermally sensitive polymers of the cell wall matrix of wood (Nguila Inari *et al*. 2007, Windeisen *et al*. 2009) and this leads to lower the Equilibrium Moisture Content (EMC) of heat treated wood (Esteves and Pereira 2009). In spite of others hypotheses issued from past studies as: (i) new toxic substances form extractives compounds degradation (Peters *et al*. 2009); (ii) modification of the lignin chemical composition preventing the fungal enzymes from degrading molecules (Vallet *et al*. 2001) and (iii) hemicelluloses degradation resulting in a decrease in nutrients for fungi (Hakkou *et al*. 2006), low EMC of heat-treated wood remains the most predominant explanation about the enhanced decay resistance of the thermally modified material. Indeed, wood colonization by fungal decay needs a Moisture Content (MC) above the fiber saturation point, so that the cell lumens contain some free-water. For native and heat treated woods, under this MC level, the cell wall is swollen and the size of nano-pores in the cell wall reaches their maximums. In addition, for heat treated woods, the role of cross-linking reactions in the wall matrix is also important (Hosseinpourpia and Mai 2016, Jalaludin *et al*. 2010) and their stiffer wood matrix is less accessible to decay fungi due to lower nano-pore sizes and a low EMC (Jalaludin *et al*. 2010). Even if the decay properties improvements of wood by thermal modification process are well known, having a heat-treated wood material of constant quality remains challenging. Most thermal treatment companies use processes by convection heat transfer (Militz 2002) which induces sometimes heterogeneous treatment temperature propagation within the oven (between the periphery and the center of the stack of wooden plates) and lead to the heterogeneity in treatment efficiency (Pétrissans *et al*. 2007). The challenge to produce final products with constant quality (durability, dimensional stability, color etc.) is also in relation with the timber species heterogeneities (within and between the species) (Willems 2013, Hamada *et al*. 2016). Inter-species property differences including parameters such as anatomy, chemical composition, moisture content and density have been reported to strongly influence degradation reactions occurring during thermal treatment and consequently the final properties of the material (Chaouch *et al*. 2010, Candelier *et al*. 2011). Thus, it is common that a treatment is not completely effective on several stack boards, in a same batch. Moreover, phenomena of cracking or delamination on the final product can be observed, causing then material and economical losses (Pétrissans *et al*. 2007).

Another challenge when qualifying heat treated timber is to use the most adequate and discriminant criteria. The durability against Basidiomycetes of heat treated wood is probably the most important to consider for a use class 3, where most end uses of heat treated wood are concentrated (cladding, …). The purpose of the standard XP Cen/TS 15083-1 (2006) is to give a method allowing a durability classification of such treated material. However, a previous ageing such as leaching is not mandatory prior to the fungal exposure, and the obligatory fungi are in limited number.

This present study is focused on the evaluation of the decay resistance variability of various European wood species thermally modified, by the French company SEFWOOD. Thermal modifications were performed in an industrial oven on about 10m^3 of 27x152x2000 mm^3 wood planks of spruce, ash, beech and poplar wood species, each wood species being treated in separated batches. For each wood species a large selection of treated wood samples were tested in order to estimate the variability of modified wood decay resistance, with and without previous leaching tests. In addition, three different brown rots and one white rot have been used on both hardwood and softwood, in order to observe if some fungal strains could be more discriminants than others. These results allow us to observe the decay resistance values of all sample repartitions according to the different durability classes relative to the European standard XP Cen TS 15083-1 (2006).

MATERIAL AND METHODS

Initial Wood samples

Industrially kiln-dried heartwood planks of ash (*Fraxinus* spp), beech (*Fagus sylvatica),* poplar (*Populus* spp) and spruce (*Picea* spp) woods were selected by the French company SEFWOOD (88640 Granges-Sur-Vologne, France).

Untreated pine (*Pinus sylvestris*) sapwood and beech (*Fagus sylvatica*) of respective density 559 and 700Kg/m^3, were used for reference material.

Heat treatment protocol

Each wood species were thermally treated separately. For each batch, 10m^3 of wood planks with dimensions of 2000-2300x155x27mm^3 [L, R, T] were placed into an oven of 20 cubic meter. Thermal treatments were performed at a temperature of 240°C, during 4 hours.

The respective densities of heat-treated ash, beech, poplar and spruce woods were 581, 598, 411 and 392Kg/m^3, respectively.

Wood sampling for decay resistance test

For each wood species, one wooden strip [800x27x15 mm^3 (L, R, T)] was taken from 30 treated wood boards selected randomly throughout the oven. All strips were conditioned to constant weight at 20±2°C and 65±5% Relative Humidity (RH) before being cut into several specimens of dimensions of 50x25x15mm^3 (L, R, T). These wood samples were again conditioned to constant weight at 20±2°C and 65±5% RH. Sampling distribution was done so that at least one piece from each treated wooden strip was used to determine the initial moisture and to evaluate the durability, with and without leaching, towards each fungus. Specimens were selected to be free of knots and other visible wood defects. 30 heat-treated wood samples coming randomly from all wooden strips were assigned to each test modality.

Leaching process

Half of the selected specimens (30 samples) from each heat treated wood species were submitted, beforehand to be subjected to the decay resistance test, to leaching according to the EN 84 (1997). Test samples were conditioned in the same way as it had been done before water impregnations (20°C, 65% RH until constant weight).

Samples were covered with deionized water (1 volume of wood for 5 volumes of water) and placed in the impregnation vessel. Samples were subjected to 4kPa vacuum for 20min. Following the vacuum, the samples stayed in the water for 2h before the water was changed for the first time. Then, the specimens were submersed in deionized water for 14 days, and 10 water changes were done regularly (including the water change after impregnation in 2h). After 14 days, the samples were removed from the water and dried until their weight is stable, at 20±2°C and 65±5% RH.

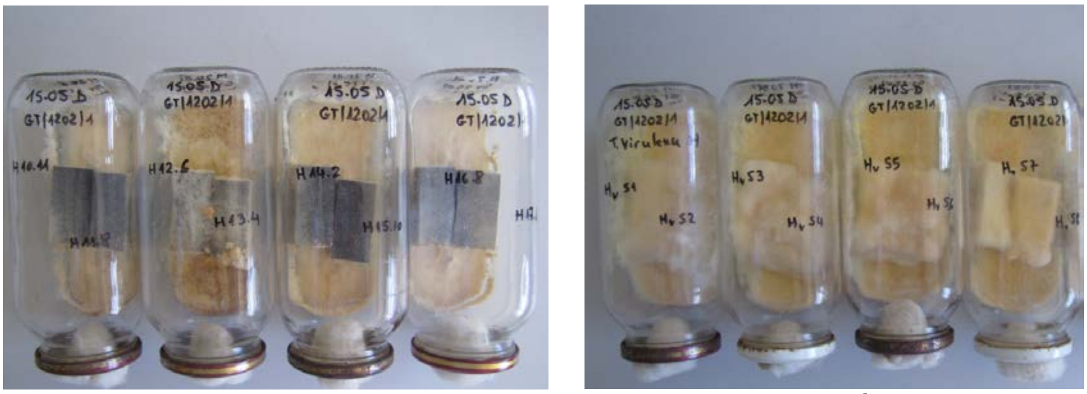
Decay resistance tests

Decay resistance tests were performed according to XP Cen/TS 15083-1 (2006). However, these tests have been conducted with more fungi than those mandatory in this standard. The following fungi were selected: three brown-rots [*Coniophora puteana* (CP) (BAM ebw. 15); *Poria placenta* (PP) (FPRL 280); *Gloeophyllum trabeum* (GT) (BAM Ebw. 109)] and one white-rot [*Coriolus versicolor* (CV) (CTB 863 A)].

The decay tests were conducted as follows: for each test fungus, 30 replicates (un-leached and leached) of each heat treated wood species (Fig. 1) were used. Beech (*Fagus sylvatica*) and Pine (*Pinus sylvestris*) sapwood samples of the same dimensions: 50x25x15mm^3 (L, R, T) were used as controls for the virulence of the strains (ten test blocks for each fungus).

Fig. 1.
Heat treated and leached Ash wood samples after exposition to Coriolus versicolor (CV).

The test specimens (two blocks per flask) were placed on stainless steel supports over the fungal mycelium (grown on 40g malt and 20g agar in 1L of distilled water) and incubated for 16 weeks in a climatic chamber (22°C, 70% relative humidity). Control, leached and un-leached wood samples were tested separately (Fig. 2). At the end of the test, all the tested specimens were removed, cleaned and oven dried (103±2°C) to a constant weight and weighed to determine their weight loss (WL %) due to their fungal degradation.



a. **b.**

Fig. 2.
Decay resistance test devices of leached heat treated Beech wood samples:
a - and Beech wood control samples during their expositions to Gloeophyllum trabeum (GT).

The classification of heat treated wood decay resistances was determined according to the median values of weight loss (X), as specify in the XP Cen/TS 15083-1 (2006) standard (Table 1). However, the analysis of all weight loss results obtained have been conducted on their minimum, maximal, average and mean values. In addition, the weight loss variability for each modality test has been evaluated, as recommended in EN 350 (2016).

Table 1

Durability rating scale according to XP CEN/TS 15083-1 (2006)

Durability class	Description	Weight loss in % (median values)
1	Very durable	X < 5
2	Durable	5 < X ≤ 10
3	Moderately durable	10 < X ≤ 15
4	Slightly durable	15 < X ≤ 30
5	Not durable	X > 30

RESULTS AND DISCUSSION

Decay resistance test validation

According to the weight loss values obtained concerning the fungal degradation of beech and Pine wood control sample, the decay resistance test performed through this study has been validated. Indeed, in accordance with Table 2, the minimal degradation level of control samples were reached.

Whatever the pre-ageing test, *Gloeophyllum trabeum* was the most aggressive rot on beech and Pine control samples, whereas *Coriolus versicolor* was the less degrading rot on these control samples (the maximal WL value reached by *Coriolus versicolor* was 26.7%, with leached Beech wood samples). These results confirm the fact that the brown rots were more degrading than white rot for native European wood species for wood biological degradation (Schwarze 2007). In addition, previous studies have shown that brown rot decay is the most destructive form of biological wood deterioration (Highley and Illman 1991), which considerably affects strength properties before that weight loss is observable (Wilcox 1878).

Table 2

Average values of weight losses of beech and pine control samples according to the four tested fungus, and decay resistance validation test conditions relative to XP CEN/TS 15083-1 (2006)

	Average weight loss		**Minimal (average) weight loss values of control samples, required in XP CEN/TS 15083-1 (2006)**
	In % (m/m)		
	Virulence Control samples associated to un-leached wood samples	Virulence Control samples associated to leached wood samples	In % (m/m)
Brown rot on *Pinus*			
Coniophora puteana	36.1	37.6	**30**
Poria placenta	44.9	43.8	**20**
Brown rot on *Fagus*			
Coniophora puteana	31.9	34.5	**30**
Poria placenta	35.6	35.4	No requirement
White rots on *Pinus*			
Coriolus versicolor	16.6	15.4	No requirement
White rots on *Fagus*			
Coriolus versicolor	25.6	26.7	**20**
Optional fungi			
Brown rot on *Pinus*			
Gloeophyllum Trabeum	45.9	43.9	20*
Brown rot on *Fagus*			
Gloeophyllum Trabeum	37.8	36.2	No requirement

* According to EN 113(1996), the minimum mass loss required is 20%

Heat treated wood durability

Durability of un-leached heat treated wood

Results of un-leached heat treated wood decay are given in Table 3. For each tested rot, a similar tendency concerning the decay resistance improvement of all wood species was observed after thermal treatments (Kamdem *et al*. 2002, Esteves and Pereira 2009). However, conferred wood durability by thermal modification depends on the treatment intensity and wood species (Momohara *et al*. 2003, Candelier *et al*. 2016a, Candelier *et al*. 2016b).

Mean values of weight loss due to fungal degradation show that *Poria placenta* was the most aggressive rot on heat treated spruce wood samples (WL = 7.8%), whereas *Coriolus versicolor* was the most degrading rot on heat treated Beech, Ash and Poplar wood samples (WL = 9.6%, WL = 4.3% and WL = 7.5% respectively). The most obvious explanation to the observed higher colonization of white rot in thermally modified hardwoods is the destructions in the cell wall due to heat, which would facilitate fungal nutrition and initial colonization of the wood (Alfredsen *et al*. 2008). The heat also degrades hemicelluloses to a greater extent than the other macromolecular components (Shafizadeh and Chin 1977), which may give easier access to the lignin for the white rot fungus *Coriolus versicolor*.

On the opposite, the main conclusion issued from previous studies conducted on the understanding of fungal colonization of modified pine wood was that the theory of moisture exclusion via the reduction of cell wall voids provides a consistent explanation for the initial inhibition of brown rot degradation in modified wood (Ringman *et al*. 2014a, Ringman *et al*. 2014b, Jellison *et al*. 2013).

These results are in agreement with the study from Elaieb *et al*. (2015) who showed that *Poria placenta* was the most aggressive fungus on 3 heat treated (230°C – with various durations) Tunisian softwood species (*Pinus halepensis*, *Pinus pinaster* and *Pinus radiata*), and previous study from Candelier *et al*. (2016a) that highlighted that *Coriolus versicolor* was the most degrading fungus of heat treated (170, 200, 215 and 228°C – 2 hours) ash wood (hardwood species). Severo *et al*. (2016) have also demonstrated that the effect of heat treatment performed on *Hevea brasiliensis* wood (hardwood) appears to be more effective, in term on decay resistance improvement, in modified wood incubated with brown-rot fungus (*Gloeophyllum trabeum*) than with white rot fungi (*Pycnoporus sanguineus*).

Table 3

Mean and median values of weight loss for all exposed heat treated wood samples, without leaching, and provisional natural durability class of the tested heat treated wood species

Heat treated wood species	Tested fungi	Without leaching				
		Minimal value of weight loss	Average value of weight loss	Maximal value of weight loss	**Median value of weight loss**	**Durability class**
		In % (m/m)	In % (m/m)	In % (m/m)	**In % (m/m)**	**Refer to Table 1**
Ash	*Coniophora puteana*	0.0 %	1.4%	7.2 %	**0.6%**	**1**
	Poria placenta	0.0 %	0.8%	13.8 %	**0.0%**	**1**
	Coriolus versicolor	0.6 %	5.6%	31.1 %	**4.3%**	**1**
	Gloeophyllum trabeum	0.0 %	0.7%	2.8 %	**0.4%**	**1**
Beech	*Coniophora puteana*	0.0 %	3.2%	15.1 %	**1.6%**	**1**
	Poria placenta	0.0 %	2.5%	13.9 %	**2.0%**	**1**
	Coriolus versicolor	2.7 %	9.8%	19.6 %	**9.6%**	**2**
	Gloeophyllum trabeum	0.4 %	1.2%	2.2 %	**1.2%**	**1**
Spruce	*Coniophora puteana*	0.0 %	4.5%	15.7 %	**4.0%**	**1**
	Poria placenta	0.0 %	10.4%	36.2 %	**7.8%**	**2**
	Coriolus versicolor	0.6 %	3.1%	8.3 %	**2.8%**	**1**
	Gloeophyllum trabeum	0.0 %	0.9%	2.2 %	**0.8%**	**1**
Poplar	*Coniophora puteana*	0.0 %	2.6%	11.3 %	**0.2%**	**1**
	Poria placenta	0.0 %	6.0%	24.1 %	**3.7%**	**1**
	Coriolus versicolor	2.4 %	8.1%	19.8 %	**7.5%**	**2**
	Gloeophyllum trabeum	0.0 %	2.3%	17.9 %	**0.6%**	**1**

To compare with our present study, Candelier *et al*. (2016a) found, by performing screening decay resistance tests [according to the method of Bravery and Dickinson (1979), during 12 weeks of incubation period] on heat treated ash wood at 228°C during 4 hours, the following median values of weight loss; 1.1%, 0.7%, 0.8% and 0.9% for the respective fungal attack towards *Coriolus versicolor*, *Poria placenta*, *Coniophora puteana* and *Gloeophyllum trabeum*, indicating thus the same durability class.

ONLINE ISSN 2069-7430
ISSN-L 1841-4737
PRO LIGNO
www.proligno.ro
Vol. 13 N° 2 2017
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Durability of leached heat treated wood

Results of leached heat treated wood decay are given in Table 4. If we focus on durability classes obtained for each treated wood species, with and without leaching, leaching process seems to have positive effect for heat treated beech wood (from durability class 2 to durability class 1 after leaching), and no effect for the other heat treated wood species.

In addition, the decay resistance of heat treated wood samples for the four tested fungi appears to be less heterogeneous, according to the Median values of weight loss (Table 3 and Table 4), after leaching. These results could be explain by the leaching process removing, from the heat treated wood surface, some extractives compound formed during the wood thermal degradation that could act as fungicides (Peters *et al*. 2009).

Even if the results are quite variable upon the wood species, slight trends concerning the weight loss values of heat treated wood after leaching can be observed: (i) an increase of weight loss values for brown-rot degradation and (ii) a decrease of weight loss values for white-rot degradations, between un-leached and leached heat treated wood submitted to decay resistance tests. This tendency could be explained by the fact that leaching process removed mainly sugar compounds forming carboxylic acids and aldehydes (Meija-Feldmane *et al*. 2015), tannic acid, acetic acid, furfural and furfural derivatives (Graf *et al*. 2005).

Table 4

Mean and median values of weight loss for all exposed heat treated wood samples, after leaching, and provisional natural durability class of the tested heat treated wood species

Heat treated wood species	Tested fungi	After leaching				
		Minimal value of weight loss	Average value of weight loss	Maximal value of weight loss	**Median value of weight loss**	**Durability class**
		In % (m/m)	In % (m/m)	In % (m/m)	**In % (m/m)**	**Refer to Table 1**
Ash	*Coniophora puteana*	0.0 %	4.6%	15.0 %	**2.6%**	**1**
	Poria placenta	0.0 %	1.2%	4.2 %	**0.8%**	**1**
	Coriolus versicolor	0.1 %	4.5%	13.5 %	**3.7%**	**1**
	Gloeophyllum trabeum	0.0 %	1.4%	5.5 %	**1.2%**	**1**
Beech	*Coniophora puteana*	0.0 %	4.7%	15.1 %	**4.3%**	**1**
	Poria placenta	0.0 %	2.8%	11.1 %	**1.8%**	**1**
	Coriolus versicolor	1.6 %	4.7%	13.7 %	**3.5%**	**1**
	Gloeophyllum trabeum	0.6 %	3.3%	22.7 %	**1.5%**	**1**
Spruce	*Coniophora puteana*	0.0 %	3.7%	13.0 %	**2.2%**	**1**
	Poria placenta	0.0 %	8.7%	34.8 %	**5.9%**	**2**
	Coriolus versicolor	0.2 %	3.3%	8.6 %	**3%**	**1**
	Gloeophyllum trabeum	0.0 %	1.2%	6.4 %	**1.0%**	**1**
Poplar	*Coniophora puteana*	0.0 %	3.8%	14.8 %	**1.9%**	**1**
	Poria placenta	0.0 %	4.7%	16.2 %	**2.1%**	**1**
	Coriolus versicolor	0.9 %	6.0%	14.7 %	**5.5%**	**2**
	Gloeophyllum trabeum	0.0 %	3.9%	24.8 %	**1.4%**	**1**

ONLINE ISSN 2069-7430
ISSN-L 1841-4737
PRO LIGNO
www.proligno.ro
Vol. 13 N° 2 2017
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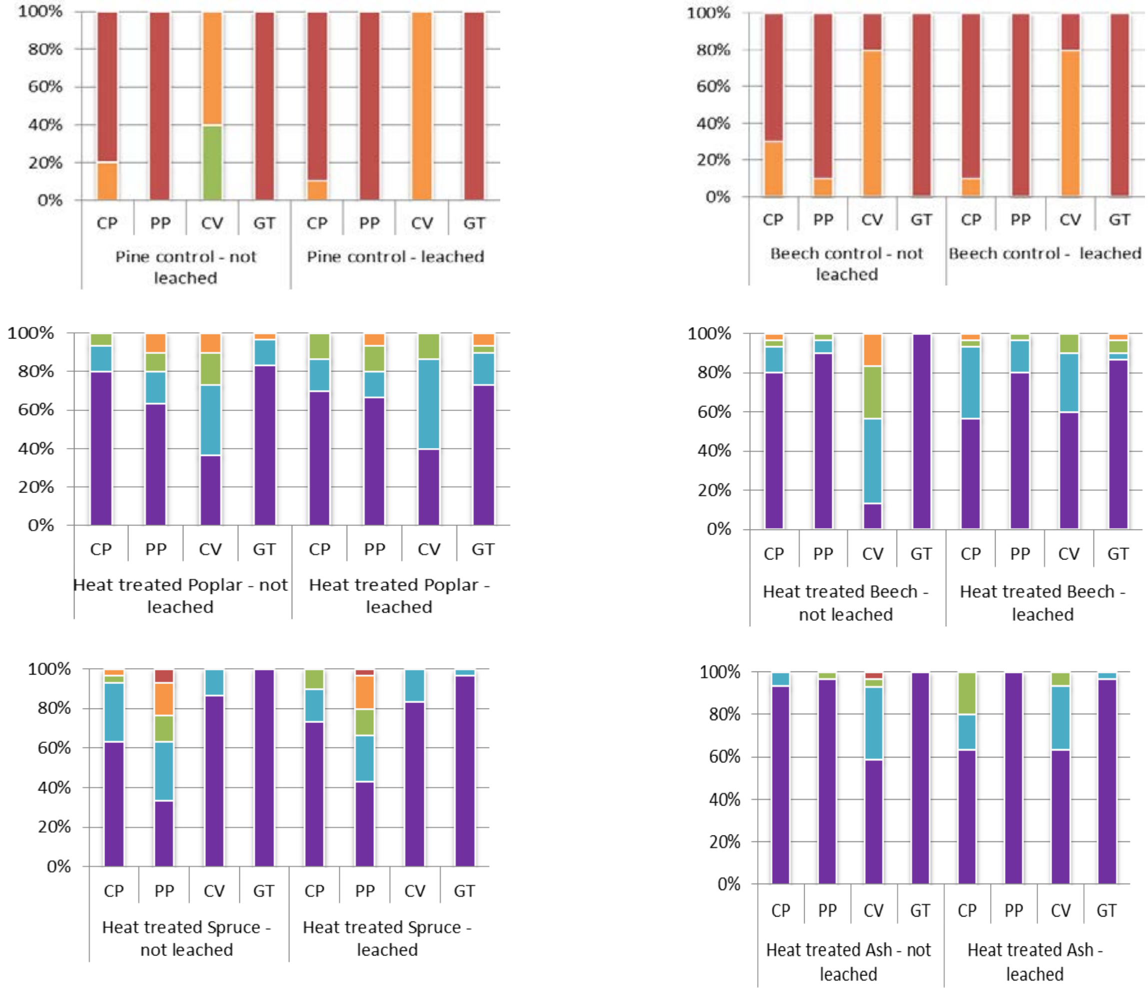
Variability of heat treated wood decay resistance

Fig. 3 illustrates the repartition (in percent) of weight loss mean values of all sample according to the different durability classes relative to the XP Cen/TS 15083-1 (2006) standard (Table 1), for all heat-tread wood species and control samples, with or not leaching.



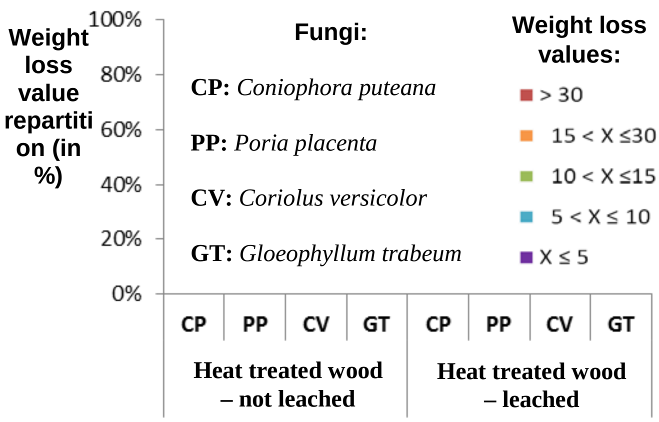


Fig. 3.
Weight loss values variability, for all treated and untreated wood species and control sample, with or not leaching.

It appears that the weight loss values of Spruce due to fungal degradation are more heterogonous than those of hardwood species and that for each test modality (with or without leaching). These results variability are more pronounced for the most aggressive fungi, whatever the heat treated wood species.

Recently, Shchupakivskyy *et al*. (2014) have highlighted a higher susceptibility of oak's Early-Wood (EW) to thermal degradation comparatively to its Late-Wood (LW) using high-frequency densitometry. Differences between EW and LW properties contribute to changes in timber dimensional stability (Kretschmann and Cramer 2007). Furthermore, differences in the chemical composition between EW and

LW exist (Kibblewhite *et al*. 2010), which may influence their thermal degradation kinetic. Through an intra-ring study on heat treated oak wood, Hamada *et al*. (2016) studied the thermal sensitivity of both Early-Wood and latewood. They observed that Early-Wood was more sensitive to thermal degradation than Late-Wood. This difference of thermal reactivity was attributed to the higher cellulose content of Late-Wood which presents larger S2 layer, rich in cellulose, in the secondary wall. Early-Wood, containing more lignin and hemicelluloses fractions than latewood, is therefore more susceptible to thermal degradation. The ratio EW/LW contained into our different heat treated wood samples, whatever the wood species, could partly explain the variability of heat treatment efficiency and of course the heterogeneity of decay resistance results.

CONCLUSION

Heat treatment improves wood durability, increasing clearly the resistance to brown and white rots. Mean values of weight loss due to fungal degradation show that *Poria placenta* was the most aggressive rot on heat treated softwood samples, whereas *Coriolus versicolor* was the most degrading rot on heat treated hardwood samples. *Coniophora puteana* was the most discriminant fungus only in the case of leached heat treated beech. In addition, the difference of decay resistance, according to the Median values of weight loss, of heat treated wood samples between the four tested fungi appears to be less pronounced, when the samples have been submitted to a pre-ageing leaching test. Finally, the weight loss results from Spruce (softwood) fungal degradation are more heterogonous than those of hardwood species, and that for each test modality (with or without leaching step). For each wood species, this results variability was also more pronounced for their respective most aggressive fungus. Along with the variability of the native timber, the variability of the heat treatment, the ratio EW/LW contained into the different heat treated wood samples could partly explain the variability of heat treatment efficiency and of course the heterogeneity of decay resistance results.

AKNOWLEDGMENTS

The authors gratefully acknowledge the French Company ‘‘SEFWOOD” and more particularly Mr. Cuny and Mr. Bousseau for their financial support and collaboration brought to this research project.

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