Resistance of thermally modified ash (*Fraxinus excelsior* L.) wood under steam pressure against rot fungi, soil-inhabiting micro-organisms and termites

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Abstract Thermal modification processes have been developed to increase the biological durability and dimensional stability of wood. The aim of this paper was to study the influence of ThermoWood® treatment intensity on improvement of wood decay resistance against soil-inhabiting micro-organisms, brown/white rots and termite exposures. All of the tests were carried out in the laboratory with two different complementary research materials. The main research material consisted of ash (*Fraxinus excelsior* L.) wood thermally modified at temperatures of 170, 200, 215 and 228 °C. The reference materials were untreated ash and beech wood for decay resistance tests, untreated ash wood for soil bed tests and untreated ash, beech and pine wood for termite resistance tests. An agar block test was used to determine the resistance to two brown-rot and two white-rot fungi according to CEN/TS 15083-1 directives. Durability against soil-inhabiting micro-organisms was determined following the CEN/TS 15083-2 directives, by measuring the weight loss, modulus of elasticity (MOE) and modulus of rupture (MOR) after incubation periods of 24, 32 and 90 weeks. Finally, *Reticulitermes santonensis* species was used for determining the termite attack resistance by non-choice screening tests, with a size sample adjustment according to EN 117 standard directives on control samples and on samples which have previously been exposed to soil bed test. Thermal modification increased the biological durability of all samples. However, high thermal modification temperature above 215 °C, represented by a wood mass loss (ML%) due to thermal degradation of 20%, was needed to reach resistance against decay comparable with the durability classes of ''durable'' or ''very durable'' in the soil bed test. The brown-rot and white-rot tests gave slightly better durability classes than the soil bed test. Whatever the heat treatment conditions are, thermally modified ash wood was not efficient against termite attack neither before nor after soft rot degradation.

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1 Introduction

Heat treatment consists of a wood pyrolysis torrefaction performed in a very poor oxygen atmosphere to avoid wood combustion. The environmental impact of this process is low, heat is introduced in the treatment system and smoke from wood thermal degradation can be retrieved, condensed and purified (Pétrissans et al. 2007). At its end of life cycle, heat-treated wood can be recycled without detrimental impact on the environment, contrary to some chemically impregnated wood containing biocidal active ingredients (CRIQ 2003). Although several processes (e.g. Plato-Process, Bois Perdure, OHT-Process, ThermoWood Process, etc.) exist due to conditions used for heat conduction (Hannouz 2014), the basic concept is common, that is to modify wood chemical structure at a defined temperature in inert atmosphere (Militz 2002). There are plenty of end-uses for thermally modified timber in many different applications, such as exterior cladding, covered decking, flooring, garden furniture, paneling, kitchen furnishing, and bathroom decor.

Thermal modification of wood, using soft pyrolysis at heating temperature ranging from 180 to 280 °C, is an attractive environmentally acceptable treatment to protect wood material against moisture content (MC) variations (Hill 2006; Pétrissans et al. 2003) and Basidiomycetes degradation (Tjeerdsma et al. 2000), to improve dimensional stability (Korkut et al. 2012), and increase the dark coloration of wood (Chen et al. 2012). Therefore, heat treatment allows to use low durability timber by making it resistant against decay for end-use in use class 2 and 3.1 [use class 3.2 has to be checked by field tests (Welzbacher and Rapp 2007), and use class 4 being excluded due to the occurrence of soft rots and termite degradation] (EN 335, 2013b) and with high economic value (Allegretti et al. 2012; Kamdem et al. 2002). These upgraded biological properties conferred to the wood are the result of chemical modifications of wood cell wall polymers occurring during treatment (Inari et al. 2007). However, the surface hardness of the heat treated wood can be improved for several wood species thermally modified at treatment temperature lower than 210 °C (Sivrikaya et al. 2015a). Low temperature and time cause the increase in the hardness value while high temperature and time decrease the hardness value of heat-treated wood (Lekounougou and Kocaefe 2014), due to higher degradation of hemicellulose and cellulose and evaporation of extractive compounds (Karamanoğlu and Akyıldız 2013; Esteves and Pereira 2009). These last improvements have an adverse effect on wood mechanical properties such as bending and compression strength, stiffness and shear strength (Dilik and Hiziroglu 2012; Candelier et al. 2013; Hannouz et al. 2015, Hermoso et al. 2015). The improved fungal resistance of thermally modified wood has also been reported by Viitanen et al. (1994), Sailer et al. (2000), Hakkou et al. (2006), Welzbacher and Rapp (2007), Mburu et al. (2006) and Boonstra et al. (2007). Sivonen et al. (2003) studied the chemical properties of thermally modified Scots pine exposed to brown and soft-rot fungi and found that, as with the untreated wood, brown-rot fungi degraded mainly hemicelluloses, while soft-rot fungus attacked cellulose more extensively. Weight loss caused by fungal attack was dependent on the thermal modification temperature and duration. Previous studies, mainly achieved at laboratory scale, have proven high correlations between mass loss issued from wood thermal degradation (ML%), treatment intensity (time and temperature) and weight loss (WL%) of heat-treated wood due to fungal decay (Hakkou et al. 2006; Chaouch et al. 2013; Candelier et al. 2016). Weiland and Guyonnet (2003) also found that in spite of strong hemicellulose degradation by thermal modification, the fungal attack still takes place. In addition, the degradation of wood components caused by decaying fungi decreases the mechanical properties of wood (Curling et al. 2002; Metsä-Kortelainen and Viitanen 2010, Råberg et al. 2012).

The impact of thermal treatment on wood termite resistance has also already been studied. However, it appears that the termite resistance of heat treated wood was impacted in a random way, was indiscriminate and even reduced in many cases, according to modification process conditions. Momohara et al. (2003) worked on Japanese cedar (*Cryptomeria japonica* D. Don) drying under saturated steam at different temperatures (from 60 to 150 °C) and with several treatment durations (from 6 to 72 h). They highlighted that Japanese cedar wood samples treated at higher temperatures (135–150 °C) showed larger weight loss due to termite attack than those treated at lower temperatures (60 °C). They observed significant differences between 135 or 60 °C and 150 °C. In the case of Japanese larch heartwood, steam treatment was also reported to increase termite attraction (Doi et al. 1998). For severe treatment temperature, Salman et al. (2016) found that Scots pine sapwood (*Pinus sylvestris* L.) treated at 220 °C for 20 h, submitted to termite attack after or without leaching, was equally degraded compared to untreated wood, although the termite mortality rate was higher for treated wood samples. On the other side, Nunes et al. (2004) studied the resistance of wood heat-treated by hot oil bath (OHT) to the termite *Reticulitermes grassei* and concluded that despite the slightly higher mortality of termites in treated samples and smaller mass loss, the difference was not significant. However, when treated and untreated counterpart wood samples were side by side during exposure, termites preferred untreated wood. These last results (Nunes et al. 2004) could be justified by the addition of oil into the wood during the thermal modification process. However, in such a treatment, the oil is sufficient to make the wood treated with it more hydrophobic, making it more resistant to the attack of fungi and termite. In another study, more similar to this present work, Sivrikaya et al. (2015b) studied the impact of Thermowood® process on *Reticulitermes grassei* termite resistance of different wood species and showed that such a heat treatment did not enhance the termite resistance of ash wood; the survival rate of the termite workers gradually increased irrespective of the increase in temperature, and that to a similar extent to the samples degradations.

The aim of this study was to measure the impact of ThermoWood® process conditions (treatment intensity) on the thermal degradation kinetic of ash wood and on its conferred durability against brown rot, white rot and during unsterile soil bed exposure. Finally, resistance to termite tests have been performed on ash wood control samples, beech wood control samples and heat-treated ash wood samples which have been submitted to soil bed tests beforehand.

2 Materials and methods

2.1 Wood samples

Industrially kiln-dried heartwood planks of ash wood (*Fraxinus excelsior* L.) were selected from a French company of Wood thermal modification (Bois Durables de Bourgogne, 71120 Vendenesse-lès-charolles, France). For the laboratory thermal modification operations, 20 planks [4000 × 25 × 110 mm^3 (L × R × T)] of test material, with only small variations in density (650 kg/m^3 ± 10%) and widths of annual rings, were selected. The planks were selected so that they do not include heartwood. All planks were split down the middle and cut into two 2 m-long pieces. One half of the planks were left as reference material and the other half was thermally modified.

2.2 Heat treatment protocols

The ThermoWood® method (Finnish Thermowood Association 2003) was used in all the thermal modification operations, by the French company Bois Durables de Bourgogne®. The temperature inside the wood and the atmosphere in the kiln were measured during the processes. The thermal modification operations were controlled by these measured temperatures (Fig. 1). The thermal modification duration, at the targeted temperature, was 2 h for every test run while the thermal modification temperature was varied from 170 to 228 °C. Four ash wood planks were treated for each temperature.

Each heat treatment was carried out simultaneously on boards of 2000 × 25 × 110 mm^3 (L × R × T). Thermal treatment was performed in a 20 m^3 industrial oven by convection under steam pressure. The oven temperature was slowly increased by 0.5 °C min^{-1} from ambient to 103 °C to dry each wood board during 48 h and to obtain their anhydrous weight (m_0). After this period, the plate's temperature was increased by about 0.2 °C min^{-1} from 103 to 150 °C and the temperature was maintained for 1 h. The temperature was then increased by about 0.2 °C min^{-1} from 150 °C to the targeted temperature to perform wood thermal modification. The heating system was then stopped and wood samples were cooled down to room temperature under an oxygen free atmosphere.

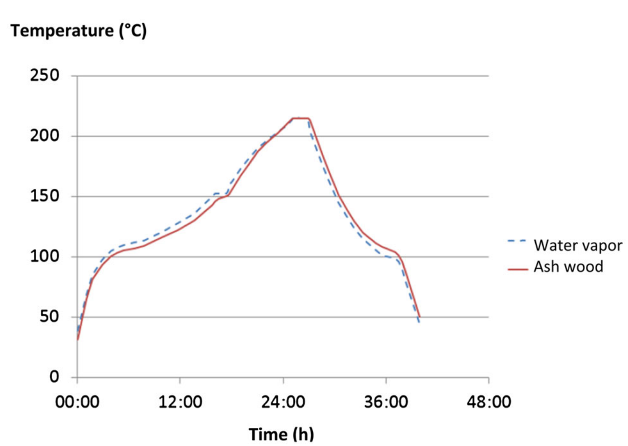


Fig. 1 Temperature kinetics of ash wood and water vapor during heat treatment performed at 210 °C for 2 h

2.3 Mass loss (ML) due to wood thermal degradation

Anhydrous mass measurements were performed on each ash wood plank before (m_0) and after thermal modification (m_1) in order to determine the mass loss (ML%) due to thermal degradation depending on treatment conditions.

$$ML\ (\%) = (m_0 - m_1)/m_0.$$

2.4 Decay resistance tests

According to an adaptation of XP CEN/TS 15083-1 (2006) standard criteria, 20 blocks of 25 × 10 × 5 mm^3 in longitudinal, radial and tangential direction were cut from untreated and from each heat-treated wood modality, and dried at 103 °C for 48 h (m_2). Petri dishes (90 mm diameter) were filled with sterile culture medium prepared by mixing 40 g malt and 20 g agar in 1 L of distilled water, inoculated with the different fungi and incubated at 22 °C and 70% relative humidity to allow full colonization of the surface by the mycelium. Decay resistance was tested on both fungi species required by the standard: *Coriolus versicolor* Quélet (CV) [Linnaeus, CTB 863 A] and *Coniophora puteana* Karsten (CP) [Schumacher ex Fries, Bam Ebw. 15], and on two other non-obligatory fungi species *Gloeophyllum trabeum* Murill (GT) [Persoon ex Fries, BAM Ebw. 109], and *Poria placenta* Coocke sensu J. Erikson (PP) [Fries, FPRL 280]. Three blocks (2 treated and one untreated ash wood sample as control) were placed in each Petri dish and incubated during 12 weeks to evaluate the effect of thermal modification (Fig. 2). For each fungus, nine untreated beech (*Fagus sylvatica* L.) wood samples were distributed into three Petri dishes and subjected to the decay resistance tests to assess the virulence of the fungi. Each experiment was triplicated. After this period, mycelia were removed and the blocks were dried at 103 °C and weighed (m_3) to determine the weight loss (WL) caused by the fungal attack.

$$WL\ (\%) = (m_2 - m_3)/m_2.$$

Weight loss was calculated and expressed as a percentage of their initial values. The results of the decay resistance test were classified into durability classes based

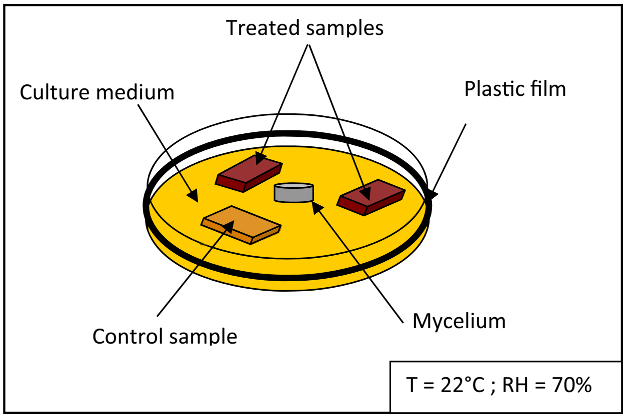


Fig. 2 Screening decay resistance tests according to Bravery and Dickinson (1979) (with some adjustments in size, number of samples and incubation duration)

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

Durability class	Description	Percent loss in mass
1	Very durable	≤5
2	Durable	>5 to ≤10
3	Moderately durable	>10 to ≤15
4	Slightly durable	>15 to ≤30
5	Not durable	>30

on the Weight Losses range, according to the criteria of XP CEN/TS 15083-1 (2006) (the criteria being adjusted to the sample size and the numbers of the tested samples). The estimated durability classes are shown in Table 1.

2.5 Soil bed tests

The natural durability against unsterile soil rot micro fungi of untreated and thermally modified ash wood was determined according to the directives of XP CEN/TS 15083-2 (2006) with adjustments concerning the number of the tested samples and incubation room conditions. Small specimens (100 × 10 × 5 mm^3), issued from treated and untreated woods, were inserted into containers with unsterile soil composed of around 80% of commercial compost (Géolia®—Universal Compost) and 20% of natural soil from Cluny-France (46°26′04″N; 4°39′33″E). PH–H_2O [1:2], determined according to NF ISO 10390 (2005) was 6.6. Water holding capacity (WHC) of the soil used for these tests, determined according to XP CEN/TS 15083-2 (2006) was 45%. The incubation times were 24, 32 and 90 weeks at 20 °C and 65% relative humidity. Each week, an amount of water was added into the containers to preserve the soil humidity.

Ten specimens from each wood material were used for each incubation duration. All samples were conditioned at 103 °C and weighed before (m_4) and after (m_5) unsterile soil incubation period in order to determine their anhydrous masses and weight losses due to soil degradation agents (WL_{sbt}%).

$$WL_{sbt}\,(\%) = (m_5 - m_4)/m_4.$$

Ten untreated beech (*Fagus sylvatica* L.) wood samples were placed in the soil bed for each tested time exposure (24, 32 and 90 weeks) in order to assess the virulence of degradation of the soil.

In order to evaluate the effect of soil degradation agents on untreated and treated ash wood, three-point bending tests (MOE, MOR) were carried out on each sample. Mass loss and mechanical loss results were then compared. An INSTRON 4467 Universal Mechanical Testing Machine was used for the measurements. After having measured anhydrous mass of the samples after unsterile soil exposure, samples were conditioned in a room with 65 ± 5% RH and 20 ± 2 °C for the time necessary to stabilize until constant weight. Three point static bending tests were carried out according to EN 408 (2012). The moving head speed and the span length were 1 mm s^{-1} and 90 mm, respectively. The load deformation data obtained were analyzed to determine the modulus of elasticity (MOE) and the modulus of rupture (MOR). Tests were replicated six times for each treatment and exposure conditions.

Weight loss and MOE loss were calculated and expressed as a percentage of their initial values. The results of the soft-rot test were classified into durability classes based on the calculated X value, with:

$$X_{Value} = \frac{\text{Median value of } WL_{sbt} \text{ for test wood specimens}}{\text{Median value of } WL_{sbt} \text{ for reference wood specimens (beech)}}$$

The durability classes are shown in Table 2.

2.6 Termite tests

Treated and untreated ash wood samples (25 × 10 × 5 mm^3—L, R, T) previously submitted or not

Table 2 Durability rating scale according to XP CEN/TS 15083-2 (2006)

Durability class	Description	Percent loss in mass
1	Very durable	≤0.10
2	Durable	>0.10 to ≤0.20
3	Moderately durable	>0.20 to ≤0.45
4	Slightly durable	>0.45 to ≤0.80
5	Not durable	>0.80

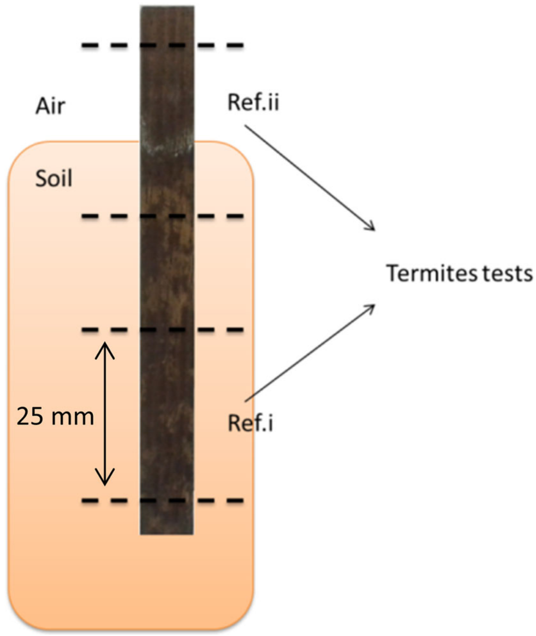


Fig. 3 Heat treated wood degraded by soil-inhabiting micro-organisms sampling to test against termite attacks

to soft rot attacks were exposed to termites (*Reticulitermes santonensis*) in non-choice screening tests. Beech and pine wood have also been tested against termites exposure as reference. Prior to being tested against termites, each sample issued from soil bed tests has been cut into two parts (Fig. 3): soil exposure (ref. i) and air/soil exposure (ref. ii).

Each specimen was placed in the middle of a Petri dish (90 mm diameter) filled with wet sand. 50 termite workers were collected and placed with each tested sample. These specimens were kept at 27 °C and >75% relative humidity for 4 weeks (Fig. 4). Observations were carried out on a weekly basis in order to add water and check termite behavior. At the end of the test, termite survivors were counted, tested sample degradation was given a visual rating according to the criteria of EN 117 (2013a) (the criteria being adjusted to the sample size) and the weight loss (WL_{term}%) was measured by difference between anhydrous masses before and after the termite exposure.

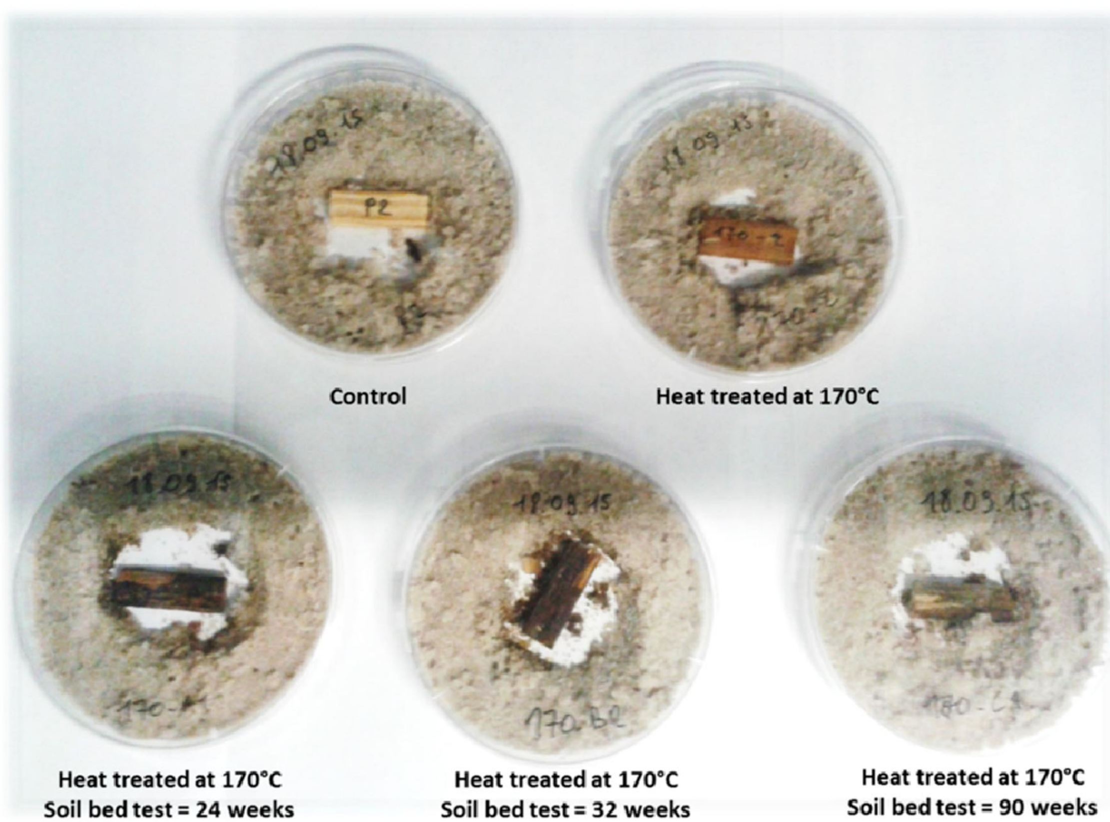


Fig. 4 Screening termites test devices, with adjustments to EN 117 standard guidelines, for control and heat-treated ash wood previously degraded by soil-inhabiting micro-organisms (Ref.i) at different incubation durations

2.7 Statistical analysis

Statistical analyses (one-way analysis of variance) using Fisher test and the JMP 10.0.2 program (SAS Institute Inc., Cary, NC, USA) have been performed. The effects of treatment intensity on mass loss, weight loss due to fungal attack, weight loss due to soil-inhabiting microorganisms, mechanical properties and termite resistance before and after soil bed tests were evaluated using ANOVA and Duncan's comparison test. Such analysis allows to class results into several categories from "a" to "g". Systems which are not connected by the same letter are largely different at the 5% level.

3 Results and discussion

3.1 Mass loss (ML) due to wood thermal degradation

Results of mass loss due to thermal degradation of wood are given in Table 3. According to literature (Candelier et al. 2011; Tenorio and Moya 2013), thermal degradation kinetics of wood, often characterized by mass loss (ML%), is mainly influenced by treatment temperature (Stamm 1956). Indeed, concerning heat treatment performed on ash wood during 2 h at 170, 200, 215 and 228 °C, mass loss of 11, 16, 20 and 25% was obtained, respectively.

Similar results have been found by Chaouch et al. (2010) in a study on the correlation between mass loss and treatment intensity (time and temperature) during the heat treatment of several wood species such as silver fir, pine, beech, poplar and ash. It is reported that wood thermal degradation kinetics strongly depends on the nature of hemicelluloses initially present in wood. In fact, acetic acid liberated during thermal degradation of hemicellulose catalyzes the thermal chemical reactions of wood degradation. This acetic acid production strongly depends on the nature of the wood species: hardwoods lead to higher amounts of acid compared to softwoods. The higher susceptibility of hardwoods to thermal degradation compared to softwoods, has thus been attributed to their higher content in acetyl groups (Sjöström 1981, Fengel and Wegener 1989) from acetylated glucoronoxylan, leading to the formation of a considerable amount of acetic acid involved in hemicelluloses and lignin degradation (Candelier et al. 2011).

3.2 Decay resistance tests

For each tested rot, similar results have been found. However, *Poria placenta* was the most aggressive rot on beech control samples (WL 49.9%), whereas *Coriolus*

Table 3 WL (%) median values and durability class according to decay tests of untreated and heat treated ash woods, according to XP CEN/TS 15083-1 (2006)

Wood species	T °C	ML (%)	CV		PP		CP		GT	
			WL (%)	Durability class	WL (%)	Durability class	WL (%)	Durability class	WL (%)	Durability class
Beech	Control	0 (0)a**	23.0 (0.13)a	4	49.9 (0.11)a	5	49.5 (0.10)a	5	21.8 (0.15)a	4
Ash	Control	0 (0)a	48.0 (0.10)b	5	39.7 (0.09)ab	5	38.2 (0.11)b	5	15.2 (0.19)ab	4
	170	11 (0.31)b	5.02 (0.05)c	2	6.2 (0.12)c	2	5.3 (0.15)c	2	5.8 (0.23)c	2
	200	16 (0.22)c	2.8 (0.13)d	1	2.2 (0.09)d	1	1.9 (0.10)d	1	2.2 (0.18)d	1
	215	20 (0.13)d	1.5 (0.19)e	1	0.9 (0.07)e	1	1.1 (0.15)e	1	1.1 (0.17)e	1
	228	25 (0.12)e	1.1 (0.10)f	1	0.7 (0.05)e	1	0.8 (0.08)f	1	0.9 (0.21)ef	1

* Coefficient of variation represented by the ratio between standard deviation and the average value

** According to one-way analysis of variance, systems not connected by the same letter are largely different at the 5% level *versicolor* was the most degrading rot on ash control samples (WL 48%). Except for ash wood heat-treated at 170 °C where *Poria placenta* was the most aggressive rot (WL 6.2%), *Coriolus versicolor* was the most degrading rot for control and other differently modified ash wood samples. Mass losses caused by *Coriolus versicolor* on ash wood treated at 200, 215 and 228 °C were 2.8, 1.5 and 1.1%, respectively. The thermal modification increased the durability of all wood materials, which is in agreement with previous studies (Kamdem et al. 2002; Esteves and Pereira 2009). These results were expected as Rousset et al. (2004) and Metsä-Kortelainen et al. (2005) also found that the thermal treatment of wood at high temperatures increases the decay resistance of wood. Moreover, Momohara et al. (2003) showed that an increase in duration and temperature during thermal treatment increased the decay resistance of wood, which is in line with the current results. Yalcin and Sahin (2015) studied the decay resistance improvement of narrow-leaved ash wood after thermal treatment under saturated steam and different treatment intensity (temperature range from 140 to 220 °C and for duration ranging between 2 and 6 h). They also found no significant change in the weight loss caused by fungi up to a treatment temperature of 180 °C, but they observed a significant decrease at higher temperatures. The decay resistance of thermally modified narrow-leaved ash increased with increasing duration of heat treatment at higher temperatures. These authors have also correlated the decrease of the weight loss due to fungal attack with the decrease in equilibrium moisture content of the wood and with the changes in the chemical composition of heat-treated wood.

It can be seen in Table 3 that *Coriolus versicolor* (CV), *Poria placenta* (PP) and *Coniophora puteana* (CP) inflicted greater damages to ash control samples compared to degradation due to *Gloeophyllum trabeum* (GT). While after thermal modification, weight loss due to the fungal attack of heat-treated ash wood, with the same treatment intensity, becomes similar whatever the rot fungi.

In most studies, an average mass loss (ML%) due to thermal degradation of 12% confers to heat-treated wood durability class 3 according to the specifications of EN 350-1 (1994) (Welzbacher and Rapp 2002; Kamdem et al. 2002; Chaouch et al. 2010; Elaieb et al. 2015). Although these authors did not use the same test method (EN 350-1, 1994) as that used in this work (XP CEN/TS 15083-1, 2006), they confirmed that a level of heat-treated wood ML higher than 12% makes it a significantly more durable material than respective untreated wood.

In this study, such a mass loss of 12% was reached by a thermal treatment performed at 200 °C (Table 3). According to Table 3, thermal treatment performed at higher temperatures than 200 °C conferred to the heat-treated wood material durability class 1 "very durable", according to the classification method of XP CEN/TS 15083-1 (2006).

This concurs with results from Paul et al. (2006) and Mazela et al. (2004), who reported limited improvement of resistance to fungal decay for heat treatment temperatures below 200 °C. A minimum heat-treatment temperature of 220 °C for a sufficient increase in the resistance to fungal decay is also recommended by Jämsä and Viitaniemi (2001), Welzbacher et al. (2007), Šušteršic et al. 2010) and Syrjänen and Kangas (2000), which also points to the significant impact of the treatment temperature on the resulting treatment intensity (Candelier et al. 2011).

3.3 Soil bed tests

Visual checking, measurements of weight loss, MOE and MOR were performed after 24, 32 and 90 weeks of exposure. The results of the unsterile soil bed test, integrating tests soil-inhabiting microorganisms degradations, highlighted a significant improvement in relative durability of all heat-treated materials compared with ash wood controls.

3.3.1 Visual appearance

According to the visual appearance of wood samples after soil bed tests (Fig. 5), it can be deducted that the soil contained mainly white rots which degrade mainly the lignin component, without causing twist stress to the wood sample (Talaei et al. 2013). Figure 5 clearly shows the influence of thermal treatment temperature on decay resistance of soil-inhabiting micro-organisms on heat-treated ash wood, according to the visual appearance of each wood sample.

3.3.2 Weight loss due to soil bed tests

As shown in Fig. 6, the degree of the value of weight loss (WL_{sbt}) increased as a function of the exposure time to soil-inhabiting micro-organisms, whatever the wood species used as reference and the thermal treatment intensity used on thermally modified ash wood. Nevertheless, as for the resistance to fungal attack, it appears that the wood weight loss due to soil exposure is considerably reduced by thermal treatment carried out on ash wood at temperatures above 200 °C.

From treatment temperature of 215 °C, Thermowood® process allows to classify modified ash wood as a "very durable" material (Table 4) according to XP CEN/TS 15083-2 (2006) (the adjusted criteria are specified in part 2.5) (Table 2). Metsä-Kortelainen and Viitaniemi (2009)

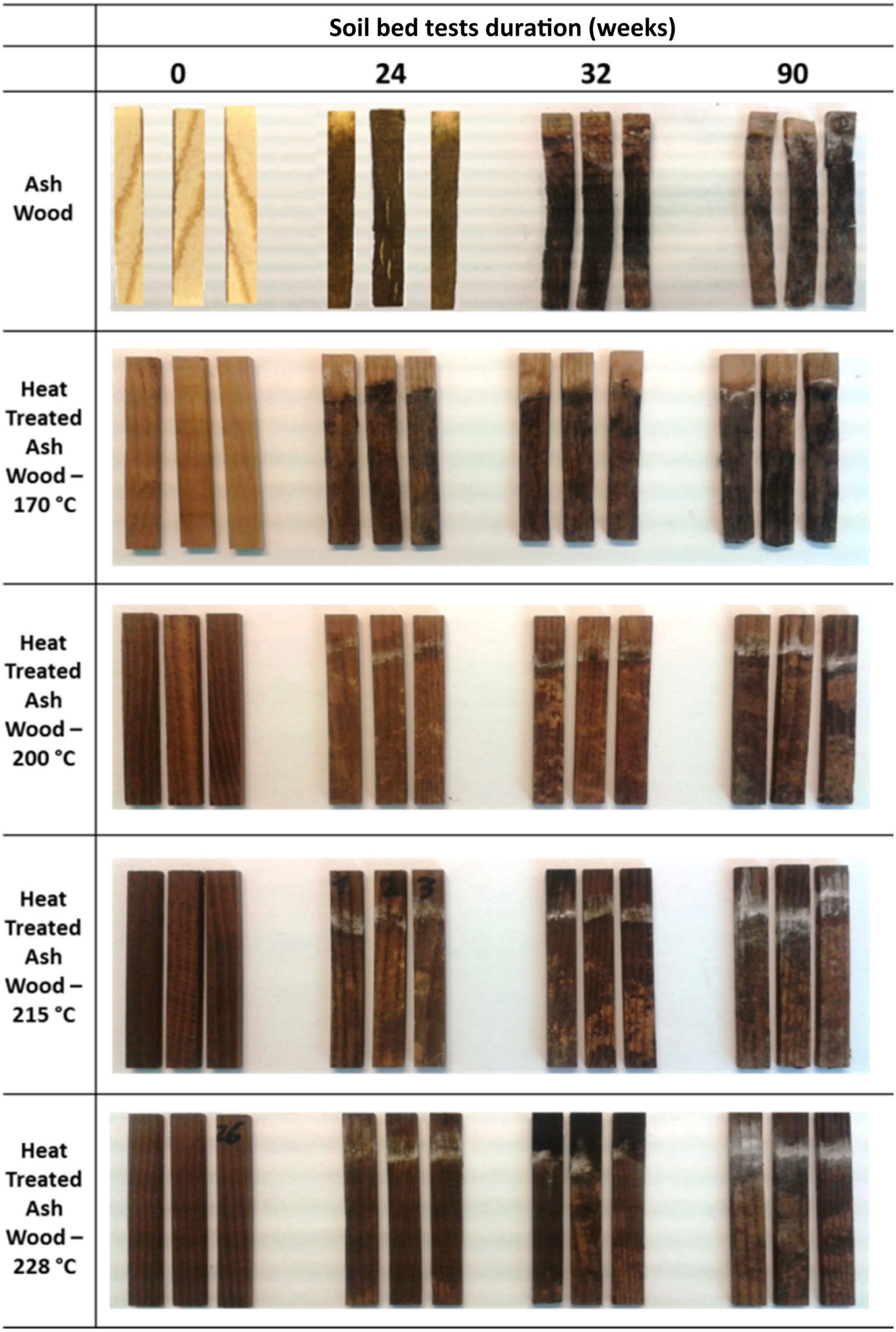
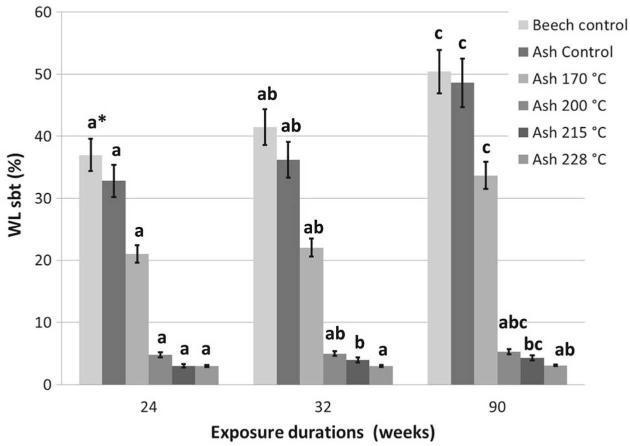


Fig. 5 Visual appearance of untreated and heat-treated ash wood after unsterile soil exposure at different durations



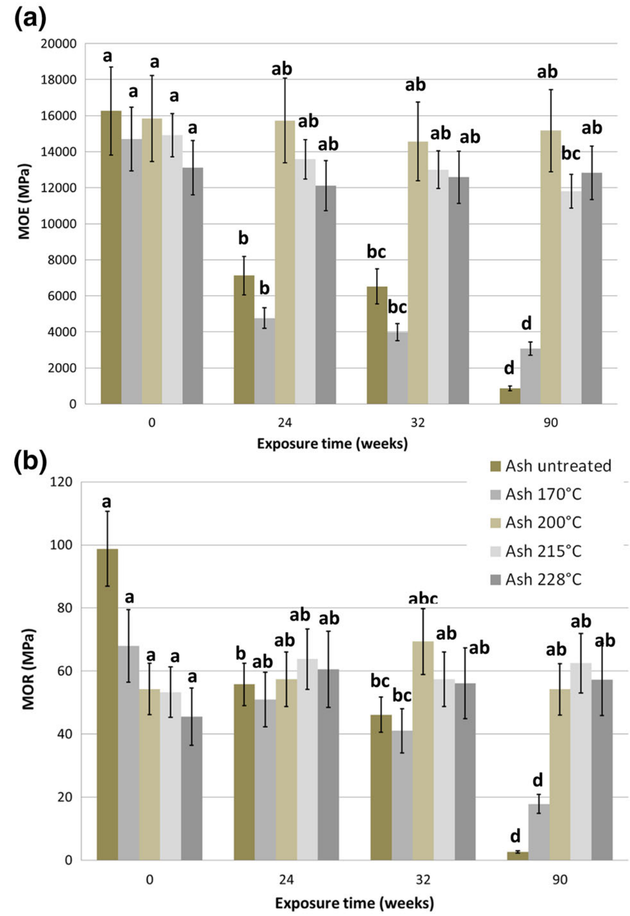
*According to one-way analysis of variance, systems not connected by the same letter are largely different, at the 5% level.

Fig. 6 Weight loss (WL_{sbt}%) due to unsterile soil exposure of untreated and heat-treated ash wood and beech wood reference

also found that pine sapwood and heartwood, thermally treated at a temperature of 230 °C and brought into unsterile soil for 32 weeks reached the classes "moderately durable" and "very durable" according to the weight and MOE loss differences, respectively.

3.3.3 *Mechanical loss after soil bed test degradation*

As previously reported in literature (Kim et al. 1998; Bal 2014), it was observed (Fig. 7) that the mechanical resistance of heat-treated ash wood, MOE and MOR in bending, decreased according to the treatment temperature. According to previous studies, it appears that weight loss greater than 10% for bio-deteriorated samples exposed to soil-inhabiting micro-organisms results in significant damages to mechanical properties (Curling et al. 2002; Venäläinen et al. 2014). Edlund and Jermer (2004) concluded that the high rate of failure was a possible consequence of the strength loss caused by the thermal modification, enhanced by wetting in the ground and further chemical degradation. In general, there was a similar trend in the mass and modulus of elasticity (MOE) loss values, and the correlation between these values was very high. The weight and MOE loss values were much greater than the MOR values, which indicate that MOE is a more sensitive measure for detecting fungal attack in the wood. This is in agreement with Humar et al. (2006) and Temiz and Yildiz (2006).



*According to one-way analysis of variance, systems not connected by the same letter are largely different, at the 5% level.

Fig. 7 Mechanical properties [MOE (a), MOR (b)] of untreated and heat-treated ash wood after unsterile soil exposure

Table 4 X* values and Durability Class according to soil bed tests of untreated and heat treated ash woods

Wood species	T °C	Soil bed tests exposure duration					
		24 weeks		32 weeks		90 weeks	
		X values*	Durability class	X values*	Durability class	X values*	Durability class
Ash	Control	0.89	5	0.87	5	0.94	5
	170	0.57	4	0.53	4	0.67	4
	200	0.13	2	0.12	2	0.11	2
	215	0.08	1	0.10	1	0.09	1
	228	0.08	1	0.07	1	0.06	1

* *X* median value of WL_{sbt} for test wood specimens/median value of WL_{sbt} for reference wood specimens (beech)

Concerning heat treatments performed on ash wood at temperatures above 200 °C, it appeared that the soil bed test duration of 24 weeks could be sufficient to estimate the resistance of modified wood. In fact, for longer exposure times (32 and 90 weeks), ash wood heat-treated at 215° and 228 °C does not seem to be more degraded. On the contrary, wood heat-treated at temperatures below 200 °C continues to be degraded after 24 weeks of soil incubation. For these treatment intensities, soil bed test should be prolonged because the modified wood continues to deteriorate significantly after 24 and 32 weeks of incubation (Junga and Militz 2005; Kamdem et al. (2002).

For each treatment intensity and for all exposure durations to soil bed test, MOR of heat-treated ash wood seems to be very slightly affected by soil-inhabiting micro-organisms. In fact, standard deviations results from MOR determination do not allow evaluating the impact of thermal treatment intensity on decay resistance of unsterile soil. Even if a small MOR increase for decayed heat-treated ash wood can be observed, which could be due to a surface densification of the wood material due to its contact with the soil during the soil bed test.

3.4 Termite resistance after unsterile soil bed test

With control, mortality of termites was less than 50% and the degree of attack was 4, which allows to validate the test.

3.4.1 Effect of heat treatment without unsterile soil incubation on termite resistance

The survival of termites reveals an effect on termite biology. Although survival rate was similar for treatments at 170 and 200 °C, it was lower above these temperatures (Table 5). Heat treatments performed at higher temperature than 200 °C cause critical changes to the wood and consequently its durability can be improved. Since wood is still degraded, a toxicity of eaten components may explain the increase in the termite mortality rate (Surini et al. 2012) according to the increase of heat treatment intensity. However, it is not totally efficient on a short time scale, as 51.33% of the termites were still alive after the test, with no significant differences between treated and untreated wood. The current results are in agreement with those of Nunes et al. (2004), who studied the resistance to termites of the species *Reticulitermes grassei* with wood treated by the German method (OHT) and concluded that in spite of the slightly higher mortality of termites in treated samples and smaller weight loss, the differences were not significant.

3.4.2 Effect of heat treatment on termite resistance after unsterile soil exposures

According to the visual appearance of wood samples after soil bed tests (Fig. 5), it can be deducted that the soil contained mainly white rots (Talaei et al. 2013). The termites with their cellulose decomposing bacteria in the gut can easily degrade and digest the cellulose of wood (Stamm 1964; Akio et al. 1990). The cellulose is the woody polymer which has been the less degraded by the heat treatment and by soil-inhabiting micro-organisms. Heat-treated ash wood previously subjected to soil bed tests remains vulnerable to termite attack (Table 5).

3.4.2.1 Soil exposure (ref. i) The exposure of treated ash wood, after soil bed test, to termite colonies showed a lower resistance to termite attack than those of the untreated samples, whatever the previous exposure duration to soil-inhabiting micro-organisms was. However, it was observed that weight losses due to termite attack stabilized (decreased slightly) after 32 weeks of soil incubation for each treatment intensity.

3.4.2.2 Air/soil exposure (ref. ii) The exposure of ash wood treated at 170 °C, after soil bed test with air contact, to termite colonies showed a lower resistance to termite attack than that of the untreated samples, whatever the previous exposure duration to soil-inhabiting micro-organisms. For thermal treatments performed at higher temperature (from 200 to 228 °C), termite resistance was improved whatever the exposure duration to soil-inhabiting micro-organisms was. However, it appears that for each treatment temperature, heat-treated ash wood incubated in the soil during 32 weeks was the most degraded sample by termite attacks, among all tested samples. This result could be explained by the fact that termites prefer rotten wood and that less cellulose is available for termites after 90 than after 32 weeks of exposure to soil-inhabiting micro-organisms.

4 Conclusion

Heat treatment improves wood durability, increasing clearly the resistance to brown and white rots and moderately the resistance to exposure to soil-inhabiting micro-organisms (for the lower treatment intensities), but it has just a little effect on the improvement in termite resistance. The downside of the treatment is the degradation of mechanical properties. The effect on MOE is small, but static bending strength (MOR) is the most weakened property. Several reasons for the improvement of rot resistance have been reported: (1) the transformations of

Table 5 Average values of mass loss (%), termite survival rates and visual rating after termite resistance tests

Treatment temperature (°C)	Soil bed exposure duration (wks)	WL_{term} (%)	SD (%)	Survival rate (%)	SD (%)	Visual rating*
Soil (Ref. i)						
Control	Ash	**4.58***f***	0.57	**69.67***abcd*	8.33	**4**
	Beech	**8.87***cde*	1.98	**69.50***abcdef*	13.10	**4**
	Pine	**11.12***c*	1.57	**64.50***bcdde*	9.43	**4**
170	Control	**11.80***cd*	2.60	**78.67***a*	4.16	**4**
	24	**19.44***b*	2.84	**72.00***ab*	8.72	**4**
	32	**18.96***b*	2.16	**61.33***cd*	1.15	**4**
	90	**29.11***a*	3.46	**70.00***b*	2.83	**4**
200	Control	**11.60***bcd*	2.12	**71.33***ab*	6.11	**4**
	24	**12.46***c*	0.77	**72.67***ab*	3.06	**4**
	32	**13.97***bc*	3.94	**72.00***ab*	3.46	**4**
	90	**13.88***c*	1.38	**72.00***ab*	9.17	**4**
215	Control	**10.86***c*	1.51	**56.67***de*	5.03	**4**
	24	**12.11***cd*	3.33	**58.00***bcdef*	12.49	**4**
	32	**14.47***bc*	5.21	**60.67***bcde*	9.45	**4**
	90	**9.98***cde*	3.13	**53.33***bcdefg*	16.77	**4**
228	Control	**9.23***cde*	1.97	**51.33***bcdefg*	13.61	**4 (67%); 3 (33%)**
	24	**10.87***bc*	1.18	**52.67***cdef*	8.08	**4**
	32	**14.24***cde*	1.40	**68.00***abc*	6.93	**4**
	90	**14.12***bc*	2.53	**69.33***abc*	7.57	**4**
Soil (ref. ii)						
Control	Ash	**4.58***e*	0.57	**69.67***a*	8.33	**4**
	Beech	**8.87***bcd*	1.98	**69.50***ab*	13.10	**4**
	Pine	**11.12***bc*	1.57	**64.50***abc*	9.43	**4**
170	Control	**11.80***bc*	2.60	**78.67***a*	4.16	**4**
	24	**23.58***a*	9.06	**74.00***a*	13.11	**4**
	32	**18.89***a*	2.67	**69.33***a*	1.15	**4**
	90	**24.95***a*	5.13	**61.00***ab*	4.24	**4**
200	Control	**11.60***b*	2.12	**71.33***a*	6.11	**4**
	24	**11.50***b*	0.89	**62.67***abcd*	10.26	**4**
	32	**13.07***ab*	3.60	**72.67***a*	4.16	**4**
	90	**10.89***abcd*	3.97	**69.33***a*	4.62	**4**
215	Control	**10.86***b*	1.51	**56.67***abcd*	5.03	**4**
	24	**1.92***ed*	0.68	**6.00***gh*	6.00	**2**
	32	**4.47***de*	2.52	**22.67***efg*	16.17	**4 (67%); 3 (33%)**
	90	**3.86***e*	1.60	**28.67***ef*	11.72	**3**
228	Control	**9.23***bcd*	1.97	**51.33***abcde*	13.61	**4 (67%); 3 (33%)**
	24	**2.43***e*	0.35	**0.00**	0.00	**2 (67%); 3 (33%)**
	32	**3.09***e*	0.84	**4.67***gh*	4.16	**2 (67%); 3 (33%)**
	90	**6.79***bcdef*	6.54	**34.00**	36.17	**3**

* ''0'' for no attack ''1'' for attempted attack, ''2'' for slight attack, ''3'' for average attack, ''4'' for a strong attack

** According to one-way analysis of variance, systems not connected by the same letter are largely different at the 5% level

hemicelluloses, which change from hydrophilic and easily digestible to hydrophobic molecules during heat treatment process; (2) the fungal enzymatic systems do not recognize the substratum and (3) the lower fiber saturation point of heat-treated wood than native wood leads to a better resistance against biological degradation and against changes in the external conditions affecting the microenvironment that affect the decay mechanism of heat-treated wood. It is also mentioned that there might be an esterification of cellulose due to acetic acid released by the thermal degradation of hemicelluloses. According to Junga and Militz (2005) and Kamdem et al. (2002), thermally modified wood material is different to untreated wood and it could be necessary to adapt different testing methods to evaluate the final decay resistance of this new material such as a prolonged test exposure (i.e. longer than 16 weeks of exposure) so as to overcome a poor estimation of the durability of the new material. This test adaptation could also be necessary for soil bed test and more particular for heat treatment performed at lower intensities (below 200 °C).

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