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THE DECAY RESISTANCE OF FOUR HYDROTHERMALLY TREATED HARDWOOD SPECIES

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ABSTRACT

Studies on the durability of European beech (*Fagus sylvatica*), alder (*Alnus glutinosa*), paper birch (*Betula papyrifera*), and Norway maple wood (*Acer pseudoplatanus*) after hydrothermal treatment in saturated water were carried out. Treatment of hardwoods was performed at two temperatures of 105 and 135°C for 6 hours. Fungal resistance was tested in the laboratory conditions with brown rot fungi *Serpula lacrymans*, *Gloeophyllum trabeum* and white-rot fungus *Trametes versicolor*. Half-specimens from each species were leached in accordance with STN EN 84 prior to the test. The results of decay resistance tests showed strong susceptibility of hydrothermally treated hardwood species to brown and white rot caused by basidiomycete fungi. In all cases, the mass loss was greater than 20%, and the moisture content significantly exceeded the fibre saturation point, above the level required for a fungal attack. Leaching had an insignificant effect on fungal resistance. All hardwoods were classified as non-durable species.

Keywords: alder, beech, birch, durability, *Gloeophyllum trabeum*, maple, saturated water steam, *Serpula lacrymans*, thermal treatment, *Trametes versicolor*

INTRODUCTION

Wood is an organic material that is sensitive to environmental conditions and micro-organisms. In principle, wood with higher moisture content – usually more than 20% - may be damaged by biological degradation processes in the presence of wood-destroying fungi, staining fungi, moulds, or bacteria (REINPRECHT 2008). Wood-decaying fungi recognise wood cell polymers as a food source and have specific enzymatic systems capable of metabolising them into simple digestible units (SMIDTH 2006; GOODELL 2020). Some wood-destroying fungi can penetrate even masonry and can transfer water and nutrition over long distances to timber. However, when the wood is not adequately protected, wood degrading fungi such as the dry rot fungus (*Serpula lacrymans*), the cellar fungus (*Coniophora puteana*), *Rhodonia* spp. and other wood-destroying fungi can cause severe damage to buildings and the greatest structural changes in wood and potentially cause human injuries (HAAS *et al.* 2019).

S. lacrymans is a well-known fungus that causes dry decay of wood. It is regarded as the most damaging destroyer of interior wooden building materials also in Slovakia (GÁPER *et al.* 2018). It grows and spreads quickly. It has low wood moisture requirements. It can produce substantial quantities of water by breaking down the wood polysaccharide. The wood rapidly loses its strength after being attacked by this fungus (REINPRECHT *et al.* 2007).

Therefore, it is one of the test organisms that comply with the standard STN EN 113-1 (2021) used to test the resistance of softwoods and hardwoods.

Gloeophyllum trabeum is a representative of the brown rot fungus, which is characterized by a special pattern of brown cubic rot of hardwoods and softwoods during which they oxidatively break down the components of the wood cell wall, causing a rapid loss of wood strength (TIRALOVÁ and REINPRECHT 2000, DANIEL *et al.* 2007). It is common in poorly stored wood and in wood exposed to the weather. Especially wooden footbridges and bridges may be encountered by these rot fungi families attacking hardwoods in Central Europe (IRBE *et al.* 2012; FOJTÍK and DĚDKOVÁ 2016).

Trametes versicolor is a well-known white-rot fungal species. It produces decay in hardwoods with simultaneous reduced lignin and carbohydrate nearly at the same rate, with a slight preference for lignin (KARIM *et al.* 2017). Although white-rot is less frequent in wooden construction, as it occurs most often in standing and felled many deciduous trees and some conifers, white-rot fungus is selected to compare it with brown rot. The wood progressively loses its strength after being attacked by a fungus, and as WITOMSKI *et al.* (2016) stated, the flexural strength and compressive strength of Scots pine wood can decrease by up to 50% with 20% mass loss, respectively with 30 % mass loss.

At the present time, the selection of naturally resistant wood species or the improvement of the less durable by means of modification processes is a common solution to minimize fungal degradation of the wood in use (HILL 2011; STN EN 350: 2017; REINPRECHT and VIDHOLDOVÁ 2019). Wood modification processes are interesting because they are implemented to improve the properties of wood including the fungal resistance and to produce new materials, and this is a good base for producing an environmentally friendly product (SANDBERG *et al.* 2021).

The hydrothermal modification of wood is one of the most important methods among methods of heat treatment. The notion of hydrothermal modification can be largely defined on the basis of a variety of temperatures and pressures. In addition, it can be performed in the presence of steam or liquid water under pressure or vacuum at different temperature levels, and it can affect the different characteristics of wood (Ali et al., 2021). The treatment softens the wooden material, releases internal stress, chemically changes some components of the wood, changes can occur in the microstructure of the cell wall, as well as changes colour of the wood (SOLÁR, 1997; TOLVAJ *et al.* 2009; TIMAR *et al.* 2016; DZURENDA 2018; SHI *et al.* 2018; WANG *et al.* 2019; GEFFERT *et al.* 2020; SANDBERG *et al.* 2021). This treatment is traditionally used in the woodworking industry, for example, in the manufacture of furniture components with solid wood bending, for the production of floors and panelling for the interior.

The aim of this paper is to determine the decay resistance of four native and hydrothermally treated hardwood species (European beech (*Fagus sylvatica*), alder (*Alnus glutinosa*), Paper birch (*Betula papyrifera*) and Norway maple wood (*Acer pseudoplatanus*)) to brown or white rot fungi and to state the influence of pre-treatment of the specimens by leaching on their durability. Based on the experimental data, the durability class will be assessed for the hydrothermally treated hardwood species.

MATERIALS AND METHODS

Wood specimens

Industrially hydrothermally treated heartwood planks of four hardwood species – European beech (*F. sylvatica* L.), alder (*A. glutinosa* L. Gaertn), Paper birch (*B. papyrifera* Marsh) and Norway maple wood (*A. pseudoplatanus* L.) were used. There were 3 to 8 growth rings per cm in each specimens and they were free from defects, The parameters of the treatment process were temperature of 105 and 135 ± 2.5 °C for 6 hours. The process is described in the works of DZURENDA (2018, 2019), DZURENDA and DUDIAK (2020) and DUDIAK and DZURENDA (2021) in more detail. Specimens were cut into 25 × 15 × 50 (axial) mm specimens. In total 32 specimens per wood species and hydrothermal treatments were used. Half of the specimens were leached, and the second half stayed without pre-treatment. Further 6 specimens from Scots pine sapwood (*Pinus sylvestris* L.) and beech (*F. sylvatica* L.) were used as controls of virulence of the fungus strain.

Leaching test

Native and hydrothermally treated specimens were leached in demineralized water according to the procedure described in STN EN 84 (2021) by some modifications. After impregnation with water (30 min at 0.8 MPa), specimens were kept submerged at room temperature. Water (5 volumes of water for 1 volume of wood) was exchanged 9 times during 14 days of test duration.

Durability test

The durability of wood was evaluated under laboratory conditions according to STN EN 113-1 (2021) and CEN/TS 15083-1 (2005).

The following fungal strains were used for the tests: the brown-rot fungi *S. lacrymans* (strain BAM 87), *G. trabeum* (Pers.) Murrill (strain BAM 115), and the white-rot fungus *T. versicolor* (Linnaeus ex Fries) Pilat (strain BAM 116).

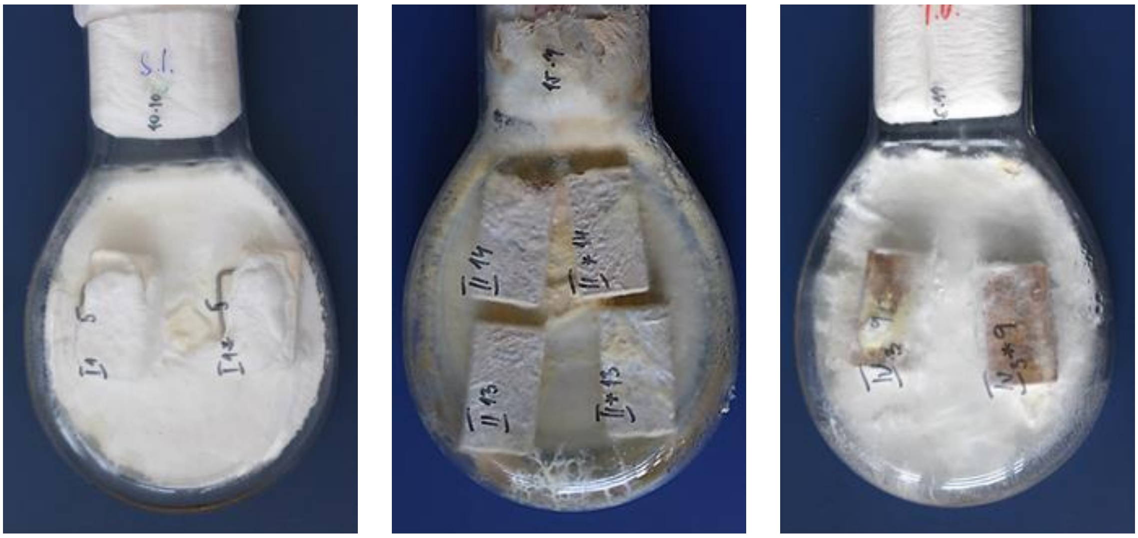
After steam sterilization of specimens in an autoclave at 120°C for 30 min, they were soaked in demineralized water for 240 min to achieve a moisture content of 25 to 30%. Finally, sets of two (four in case fungus *G. trabeum*) specimens of the same species were deposited on stainless steel grids under which a fungal mycelium had already been grown on a sterilized 4.5 weight% malt agar medium (HiMedia, Ltd., Mumbai, India) with a thickness from 3 to 4 mm in Kolle flasks. All specimens were incubated for 16 weeks at 22 ± 2°C and 70 ± 5% RH in a culture chamber.

After fungal exposure (Fig. 1), specimens were cleaned from adhering fungal mycelium. Their moisture content (MC_F) and mass loss by fungal decay (ML_F) were calculated in percentages using the following equations:

$$MC_F = \frac{m_{w\ decayed} - m_{0\ decayed}}{m_{0\ decayed}} \cdot 100 \tag{1}$$

$$ML_F = \frac{m_0 - m_{0\ decayed}}{m_0} \cdot 100 \tag{2}$$

where: $m_{w\ decayed}$ is the moisture mass after fungal exposure [g], m_0 is the initial oven-dry mass before fungal exposure [g] and $m_{0\ decayed}$ is the oven-dry mass after fungal exposure [g]. Specimens were oven-dried at 103°C until constant mass.



(a) (b) (c)

Fig. 1. The specimens with the mycelium grown on the end of decay resistance test (after 16 weeks) – brown-rot fungi *Serpula lacrymans* (a) and *Gloeophyllum trabeum* (b), and white-rot fungus *Trametes versicolor* (c).

(Note: left specimen - specimen without leaching, right specimen - specimen with leaching)

Classification of durability

Durability classes (DC) were derived from median ML_F according to CEN/TS 15083-1 (2005) and STN EN 350 (2017), as shown in Tab. 1.

Tab. 1. Durability classes (DC) based on median mass loss ($ML_{F,\ med}$) (according to CEN/TS 15083-1: 2005 and STN EN 350:2017).

Durability class (DC)	Description	$ML_{F,\ med}$ [%]
1	Very durable	<5
2	Durable	$5 < ML_F \leq 10$
3	Moderately durable	$10 < ML_F \leq 15$
4	Less durable	$15 < ML_F \leq 30$
5	Non-durable	>30

Statistical evaluation

The data are presented as the median value and the standard deviation (SD). Statistical significance of the differences in the weight loss in the leached and u leached wood was tested by the nonparametric Kruskal-Wallis median ANOVA test at an 0.05 significance level was used.

RESULTS AND DISCUSSION

Durability against brown rot causing basidiomycetes

The mass loss (ML_F) of tested specimens against brown-rot fungi *S. lacrymans* and *G. trabeum* is given in Tab. 2. Following the SMITH and ORSLER (1994) and STIRLING *et al.* (2016) studies and with the standard STN EN 350 (2017) it is proposed, that the median (central trend) can be better determined than the average. The impact of certain aberrant data with extreme value deviations was eliminated by the use of the median value.

Tab. 2. Mass loss due to fungal decay (*ML_F*) in durability tests against basidiomycetes.

Wood species	Treatment	Leaching	*S. lacrymans*					*G. trabeum*					*T. versicolor*				
			Med. [%]		Avg. [%]	SD [%]	DC	Med. [%]		Avg. [%]	SD [%]	DC	Med. [%]		Avg. [%]	SD [%]	DC
European beech																	
	None	No	30.22	d	30.85	3.25	5	42.85	d	42.03	3.07	5	29.95	d	29.16	5.82	4-5
		Yes	24.30		24.24	4.18	4	38.16		38.39	7.14	5	28.65		28.74	3.90	4-5
	HTT-105°C	No	41.72	d	40.51	5.03	5	44.65	d	45.52	2.79	5	28.41	d	27.58	3.39	4-5
		Yes	37.05		35.98	4.02	5	38.56		36.96	5.24	5	31.46		30.43	3.19	5
	HTT-135°C	No	41.94	d	41.21	5.56	5	48.85	d	47.02	4.92	5	31.48	d	31.88	2.04	5
		Yes	39.36		36.63	8.15	5	42.23		42.60	4.39	5	29.65		29.50	1.95	4-5
Alder																	
	None	No	36.08	d	36.17	4.69	5	31.07	d	31.80	3.20	5	35.27	d	35.45	6.18	5
		Yes	30.28		31.55	3.89	5	37.82		36.99	4.22	5	33.28		29.83	7.73	5
	HTT-105°C	No	36.20	d	37.15	5.37	5	32.74	d	33.16	5.56	5	35.87	d	37.03	3.99	5
		Yes	34.58		34.02	2.41	5	31.52		32.40	4.33	5	32.66		33.25	3.15	5
	HTT-135°C	No	40.29	d	41.25	5.97	5	35.05	d	35.07	4.73	5	37.58	d	37.37	4.40	5
		Yes	40.01		38.77	4.48	5	34.90		35.76	3.22	5	32.01		31.80	3.14	5
Paper birch																	
	None	No	32.12	d	32.77	5.51	5	30.96	d	31.07	1.93	5	35.54	d	34.29	3.46	5
		Yes	32.50		32.59	0.83	5	30.85		31.17	3.31	5	32.68		33.35	2.81	5
	HTT-105°C	No	33.63	d	32.63	4.61	5	31.37	d	30.87	3.94	5	33.13	d	32.93	3.58	5
		Yes	29.65		28.78	4.22	4-5	29.15		29.08	2.22	4-5	31.12		29.71	3.10	5
	HTT-135°C	No	34.61	d	35.40	7.52	5	34.09	d	33.92	8.21	5	36.58	d	37.47	5.16	5
		Yes	24.57		24.25	2.98	5	33.07		34.10	6.06	5	31.36		32.01	4.34	5
Norway maple																	
	None	No	31.36	d	29.50	6.53	5	36.32	d	36.43	0.53	5	35.93	d	36.54	3.59	5
		Yes	32.20		32.41	4.64	5	32.58		33.13	2.79	5	32.87		33.41	5.86	5
	HTT-105°C	No	36.16	d	35.65	1.83	5	35.35	d	35.53	2.86	5	30.87	d	31.28	2.47	5
		Yes	30.22		29.09	4.99	5	30.52		30.84	2.10	5	35.06		34.84	5.53	5
	HTT-135°C	No	27.05	d	26.93	2.77	4-5	44.92	d	45.34	3.33	5	32.28	d	35.02	8.51	5
		Yes	23.99		24.00	4.36	4	39.37		39.82	3.31	5	23.52		25.06	6.16	5

NOTE: Med. = Median, Avg.= Average, SD = Standard deviation, DC = Durability class
d = No significant effect of the leaching procedure (Kruskal-Wallis test, $p > 0.05$).

The validity of the test was confirmed by the satisfactory virulence of the fungus strain, which in the specimens of *P. sylvestris* sapwood caused a mass loss of 23.28% by *S. lacrymans* and 26.90%, by *G. trabeum* – i.e., more than the level of 20% required for this reference wood species, according to STN EN 113-1 (2021), although it was very close to threshold (more than the level of 30%) according to CEN/TS 15083-1 (2005).

All native (untreated) hardwoods – European beech, alder, Paper birch, and Norway maple – showed a mass loss of over 30% against two brown-rot fungi tested. The highest mass loss value was obtained with *G. trabeum* on beech wood. Higher mean mass loss of birch wood with *C. puteana* was observed in a study by KLARSSON *et al.* (2011).

The durability of all hardwood species against brown rot causing basidiomycetes slightly decreased after hydrothermal modification at 105 °C and 125 °C. The difference between the median mass losses of the treatment varies is insignificant for almost every species. This result is in accordance with the work of OHNESORGE *et al.* (2009), by which the resistance of hardwood species exposed to the brown-rot fungus *S. lacrymans* where the medium treatment level (160 °C) showed higher weight losses than the untreated ones. This could be explained by the degradation of hemicelluloses due to dehydration reactions that took place during the hydrothermal process (GEFFERT *et al.* 2020; DUDIAK and DZURENDA, 2021). At low hydrothermal temperatures (60–80 °C), there are negligible chemical and structural changes in the basic wood components. Increasing the temperature of the hydrothermal reaction from 100 °C to 150 °C deepens the chemical and physicochemical changes of all components of the wood substance (SOLÁR 1997). The durability of wood against decaying fungi could be improved due to thermal modification methods, mainly at temperatures higher than 180 °C depending on wood species, process conditions such as duration, oxygen or inert atmosphere, and others as was documented in many studies, for example, SAILER *et al.* 2000; SCHWARZE and SPYCHER 2005; METSÄ-KORTELAINEN and VIITANEN 2009; KLARSSON *et al.* 2011; WANG *et al.* 2019, BAAR *et al.* 2021; REINPRECHT and REPÁK 2019 and 2022; VIDHOLDOVÁ *et al.* 2022 and other.

On the other hand, pre-treatment with leaching has led to a slight decrease in mass loss. No significant effect of the leaching procedure on mass loss was confirmed by the median Kruskal-Wallis test with *p*-value was lower than 0.05 (Tab. 2). Similar results related to the effect of the leaching procedure on mass loss of native both hardwoods (European beech, English oak, Common juniper, Black cherry, Black locust, Rowan) and softwoods (Scots pine sapwood, English yew) were obtained in studies BRISCHKE *et al.* (2014, 2018). They observed no overall effect of leaching on the decay test results.

Durability against white rot causing basidiomycetes

The mass loss in the white-rot test with *T. versicolor* is presented in Tab. 2. The validity of the test was again confirmed by the satisfactory virulence of the fungus strain, which in the specimens of *F. sylvatica* sapwood caused a mass loss of 36.32% – i.e., more than the level of 20% required for this reference wood species according to STN EN 113-1 (2021) and CEN/TS 15083-1 (2005). Native alder, Paper birch, and Norway maple showed higher than 30% mass loss (ML_F) against tested fungus. The durability of these hardwoods slightly changed after hydrothermal modification however it was still lower. It might be explained by the creation of easily accessible simple hemicelluloses due to the hydrothermal process (GEFFERT *et al.* 2020). In the case of beech wood, the mass loss closed to 30 % illustrates the variations in their decay capabilities. *T. versicolor* was associated with the highest weight losses, which tends to be more aggressive on hardwoods such as beech (BARI *et al.* 2021). SALIMAN *et al.* (2017) studied the effects of hydrothermal treatment parameters on the decay resistance of oil palm wood. They found that the weight loss of the treated wood caused by white-rot fungus reduced as the temperature and time increased. However, the influence of treatment time on decay resistance enhancement was less than treatment temperature. For example, specimens treated at 110 °C for 147 min showed higher weight loss compared to specimens treated at 160 °C for 80 min (18.24% versus 9.00%). The improvement in decay resistance could be attributed to the reduction of equilibrium moisture content in treated specimens. The reduction of equilibrium moisture content is directly proportional to the increase in temperature and time (ČERMÁK *et al.* 2022).

Similar results were obtained regarding the effect of pre-treatment by leaching on the loss of mass, as in the case of the action of brown-rot fungi. No significant effect of the leaching procedure on mass loss was confirmed by Kruskal-Wallis median test which the *p*-value was lower than 0.05 (Tab. 2).

Moisture content after durability tests

After the decay resistance test, all native and hydrothermally treated hardwoods reached a moisture content considerably exceeding the fibre saturation point depending on the species of the tested fungus (Tab. 3).

The average wood moisture after the tests ranged from 49 to 103% for *S. lacrymans.* It was higher for *G. trabeum* from 102 to 169%. As was be expected, these fungi showed the ability to produce considerable amounts of free water by decomposing native and hydrothermally treated wood polysaccharide components. The increased moisture content of degraded hydrothermally treated wood was also due to structural changes created during hydrothermal treatment, which increase a water absorption and migration. WANG *et al.* (2019) treated the Chinese sweetgum wood specimens in water bath with temperatures of

60, 80, and 100°C for 4 hours and investigated the increase in water absorption and the changes in microstructure, mainly pit membrane was ruptured after treatment. Also, in the study REZAYATI *et al.* (2007), a better anti-swelling efficiency value of beech wood was achieved at a higher temperature of 170°C.

Tab. 3. Moisture content (*MC_F*) after durability tests against basidiomycetes.

			S. lacrymans		*G. trabeum*		*T. versicolor*	
Wood species	Treatment	Leaching	Avg. [%]	SD [%]	Avg. [%]	SD [%]	Avg. [%]	SD [%]
European beech								
	None	No	79.12	27.71	151.06	9.75	65.87	17.17
		Yes	79.06	16.34	138.92	30.23	55.17	9.71
	HTT-105°C	No	58.60	11.21	155.37	25.77	100.49	59.51
		Yes	54.78	11.44	119.77	50.60	81.97	71.70
	HTT-135°C	No	74.00	19.77	164.66	15.72	58.99	18.31
		Yes	81.94	19.55	148.44	19.31	80.48	13.71
Alder								
	None	No	116.04	64.14	104.38	30.42	105.49	34.79
		Yes	81.17	36.07	133.75	52.04	88.02	27.71
	HTT-105°C	No	84.13	35.94	133.48	23.99	138.67	28.67
		Yes	75.56	23.15	119.41	10.70	113.97	23.68
	HTT-135°C	No	81.22	32.35	160.54	27.72	152.64	33.85
		Yes	83.25	30.71	148.87	10.32	124.58	36.36
Paper birch								
	None	No	58.61	24.38	104.54	44.88	97.76	58.37
		Yes	82.14	60.91	123.14	14.89	68.76	10.36
	HTT-105°C	No	49.37	7.69	113.02	7.57	73.78	17.80
		Yes	60.45	19.60	111.37	9.94	66.19	13.98
	HTT-135°C	No	59.28	17.91	126.29	24.68	91.18	23.90
		Yes	60.29	11.71	136.80	17.68	103.46	8.28
Norway maple								
	None	No	56.98	11.31	124.25	13.27	99.78	60.06
		Yes	56.80	3.18	113.31	13.53	88.24	43.93
	HTT-105°C	No	52.28	5.89	127.97	11.54	49.86	3.53
		Yes	58.04	12.83	106.83	10.68	60.51	17.38
	HTT-135°C	No	51.47	21.91	168.71	12.84	96.78	67.11
		Yes	55.90	53.06	130.30	6.64	54.56	11.13

NOTE: Avg.= Average, SD = Standard deviation

The average moisture content of the wood (*MC_F*) at the end of the white-rot fungus exposure period ranged from 50% to 153% and was above the levels required for fungal attack (Tab. 3). This fungus typically forms a compact mycelial film on the specimens in decay tests, which helps to increase the moisture level in the wood (BARI *et al.* 2021).

Some cell wall damage was observed for heat treated black alder at exposed to a temperature of 190 °C for 6 hours (SALCA and HIZIROGLU 2014). Slight deformations of scalariform perforation plates of the vessel such are axial cracks and the separation of S3 layer of the secondary wall were observed after processing at 100 °C and 4 MPa (BEKHTA *et al.* 2016). Cracks increase in the proportion of intercellular spaces of wood caused by heat treatment, which may increase the capillary water flow along the longitudinal direction (ZAUER *et al.* 2014). LIN *et al.* (2016) found that the removals of extractives which was situated on the cell wall would block the pits of alder birch wood played an important roles in improving the permeability of wood after heat treatment. As noted above, the pit membrane, which was ruptured after treatment, can influence moisture migration and wood permeability (WANG *et al.* 2019).

Durability classification

The results from resistance tests against individual brown and white rot causing basidiomycetes were used to classify the four wood species according to their durability according to EN 350 (2016) as shown in Tab. 2.

As expected from the high mass loss determined for native alder (*A. glutinosa*), Paper birch (*B. papyrifera*) and Norway maple wood (*A. pseudoplatanus*) were classified as non-durable. In the case of native beechwood the potentially high variation in the mass loss in *T. versicolor* on beech and its value which was very close to 30%, led to the determination of DC 5.

In addition, on the base of the high mass loss determined for all hydrothermally treated hardwoods at temperatures of 105 and 135 ± 2.5 °C, it was classified as non-durable material (Durability class DC 5).

However, there was no uniform leaching effect, i.e. in one case improving, in two cases decreasing the durability class. Consequently, in summary, no significant effect of the leaching procedure according to STN EN 84 (2021) on the durability classification could be established. Similar findings were noted by BRISCHKE *et al.* (2014, 2018) and OHNESORGE *et al.* (2009).

CONCLUSION

The results of this laboratory study on decay resistance were as follows:

- Native and hydrothermally treated European beech (*F. sylvatica*), alder (*A. glutinosa*), Paper birch (*B. papyrifera*) and Norway maple wood (*A. pseudoplatanus*) were highly susceptible to brown and white rot causing basidiomycetes (S. *lacrymans, G. trabeum* and *T. versicolor*) – in all cases the mass loss was higher than the 20% and the moisture content considerably exceeded the fibre saturation point, so it was above the level required for a fungal attack;
- Leaching pre-treatment had a negligible impact on decay resistance;
- Native and hydrothermally treated hardwood species have been classified as non-durable species (Durability class DC 5).

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