Thermowood® vs Termovuoto process – comparison of thermally modified timber in industrial conditions

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ABSTRACT

The study aimed at treating Scots pine (*Pinus sylvestris* L.), spruce (*Picea abies* Karst.) and two hardwoods, metil (*Sterculia appendiculata* K. Schum) and neem (*Azadirachta indica* A. Juss) timbers in *industrial conditions* by steam (Thermowood®) and vacuum (Termovuoto) thermal modifications. Matched boards were treated identically and alterations in spectral characteristics and chemical composition of wood were compared. Mass loss, selected mechanical properties and wood durability were also traced and related to the chemical alterations. The physical nature of the two processes induced similar changes in chemistry and structure of softwoods but at different magnitudes for the hardwoods. The applied vacuum removed partly the acetic acid, which catalysed carbohydrate degradation, i.e. heat applied under vacuum was less destructive. Wood permeability was found important in the thermo-vacuum process.

Mass loss was significantly higher after Thermowood® process namely, 14.1% vs. 9.9% for metil and 14.2 and 12.1% for neem wood. No significant mass loss differences were found between pine and spruce wood after the two treatments. Mass loss correlated with the decrease of shear strength, rupture and elasticity moduli and increase of wood decay resistance. As more permeable, metil demonstrated significant difference between the properties above; thermo-vacuum process was less destructive but ensured lower improvement of durability compared to Thermowood® treatment.

The entire study allows concluding that Thermowood® and thermo-vacuum treatment according to Termovuoto technology produce similar final products with regard to chemical, physical-mechanical properties and durability with clear difference in the appearance (colour). Thermo-vacuum modification of wood can be advantageous where the end use of the product demands retaining the mechanical properties closer to those of the untreated material.

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INTRODUCTION

Termovuoto (TV) process is a new industrial thermal modification (TM) technology that has been developed by the National Research Council of Italy and SLU, Sweden, based

on the combination of an efficient vacuum drying process with thermal treatment process (Allegretti *et al*. 2012, Ferrari *et al.* 2013). It is actually a thermo-vacuum process where the wood is dried initially in air at 100℃ until the moisture content reaches 0%. Thereafter, TM is performed in the same chamber by increasing the temperature in the range of 160-220℃. A vacuum pump is used at this time to remove the residual air and maintain the vacuum. Compared to other TM processes, TV is a promising method with several advantages such as shorter duration and lower energy consumption, easier and cheaper management of the volatile waste and less corrosive. Lower mass loss of wood and odourless final product that might be affected by the action of the vacuum pump that continuously removes volatile compounds are other important features of TV (Allegretti *et al.* 2012, Candelier *et al.* 2013a,b). Several TMWs produced by TV process from softwoods and hardwoods have already been found to yield satisfactory mechanical properties and decay resistance. Commercially treated wood is already available on the market under the name *VacWood.*
The objective of the study was to compare selected chemical, physical-mechanical properties and durability of Scots pine (*Pinus sylvestris* L.), Norway spruce (*Picea abies* Karst.), metil (*Sterculia appendiculata* K. Schum) and neem (*Azadirachta indica* A. Juss) timber modified in *industrial conditions* by steam according to Thermowood® technology (TW) and under vacuum by Termovuoto process (TV). The comparison was intended to find out the features of modified wood and reveal eventual differences that may be caused by the nature of the applied processes. The choice of the wood species reflects the most commercially used and thermally modified timbers in Europe but also example of two tropical hardwood timbers.

EXPERIMENTAL

Sampling and thermal modification: Three logs of Scots pine, spruce, metil and neem were sawn to boards (28×120×3100 mm) that were stored and dried in room climate for 2 months. Six boards of each wood species were chosen for the present experiment and cut to 3 sub-samples (28×120×1000 mm); the first two sub-samples were treated by TW and TV methods while the third sub-sample served as untreated control. Wood density at room climate was determined and a 10-mm thick slice was cut from each sub-sample for initial moisture content (MC). In order to ensure comparability between the two treatments, both TW and TV thermal modification consisted of *a) initial drying* to achieve very low MC prior to the real thermal treatment; *b) heating phase from 100°C and up* to the target temperature set value with an approximate increase of 10ºC/h; *c) thermal modification phase* at constant temperature of 212°C and duration of 3 h; and *d) cooling phase* to slow down the wood temperature to 50°C. The experiments were carried out in *industrial TM chambers* of ca. 10 m^3 volume.
Lignin, monosaccharides and acetyl content: Treated and untreated samples were analyzed for soluble lignin (L), monosaccharides and acetyl content (Ac) based on acid hydrolysis. L was determined by Hitachi U-2910 spectrophotometer with an absorptivity of 110 L/g cm at 205 nm. The monomeric carbohydrates were determined by a HPLC system equipped with an evaporative light scattering detector (ELSD-90), and a Metacarb 87P column (300×6.5 mm) with a guard column (Metacarb 87P 50×4.6 mm). The ELSD-90 was operated at 50ºC, 2.5 bars and N_2 was used for spray forming. The sugars were eluted by ultra-pure water as mobile phase at a constant flow rate of 0.5 mL/min at 85ºC. The acetyl content was determined by means of a diode array detector (DAD) operated at 210 nm, and a Metacarb 87H column combined with a guard column

(MetaCarb 87H 50×4.6 mm). The mobile phase was 0.005 mol/L H_2SO_4 (pH 2.1), with a flow rate of 0.6 mL/min at 30ºC.

Mass loss (ML) and colour evaluation: All boards were weighed after treatments and ML was calculated (Table 2). Five boards of each species and treatment were randomly selected and color measurements carried out on board areas without defects. Konica Minolta CM-2500d surface reflectance spectrophotometer was used, the colour space coordinates $L*$, $a*$ and $b*$ measured and colour difference ($\Delta E*$) calculated.

Mechanical properties: Untreated and treated samples were conditioned to the same MC (*ca*. 8%) prior to the mechanical tests which were carried out by a testing machine (Shimadzu AG-X 50 KN) with accuracy of ±0.01 mm for position, ±0.1% for speed, and ±0.5% for loading. The measured properties were modulus of elasticity (MOE), modulus of rupture (MOR), static hardness perp. and parallel to grain, compression stress perp. and parallel to grain and shearing strength parallel to grain, all measured according to ISO standards.

Durability test with basidiomycetes: Wood durability classes were obtained by laboratory tests with white and brown rot fungi (EN 113 2004). Decay fungi: *Coniophora puteana* BAM Ebw. 15, *Gloeophyllum trabeum* BAM Ebw. 109, *Postia placenta* FPRL 280 and *Trametes versicolor* CTB 863A. Prior to the test, the treated samples were leached in water. The durability class is derived from the ratio of $ML_{sample}/ML_{reference}$.

RESULTS AND DISCUSSION

Compared to the untreated wood, the sum of simple sugars from hemicelluloses decreased significantly after both treatments (Table 1).

Table 1: Chemical constituents (%) of untreated and thermally modified soft- and hardwood samples (UN – untreated, TW – Thermowood treated, TV – thermo-vacuum treated)

Species	UN	TW	TV	Difference TW-TV
			Hemicelluloses	
Neem	17.2	9.0	8.5	0.5
Metil	14.7	5.1	9.1	-4.0
Pine	24.3	16.9	18.2	-1.3
Spruce	23.5	15.4	18.6	-3.2
			Acetyl content	
Neem	7.0	4.4	5.2	-0.8
Metil	8.2	4.4	5.7	-1.3
Pine	2.6	2.3	2.4	-0.1
Spruce	2.9	2.6	2.8	-0.2
			Lignin	
Neem	28.2	39.9	31.2	8.7
Metil	24.1	33.8	24.2	9.6
Pine	26.6	32.5	31.2	1.3
Spruce	27.8	31.0	29.8	1.2

The decrease caused by TW was larger than that of TV; the hemicellulose difference between TW and TV was particularly large for spruce and metil, i.e. TV causes less degradation of the hemicelluloses (Table 1). Similar results (Candelier *et al.* 2013a) were reported for thermally modified beech in nitrogen under vacuum. The degree of hemicelluloses degradation is proportional to the Ac decrement, which was clearly seen for all species and both treatment methods; the decrease was more pronounced after the TW process and thus, the hypothesis of the effect of vacuum during TM can be confirmed. As shown by Fengel and Wegener (1989), wood polysaccharides are affected by the presence of acetyl groups that are thermally labile and lead to formation of acetic acid, thereby causing acid-catalysed degradation of the polysaccharides during TM. The results showed neither significant decrease in Ac between untreated and modified and spruce, nor between the TW and TV treatments. On the other hand, hardwoods, particularly metil, were more liable to acid-catalysed degradation. This is explained by the fact that softwoods have lower hemicellulose content than hardwoods and the hemicelluloses of softwoods have a lower Ac compared to hardwoods. The thermal degradation of softwoods at defined temperature and duration is expected to be less compared to hardwoods (confirmed by the ML in Table 2). L content for all species is elevated (Table 1), while the L increment in case of TW process is significantly higher for the hardwood species. Material's ML is reflection of the chemical changes caused by the TM; in this study the effect of TM was proven by high ML (>7%) for all tested species (Table 2).

Table 2: Density (kg/m3), mass loss (ML, %) of thermally modified soft- and hardwood boards and colour difference ΔE^* (UN – untreated, TW – Thermowood treated, TV – thermo-vacuum treated).

Species	Density UN	ML_{TW}	ML_{TV}	Difference TW-TV	ΔE^* (TW-TV)
Neem	702	14.2	12.1	2.1*	3.7
Metil	589	14.1	9.9	4.2*	6.0
Pine	409	9.3	9.0	0.3	3.0
Spruce	402	7.1	7.5	-0.4	5.7

***** *Statistically significant difference (t-tests at α = 0.01)*

TW caused significantly higher ML than TV in both hardwoods, while the ML difference in the softwoods was insignificant (Table 2), i.e. the result does not support earlier reports (Allegretti *et al.* 2012) and results (Candelier *et al.* 2013a,b). In the case of softwoods (pine and spruce), we presume that during TM, the permeability of pine and spruce is similar and low since neither pine nor spruce was subjected to stronger thermal degradation by the TW process compared to TV (Table 2).

The ML of TW metil and neem are similar (14.1 and 14.2%, respectively). The effect of vacuuming away the acetic acid during the course of TM is expected to be stronger in more permeable wood species (i.e. less ML in metil than neem) confirmed by the results in Table 2 (9.9% for metil vs. 12.1% for neem). Metil is diffuse-porous wood with 371 µm average vessel diameter at a frequency of 5 per mm^2; it lacks tyloses or any visible incrustations in the vessels. Its thin-walled axial and ray parenchyma constitutes the bulk of the ground tissue. Thus, metil wood is lighter than neem and has a high impregnability as demonstrated by a high liquid uptake of 463 kg/m^3. Neem is also diffuse-porous wood with significantly smaller vessel diameters (up to 80 µm) and the vessels dispose of simple perforated plates, on which reddish-brown deposits of gum are visible and plug the perforations. The decrease in permeability in some hardwoods is caused by deposition of extractives on vessel perforations and cell walls blocking fluid transfer through the porous media. Higher density of neem and its smaller vessels with deposits on the perforation plates indicate a lower permeability than in metil. This

explains why the degradation products are removed easier from metil than from neem. Consequently, neem suffers higher ML in TV treatment than metil while in TW the ML of the two species are similar (Table 2).
TW treated wood is visible darker than the TV wood for all tested species. The colour difference ΔE^* is larger for metil and spruce than for neem and pine, as seen by naked eyes (ΔE^*> 3). The lighter colour of TV wood correlates well with its lower ML (Table 2). Gonzalez-Pena and Hale (2009) found that ΔE^* is a good predictor to describe even reduction of a number of mechanical properties of TM beech, Scots pine and spruce, contrary to Johansson and Moren (2006) who found weak colour-to-strength relationship. When TW and TV methods are compared by the mechanical properties, it is apparent that only MOR of pine differs significantly (Table 3).

Table 3: Mechanical properties (N/mm2) of untreated and treated samples that are significantly different between TW and TV treated wood. (UN – untreated, TW – Thermowood treated, TV – thermo-vacuum treated).

Species	UN	TW	TV	Difference TW-TV
			MOE	
Metil	9617	6396	7262	-866
			Shear strength	
Metil	11.2	6.7	8.7	-2.0
			MOR	
Metil	99	46.7	57.6	-10.9
Pine	85.1	62.8	73	-10.2

The value is larger for wood treated by TV than TW. The ML of the softwoods in this study is similar after TW and TV treatment processes and thus, there is no reasonable explanation why MOR deviates from the general trend. For the hardwoods, only some features of metil demonstrated significant difference between the methods; MOE, shear strength and MOR decreased more for TW treated wood than that treated by TV. Permeability of the treated wood species probably matters significantly when TV is applied; metil is a permeable species, and thus the acetic acid produced is easily removed leading to less ML at the end of the treatment. In absence of acetic acid, the MOE, MOR and shear are higher than after TW. Durability improvement of metil (DC 3) after TV is also less than after TW treatment (Table 4).

Table 4: Durability classes (DC) of untreated and treated samples exposed to basidiomycete fungi in a standard EN 113 test. (UN – untreated, TW – Thermowood treated, TV – thermo-vacuum treated).

Species	DC_{UN}	DC_{TW}	DC_{TV}
Neem	3	1	1
Metil	5	2	3
Pine	4	2	2
Spruce	4	2	2

The effect of TM on wood durability does not vary between pine, spruce and neem. TV modification shifts up the wood durability with two classes above the initial natural DC (Gao *et al.* 2016) which is also confirmed in the present study.

CONCLUSIONS

Thermowood® and TV treatment Termovuoto of Scots pine, Norway spruce, metil and neem timber ensure similar final products. Performed at identical temperature and duration, Thermowood® and TV process caused similar mass loss of the softwood species. Chemical analyses showed decreased hemicellulose content in both softwood species, while no significant effect on the acetyl content was observed. Hardwood (metil), mechanical and durability tests confirmed the basic role of the ML caused by the TM as an indicator of wood's physical and mechanical properties and durability. Wood permeability is an important factor in the TV process that should be considered when wood species are selected for treatment. The study concludes that Thermowood® and Termovuoto technology produce similar industrial products regarding chemical, physical-mechanical properties and durability with some difference in the appearance.

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