Effect of Ethylene Oxide Sterilization and Accelerated Ageing on the Physical and Mechanical Properties of Beech, Oak, and Elm Wood: Part 1

Daniela Tesařová,[a] Alena Capíková,[a] Eva Jeřábková,[a] Petr Čech,[a] Adam Ekielski,[c] , and Pawan Kumar Mishra [b,]*

Effects of ethylene oxide (EO) treatment on library and museum furniture were investigated. Three treatments (accelerated ageing, EO treatment, and EO treatment followed by accelerated ageing) were applied to understand the effects on physical and mechanical properties of the common furniture woods. The Buchholz indentation test/micro-hardness (MH), color and gloss, Brinell hardness/macro-hardness (BH), and surface roughness were tested. The MH of oak and beech samples was not noticeably affected by any of the treatments, whereas elm samples showed some effects. For all types of wood, color was affected by EO treatments, but not considerably affected by ageing. Gloss significantly decreased with EO and accelerated ageing treatments for all samples. In BH measurements, ageing caused a decrease in hardness for both the control and EO treated samples, although this affect was not marked in beech samples. The EO treatment caused a decrease in hardness for beech and elm samples, but no major effect was observed in oak samples. There was a significant effect on the R_a values (arithmetic average of the absolute values of the roughness profile ordinates) in the aged groups of beech and oak samples, as well as oak samples that received both EO treatment and ageing.

Keywords: Ethylene oxide; Wood sterilization; Surface characterization; Oak wood; Elm wood; Beech wood

*Contact information: a: Department of Furniture, Design and Habitat, Mendel University In Brno, Zemědělská 1665/1, 613 00 Brno-sever-Černá Pole, Brno, Czech Republic; b: Department of Wood Processing Technology, Mendel University In Brno, Zemědělská 1665/1, 613 00 Brno-sever-Černá Pole, Brno, Czech Republic; c: Department of Production Management and Engineering, Warsaw University Of Life Sciences, Nowoursynowska 166, 02-787 Warszawa, Poland; *Corresponding author: pawan.mishra@mendelu.cz*

INTRODUCTION

Ethylene oxide (EO) treatment has been a traditional field of study in wood science. This treatment has two main uses: sterilization and modification. Both categories have seen a change in necessary conditions with change in time (Mishra *et al*. 2018). A number of studies have been conducted on EO as a sterilizing agent for different wood species (Smith and Sharman 1971; Tohmura *et al.* 2012). Smith (1968) compared the effects of EO and propylene oxide as sterilizing agents in spruce heartwood blocks in the presence of moisture and found that bacteria are more resistant than fungi and that ascomycetes are more resistant to propylene oxide sterilization when compared with basidiomycetes (Smith 1965, 1968). EO was also studied as a sporicidal decontaminant of pine wood, archival paper, and painted canvas for *Bacillus anthracis* and *Bacillus atrophaeus* under optimal conditions (Whitney *et al*. 2003; United States Environmental Protection Agency 2013). The role and practical application of EO as a fumigant in library and museums is relevant because of its ease of application and compatibility with lignocellulosics (wooden furniture and cellulose in paper), leather, silk, and other commonly found materials. At the chemical level, EO reacts with hydroxyl groups present in various components (lignin, cellulose, and hemicelluloses) of wood. Specifically, a nucleophilic attack by hydroxyl groups on the highly strained EO leads to formation of an additional hydroxyl group, which is in turn available for the next EO attack and polymerization reaction (Cetin and Hill 1999; Kumar 2007; Kral *et al*. 2015). Hence, an effect on surface properties can be easily anticipated. A number of studies have been performed on the effects of ageing (natural, storage, accelerated) on color and other surface properties of wood (Tsuchikawa *et al*. 2005; González-Peña and Hale 2009; Ganne-Chédeville *et al*. 2012).

Library and museum furniture is relatively more exposed to the EO due to regular EO fumigation for preservation of the specimens, manuscripts, and textbooks as compared to any other furniture used for other purposes. Therefore, library and museum furniture gets affected by two separate processes, *i.e.* EO treatment and normal ageing process of wood. There have been several reports of ageing (accelerated and normal) of wood and its impact on different properties of wood. This data can be used for understanding the ageing the normal furniture. Additionally, the interaction of EO with wood as a wood modifying agent already well known. However, the combination of these two parameters is highly relevant for library and museum furniture and relatively less studied. This study tries to understand the combined effect of these two treatments on library and museum furniture.

In this study, beech, oak, and elm wood samples were used to determine the impacts of EO treatment and ageing (accelerated testing) on surface properties common furniture species from central Europe. This work, supported by Slovak National library, aimed to understand the immediate and long-term (using accelerated ageing methods) effects of EO treatment on the properties of wooden library furniture. The study was divided into two parts: surface properties and mechanical properties. This paper covers the impact on surface properties only.

EXPERIMENTAL

Materials

Three different types of wood were considered: beech (*Fagus sylvatica*), oak (*Quercus robur*), and elm (*Ulmus minor*). Because of the prevalence of Dutch elm disease, samples of solid/bulk elm wood samples of required thickness (9 mm and 0.9 mm) were not available in the central European market and hence were not used in this study. The available samples of 0.6 mm thickness were used in the case of elm. The various samples with their associated specifications, treatments, and sample codes are presented in Table 1.

Methods

Ethylene oxide treatment

A sterilization chamber Model DLOG, Overpress gas, (DE LAMA, San Martino, Italy, installed by Mac spol. s.r.o, Slovakia) with a usable volume of 1.8 m^3 was used to sterilize the samples. As a sterilization medium, the device employed a gaseous mixture of ethylene oxide and carbon dioxide in a ratio of 10:90. During the sterilization process, the P5 (pre-program setting) sterilization program was used at an operating temperature of 25

°C, an overpressure of 3.8 bar, and 50% wetting of the material. The sterilization process itself took 720 min, and after the completion of the entire sterilization program, wood samples were subjected to the aeration program P98 to remove ethylene oxide residues. With the P98 venting program, the air was exchanged 88 times over 22 h.

Table 1. Explanation of Sample Codes Used in the Study

Wood	Sample type	EO treatment	Accelerated ageing	Code
Oak	Bulk wood, 200x150x9 (mm) (B)	Untreated (U)	No - (U_g)	**OBUU**$_g$
Oak	Bulk wood, 200x150x9 (mm) (B)	Untreated (U)	Yes (A_g)	**OBUA**$_g$
Oak	Bulk wood, 200x150x9 (mm) (B)	Treated (E)	No - (U_g)	**OBEU**$_g$
Oak	Bulk wood, 200x150x9 (mm) (B)	Treated (E)	Yes (A_g)	**OBEA**$_g$
Oak	Veneer 200x150x0.9 (mm) (V)	Untreated (U)	No - (U_g)	**OVUU**$_g$
Oak	Veneer 200x150x0.9 (mm) (V)	Untreated (U)	Yes (A_g)	**OVUA**$_g$
Oak	Veneer 200x150x0.9 (mm) (V)	Treated (E)	No - (U_g)	**OVEU**$_g$
Oak	Veneer 200x150x0.9 (mm) (V)	Treated (E)	Yes (A_g)	**OVEA**$_g$
Beech	Bulk wood, 200x150x9 (mm) (B)	Untreated (U)	No - (U_g)	**BBUU**$_g$
Beech	Bulk wood, 200x150x9 (mm) (B)	Untreated (U)	Yes (A_g)	**BBUA**$_g$
Beech	Bulk wood, 200x150x9 (mm) (B)	Treated (E)	No - (U_g)	**BBEU**$_g$
Beech	Bulk wood, 200x150x9 (mm) (B)	Treated (E)	Yes (A_g)	**BBEA**$_g$
Beech	Veneer 200x150x0.9 (mm) (V)	Untreated (U)	No - (U_g)	**BVUU**$_g$
Beech	Veneer 200x150x0.9 (mm) (V)	Untreated (U)	Yes (A_g)	**BVUA**$_g$
Beech	Veneer 200x150x0.9 (mm) (V)	Treated (E)	No - (U_g)	**BVEU**$_g$
Beech	Veneer 200x150x0.9 (mm) (V)	Treated (E)	Yes (A_g)	**BVEA**$_g$
Elm	Veneer 200x150x0.6 (mm) (V)	Untreated (U)	No - (U_g)	**EV**$_1$**UU**$_g$
Elm	Veneer 200x150x0.6 (mm) (V)	Untreated (U)	Yes (A_g)	**EV**$_1$**UA**$_g$
Elm	Veneer 200x150x0.6 (mm) (V)	Treated (E)	No - (U_g)	**EV**$_1$**EU**$_g$
Elm	Veneer 200x150x0.6 (mm) (V)	Treated (E)	Yes (A_g)	**EV**$_1$**EA**$_g$

Accelerated ageing testing

The accelerated ageing process included 25 cycles. Each cycle consisted of two stages. Conditions of the first stage were 50 °C and 75% relative humidity (R.H.) for 12 h, while conditions of the second stage were −30 °C and 0% R.H. for 12 h. The air conditioning chamber used was an MEMMERT HPP 108 (Memmert GmbH+ Co. KG, Büchenbach, Germany) with an operating temperature range of 5 to 70 ± 1 °C and R.H. range of 10% to 90% ± 1.5%. The freezer used was an Elcold (HPP108, Hobro, Denmark) with an operating range of 0 °C to −40 ± 0.5 °C and a setting accuracy of 1 °C.

Buchholz indentation test

The Bucholz indentation test, also called the microhardness (MH) test, was used to compare surface hardness at low applied force (approximately 2 N). The MH of all samples was measured using a Buchholz FL - 2000 H (Melit GmbH Mess- und Industrietechnik, Hinwil, Switzerland) instrument and per ČSN EN ISO 2815 (2004) standards. In this method, test samples were placed on a horizontal surface on the measuring device. The device was set, and the tip of the device struck against the sample surface. The device displayed the value of penetration depth in millimeters (mm), which was later converted to micrometers (μm).

Color and gloss

Color and gloss are an integral part of furniture properties. The changes in color and gloss were used to compare the effects of EO treatment and ageing. The color and gloss of all samples were measured using spectro-guide portable spectrophotometer (Spectro guide 45/0, gloss with white standard, Byk –Gardner, USA) (Byk-Gardner, Germany) and in accordance with the ČSN 673068 (1984) standard. The CIELAB color description system of the Commission Internationale de l'Eclairage (International Commission on Illumination) was used (Ekielski *et al*. 2018). Color and gloss were measured as the difference from a standard color and gloss, Bila_232 (ΔE^*) and D65/10° (ΔG^*), respectively.

Brinell hardness test

Brinell hardness test, also called the macrohardness test, was used to compare the change in surface hardness at relatively higher applied forces (29.42 kN). The Brinell hardness was measured using a Brinell hardness tester, ZD 10/90, (VEB WPM) (FRITZ HECKERT, Germany), and in accordance with the test standard ČSN EN ISO 6506 – 1,4 (2015). This method is based on the penetration of a tungsten carbide ball (indenter) into the test sample with some force (applied load). The Brinell hardness number (designated as HBW in the standard) was calculated using the applied load, diameter of the indenter, and diameter of the indentation using the formula given in the test standard (ČSN EN ISO 6506 - 1,4 (2015)).

Surface roughness

Surface roughness indicates the mechanical performance of the material, as it may lead to the formation of nucleation sites for cracks. Changes in surface roughness caused by EO treatment and ageing were studied for this reason. Surface roughness was measured using a Mitutoyo SJ – 201 (Mitutoyo Europe GmbH, Germany) and in accordance with the ČSN EN ISO 4287 (1999) standard. From the measured profile, the R_a (arithmetical mean roughness value, measured in μm) was calculated, which can be defined as the arithmetical mean of absolute values of the profile deviations from the mean line of the roughness profile. This is also known as the arithmetic average (AA) and center line average (CLA).

RESULTS AND DISCUSSION

Micro-hardness parameter values (depth measured in μm) of the tested samples are listed in Table 2, and their interactions are plotted in Fig. 1. In the case of beech samples, no significant effect of aging on MH values was observed in either type of sample (bulk wood and veneer).

A similar result was observed in both types of samples of oak wood, in that ageing and/or EO treatment had no significant effect on the observed MH values. The elm veneer samples showed a significant change in MH values in aged samples, both untreated and EO-treated. Nevertheless, there was no significant effect found on MH values from EO treatment alone for elm samples.

Table 2. MH Values Measured in µm for Samples Tested

Sample code	Mean (µm)	Std. Dev.	P-value
BBUUg	-0.019[a,b,c]	0.004	control
BBUAg	-0.028[a,d,e]	0.004	•
BBEUg	-0.018[b,d,f]	0.002	•
BBEAg	-0.023[c,e,f]	0.003	•
BVUUg	-0.070[g,h,i]	0.021	control
BVUAg	-0.158[g,j]	0.055	•
BVEUg	-0.025[h,k]	0.005	•
BVEAg	-0.086[i,j,k]	0.013	•
OBUUg	-0.026[L,m,n]	0.002	control
OBUAg	-0.034[L,o,p]	0.003	•
OBEUg	-0.025[m,o,q]	0.005	•
OBEAg	-0.026[n,o,q]	0.007	•
OVUUg	-0.146[r,s,t]	0.025	control
OVUAg	-0.106[r,u,v]	0.029	•
OVEUg	-0.127[s,u,w]	0.036	•
OVEAg	-0.118[t,v,w]	0.018	•
EV1UUg	-0.127[x]	0.035	control
EV1UAg	-0.454	0.241	•
EV1EUg	-0.157[x]	0.060	•
EV1EAg	-0.264	0.195	•

*Values are significantly different from the control (ANOVA; $p < 0.05$). Values having the same superscript letter are not significantly different from each other (Duncan post-hoc).

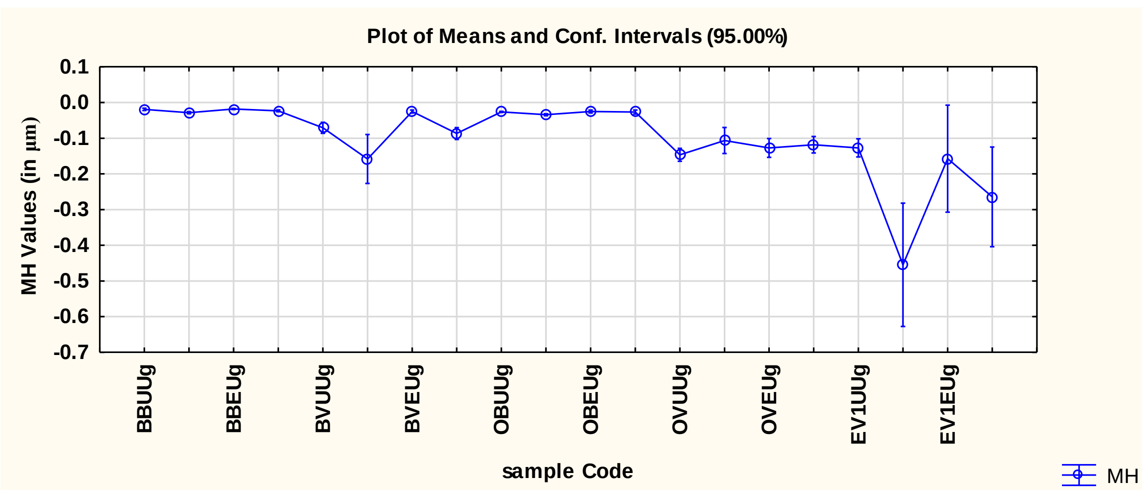

Fig. 1. Interaction plot of measured MH values

A change in the color of a material represents a change in the chemistry of the material. Based on the work of Hikita *et al.* (2001), a change in the ΔE^* value that is less than 0.5 can be assumed as negligible, that between 0.5 and 1.5 is slightly perceivable, and that between 0.5 and 3.0 can be taken as noticeable difference. In the case of bulk beech samples, a negligible change (also statistically insignificant) in color with respect to the control was observed for aged wood samples. The EO-treated wood samples showed significantly lower values of ΔE^* compared with the control. However, the ageing of the

EO-treated wood sample caused a significant increase in ΔE^* values and a slightly perceivable change in color. In the case of bulk oak samples, ageing had no significant effect on color. However, EO treatment resulted in a significant change and noticeable difference with respect to the control.

Table 3. Value of ΔE^* Measured for Various Samples

Sample	ΔE^*(mean)	ΔE^*(std. dev.)	P-value
BBUUg	$29.24^{a,b}$	1.74	control
BBUAg	28.82^{a}	0.57	•
BBEUg	28.26	1.19	•
BBEAg	29.71^{b}	1.47	•
BVUUg	33.55	1.08	control
BVUAg	35.02^{c}	1.18	•
BVEUg	$35.52^{c,d}$	1.20	•
BVEAg	35.85^{d}	1.27	•
OBUUg	35.17^{e}	1.66	control
OBUAg	35.23^{e}	1.72	•
OBEUg	37.96^{f}	4.93	•
OBEAg	37.68^{f}	2.09	•
OVUUg	34.47	0.86	control
OVUAg	37.50	1.01	•
OVEUg	35.89^{g}	1.45	•
OVEAg	36.02^{g}	1.32	•
EV1UUg	$38.91^{h,i}$	1.23	control
EV1UAg	$38.76^{h,j,k}$	1.58	•
EV1EUg	$38.89^{i,j,L}$	1.73	•
EV1EAg	$38.42^{i,k,L}$	1.68	•

*Values are significantly different from the control (ANOVA; $p < 0.05$). Values having the same superscript letter are not significantly different (Duncan post-hoc).

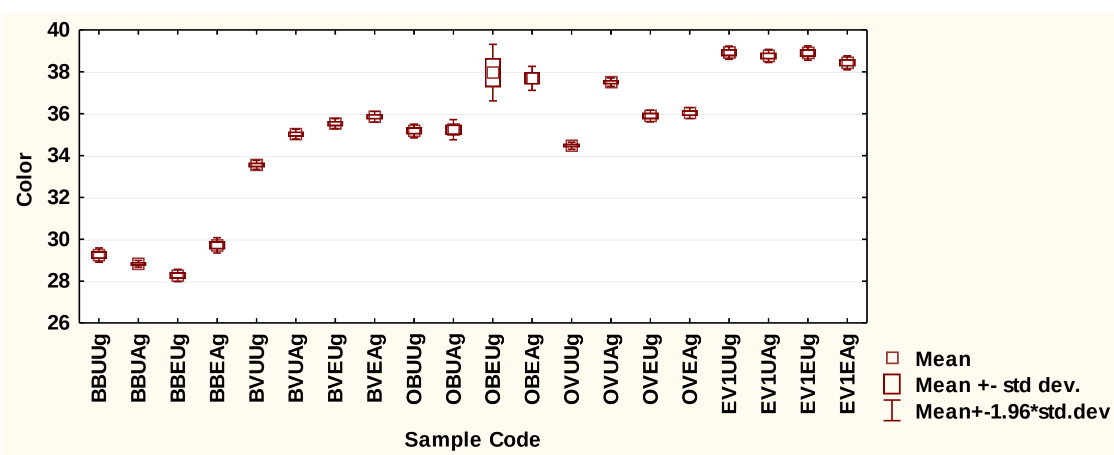

Fig. 2. Interaction plot of estimated Color (ΔE^*) values

There was almost no marked effect of ageing. In the case of oak and beech veneer samples, ageing resulted in a clear and significant effect on the color of samples. The effect of EO treatment on color for both oak and beech samples was also marked when compared with the control. However, the effect of ageing on EO treated wood was not significant in these samples. The difference in behavior of veneer and bulk wood samples may have been due to differences in the thickness of samples, which affects the availability of reactive sites in bulk wood. For elm veneer samples, no significant effect of EO and/or ageing was observed with respect to color change.

The gloss of wood is an important parameter for furniture quality estimation. The impact that wood treatments have on wood gloss has not been widely studied, although some reports can be found, such as in the paper by Aksoy *et al*. (2011). Gloss values, presented as the difference from the standard (ΔG^*), can be found in Table 4 and Fig. 3. The negative sign signifies a higher gloss value than that of the standard used. In the case of both beech and oak wood, the bulk wood gloss was significantly affected by ageing, EO treatment, and EO treatment followed by ageing. However, ageing of beech veneers did not show any significant effect on gloss compared with the control sample, although EO treatment did show a marked effect. The effect of ageing on EO treated wood was also found to be significant in beech veneer samples. For oak veneer samples, gloss values were significantly affected by ageing, although EO treatment did not have a significant effect. Moreover, EO treatment followed by ageing showed a marked effect on gloss. In the case of elm samples, no significant effect of ageing and EO treatment was observed on gloss values. However, EO treatment followed by ageing affected the gloss significantly.

Table 4. Gloss Values (ΔG^*) in GU for Various Samples

Code	Mean	Std. Dev.	P-value
BBUUg	-1.52	0.186606	control
BBUAg	-2.24	0.403665	•
BBEUg	-1.39	0.218004	•
BBEAg	-1.83	0.307369	•
BVUUg	$-2.02^{a,b}$	0.232913	control
BVUAg	$-2.03^{a,c}$	0.175226	•
BVEUg	-1.63	0.221002	•
BVEAg	$-1.97^{b,c}$	0.226368	•
OBUUg	-1.65	0.246909	control
OBUAg	-1.87	0.308152	•
OBEUg	-1.80	0.338104	•
OBEAg	-2.38	0.203710	•
OVUUg	-2.09^{d}	0.142736	control
OVUAg	-2.28^{e}	0.121637	•
OVEUg	-2.15^{d}	0.126914	•
OVEAg	-2.23^{e}	0.117502	•
EV1UUg	$-1.95^{f,g}$	0.208540	control
EV1UAg	$-1.94^{f,h}$	0.210583	•
EV1EUg	$-1.92^{g,h}$	0.271332	•
EV1EAg	-2.16	0.146047	•

*Values are significantly different from the control (ANOVA; $p < 0.05$). Values having the same superscript letter are not significantly different (Duncan post-hoc).

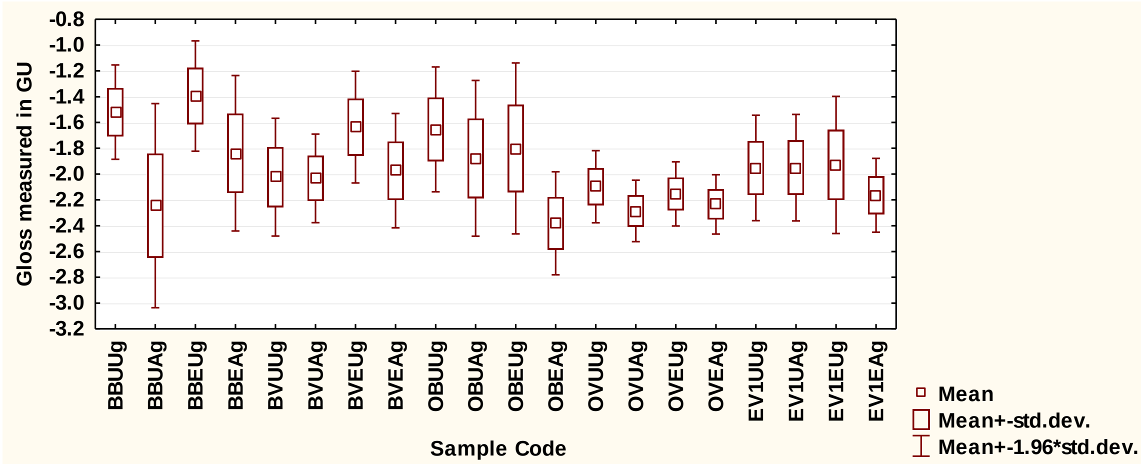

Fig. 3. Interaction plots of estimated gloss ΔG^* values

Brinell hardness is represented by BHN. In general terms, higher HBW or BHN values reflect harder wood. A number of reports have used BHN as a characteristic feature in their study (Holmberg 2000; Bektas *et al*. 2001; Doyle and Walker 2007). The measured HBW values are presented in Table 5, and the interaction plot is shown in Fig. 4.

Table 5. HBW Values Measured in kg/m^2

Sample Code	Mean	Std. Dev.	P values
BBUUg	2.79[a]	0.22	control
BBUAg	2.56[a]	0.30	•
BBEUg	4.13	0.44	•
BBEAg	3.66	0.29	•
OBUUg	3.25[b]	0.39	control
OBUAg	2.44	0.11	•
OBEUg	3.39[b]	0.56	•
OBEAg	2.00	0.25	•

*Values are significantly different from the control (ANOVA; p < 0.05). Values having the same superscript letter are not significantly different (Duncan post-hoc).

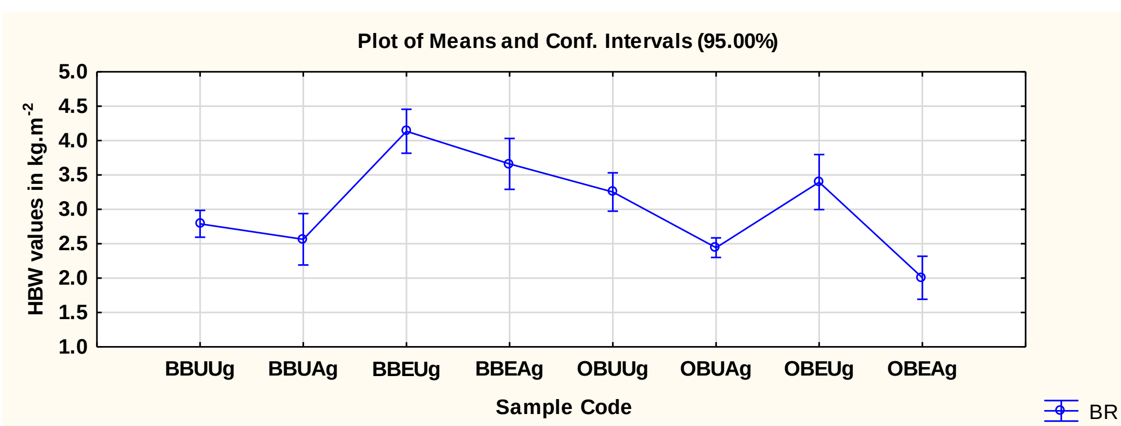

Fig. 4. Interaction plot of estimated HBW values

In the case of beech samples, ageing did not have a significant effect on HBW values. However, EO treatment caused an increase in hardness, which could have been due to chemical reactions between the EO and wood components. The ageing of EO treated samples caused a loss in hardness values. In the case of oak samples, ageing of control and EO treated caused a decrease in Brinell hardness in both samples, but the effect of EO treatment was not significant on HBW values compared with the control.

Surface roughness parameter values of bulk wood samples of beech and oak wood are presented in Table 5 and Fig. 4. Similar studies on surface roughness using accelerated ageing (UV and water spray based) of preservative-treated wood were performed by Temiz *et al*. (2005). In beech samples, a significant change in R_a value caused by ageing was observed in untreated samples, whereas this effect was not significant in EO-treated samples, which may have been due to EO reactions with wood components. In oak sample, ageing had a significant effect on the R_a value, and a similar effect could be observed in EO-treated samples. The difference in behavior of beech and oak samples can be attributed to the difference in relative composition of the two types of wood (lignin, holocellulose, and extractives contents) (Grønli *et al*. 2002).

Table 6. Roughness Parameter R_a Values Expressed in μm

Sample code	R_a	Std. dev.	P-value
BBUUg	$2.62^{a,b}$	0.27	control
BBUAg	6.52^{c}	1.57	•
BBEUg	$2.74^{a,d}$	0.25	•
BBEAg	$5.27^{b,c,d}$	1.07	•
OBUUg	4.98^{e}	0.85	control
OBUAg	9.84^{f}	3.81	•
OBEUg	3.70^{e}	0.54	•
OBEAg	9.84^{f}	3.81	•

*Values are significantly different from the control (ANOVA; p < 0.05). Values having the same superscript letter are not significantly different (Duncan post-hoc).

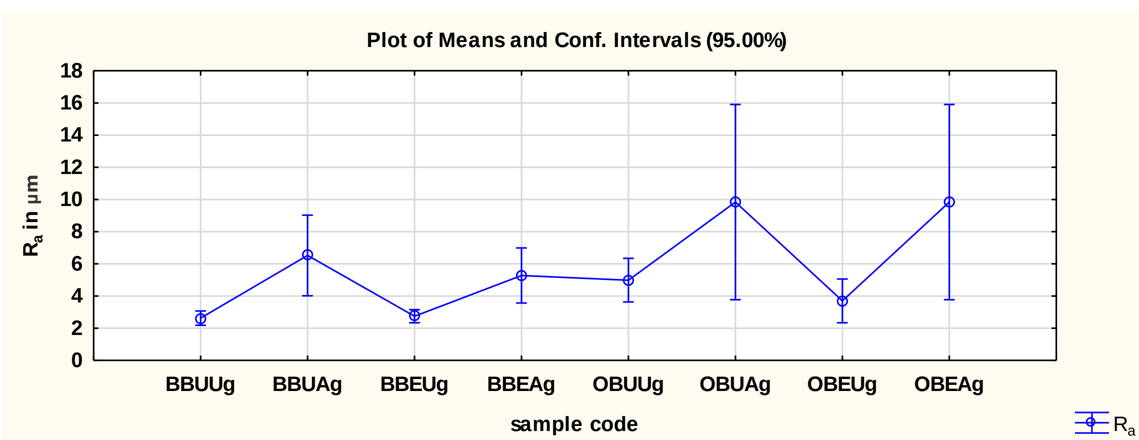

Fig. 5. Interaction plot of R_a values

CONCLUSIONS

1. The MH values of oak and beech samples of both types (bulk and veneer) were not significantly affected by EO treatment and/or ageing. A marked effect of ageing and EO treatment followed by ageing was observed in elm samples, with no significant effect observed in EO treatment only samples.
2. For color measurements, the effect of ageing was negligible and insignificant in bulk wood of both beech and oak. However, the effect of EO treatment and EO treatment followed by ageing was significant in beech samples. In the case of oak and beech veneers, ageing and EO treatment alone had a significant effect on color values. However, EO treatment followed by ageing had no effect on color parameter. For elm veneers, the effect of EO and/or ageing was observed to be insignificant.
3. In gloss measurements, all three kinds of treatments (ageing, EO treatment, and EO treatment followed by ageing) had a significant effect on bulk beech and oak samples. Beech and elm veneer gloss measurements had no effect from ageing, while oak veneer resulted in a significant decrease. In all samples, EO treatment followed by ageing showed a decrease in gloss value (ΔG^*).
4. In Brinell hardness measurement, ageing caused decrease in hardness for both control and EO treated samples, although this effect was not significant in beech samples. The EO treatment caused a decrease in hardness values for both (Oak and Elm) samples, though not significantly in oak samples.
5. In roughness measurements, ageing of both bulk wood types resulted in a significant increase in R_a values. In contrast, the effect of EO treatment was insignificant. Ageing of EO treated wood showed no marked effect on R_a values of beech, while the effect was significant in oak.
6. Librarians and museum curators can use the reported results of this study for planning the fumigation and maintenance of the wooden furniture and artifacts.

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