Effect of Ethylene Oxide Sterilization and Accelerated Ageing on the Physical and Mechanical Properties of Beech, Oak, and Elm Wood: Part 2

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The effect of ethylene oxide (EO) sterilization and/or accelerated ageing on impact bending strength (IBS), tensile strength along and across the fiber (TSALF and TSACF), ultimate flexural strength along and across the fiber (FSAL and FSAC), and splitting resistance (SR) was tested in bulk and veneer wood. The IBS was greatly increased in beech but decreased in some oak samples. The EO treatment followed by ageing caused the TSACF to increase in bulk beech and to decrease in bulk oak. The EO treatment decreased the TSALF values in bulk oak, beech, and elm veneer. However, beech veneer samples exhibited increased TSALF. The ageing of EO treated samples decreased the TSALF values for veneers (oak and beech), while both bulk samples increased. A notable decline was observed due to EO treatment of oak and beech, along with post ageing decline for oak samples in FSAC values only. The FSAL values decreased after ageing of EO treated and untreated samples (oak and beech), while a post EO treatment decline was observed in oak samples only. The SR (R_w) values decreased in EO-only treated oak wood and increased in EO treated and aged samples (oak and beech).

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

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INTRODUCTION

Ethylene oxide (EO), also known as oxirane, is the simplest epoxide compound, and it is most commonly used as a fumigant for sterilization purposes. The advantage of EO as a sterilizing agent for porous materials (such as wood) is its gaseous nature, which helps its quick diffusion into pores and easy removal after sterilization. A number of studies have been reported on the sterilizing action of EO for wood based materials (Smith and Sharman 1971; Tohmura *et al.* 2012). A comparative study on the sterilizing actions of EO and propylene oxide (PO) on spruce heartwood blocks reported that in the presence of moisture bacteria was more resistant to PO than fungi, and ascomycetes were more resistant to PO than EO (Smith 1965; 1968). In another study, EO was studied as a sporicidal sterilizing agent for pine wood, archival paper, and painted canvas, and has also been studied for *B. anthracis* and *B. atropheaus* under optimal conditions (Whitney *et al.* 2003; US EPA 2013). Along with sterilization of wood, the tendency of EO to react with various chemical components of wood (cellulose, lignin, and hemicellulose) is due to the highly strained cyclic structure of EO, which favors a nucleophilic attack by hydroxyl groups, leading to the generation of an additional hydroxyl group that is available for further polymerization reaction (Kumar 1994; Cetin and Hill 1999; Kral *et al.* 2015). These epoxides have also been studied as a wood modifying agent because the chemical reaction between wood components and EO leads to polymer formation (Kumar 1994). Ageing (normal and storage) has been widely studied as an effect on the property of wooden material since it affects the mechanical properties of wood (Ganne-Chédeville *et al.* 2012; Kránitz 2014; Mishra *et al*. 2018). However, the effect of ageing on EO treated wood is relatively less studied. Ethylene oxide is the fumigant of choice for libraries and museums due to its ease of application and practicality.

This manuscript represents the second part of a two-stage study. The first part reported the effect of EO and/or accelerated ageing on the surface characteristics of beech, oak and elm wood species (Tesařová *et al*. 2018). In this study, beech, oak, and elm species were studied to gauge the impact of EO treatment and/or ageing (accelerated testing) on mechanical properties. Two different types of thicknesses (veneer and solid wood) were used as samples. The goal was to understand the immediate and long-term effects (using accelerated ageing methods) of EO treatment on the properties of wood. The derived aim of this study was to understand the mechanical performance of library and museum furniture (immediate and long term) following EO treatment.

EXPERIMENTAL

Materials

Three different species of wood were considered, beech, oak, and elm. Due to Dutch elm disease, samples of solid wood/bulk wood in the required thickness were not available in the central European market, and they were not used in this study. Instead, elm samples of a thickness of 0.6 mm were used. Different samples with specifications, treatments, and sample codes are presented in Table 1. All wooden boards were supplied Potrusil s.r.o (Brno, Czech Republic) and sampling was done was at university laboratories.

Methods

Ethylene oxide treatment

The effect of the EO treatment was similar to what was reported in Part 1 of this study. A sterilization chamber (DeLama, San Martino Siccomario, Italy) with a usable volume of 1.8 m^3 was used to sterilize the samples. As a sterilization medium, the device employs a gaseous mixture of ethylene oxide and carbon dioxide in a ratio of 10:90. During the sterilization process, the P5 sterilization program (Inbuilt in the instrument) was used that involved an operating temperature of 25 °C, an overpressure of 3.8 bars, and a 50% wetting of the material (by EO).

The whole process lasted 720 min. After completion of the sterilization program, wood samples were subjected to the P98 aeration program (Inbuilt in the Instrument) to remove ethylene oxide residues. With the P98 venting program, the air was exchanged 88 times in 22 h.

Accelerated ageing

Accelerated ageing was performed using a modified method based on ČSN 673098 (1988). The accelerated ageing process was done for 25 cycles. Each cycle consisted of two stages: one at 50 °C and a relative humidity (RH) of 75% for 12 h, followed by the second stage at -30 °C and a RH of 0% for 12 h. The Memmert HPP 108 air conditioning chamber (Swabach, Germany) was used at an operating temperature range of 5 °C to 70 °C ± 1 °C and RH range of 10 to 90% ± 1.5%. The freezer used was an Elcold (HPP108, Hobro, Denmark) with operating range of 0 °C to -40 °C ± 0.5 °C, with setting gradations of 1 °C.

Table 1a. Explanation of Different Sample Codes Used in the Study

Wood	Sample Type	EO Treatment	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_1UU_g**
Elm	Veneer 200x150x0.6 (mm) (V)	Untreated (U)	Yes (A_g)	**EV_1UA_g**
Elm	Veneer 200x150x0.6 (mm) (V)	Treated (E)	No-(U_g)	**EV_1EU_g**
Elm	Veneer 200x150x0.6 (mm) (V)	Treated (E)	Yes (A_g)	**EV_1EA_g**

Impact bending strength (IBS)

Impact bending strength tests (Charpy impact test) were completed to compare the effects of EO treatment and ageing on the fracture resistance of the wood samples. All tests were done using an Instron CEAST 9050 Impact Pendulum (Norwood, MA, USA) per ČSN 490117 (1982) standards. The shape of test body was a rectangular prism. The IBS (A_w) was calculated using the formula,

$$A_w = \frac{Q}{b \times h} \tag{1}$$

where Q is the work done in splitting/disturbing the sample in joules, and b and h are dimensions (cm) of the sample in radial and tangential directions. Usually, IBS is calculated at fixed humidity but the effect of humidity was ignored due to comparative nature of study (all samples were measured at same humidity level).

Tensile strength along and across the fibers (TSALF and TSACF)

For the tensile strength measurements an Instron UTM 3365 (Norwood, MA, USA) was used. ČSN 490113 (1992) and ČSN 490114 (1992) standards were employed for the tensile strength measurement along and across the grain, respectively. The modulus of elasticity parallel to grain was calculated from the linear part of the load curve using the strain gauge. Test bodies for both tests were prepared according to ČSN 490123 (2002). TSALF (σ_{tw}) was calculated using the following formula,

$$\sigma_{tw} = \frac{F_{max}}{a \times b} \tag{2}$$

where F_{max} is the break force in newtons (N), and a and b are width and thickness (mm) of test body/samples.

Ultimate flexural strength along and across the fibers (FSAL and FSAC)

Ultimate flexural strength was calculated using a three-point bending test. Dimensions of the test specimens were 20 × 20 mm in cross section, with a length of 300 mm. All measurements were done using an Instron UTM 3365 device (Norwood, MA, USA) per the ČSN 490115 (1979) standard. Fractural strength (σ_w) was calculated using the following formula,

$$\sigma_w = \frac{3F_{max} \times l}{2b \times h^2} \tag{3}$$

where F_{max} is the fracture load in N, l is the distance between the centers of the supports in mm, *(h)* is the height in mm, and *(b)* is the width of the test body in mm.

Splitting resistance

Splitting resistance was calculated using a ZWICK Z 0500 / TH3A Splitting Testing Device (Ulm, Germany) per the ČSN 490119 (1984) standard. In this test, the sample is placed in clamping jaws to firmly secure the sample in grooves. Maximum force is measured for splitting the sample; two different coefficients can be calculated with respect to width of test body and nominal area of splitting. Splitting resistance (R_w) was calculated using the following formula,

$$R_w = \frac{F_{max}}{A} \tag{4}$$

where F_{max} is the maximum force in N, and A is the area of the test body in mm^2 ($A=b \times l$, where b is the width of test sample, and l is the length of the split in mm.)

RESULTS AND DISCUSSION

Ageing generally can be assumed to involve the decomposition of hemicellulose and cellulose, cross-linking of lignin, rearrangement of polysaccharides, and hornification (Obataya 2017). However, all artificial ageing cannot be assumed similar to the natural ageing, as different ageing parameters (maximum temperature and presence or absence of UV light) affect different ageing processes differently. The impact bending strength of all tested samples is listed in Table 2, and Fig. 1 shows a mean interaction plot. In bulk beech samples, ageing and EO treatment followed by ageing showed a significant increase of 17.4% and 9.1% respectively, while EO-only treatment reflected an insignificant increase in IBS when compared with the control. In the case of beech and oak veneer samples, EO treatment and ageing caused an insignificant decrease in IBS values as compared to the control.

Table 2. IBS (Impact bending strength) Values Measured in J × cm^{-2}

Code	Number of samples	Mean Values	Standard Deviation	Description
BBUUg	23	98.99^{a}	24.47	Control
BBUAg	20	116.31	6.17	•
BBEUg	23	104.74^{a,b}	15.34	•
BBEAg	20	108.03^{b}	13.47	•
BVUUg	22	39.43^{c,d,e}	5.21	Control
BVUAg	23	36.58^{c,f,g}	5.24	•
BVEUg	22	34.01^{d,f,h}	5.42	•
BVEAg	23	36.57^{e,g,h}	3.09	•
OBUUg	23	81.22^{i,j}	25.24	Control
OBUAg	20	87.41^{i,k}	25.64	•
OBEUg	23	85.61^{j,k}	18.25	•
OBEAg	20	67.53	17.46	•
OVUUg	22	27.12^{l,m,n}	7.74	Control
OVUAg	23	28.48^{l,o,p}	4.91	•
OVEUg	22	28.17^{m,o,q}	3.25	•
OVEAg	23	30.31^{n,p,q}	5.39	•
EV1UUg	22	29.71^{r,s,t}	4.55	Control
EV1UAg	23	31.47^{r,u,v}	4.52	•
EV1EUg	22	28.70^{s,u,w}	5.28	•
EV1EAg	23	29.25^{t,v,w}	5.55	•

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

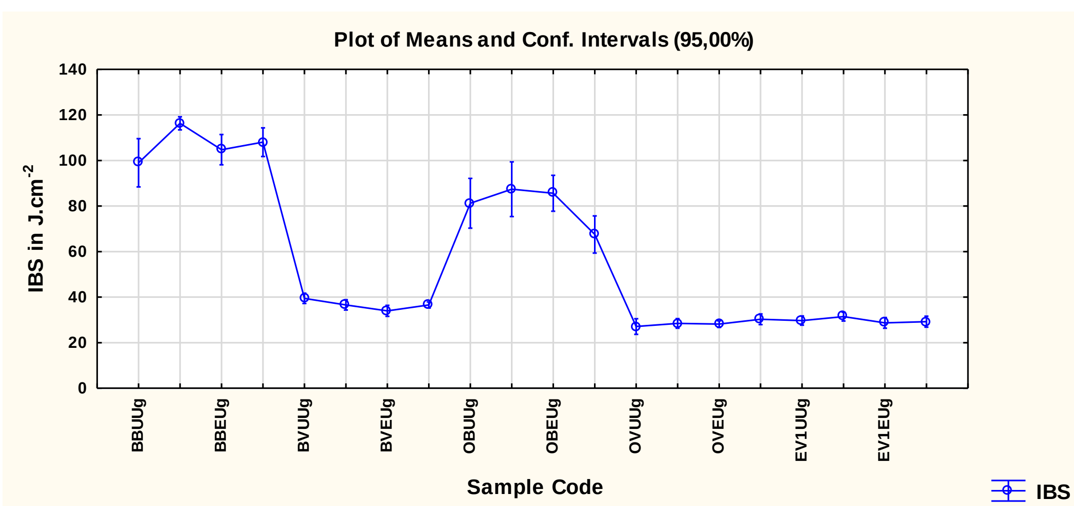

Fig. 1. Mean interaction plot of measured IBS (Impact bending strength) values

A significant increase in IBS by ageing of EO treated oak veneer samples was observed; however this effect was insignificant in the case of beech veneer samples. The bulk oak wood samples showed an insignificant increase in IBS due to EO treatment and ageing separately; however a considerable decrease (significant) in IBS of the EO treated sample was observed because of ageing. The reason for the decreased IBS is unclear. The elm samples also behaved similarly to the oak and beech veneer samples, where an insignificant increase in IBS values was observed. However, a decrease in IBS and fungal attack on heat treated wood was also reported (Henningsson 1967; Kubojima *et al.* 2000). The decline in bending properties caused by ageing has also been reported in eastern white pine (Attar-Hassan 1976). The effect of ageing on Oak wood was also generally reported as negative (IBS value decrease with age) by previous reports (Sonderegger *et al.* 2015); however an inherent variability in wood has precluded a definitive conclusion (Cavalli *et al.* 2016).

The TSACF of the tested samples is presented in Table 3 with a mean interaction plot in Fig. 2. Measurements for the oak and elm veneers were not possible due to sample fragility. In case of bulk samples (beech and oak), the ageing caused a significant decline (12.13 and 31.5% respectively) in TSACF values with respect to control. The decline in TSCAF values after EO treatment was great 14.09% for bulk beech and insignificant for bulk oak samples as compared to the control.

Table 3. TSACF (Tensile strength across the fibers) Measured in N × mm^{-2}

Code	Number of samples	Average	Standard Deviation	P-value
BBUUg	16	10.22	1.130518	control
BBUAg	12	8.98^{a}	2.062104	•
BBEUg	16	8.78^{a}	0.713420	•
BBEAg	13	11.51	1.348813	•
BVUUg	12	1.98^{b,c,f}	0.656736	control
BVUAg	10	2.36^{b,e,f}	0.827222	•
BVEUg	10	2.46^{c,e,g}	0.864463	•
BVEAg	10	2.53^{d,f,g}	0.734975	•
OBUUg	14	10.96^{h}	0.660462	control
OBUAg	12	7.50^{g}	1.718999	•
OBEUg	16	10.64^{h}	1.615877	•
OBEAg	10	7.31^{g}	1.441819	•

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

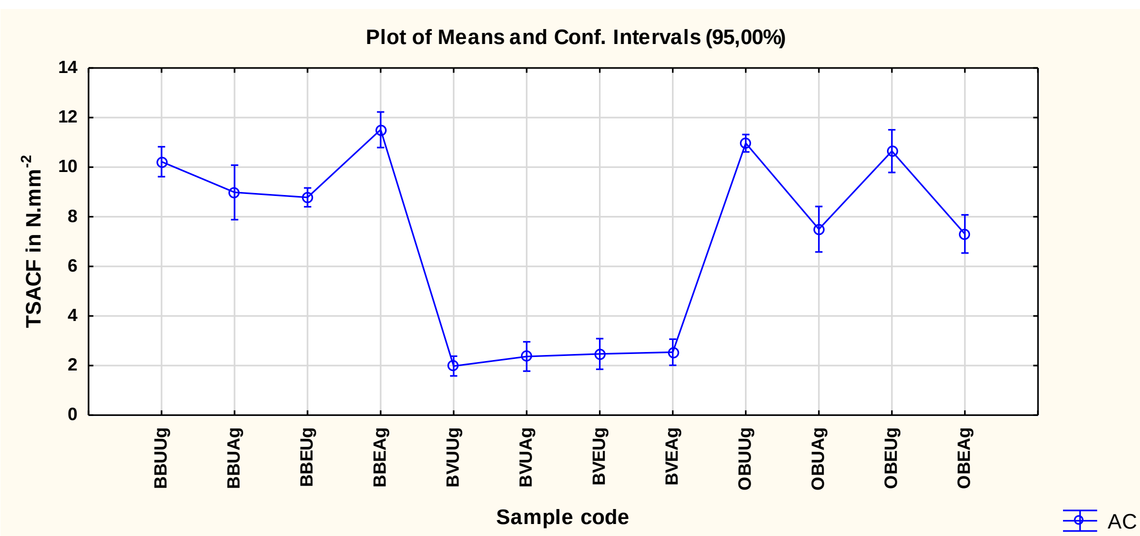

Fig. 2. Mean interaction plot of the measured TSACF (Tensile strength across the fibers) values

The tested samples of beech veneer showed an effect, which was mostly insignificant for every treatment. The EO treatment followed by ageing caused a significant increase in TSCAF values for beech and a significant decline for oak wood samples. The difference in behavior of these two types of wood can be attributed to the difference in relative chemical composition, and variability with in wood samples makes it difficult to generalize behavior as both type of effects have been reported in literature (Cavalli *et al.* 2016).

The TSALF values of measured samples are presented in Table 4 with a mean interaction plot in Fig. 3. In the case of beech samples (both bulk and veneer), ageing showed a significant increase (12.8 and 1.95%, respectively) in TSALF values; however, the effects of the EO treatment caused a significant decrease in bulk beech, and this effect was reversed in veneer samples (significant decrease). The EO treatment followed by ageing led to an increase and decrease (both significant) in TSALF values for bulk and veneer samples, respectively.

It was noticeable that sample thickness and EO treatment reversed the behavior of beech samples. For oak samples, the ageing caused a decrease and increase (both significant) in TSALF values for bulk and veneer samples, respectively. However, the decline (significant) in TSALF values by EO treatment was noticeable in bulk oak but insignificant in veneer oak. The EO treatment followed by ageing led to a visible rise in TSALF values of bulk oak samples but veneer samples showed a decline (both significant). In elm samples, three of the treatments led to a decline in TSALF values, out of which in EO treatment followed by ageing reflected an insignificant decline.

Table 4. TSALF (Tensile strength along the fibers) Measured in N × mm^{-2}

Code	Number of samples	Means	Std. Dev.	P-value
BBUUg	10	117.87	20.25535	control
BBUAg	13	132.68^{a}	19.75074	•
BBEUg	10	114.60^{a}	15.56589	•
BBEAg	13	119.99	27.80902	•
BVUUg	19	102.53	20.50000	control
BVUAg	14	104.64^{b}	30.98362	•
BVEUg	20	109.43$^{\mathbf{b}}$	28.83092	•
BVEAg	12	89.14	11.82719	•
OBUUg	10	88.11	20.44279	control
OBUAg	13	84.61	12.95583	•
OBEUg	14	72.81^{c}	21.12175	•
OBEAg	13	101.10^{c}	33.96435	•
OVUUg	16	54.98^{d}	19.71704	control
OVUAg	12	57.41^{e}	12.49035	•
OVEUg	16	24.81^{d,e,f}	8.18386	•
OVEAg	12	50.95^{f}	15.99739	•
EV1UUg	14	73.8943^{g}	24.51469	control
EV1UAg	12	65.2449	26.70605	•
EV1EUg	22	65.6946	28.49201	•
EV1EAg	12	51.4813^{g}	23.72196	•

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

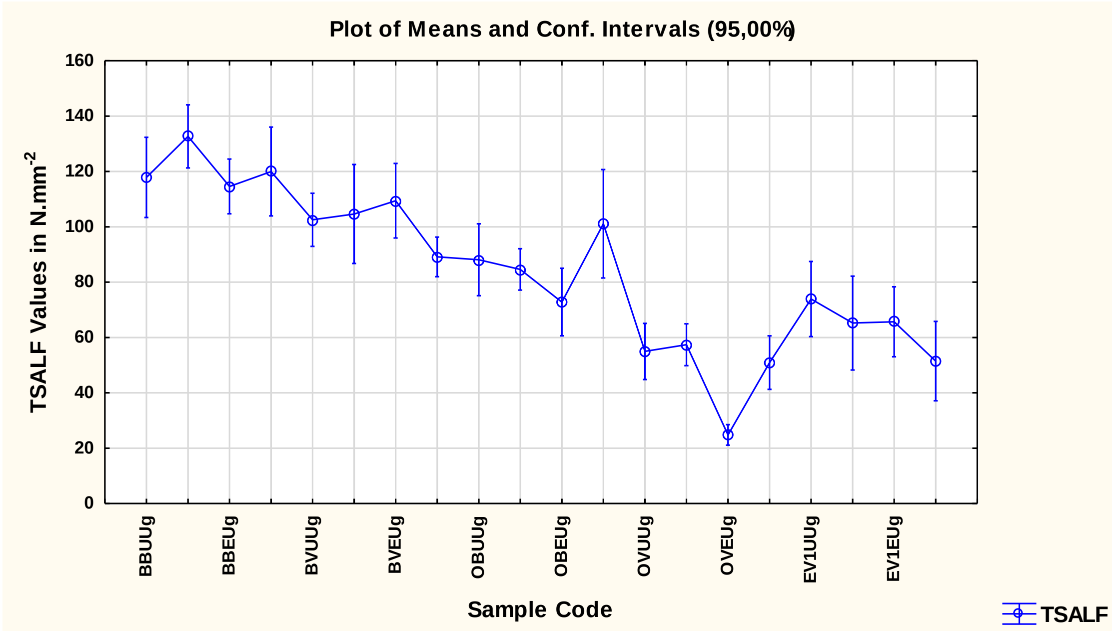

Fig. 3. Mean interaction plot of estimated TSALF (Tensile strength along the fibers) values

The FSAL values of the tested samples can be found in Table 5 with a mean interaction plot in Fig. 4. In both cases of bulk samples (oak and beech) the ageing caused a significant decline in FSAL values. The effect of EO treatment was insignificant in beech samples; however, a significant decrease in FSAL value was observed in oak samples. The EO treatment followed by ageing led to a significant decrease in FSAL values, but these values were 10.6 and 10.9%. When compared to the EO treated beech and oak sample (4.15 and 22.6%), respectively. These decrease in FSAL value showed similar values as the result of ageing, which was relatively in only EO samples.

The FSAC values for the tested samples can be found in Table 6, with their mean interaction plot in Fig. 5. Like the FSAL values, the ageing caused a decline in FSAC values (insignificant in the case of beech). However, the decline shown in FSAC values by EO treatment was significant in both (beech and oak) types of samples. In the samples that underwent EO treatment followed by ageing the decline in FSAC values was visible (significant) in the beech samples and insignificant in the oak samples.

Table 5. FSAL (Ultimate flexural strength along the fibers) Measured in N × mm^{-2}

Code	Number of samples	Means	Standard Deviation	P-value
BBUUg	12	149.18^{a}	16.73	control
BBUAg	12	110.63	15.16	•
BBEUg	12	155.66^{a}	7.19	•
BBEAg	12	134.47	6.75	•
OBUUg	12	119.27	21.20	control
OBUAg	12	86.16^{b}	13.40	•
OBEUg	12	92.02^{b}	8.89	•
OBEAg	12	106.72	16.08	•
•Values are significantly different to the control (ANOVA; $p < 0.05$). Values having the same letter in superscript are not significantly different (Duncan post-hoc).				

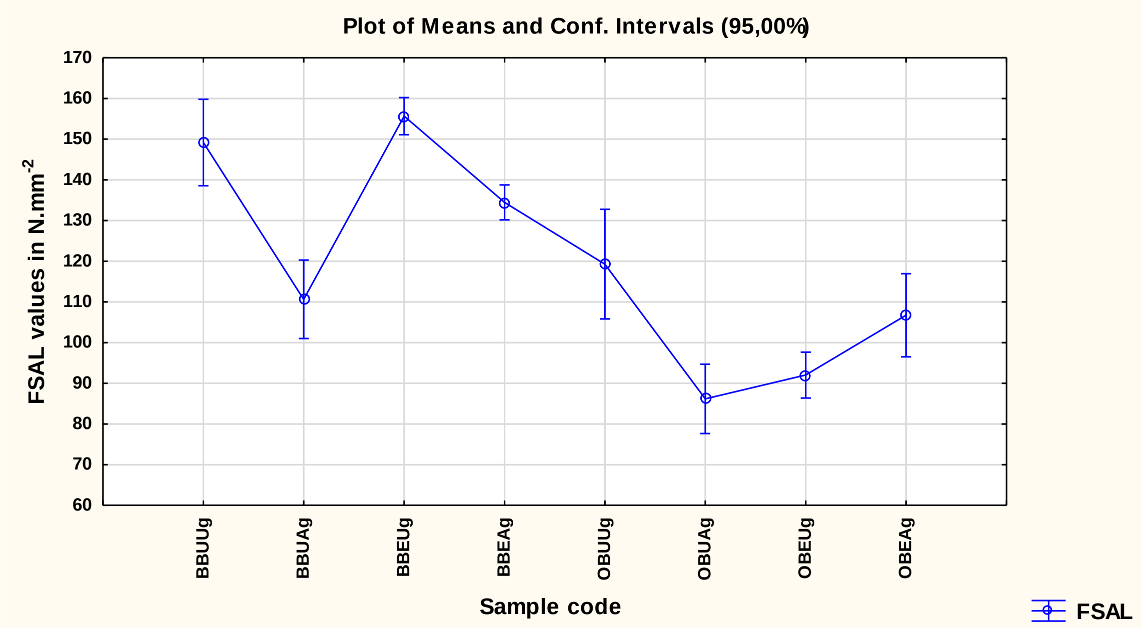

Fig. 4. Mean interaction plot of measured FSAL (Ultimate flexural strength along fibers) values

Table 6. FSAC (Ultimate flexural strength across the fibers) measured in N×mm^{-2}

Code	Number of samples	Means	Standard Deviation	P-value
BBUUg	12	15.25^{a,b}	2.79	control
BBUAg	12	11.09^{b,c}	1.77	•
BBEUg	12	14.34^{c,d}	2.35	•
BBEAg	12	10.10^{b,d}	4.69	•
OBUUg	12	12.79^{e}	1.34	control
OBUAg	12	11.66^{f}	3.32	•
OBEUg	12	10.00	3.25	•
OBEAg	12	8.47^{e,f}	1.16	•

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

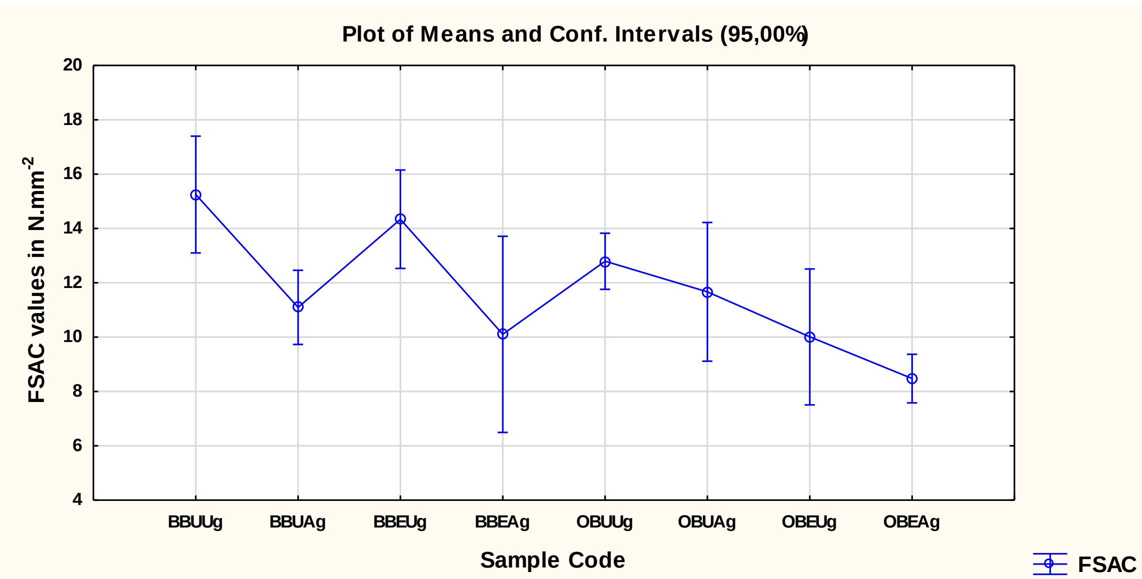

Fig. 5. Mean interaction plot of measured FSAC (Ultimate flexural strength along the fibers) values

Splitting resistance (R_w) values of the tested samples are shown in Table 7, and their mean interaction plot is shown in Fig. 6. There was no significant effect of ageing on R_w values in either (beech or oak) samples. However, EO treatment caused a significant increase and insignificant decrease in R_w values of beech and oak samples, respectively. The EO treatment followed by ageing, showed a significant increase in R_w values for both types of samples. The post ageing R_w values of EO treated samples, when compared to EO-only treated samples, showed an insignificant increase in the case of oak samples and significant decrease in case of Bulk beech samples.

Table 7. R_w (Splitting resistance) measured in N × mm^{-2}

Code	Number of samples	Means	Std. Dev.	P-value
BBUUg	17	1.024^{a}	0.148495	control
BBUAg	20	1.279^{a,b,c}	0.236231	•
BBEUg	20	1.112^{b}	0.129680	•
BBEAg	20	1.030^{c}	0.116212	•
OBUUg	20	1.099^{d,e}	0.125568	control
OBUAg	20	0.816^{d,f,g}	0.100120	•
OBEUg	20	0.916^{e,f,h}	0.238554	•
OBEAg	20	1.168^{h,g}	0.108301	•

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

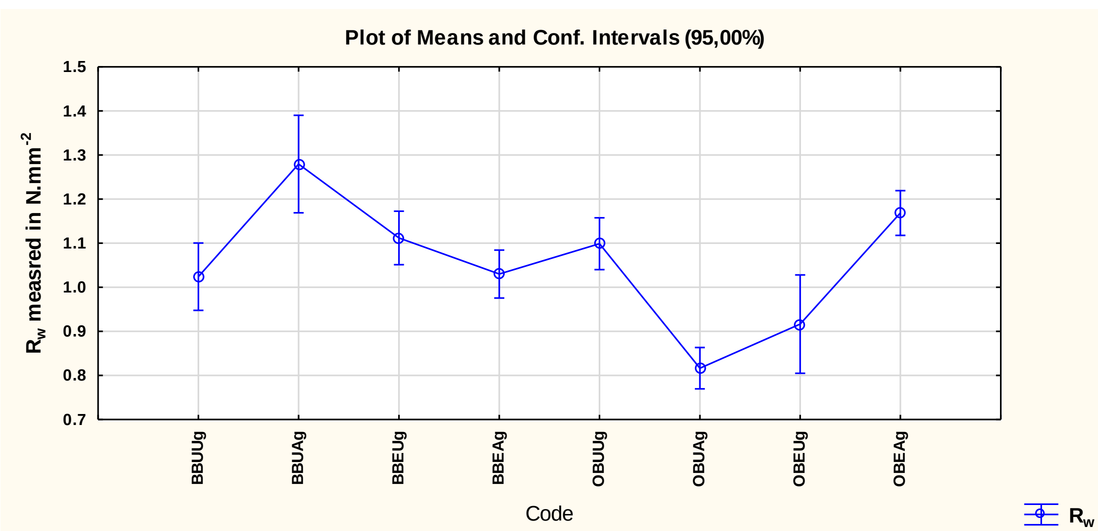

Fig. 6. Mean interaction plot of the measured R_w (Splitting resistance) values

CONCLUSIONS

1. In impact bending strength (IBS) measurements, the veneer samples for all three wood species did not show any significant effect of ageing, ethylene oxide (EO) treatment and EO treatment followed by ageing. However, in bulk beech showed a significant increase in IBS upon ageing and EO treatment followed by ageing samples. In oak samples (bulk), the effect of ageing and EO treatment were found to be insignificant; however, a significant decrease was observed in aged EO treated samples.

2. In the measured values of tensile strength across the fiber (TSACF), the bulk samples of both types (oak and beech) showed a significant decrease with ageing. Those subjected to EO treatment followed by ageing reflected different behaviors: a significant increase in TSACF values in beech samples and a significant decrease in oak samples. In tensile strength along the fiber (TSALF) values, ageing caused a significant increase for beech (bulk and veneer samples) and oak veneer samples, while a significant decrease was registered in bulk oak and elm veneer samples. The EO treatment reflected a significant decrease in bulk (oak and beech) and elm veneer samples. However, a significant increase was registered for beech veneer samples. The ageing of EO treated samples caused a significant decline in the TSALF values for veneers (oak and beech), while both bulk samples showed a significant increase.
3. In the ultimate flexural strength across the fiber (FSAC) value measurements, a significant decline was observed with EO treatment of both (oak and beech) samples, along with post ageing decline for oak samples only. In measured values of ultimate flexural strength along the fiber (FSAL), with ageing of EO treated and untreated, both samples showed a significant decline for both types (oak and beech) except ageing of EO treated oak (significant increase) and insignificant decrease ageing of EO treated Beech.
4. In the measured values of splitting resistance (R_w), EO treatment followed by ageing led to a significant increase in splitting resistance (R_w) values for both samples with exception to the EO treated bulk oak.

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