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Effect of Water Repellents on Hygroscopicity and Dimensional Stability of Densified Fir and Aspen Woods

Utjecaj vodoodbojnih sredstava na higroskopnost i dimenzijsku stabilnost ugušćenog drva jele i drva jasike

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ABSTRACT • *This study investigated the effect of pre-impregnation with water-repellent agents on the hygroscopicity and dimensional stability of fir (Abies bornmulleriana Mattf.) and aspen (Populus tremula L.) woods. After pre-vacuum treatment, the samples were impregnated at atmospheric pressure with paraffin, linseed oil and styrene, and then densified at compression rates of 20 % and 40 % at 120, 150 and 180 °C. The results showed that water repellents significantly affected the hygroscopicity and dimensional stability of the densified wood samples. Compression recovery rate (CRR), thickness swelling (TS), equilibrium moisture content (EMC), and water absorption (WA) values of the densified samples decreased with impregnation pretreatments. The linseed oil treatment gave more positive CRR and TS results than paraffin. Lower EMC and WA values were found in the paraffin-treated samples. However, the most successful results for all tested properties were determined in the styrene pretreated samples in which hygroscopicity decreased and dimensional stability increased (especially for aspen) due to increases in the compression rate and temperature related to densification conditions. In the styrene pretreated samples, the high temperature (180 °C) and compression rate (40 %) significantly reduced CRR, TS, EMC and WA, total dimensional stability was nearly achieved and the water repellent effectiveness was close to 100 %.*

Keywords: *densification; dimensional stability; hygroscopicity; impregnation; water repellents*

SAŽETAK • *U radu se prikazuje istraživanje utjecaja predimpregnacije drva jele (Abies bornmulleriana Mattf.) i jasike (Populus tremula L.) vodoodbojnim sredstvima na njihovu higroskopnost i dimenzijsku stabilnost. Nakon vakuumske obrade uzorci su pri atmosferskom tlaku impregnirani parafinom, lanenim uljem i stirenom. Zatim je provedeno ugušćivanje sa stupnjevima ugušćenja 20 i 40 % pri temperaturi od 120, 150 i 180 °C. Rezultati su pokazali da vodoodbojna sredstva značajno utječu na higroskopnost i dimenzijsku stabilnost uzoraka ugušćenog drva. Predimpregnacijom uzoraka smanjili su se stupanj povrata ugušćenja (CRR), debljinsko bubrenje (TS), ravnotežni sadržaj vode (EMC) i apsorpcija vode (WA) ugušćenih uzoraka. Postupkom s lanenim uljem smanjeni*

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su povrat ugušćenja i debljinsko bubrenje u usporedbi s postupkom impregnacije parafinom. Na uzorcima impregniranim parafinom utvrđene su niže vrijednosti ravnotežnog sadržaja vode i apsorpcije vode. Međutim, najbolji rezultati svih istraživanih svojstava dobiveni su na uzorcima impregniranima stirenom, kojima se zbog povećanja brzine i temperature ugušćivanja smanjila higroskopnost i povećala dimenzijska stabilnost (posebice drva jasike). Na uzorcima impregniranima stirenom i ugušćenima pri temperaturi od 180 °C sa stupnjem ugušćenja 40 % znatno su se smanjili stupanj povrata ugušćenja, debljinsko bubrenje, ravnotežni sadržaj vode i apsorpcija vode te je postignuta gotovo potpuna dimenzijska stabilnost i učinkovitost odbijanja vode (blizu 100 %).

Ključne riječi: *ugušćivanje; dimenzijska stabilnost; higroskopnost; impregnacija; vodoodbojna sredstva*

1 INTRODUCTION
1. UVOD

Due to its superior properties, wood is used in many structural and non-structural applications. However, due to the difficulties in supplying high-quality wood and the increase in younger, fast-growing and less durable trees, studies on modifications for improving the properties of wood have been accelerated (Rowell, 2012). Many modification processes are currently applied, mainly to improve wood properties such as hygroscopicity, dimensional stability, mechanical strength, biological resistance and UV resistance. Various new technologies such as thermal modification, acetylation, furfurylation and different impregnation and densification processes have been successfully introduced to the market as modern technologies (Sandberg *et al.,* 2017; Lunguleasa *et al.,* 2018).

In order to increase the service life of wood, it is necessary to protect it from damp conditions and prevent dimensional changes (Koski, 2008). Unless necessary measures are taken with wood, high humidity causes undesirable physical changes such as bending, buckling and cracking. In addition, biological pests that require excess moisture to live and develop cause wood to break down. For this reason, it is important to remove excess moisture from the wood and keep its equilibrium moisture content at lower levels. The water absorption rate can be significantly reduced by forming a water repellent barrier in the wood (Williams and Feist, 1999; Koski, 2008).

On the other hand, the dimensional stability of wood can be achieved by blocking the wood cell lumen and reducing the water absorption or bulking and swelling of the cell wall. The impregnation of water-insoluble materials into the cell wall structure of wood is an effective method for keeping the wood cell wall in a swollen state. This can be achieved by impregnation with a variety of suitable agents, such as natural or synthetic resins, paraffin / wax and vinyl monomers. In this way, the dimensional changes due to moisture differentiation in the wood are significantly reduced (Stamm and Tarkow, 1947; Deka and Saikia, 2000; Kocaefe *et al.,* 2015).

Most properties of wood are closely related to its density. The hardness, wear resistance and other mechanical properties can be improved by modifying the wood via densification. Densification modification is important, especially in order to increase the strength properties of low-density wood species and to enable use in a larger area (Laine *et al.,* 2013; Sandberg *et al.,* 2013). Densification is based on increasing the material density by decreasing the void volume in the wood. The main aim of wood densification is to improve the mechanical properties of wood (Laine *et al.,* 2013). Wood is generally densified using three different methods. The first is mechanical compression of wood using high pressure under the effect of heat or steam. The second is filling the wood cell cavities with synthetic or natural resins via impregnation. The third is a combination of mechanical compression and impregnation methods (Rowell and Konkol, 1987; Kamke, 2006; Kutnar *et al.,* 2008). The main issue associated with mechanically densified wood is the fixation of the compressed thickness. When exposed to water or heat, mechanically densified wood tends to return to its original dimensions prior to compression (Navi and Heger, 2004; Laine *et al.,* 2013). In order to increase the dimensional stability of densified wood, thermal processes are applied at the time of compression or prior to and following compression. Due to these thermal processes, the dimensional stability of densified wood is significantly increased (Dwianto *et al.,* 1997; Navi and Girardet, 2000; Kamke and Sizemore, 2008; Welzbacher *et al.,* 2008; Fang *et al.,* 2012; Kutnar and Kamke, 2012; Pelit *et al.,* 2016; Kariz *et al.,* 2017). However, thermal processes generally have a negative effect on the hardness or mechanical strength properties of densified wood (Welzbacher *et al.,* 2005; Gong *et al.,* 2010; Kwon *et al.,* 2014; Dubey *et al.,* 2016; Pelit *et al.,* 2018). Another important method for fixing the compressed thickness in densified wood is impregnation with phenolic resins. In the case of wood treated with resins such as phenol and melamine formaldehyde prior to compression, dimensional stability is almost completely achieved. In addition, the mechanical properties of these materials are significantly increased. However, due to the resin properties, the friability of densified wood is also increased (Seborg *et al.,* 1962; Kollmann *et al.,* 1975). In addition, the cost of phenolic resin treatment is high, and the color change in the material to which it is applied is also an important disadvantage (Morsing, 2000).

The aim of this study was to contribute to the literature by determining the effect of pre-impregnation with water-repellent substances (linseed oil, paraffin) and a vinyl monomer (styrene) on the hygroscopicity and dimensional stability of fir (*Abies bornmulleriana* Mattf.) and aspen (*Populus tremula* L.) wood densified at different temperatures and compression rates.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Wood samples
2.1. Uzorci drva

This study used fir (*Abies bornmuelleriana* Mattf.) and aspen (*Populus tremula* L.) wood, both of which have relatively low densities (fir: 0.45 g/cm^3, aspen: 0.37 g/cm^3). The wood was selected randomly from a timber company in Düzce, Turkey. Fir and aspen samples were cut from sapwood in rough sizes, in accordance with the study methodology. Attention was paid to ensure that no rot, knots, cracks, or density differences were present in the samples (ISO 3129, 2012). The samples were subjected to natural drying to approximately 12 % moisture content, and then cut to the dimensions of 300 mm × 20 mm (longitudinal direction × tangential direction) and three different thicknesses 20 (for undensified samples), 25 and 33.3 mm (radial direction). Before impregnation, the samples were held in a drying oven at 70 °C until they reached a stable weight. The samples were then weighed using an analytical balance and their weights were recorded.

2.2 Impregnation of wood samples
2.2. Impregnacija uzoraka drva

As water repellents, paraffin (oil ratio: 25-35 %, melting point: 61-63 °C), linseed oil (density: 0.93-0.95 g/cm^3, drying time: 18-20 h at 20 °C, packing: in 17 kg tin) and styrene (density: 0.909 g/cm^3, boiling point: 140 °C, packing: in 15 kg tin) were used as impregnating agents. Pre-treatment procedures were applied to the water-repellent materials before impregnation. The paraffin in solid form was placed in a metal container and melted by exposure to heat. The linseed oil, which was at its packaged viscosity, was thinned by adding 100 % synthetic thinner. The styrene monomer was mixed with 1 % catalyst (methyl ethyl ketone peroxide) to allow polymerization to take place. Thus, the water repellents were made ready for impregnation. A cylindrical tank assembly with a vacuum holder was used in the impregnation of the wood samples in accordance with ASTM D 1413-76 (1976) standard. With this arrangement, a pre-vacuum equivalent pressure of 760 mmHg^{-1} was applied to the samples for 60 min. The impregnation solutions were then diffused into the samples by holding at atmospheric pressure for 24 h. In order to prevent the melted paraffin from solidifying again, the samples in the paraffin solution were held at 80 °C for 24 h.

Following the impregnation processes, the excess impregnation solutions were wiped from the samples and they were weighed again and their weights recorded. Afterwards, the paraffin- and linseed-oil treated samples were kept at a constant temperature of (20±2) °C and relative humidity (RH) of (65±3) % in accordance with ISO 13061-1 (2014). The samples treated with styrene monomer were wrapped in aluminum foil and then incubated in an oven at 90 °C for 2 h to initiate the polymerization process. These samples were then removed from the oven and immediately densified.

2.3 Densification process of wood samples
2.3. Proces ugušćivanja uzoraka drva

The impregnated samples were densified using special metal molds in a hydraulic test press having table dimensions of 60 cm × 60 cm (Figure 1a). Densification was carried out at three different temperatures and two

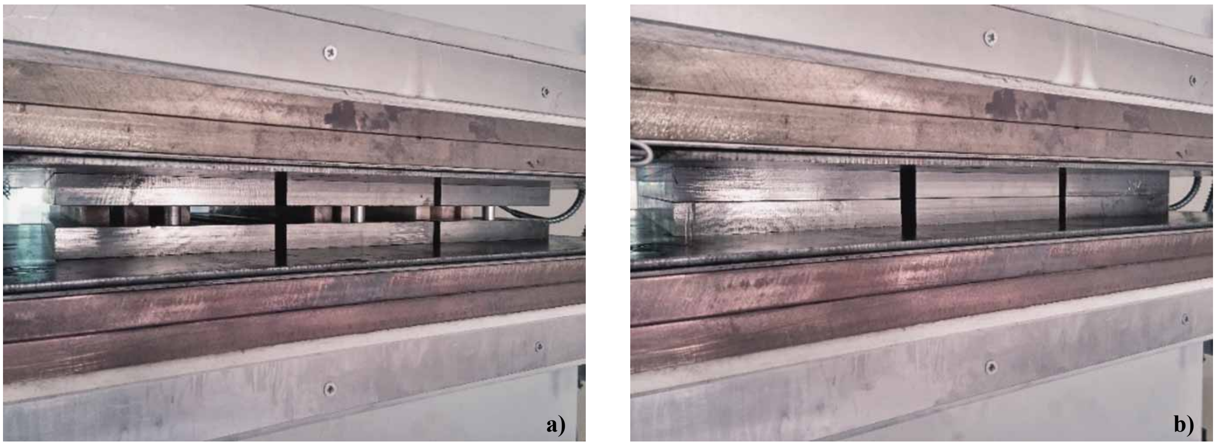


Figure 1 Densification of samples in hot press using metal molds
Slika 1. Ugušćivanje uzoraka u vrućoj preši uz pomoć metalnih kalupa

Table 1 Densification parameters
Tablica 1. Parametri ugušćivanja

<table>
<tr><th>Code in study
Oznaka u istraživanju</th><th>Pressing temperature, °C
Temperatura prešanja, °C</th><th>Compression ratio, %
Stupanj ugušćenja, %</th><th>Duration
Trajanje</th></tr>
<tr><td>A1</td><td>120</td><td>20</td><td rowspan="6">10 min heating
+
20 min pressure

10 min zagrijavanje
+
20 min prešanje</td></tr>
<tr><td>A2</td><td>120</td><td>40</td></tr>
<tr><td>B1</td><td>150</td><td>20</td></tr>
<tr><td>B2</td><td>150</td><td>40</td></tr>
<tr><td>C1</td><td>180</td><td>20</td></tr>
<tr><td>C2</td><td>180</td><td>40</td></tr>
</table>

different compression rates. The parameters applied in the densification of the samples are given in Table 1.

Channels 10 mm in depth and 20 mm wide were opened in the metal molds used for densification. The 25 and 33.3 mm thick samples were placed in the channels, the tables had been pre-heated at the specified temperatures for 10 min and the compression of the samples was then carried out in the radial orientation with a loading speed of 60 mm/min. In order to achieve the targeted thickness (20 mm), the load was maintained until the metal molds came into contact with each other (Figure 1b).

The compressed samples were kept under pressure for 20 min and then were removed from the press together with the molds and cooled to room temperature under an average pressure of 0.5 MPa in order to minimize the spring-back effect. According to ISO 13061-1 (2014), after the densification process, samples remained in a conditioning cabin at (20±2) °C and (65±3) % RH until they reached a stable weight. The impregnated and densified samples were then cut into smaller samples according to the standard of the selected tests. The test samples were prepared in a number sufficient to accommodate eight repetitions (n = 8) for each variable.

2.4 Determination of retention
2.4. Određivanje retencije

The retention values of the wood samples impregnated with water repellents were determined using Eq. 1,

$$Retention\ (\mathrm{kg/m^3}) = (G \times C) / V \times 10 \quad (1)$$

where G is the amount (g) of water repellent absorbed by the samples, C is the concentration of the water repellent solution, and V is the volume (cm^3) of the wood samples.

2.5 Determination of dimensional changes
2.5. Određivanje dimenzijskih promjena

Compressed wood has a tendency to partially regain its original shape after the removal of applied pressure due to elastic recovery. This behavior is known as spring-back and results in changes in the compressed dimensions (Garcia-Romeu *et al.,* 2007). The spring-back (SB) values of the samples were calculated using Eq. 2. In addition, compression recovery rate (CRR) (or set recovery) of the densified samples after immersion in water was determined using Eq. 3 (Pelit *et al.,* 2014),

$$SB\ (\%) = [(T_3 - T_2) / T_2] \times 100 \quad (2)$$

$$CRR\ (\%) = [(T_4 - T_3) / (T_1 - T_3)] \times 100 \quad (3)$$

where T_1 is the initial thickness of samples before pressing, T_2 is the thickness of samples under pressure (load), T_3 is the thickness of samples conditioned at (20±2) °C and (65±3) % RH for eight weeks after pressing, and T_4 is the thickness of samples after submersion in water for three weeks.

Thickness swelling (TS) of wood samples was determined according to ISO 13061-15 (2017). The TS value were calculated using Eq. 4,

$$TS\ (\%) = [(T_4 - T_0) / T_0] \times 100 \quad (4)$$

where T_0 is the oven-dry thickness of samples after holding at 55 °C for 72 h.

2.6 Determination of hygroscopicity
2.6. Određivanje higroskopnosti

Equilibrium moisture content (EMC) of the samples was determined according to ISO 13061-1 (2014). The EMC was calculated using Eq. 5,

$$EMC\ (\%) = [(W_1 - W_0) / W_0] \times 100 \quad (5)$$

where W_1 is the weight of samples conditioned at (20±2) °C and (65±3) % RH for eight weeks and W_0 is the oven-dry weight of samples after holding at 55 °C (a temperature below the melting point of paraffin) for 72 h.

Water absorption (WA) of the samples was determined in parallel with the thickness swelling test. The WA was calculated using Eq. 6. In addition, water repellent effectiveness (WRE) values calculated for impregnated samples as compared to untreated samples were determined using Eq. 7,

$$WA\ (\%) = [(W_2 - W_0) / W_0] \times 100 \quad (6)$$

$$WRE\ (\%) = [(WA_c - WA_t) / WA_c] \times 100 \quad (7)$$

where W_2 is the weight of samples after submersion in water for three weeks, WA_c is the percentage of water absorption of the untreated samples and WA_t is the percentage of water absorption of the impregnated samples.

2.7 Statistical analyses
2.7. Statistička analiza

Analysis of variance (ANOVA) tests were performed to determine the effect of water repellents on the hygroscopicity and dimensional stability of the densified fir and aspen wood at the 0.05 significance level. Significant differences between the groups were compared using Duncan's test.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Retention
3.1. Retencija

The retention values of the fir and aspen wood samples determined after impregnation with water repellents are shown in Table 2. According to the findings, the highest retention value for both wood species

Table 2 Retention values of fir and aspen wood (kg/m^3)
Tablica 2. Vrijednosti retencije drva jele i drva jasike (kg/m^3)

Wood species *Vrsta drva*	**Water repellents** *Vodoodbojno sredstvo*		
	Paraffin *Parafin*	**Linseed oil** *Laneno ulje*	**Styrene** *Stiren*
Fir / *jelovina*	148.08 (9.47)	230.09 (20.38)	300.37 (18.17)
Aspen / *jasikovina*	155.35 (10.04)	230.16 (21.41)	311.56 (21.71)

Values in parenthesis are standard deviations. / *Vrijednosti u zagradama standardne su devijacije.*

was obtained in samples impregnated with styrene and the lowest in paraffin-impregnated samples.

3.2 Spring-back, compression recovery rate and thickness swelling
3.2. Povrat, stupanj povrata ugušćenja i debljinsko bubrenje

According to ANOVA results, the effect of water repellent type and densification condition factors on the spring-back (*SB*), compression recovery rate (*CRR*) and thickness swelling (*TS*) for fir and aspen woods was statistically significant ($p \leq 0.05$). Duncan's one-way comparison results conducted for the factors of water repellents and densification are given in Table 3.

Regarding water repellents, for the fir wood, the highest *SB* average was determined in the non-impregnated (untreated) samples (5.49 %), and the lowest in the samples impregnated with paraffin (3.09 %). For aspen wood, the highest SB was obtained in the samples impregnated with linseed oil (5.42 %) and the lowest in the paraffin-impregnated samples (3.58 %) (Table 3). The *SB* values were higher in both wood species impregnated with styrene and compressed at 120 °C (under conditions *A1* and *A2*) (Figure 2a). However, the SB values were significantly reduced in samples that were impregnated with styrene and compressed at elevated temperatures (especially at 180 °C). Compared to the untreated samples condensed under *C2* conditions, the SB values in the styrene-impregnated fir and aspen wood samples decreased by 80.9 % and 81.1 %, respectively. The styrene monomer absorbed by the wood samples was more likely to evaporate from the material at a high compression temperature. As a result of this situation, it can be said that the SB results were affected by the decrease in high internal stresses formed in the compressed wood due to the excess impregnation chemical. In addition, the decrease in the *EMC* ratios of the samples due to the increase in the compression temperature may have affected the SB results. For fir wood, in general, the SB values were decreased in samples impregnated with paraffin and linseed oil compared to untreated samples. In fir wood impregnated with paraffin and densified under *C2* conditions, SB results close to those of the styrene-treated samples were obtained. For aspen wood, the SB value of samples impregnated with paraffin was found to be near to or lower than that of the untreated samples. However, compared to untreated samples, the SB values were higher in aspen samples impregnated with linseed oil. Generally, in the evaluation of water repellents, the untreated and paraffin-impregnated samples yielded more positive results with densification applications at a low temperature (120 °C), while quite successful results were obtained in samples impregnated with styrene via densification applications at a high temperature (180 °C). Moreover, in both tree species impregnated with paraffin, SB values were lower than those of linseed oil (Figure 2a).

With respect to densification variables, the highest SB average for fir wood was found in samples

Table 3 Duncan's test results for mean values of *SB*, *CRR*, and *TS*
Tablica 3. Rezultati srednjih vrijednosti za *SB*, *CRR* i *TS* prema Duncanovu testu

Wood specie *Vrsta drva*	**Factor** *Čimbenik*	***SB*, %**		***CRR*, %**		***TS*, %**	
		Mean	SG	Mean	SG	Mean	SG
Fir wood *drvo jele*	Water repellents / *Vodoodbojna sredstva*						
	Untreated / *netretirano*	5.49	a	93.32	a	34.43	a
	Paraffin / *parafin*	3.09	c	87.27	b	31.51	b
	Linseed oil / *laneno ulje*	4.45	b	71.42	c	26.32	c
	Styrene / *stiren*	4.44	b	31.26	d	10.56	d
	Densification / *Ugušćivanje*						
	Undensified / *neugušćeno*	-	-	-	-	3.37	g
	A1	3.14	e	85.36	a	20.93	d
	A2	5.28	b	75.79	c	48.49	a
	B1	4.21	d	81.29	b	18.65	e
	B2	5.66	a	68.84	d	42.28	b
	C1	3.22	e	64.03	e	15.01	f
	C2	4.70	c	49.60	f	31.21	c
Aspen wood *drvo jasike*	Water repellents / *Vodoodbojna sredstva*						
	Untreated / *netretirano*	4.40	b	88.59	b	32.62	a
	Paraffin / *parafin*	3.58	d	91.37	a	32.20	a
	Linseed oil / *laneno ulje*	5.42	a	80.07	c	27.69	b
	Styrene / *stiren*	3.99	c	30.39	d	8.43	c
	Densification / *Ugušćivanje*						
	Undensified / *neugušćeno*	-	-	-	-	3.11	g
	A1	3.99	d	92.29	a	21.71	d
	A2	5.53	a	75.02	c	47.12	a
	B1	3.57	e	80.97	b	17.20	e
	B2	5.25	b	67.02	d	40.10	b
	C1	3.37	f	67.09	d	14.85	f
	C2	4.37	c	53.25	e	32.55	c

SG: statistical group (different letters denote a significant difference). / *SG: statistička grupa (različita slova označavaju značajnu razliku).*

densified under *B2* conditions (5.66 %) and the lowest in the samples densified under *A1* and *C1* conditions (3.14 % and 3.22 %). The highest SB for aspen wood was determined in the samples densified under *A2* conditions (5.53 %), whereas the lowest was for samples densified under *C1* conditions (3.37 %) (Table 3). The SB values of the densified fir and aspen samples varied depending on the compression rate and temperature. For both tree species, SB values were higher in the samples densified at the compression rate of 40 % compared to the 20 % compression rate (Figure 2a). In other words, the SB values had increased due to the

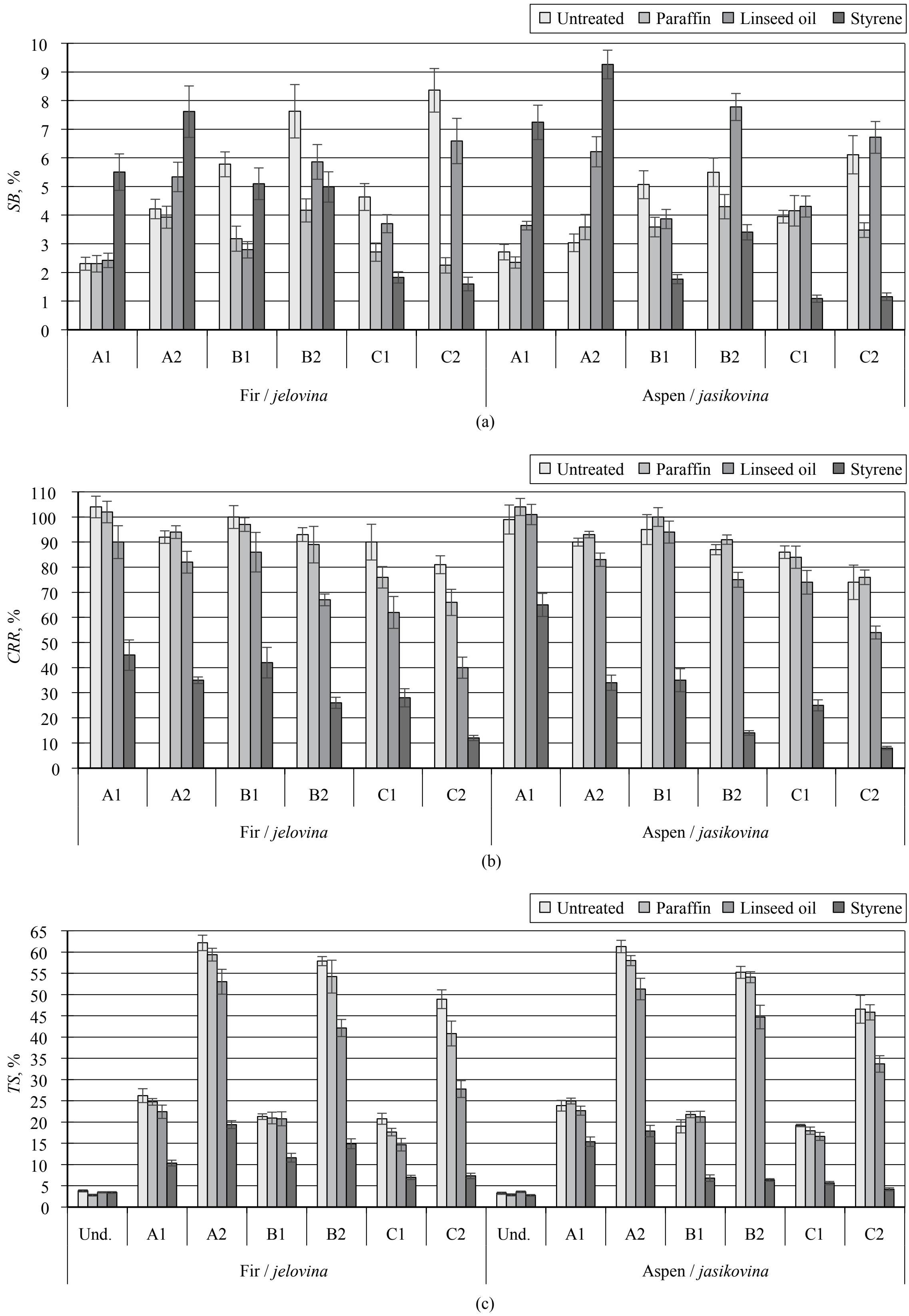


Figure 2 *SB*, *CRR* and *TS* for fir and aspen wood depending on densification conditions
Slika 2. *SB*, *CRR* i *TS* za drvo jele i drvo jasike u ovisnosti o uvjetima ugušćivanja

increase in the compression rate. In the literature, it has been reported that increased internal stress in the material during the wood densification process resulting from increase in the compression rate leads to higher *SB* values (Wolcott *et al.,* 1989; Nairn, 2006; Pelit *et al.,* 2016). In the untreated samples and in those impregnated with linseed oil, SB values increased due to an increase in the compression temperature. However, especially in samples impregnated with styrene, as a result of the higher compression temperature, SB values decreased significantly.

With respect to water repellents, the highest *CRR* average for fir wood was found in untreated samples (93.32 %), and for aspen wood in samples impregnated with paraffin (91.37 %). The lowest *CRR* average was determined in styrene-impregnated samples for both wood species (31.26 % for fir and 30.39 % for aspen) (Table 3). In both species impregnated with water repellent agents (except for the paraffin-impregnated aspen samples), the *CRR* values were reduced (Figure 2b). The *CRR* values in the samples impregnated with linseed oil were lower than in the untreated samples. However, the most significant reduction in *CRR* values was detected in the styrene-treated samples. The *CRR* values of the styrene-impregnated fir and aspen woods were reduced by 85 % and 89 %, respectively, compared to untreated samples densified under the same conditions. With the styrene pretreatment, the compressed thickness of the densified samples exposed to water over a long period was almost completely retained (Figure 2b). As for the results, it can be said that the significantly reduced water absorption of the styrene-treated samples was effective (Figure 3b). Chao and Lee (2003) reported that the polymerized hydrophobic styrene monomer remained in the cell wall and lumen of the impregnated wood and formed a barrier on the wood surface, resulting in less water entering the wood. This greatly reduced the water absorption and dimensional changes of wood. Furthermore, water repellency and dimensional stability have been reported to increase significantly in wood treated with vinyl monomers compared to untreated wood (Rowell, 2012).

Regarding densification conditions, the highest *CRR* average was determined for both tree species in samples densified under *A1* conditions (85.36 % for fir and 92.29 % for aspen) and the lowest for samples densified under *C2* conditions (49.60 % for fir and 53.25 % for aspen) (Table 3). At all temperature levels, *CRR* values were significantly lower in the fir and aspen samples compressed at a higher rate (40 %) (Figure 2b). In the densification process, with the higher compression rate, deformations such as fractures, cracking and collapse of the wood cell wall also increased (Tabarsa and Chui, 1997; Dogu *et al.,* 2010; Budakci *et al.,* 2016; Bekhta *et al.,* 2017). It can be said that the increase in the amount and size of deformation in the densified wood reduced the tendency of the cell to recover its initial shape and this situation had an effect on the *CRR* results. In addition, at the same compression rate (20 % or 40 %), the *CRR* values were lower in the samples which were densified at a higher temperature. The increase in densification temperature had a positive effect on the *CRR* results of both untreated and impregnated samples, especially in those treated with styrene.

Regarding water repellents, the highest *TS* average in the untreated samples was 34.43 % for fir and 32.62 % aspen, while the lowest was obtained in the styrene-impregnated samples with 10.56 % for fir and 8.43 % for aspen (Table 3). It was observed that impregnation with water-repellents did not have much impact on the *TS* values of the undensified samples (Figure 2c). However, the pre-impregnation processes in the densified samples generally reduced the *TS* values. More positive results were obtained in linseed oil-treated samples compared to paraffin-treated samples. Positive results for the *TS* values were mainly determined in the styrene-treated samples. The *TS* values decreased significantly in these samples depending on the compression rate and temperature. In particular, under *C2* conditions, the *TS* values of the densified styrene-pretreated samples were close to those of the control (undensified) samples. The *TS* values of styrene-treated fir and aspen samples were reduced by 85 % and 91 %, respectively, compared to the untreated samples densified under the same conditions. In the literature, it was reported that in agathis wood impregnated before compression with vinyl monomers (styrene and methyl methacrylate), dimensional stability increased due to changes in the cellular structure and chemical components (i.e., cellulose crystallinity, microfibril angle and preferred orientation of fibers) as well as degradation of cellulose (Khalil *et al.,* 2014). In addition, it was reported that polymerization of styrene in wood can result in the grafting of the styrene to cellulose, lignin, and pentosans (Rowell, 2012). Moreover, it was found that water-repellent efficacy and dimensional stability properties improved as a result of in situ polymerization of vinyl monomers filling wood cell voids (Rowell and Konkol, 1987).

With respect to densification conditions, the highest *TS* average was found in densified samples under *A2* conditions (48.49 % for fir and 47.12 % for aspen), whereas the lowest was in undensified samples (3.37 % for fir and 3.11 % for aspen) (Table 3). According to Figure 2c, the *TS* values of all the densified samples were higher than those of the undensified samples. In densified samples, *TS* values increased with the higher compression rate. In general, a higher *TS* was obtained at the compression rate of 40 % than at 20 %. The fact that the densified samples exposed to water tended to return to their initial pre-compression dimensions can be said to affect the results (Seborg *et al.,* 1962; Kollmann *et al.,* 1975; Pelit *et al.,* 2014). As an exception, *TS* values were found to be lower in the styrene-pretreated aspen samples densified under *B2* and *C2* conditions. This can be explained by the fact that the polymerization of the styrene monomer within the densified wood was more effective at high temperatures (150 °C and 180 °C). In terms of compression temperature, the highest *TS* value was determined in the samples densified at 120 °C and the lowest in the samples densified at 180 °C. In the densified fir and aspen samples, the *TS* values were reduced due to the increase in compression temperature.

3.3 Equilibrium moisture content, water absorption and water repellent effectiveness
3.3. Ravnotežni sadržaj vode, upojnost uzoraka i učinkovitost vodoodbojnih sredstava

The effect of water repellent type and densification condition factors on the equilibrium moisture content (*EMC*), water absorption (*WA*), and water repellent effectiveness (*WRE*) for both wood species was statistically significant. Duncan's one-way comparison results conducted for mean values of *EMC*, *WA*, and *WRE* are shown in Table 4.

Regarding water repellents, the highest mean *EMC* was found for untreated samples (10.22 % for fir and 9.77 % for aspen) and the lowest for styrene-impregnated samples (6.63 % for fir and 5.43 % for aspen) (Table 4). The *EMC* decreased in all samples impregnated with water repellents (Figure 3a). A lower *EMC* was determined in the paraffin-treated samples compared to the linseed oil-treated samples. The lowest *EMC* for both wood species was measured in the styrene-treated samples. Due to the increase in the compression rate and temperature, the *EMC* was significantly reduced in the styrene-pretreated wood samples, while the hygroscopicity resistance of the samples increased. In the styrene pre-treated fir and aspen wood densified under *C2* conditions, the *EMC* was reduced by 53 % and 67 %, respectively, relative to untreated samples.

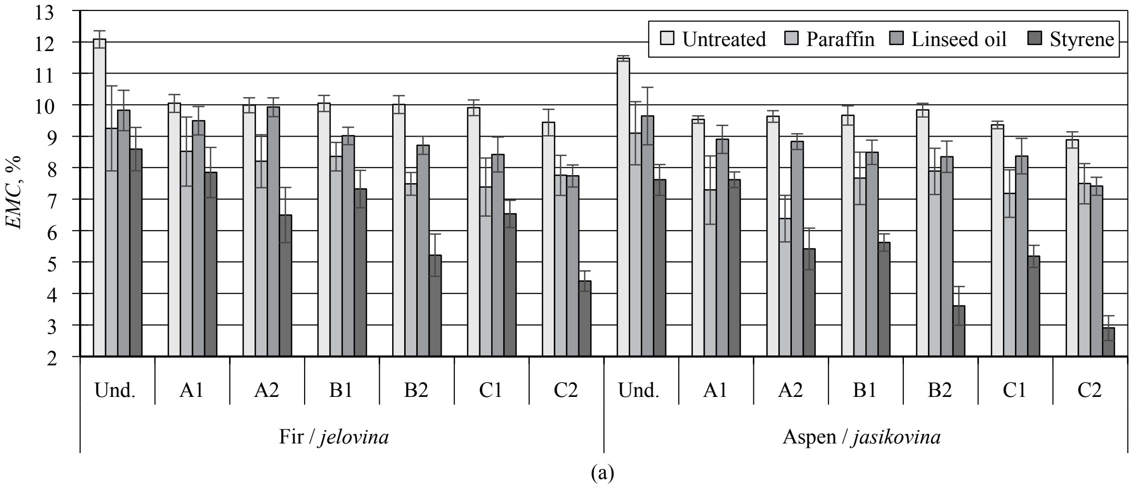
Regarding densification variables, the highest *EMC* average was determined in undensified samples (48.49 % for fir and 47.12 % for aspen), while the lowest was in densified samples under *C2* conditions (3.37 % for fir and 3.11 % for aspen) (Table 4). According to Figure 3a, the *EMC* value of the densified samples was generally lower than that of the undensified samples. The effect of the compression rate on *EMC* was not significant in the paraffin- and linseed-treated samples. However, the *EMC* decreased in the styrene-treated samples due to a higher compression rate. Similarly, the effect of the compression temperature on *EMC* in the paraffin- and linseed oil-treated samples was not apparent, while in the styrene-treated fir and aspen samples, the *EMC* was decreased due to a high compression temperature. The lowest *EMC* was determined in the samples compressed at 180 °C.

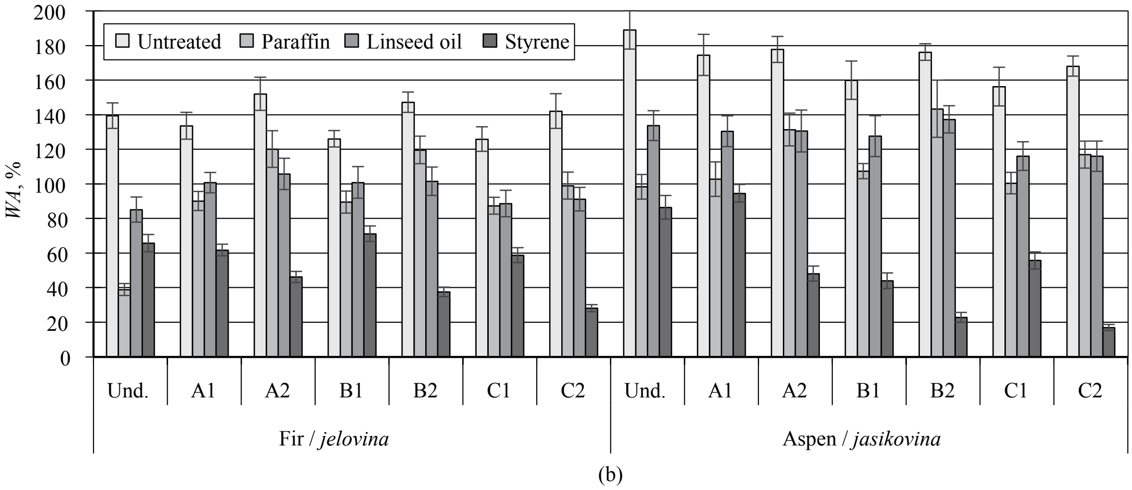
With respect to water repellents, the highest *WA* average was found in untreated samples (137.98 % for fir and 171.61 % for aspen) and the lowest in the styrene-impregnated samples (52.67 % for fir and 52.59 % for aspen) (Table 4). The *WA* was also reduced in both tree species impregnated with water repellents (Figure 3b). Depending on the substances used and their quantities, water repellents fill the cell cavities and are stored on the outer surfaces and partially on the inner surfaces. Thus, the wood surface exhibits hydrophobicity and the water intake rate is reduced (Dizman Tomak and Yıldız, 2012). In general, the *WA* was found to be

Table 4 Duncan's test results for mean values of *EMC*, *WA*, and *WRE*
Tablica 4. Rezultati srednjih vrijednosti za *EMC*, *WA* i *WRE* prema Duncanovu testu

Wood species *Vrsta drva*	**Factor** *Čimbenik*	***EMC*, %**		***WA*, %**		***WRE*, %**	
		Mean	SG	Mean	SG	Mean	SG
Fir wood *drvo jele*	Water repellents / *Vodoodbojna sredstva*						
	Untreated / *netretirano*	10.22	a	137.98	a	-	-
	Paraffin / *parafin*	8.14	c	92.01	c	33.25	b
	Linseed oil / *laneno ulje*	9.02	b	96.17	b	29.94	c
	Styrene / *stiren*	6.63	d	52.67	d	61.07	a
	Densification / *Ugušćivanje*						
	Undensified / *neugušćeno*	9.94	a	82.23	e	54.55	a
	A1	8.98	b	96.46	c	36.82	d
	A2	8.66	c	105.97	a	40.14	cd
	B1	8.69	bc	96.81	c	30.81	e
	B2	7.86	d	101.39	b	41.32	c
	C1	8.06	d	90.09	d	37.69	d
	C2	7.33	e	90.01	d	48.59	b
Aspen wood *drvo jasike*	Water repellents / *Vodoodbojna sredstva*						
	Untreated / *netretirano*	9.77	a	171.61	a	-	-
	Paraffin / *parafin*	7.57	c	114.29	c	33.12	b
	Linseed oil / *laneno ulje*	8.57	b	127.29	b	25.59	c
	Styrene / *stiren*	5.43	d	52.59	d	69.44	a
	Densification / *Ugušćivanje*						
	Undensified / *neugušćeno*	9.46	a	126.77	a	43.76	b
	A1	8.34	b	125.50	ab	37.17	c
	A2	7.57	d	121.87	bc	41.84	b
	B1	7.86	c	109.64	d	41.54	b
	B2	7.42	d	119.87	c	42.58	b
	C1	7.53	d	107.04	de	41.66	b
	C2	6.68	e	104.42	e	50.47	a

SG: statistical group (different letters denote a significant difference). / *SG: statistička grupa (različita slova označavaju značajnu razliku).*





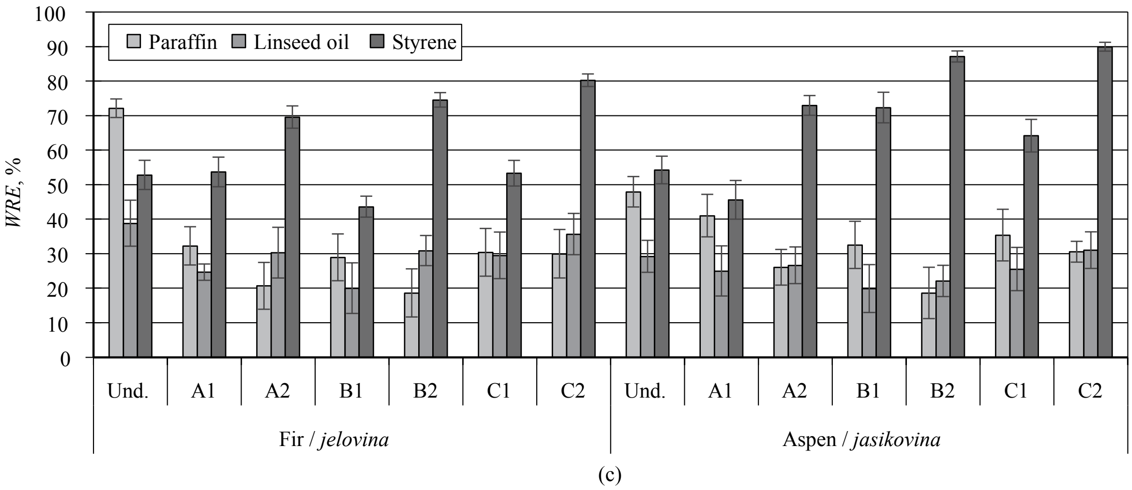


Figure 3 *EMC*, *WA* and *WRE* for fir and aspen wood depending on densification conditions
Slika 3. *EMC*, *WA* i *WRE* za drvo jele i drvo jasike u ovisnosti o uvjetima ugušćivanja

lower in paraffin-treated samples compared to linseed oil-treated samples. However, the most positive results for *WA* were determined in the styrene-treated samples. In these samples, the *WA* was significantly reduced, especially after high temperature (180 °C) and high compression (40 %). Compared to untreated samples, the *WA* decreased 80 % and 90 %, respectively, in styrene pre-treated fir and aspen woods densified under *C2* conditions (180 °C / 40 %). When wood is impregnated with vinyl monomers, the polymer is located almost completely in the lumen of the wood and only a small amount is polymerized in the cell wall. Consequently, the *WA* is significantly reduced in woods with polymers filling the volume of the cavities (Baysal *et al.,* 2007; Rowell, 2012). In previous studies, it was reported that water absorption decreased significantly in styrene-treated wood compared to untreated wood. (Yalinkilic *et al*. 1998; Chao and Lee, 2003; Baysal *et al.,* 2006).

Regarding densification factors, the highest *WA* average for fir wood was found in samples densified under *A2* conditions (105.97%) and for aspen wood in undensified samples (126.77 %). The lowest *WA* average was determined in undensified samples for fir wood (82.23 %) and for aspen wood in samples densified under *C2* conditions (104.42 %) (Table 4). In densified samples (except for styrene-pretreated samples), the *WA* values increased due to a higher compression rate. The *WA* was generally higher at 40 % compression rate than at 20 % (Figure 3b). It can be said that the increase in the amount of water-repellent agents moving away from the wood with the increase of compression rate affect the results. Depending on the amount of water repellent impregnated into wood, hydrophobic properties of wood increase (Koski, 2008). The effect of the compression temperature on the *WA* in the untreated, paraffin-treated and linseed oil-treated samples was not very pronounced. However, in the styrene-treated samples, the *WA* was reduced as a result of a higher compression temperature.

Regarding water repellents, the highest *WRE* average was found in samples impregnated with styrene (61.07 % for fir and 69.44 % for aspen), and the lowest in samples impregnated with linseed oil (52.67 % for fir and 52.59 % for aspen) (Table 4). In samples impregnated with water repellents, the *WRE* was the highest with styrene, followed by paraffin and linseed oil, respectively, compared to the untreated samples. After densification, the *WRE* decreased in the samples with paraffin and linseed oil treatment. On the other hand, depending on the compression rate and temperature, the *WRE* was significantly increased in the styrene-treated samples. After impregnation with styrene, the *WRE* was found to be close to 100 % in the wood samples (especially aspen) densified under *C2* conditions (Figure 3c).

With respect to densification conditions, the highest *WRE* average was obtained in undensified samples for fir wood (54.55 %) and in samples densified under *C2* conditions for aspen wood (50.47 %). The lowest *WRE* average was determined in the samples densified under *B1* conditions for fir wood (30.81 %) and in the samples densified under *A1* conditions for aspen wood (37.17 %) (Table 4). According to Figure 3c, the *WRE* was found to be higher at the 20 % compression rate in the paraffin-treated samples. However, the *WRE* was higher in the linseed- and styrene-treated samples at the 40 % compression rate. The *WRE* increased significantly depending on the compression rate, especially in the styrene-treated samples. On the other hand, while the effect of compression temperature on *WRE* was insignificant in the paraffin- and linseed oil-treated samples, the *WRE* decreased in the styrene-treated samples due to a higher compression temperature.

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigated the effect of pre-impregnation with water-repellent agents on dimensional stability and hygroscopicity in densified wood compressed at different rates and temperatures. Water repellents significantly influenced the physical properties of densified fir and aspen wood. In the densified samples, the *CRR* and *TS* values were generally reduced with impregnation pretreatments. More positive *CRR* and *TS* results were obtained from the linseed oil-treated samples than from those treated with paraffin. However, the most successful results were determined in the styrene-pretreated samples. The *CRR* values of the styrene-treated fir and aspen woods decreased by 85 % and 89 %, respectively, compared to the undensified (untreated) samples under the same conditions. In the same samples, the *TS* values were decreased by 85 % and 91 %, respectively. Thus, the dimensional stability of the styrene pre-treated and densified samples was almost completely achieved depending on the compression conditions. These results can be shown as one of the most important outcomes of the study.

The *EMC* and *WA* were reduced in both wood species impregnated with water repellents. The *EMC* and *WA* were lower in the paraffin-treated samples than in the linseed oil-treated samples, while the lowest values for both wood species were found in the styrene-treated samples. In the densification process, due to the increased compression rate and temperature, the *EMC* and *WA* were significantly reduced in the styrene-pretreated wood samples, whereas the *WRE* of the samples increased. After impregnation with styrene, the *WRE* was close to 100 % in the wood samples (especially aspen) densified under *C2* conditions (180 °C / 40 %).

Regarding densification conditions, the most positive results on the physical properties of the wood samples were obtained at a high temperature (180 °C) and high compression rate (40 %). Due to the increased compression rate and temperature, the hygroscopicity was reduced and the dimensional stability increased in the fir and aspen samples.

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