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Physical and mechanical properties of plywood produced with 1.3-dimethylol-4.5-dihydroxyethyleneurea (DMDHEU)-modified veneers of *Betula sp.* and *Fagus sylvatica*

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Abstract The dimensional stability and some mechanical properties were tested in plywood produced with veneers modified with 1.3-dimethylol-4.5-dihydroxyethyleneurea (DMDHEU). The experimental design included *Betula sp.* and *Fagus sylvatica* impregnated with 0.8 M, 1.3 M, and 2.3 M DMDHEU. The plywood consisted of five veneers glued with a phenolic resin. Dimensional stability tests were conducted after 10 cycles of soaking/oven-drying to determine volume changes and anti swelling efficiency (ASE). The mechanical properties tested were hardness (Brinell), modulus of elasticity in bending (MOE), bending strength (BS) and work to maximum load in bending (WMLB). The modified samples for both species were considerably more dimensionally stable than the untreated samples. The samples of *Betula sp.* and *F. sylvatica* modified with DMDHEU presented a MOE and a BS unaffected by the treatment. The WMLB was consistently lower in the modified samples than in the unmodified samples. As determined by the Brinell method, the DMDHEU-modified plywood of the *Betula sp.* and *F. sylvatica* was harder than the unmodified plywood.

Physikalische und mechanische Eigenschaften von Sperrholz aus mit 1.3-Dimethylol-4.5-Dihydroxyethyleneurea (DMDHEU) modifizierten Birken- und Buchenfurnieren

Zusammenfassung Die Dimensionsstabilität und einige ausgewählte mechanische Festigkeiten wurden an Sperrhölzern getestet, die aus mit 1.3-Dimethylol-4.5-Dihydroxyethyleneurea (DMDHEU) modifizierten Furnieren hergestellt wurden. Es wurden Birken- (*Betula sp.*) und Buchenfurniere (*Fagus sylvatica*) verwendet, welche mit 0,8 M, 1,3 M sowie 2,3 M DMDHEU Lösung imprägniert wurden. Die aus 5 Lagen bestehenden Sperrhölzer wurden mit einem Phenolharz verklebt. Zur Untersuchung der Dimensionsstabilität wurden die Prüfkörper über 10 Zyklen wassergesättigt und anschließend bei 103 °C getrocknet. Aus den Volumenänderungen wurde anschließend die Quellungsvergütung (ASE) bestimmt. Des Weiteren wurden die Härte nach BRINELL, der Elastizitätsmodul während der Biegeprüfung (MOE), die Biegefestigkeit (BS) sowie die Biegearbeit (WMLB) bestimmt. Die Prüfkörper aus den modifizierten Sperrhölzern beider Holzarten zeigten eine höhere Dimensionsstabilität als die Prüfkörper aus unmodifizierten Sperrhölzern. Der Elastizitätsmodul und die Biegefestigkeit wurden durch die Modifizierung nicht beeinflusst. Die Biegearbeit wurde durch die Modifizierung verringert, die Härte erhöht.

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1 Introduction

The chemical modification of wood has been significantly studied in the last decades, but only recently the increasing demand for renewable materials has allowed this research to be put into commercial use (Hill 2006). 1.3-dimethylol-4.5-dihydroxyethyleneurea (DMDHEU) is a resin originally used in the textile industry, but the technology has successfully been transferred to wood processing (Krause et al. 2003, Schaffert et al. 2005, Wepner and Militz 2005, Hill 2006). DMDHEU-modified veneers are already used to produce plywood in Germany (Becker 2005). It is known that DMDHEU improves the durability and the dimensional stability of solid wood (Nicholas and Williams 1987, Militz 1993, Krause et al. 2003, Schaffert et al. 2005, Hill 2006), veneers (Wepner and Militz 2005) and plywood (Wepner et al. 2006). Nevertheless, it has been reported that the treatment with DMDHEU reduces the bending strength of solid wood, due to the acid degradation of the cell wall (Nicholas and Williams 1987, Hill 2006). A reduction of the tensile strength of veneers, determined by a thin-veneer strip technique, is attributed to the modification with DMDHEU, explained by 1) the acidic hydrolysis of the polysaccharides as a result of the use of magnesium chloride as catalyst and 2) by the movement limitation of the cell wall due to the cross-linking with DMDHEU (Mai et al. 2007, Xie et al. 2007). Similar causes are reported to affect the mechanical properties of solid wood treated with formaldehyde in a phenomenon described as embrittlement (Rowell 1996).

The objective of this work was to investigate the effect of a modification with DMDHEU on the physical and mechanical properties of plywood.

2 Materials and methods

Rotary-cut *Betula sp*. and *Fagus sylvatica* veneers were impregnated in an autoclave at 80 mbar for 30 min and at 12 bar for 2 h with solutions of DMDHEU in the following concentrations: 0.8 M, 1.3 M, and 2.3 M, using 4% magnesium nitrate relative to the mass of DMDHEU of the solution as catalyst. After impregnation, the solution uptake was determined. Then, the veneers were dried in an oven to approximately 10% moisture content prior to curing in a hot press at 160 °C and 1 Nmm^{-2} for 10 min. The modified veneers were used to produce hot-pressed 5-layer plywood glued with 150 gm^{-2} of a phenolic resin (Prefere 4976). The press parameters were 140 °C and 1 Nmm^{-2} for 10 min.

2.1 Dimensional tests

The dimensional tests were performed according to EN 317 (1993a) and following previous work on soak/oven-dry cycling test (Hill and Jones 1996). Five 7.5 × 250 × 250 mm^3 *Betula sp*. and *F. sylvatica* plywood samples were constructed each with veneers untreated (control) and treated with each concentration, and from these eight 50 × 50 mm^2 samples were obtained, resulting in 160 samples for each species (total number of samples = 3 DMDHEU concentrations and 1 control × 5 plywood × 8 samples = 160). The samples were cyclically immersed in water and dried to 0% MC. The samples were saturated for 1 h with water at 80 mbar, and then stored under water for 16 h. After each cycle, the mass, thickness, length (in the grain direction), and width (direction perpendicular to the grain) of the samples were determined. A complete cycle ended when the samples were anhydrous. The samples were oven-dried at 50 °C for 24 h, then at 80 °C for 24 h, and finally at 103 °C for 24 h. After drying, the samples were cooled down to room temperature in a dessicator containing silica gel. The cycle was repeated ten times. The data was used to calculate swelling, anti-swelling efficiency in thickness and weight percent gain.

The thickness swelling coefficient values (*S*) were calculated using the following equation,

$$S = \frac{T_\mathrm{w} - T_\mathrm{d}}{T_\mathrm{d}} \times 100 \quad [\%] \tag{1}$$

where S is the thickness swelling, T_d is the dry thickness, and T_w is the wet thickness.

The ASE in thickness was calculated using the following equation

$$\mathrm{ASE} = \frac{S_\mathrm{u} - S}{S_\mathrm{u}} \times 100 \quad [\%] \tag{2}$$

where ASE is the anti-swelling efficiency in thickness, S_u is the thickness swelling of untreated wood and S is the thickness swelling of treated wood.

The weight percentage gain (WPG) of the plywood during the test was calculated using the following equation

$$\mathrm{WPG} = \frac{M_\mathrm{m} - M_\mathrm{u}}{M_\mathrm{u}} \times 100 \quad [\%] \tag{3}$$

where M_m is the oven dry mass of the modified samples at the end of every cycle and M_u is the oven dry mass of the unmodified samples (Hill 2006).

2.2 Mechanical tests

The determination of hardness was done according to EN 1534 (2000). Samples of 50 × 50 × (plywood thickness) mm^3 were conditioned to constant weight at a relative humidity of $65 \pm 5\%$ and at a temperature of 20 ± 2 °C. A metal sphere of diameter 10 mm was forced into the sample until a maximum force of 1000 N was achieved. Then, after the metal sphere was retired, the material was allowed to spring back, and the depth of penetration was measured to calculate the hardness according to Eq. 4:

$$\mathrm{BH} = \frac{F}{2\pi r h} \quad \left[\mathrm{Nmm}^{-2}\right] \tag{4}$$

where BH is the Brinell hardness (Nmm^{-2}); h is the depth of penetration (mm); r is the radius of the metal sphere (mm); and F is the force (N).

The determination of MOE, bending strength and WMLB were performed according to EN 310 (1993b). Two types of samples of $7.5 \times 50 \times 200\,\text{mm}^3$ were sawn from the plywood: samples with the grain of the upper layer parallel to the longitudinal axis and samples with the grain of the upper layer perpendicular to the longitudinal axis. The samples were conditioned to constant weight at a relative humidity of $65 \pm 5\%$ and at a temperature of $20 \pm 2\,°\text{C}$.

The statistical model utilized to test the effect of the experimental treatments on the mechanical properties was a factorial design presented in Eq. 5

$$y_{ijk} = \mu + \tau_i + \beta_j + (\tau\beta)_{ij} + \zeta_k + \varepsilon_{ijkl} \quad (5)$$

where y_{ijk} is the estimation for each variable; μ is the overall mean effect; τ_i is the effect of the species; β_j is the effect of the DMDHEU concentration; $(\tau\beta)_{ij}$ is the effect of the interaction between species and DMDHEU concentration, ζ_k is the orientation of the first layer, and ε_{ijk} is a random error component (Sokal and Rohlf 1995).

The analysis performed was a factorial ANOVA and the comparison of means of the hardness (Brinell) was performed with the Tukey's Studentized Range at the 0.05 level.

3 Results

3.1 Dimensional tests

The plywood made with DMDHEU-modified veneers was more dimensionally stable than the plywood constructed with unmodified veneers. The results are summarized in Figs. 1, 2, and 3. Furthermore, the correlation between ASE in thickness and WPG after the first cycle was studied for each species at different concentrations of DMDHEU. The correlation proved to be high for *Betula sp.*, meaning that a higher WPG rendered a higher ASE in thickness. The correlation between the two variables was lower for *F. sylvatica*, but still showed a similar trend to *Betula sp*. The results for the first soaking and drying cycle are presented in Table 1.

Betula sp. presented the highest swelling in thickness, both for treated and untreated samples. The untreated samples swelled approximately twice as much as the treated samples. A higher concentration of DMDHEU did not imply a higher weight loss during the cycling, measured as decline of WPG. On the contrary, the weight loss relative to the original WPG was lowest for the higher concentrations, namely DMDHEU 2.3 M 14%, DMDHEU 1.3 M 17% and

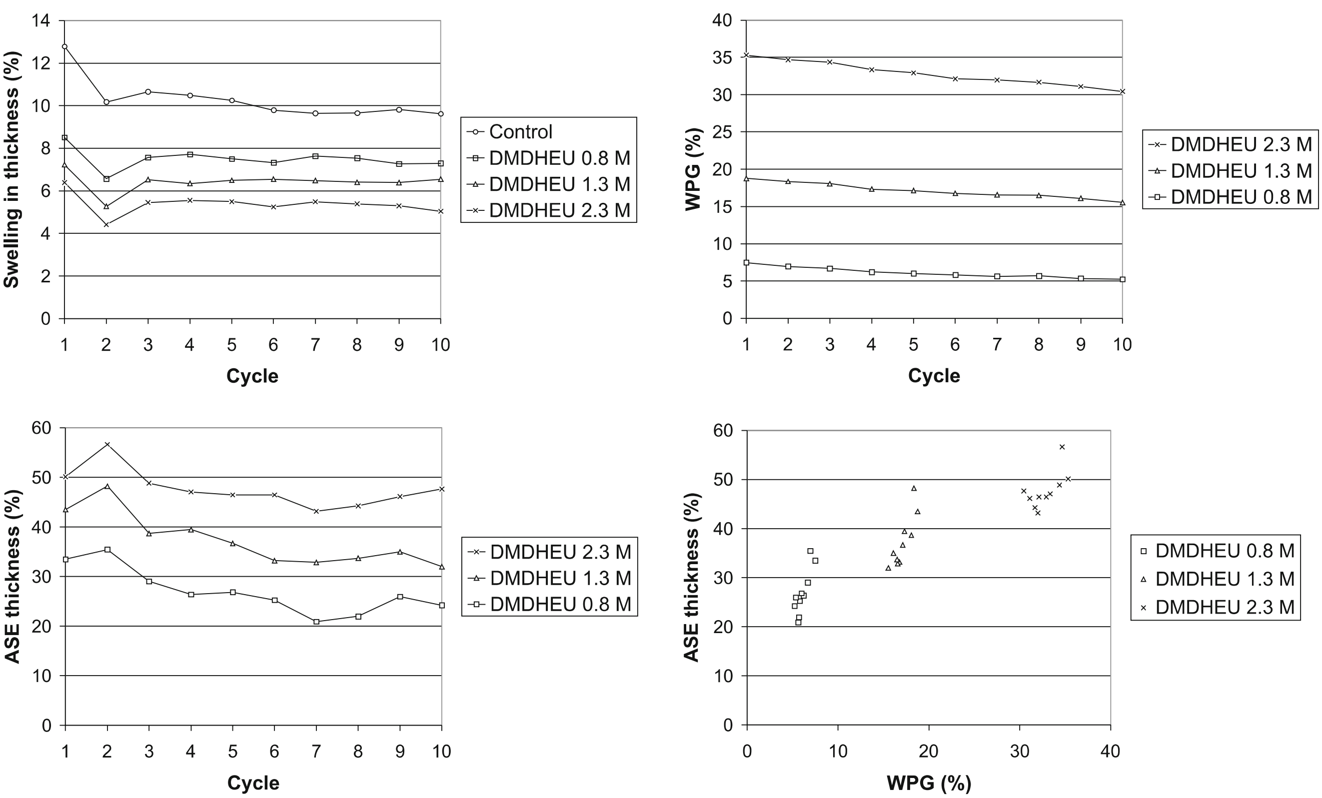

Fig. 1 Thickness swelling, WPG and ASE in thickness of *Betula sp.* plywood treated with different DMDHEU concentrations (averaged over 10 cycles of soaking and drying)

Abb. 1 Dickenquellung, WPG und ASE von Prüfkörpern, die aus behandelten sowie unbehandelten Sperrhölzern (*Betula sp.*) hergestellt wurden

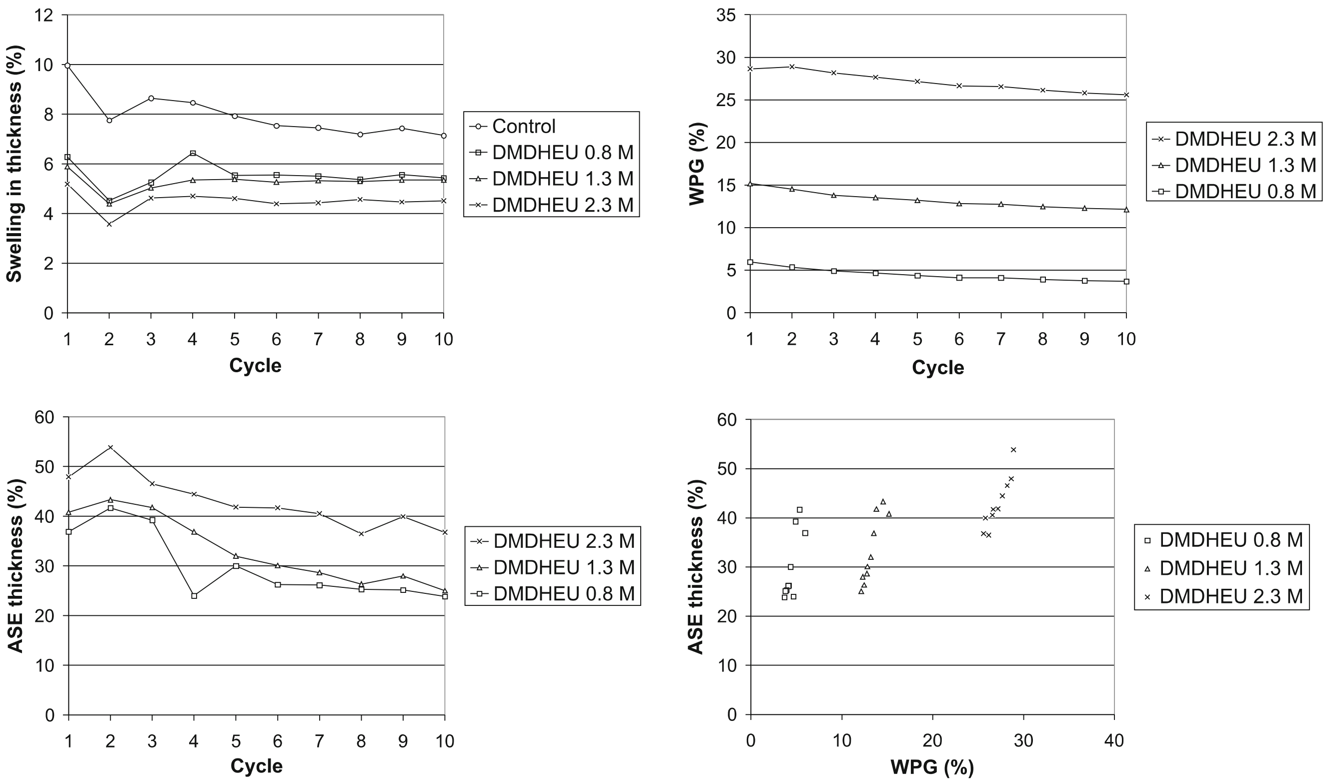

Fig. 2 Thickness swelling, ASE in thickness and WPG of *F. sylvatica* plywood treated with different DMDHEU concentrations (averaged over 10 cycles of soaking and drying)

Abb. 2 Dickenquellung, WPG und ASE von Prüfkörpern, die aus behandelten sowie unbehandelten Sperrhölzern (*F. sylvatica*.) hergestellt wurden

Table 1 Correlation between ASE thickness and WPG for the DMDHEU-modified plywood of *Betula sp.*, and *F. sylvatica* after the first cycle

Tabelle 1 Korrelation zwischen Vergütung der Dickenquellung (ASE) und der Gewichtszunahme (WPG) des DMDHEU-modifizierten Sperrholzes aus *Betula sp.* und *F. sylvatica* nach einem Zyklus

Species	DMDHEU concentration	*n*	ASE thickness (%)	WPG (%)	*r*
Betula sp.	0.8 M	40	33.48	7.48	0.841
	1.3 M	40	43.49	18.76	
	2.3 M	40	50.10	35.32	
F. sylvatica	0.8 M	40	36.92	5.97	0.559
	1.3 M	40	40.81	15.18	
	2.3 M	40	47.95	28.64	

DMDHEU 0.8 M 30%. The results are presented in the WPG diagram of Fig. 1. The thickness ASE was positively affected by the DMDHEU concentration: higher DMDHEU concentration implied higher ASE. A short peak was observed at the second cycle, but during the following cycles the ASE did not show a large variation. The samples treated with a DMDHEU concentration of 0.8 M, 1.3 M and 2.3 M presented a thickness ASE that oscillated around 25%, 35% and 45%, respectively. The plotting of the WPG versus the ASE for each of the 10 cycles showed a clear correlation between both variables, shown by the correlation coefficients 0.852, 0.872, and 0.649 for 0.8 M, 1.3 M and 2.3, respectively. This phenomenon implied that higher concentrations of DMDHEU rendered higher ASE in thickness. The data is presented in Fig. 1.

The treated samples constructed with *F. sylvatica* presented a lower thickness swelling than in *Betula sp*. The untreated samples swelled in thickness twice as much as the treated samples, but did not present a large variation for the different DMDHEU concentration, ranging from 5 to 6%. As expected, higher concentrations of DMDHEU implied higher WPG, but the WPG was lower than in *Betula sp.* During the cycling, there was a constant decline in WPG comparable to the one observed for *Betula sp*. Thus, due to the fixation of the chemical, a higher concentration of DMDHEU did not render a higher weight loss during the cycling. The values of ASE in thickness generally oscillated between 20 and 50%. The correlation coefficients between WPG and ASE in thickness calculated for each cycle were similar to those observed for *Betula sp*: 0.817, 0.922, and 0.914 for 0.8 M, 1.3 M and 2.3, respectively. Therefore, higher WPG implied higher ASE. It was observed that during the cycling the ASE for *F. sylvatica* was not as stable as for *Betula sp*.

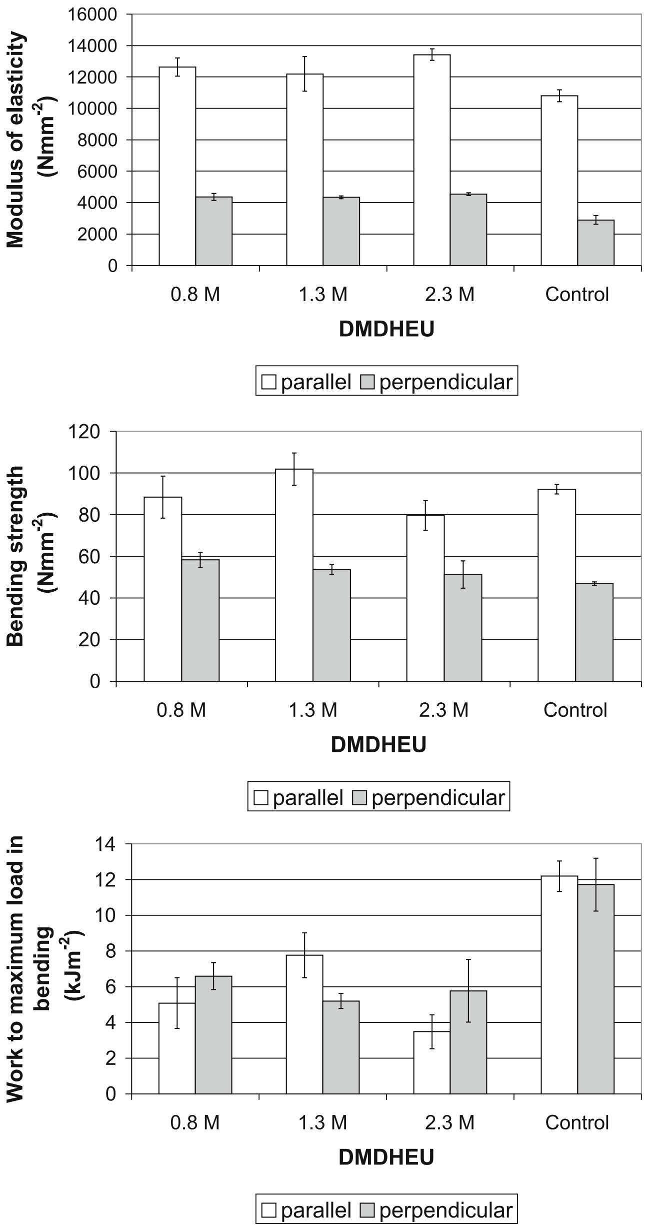

Fig. 3 Determination of modulus of elasticity, bending strength and work to maximum load in bending for DMDHEU-modified plywood of *Betula sp.* according to the grain orientation of the first layer. The error bars represent the confidence with $\alpha = 0.05$

Abb. 3 Elastizitätsmodul, Biegefestigkeit, Biegearbeit von modifizierten und unmodifizierten Sperrhölzern, hergestellt aus *Betula sp.* Der Fehlerindikator zeigt das 95%ige Konfidenzintervall

The results showed that DMDHEU has penetrated the cell wall and that it remained fixed, even after leaching. The mechanism of fixation, whether cross-linking with the cell wall, condensation with NH groups, or condensation with OH groups, are not evident from these results (Krause et al. 2008). An improvement of ASE was observed, even at low levels of DMDHEU treatment, but a higher WPG produced relatively minor changes in ASE (Fig. 2).

Table 2 Determination of Brinell hardness (maximal force 1000 N) for 5-layer plywood constructed with DMDHEU-modified veneers of *Betula sp.* and *F. sylvatica*. Same letter indicates no significant differences ($\alpha = 0.05$)

Tabelle 2 BRINELL-Härte (maximale Kraft 1000 N) von behandelten sowie unbehandelten Sperrhölzern aus *Betula sp.* und *Fagus sylvatica.* Ein signifikanter Unterschied wird durch verschiedene Buchstaben ausgedrückt

Species	DMDHEU concentration	*n*	Hardness Nmm^2	
Betula sp.	Control	40	29.74 ± 0.97	a
	0.8 M	40	47.88 ± 1.58	b
	1.3 M	40	43.72 ± 1.55	c
	2.3 M	40	61.99 ± 2.51	d
F. sylvatica	Control	70	35.27 ± 1.98	a
	0.8 M	40	51.51 ± 0.89	b
	1.3 M	40	50.56 ± 2.65	b
	2.3 M	40	61.89 ± 2.48	c

3.2 Determination of hardness (Brinell)

The Brinell hardness of unmodified early-wood of *Betula pendula* and *Betula pubescens* is reported at 23.37 and 20.53 Nmm^2, respectively (Heräjärvi 2001), which is lower than the results of this work.

The modification with DMDHEU improved the hardness of the plywood. The higher concentration of DMDHEU, namely 2.3 M, rendered the harder plywood, as presented in Table 2.

3.3 Determination of MOE, bending strength and WMLB

The model proposed in Eq. 5 acceptably explained that the experimental factors considered had an effect on the dependent variables. According to the coefficient of determination, MOE was the variable to which the model best adapted ($R^2 = 0.920$). As it has been previously reported (Wilcox et al. 1991), the orientation of the first layer was critical for the three dependent variables studied (Table 3).

It was observed that the values of both MOE and bending strength were higher when the longitudinal axis of the sample was parallel to the grain. Thus, three out of five layers were stiffer, rendering higher values of modulus of elasticity and bending strength as shown in Figs. 3 and 4. It is reported that for 5-layer beech plywood the difference for MOE, bending strength and work to maximum load due to the orientation of the grain is significant (Hrazsky and Kral 2005), while it is rendered to be insignificant when the studied plywood is constructed with 9 layers (Sonderegger and Niemz 2006).

The results of MOE for the untreated wood were in agreement with previous work done on 5-layer beech plywood oriented along the grain, namely a MOE of 9369 Nmm^{-2} (Hrazsky and Kral 2005). No data was found in the litera-

Table 3 General linear model analysis using factorial ANOVA of the dependent variables MOE, bending strength and WMLB considering the experimental factors presented in Eq. 4
Tabelle 3 Varianzanalyse (ANOVA) von MOE, Biegefestigkeit und Biegearbeit in Abhängigkeit von den Faktoren aus Gl. 4

Experimental factors	MOE		Bending strength		WMLB	
	F value	*Pr* > *F*	*F* value	*Pr* > *F*	*F* value	*Pr* > *F*
Species (τ_i)	57.31	< 0.0001	10.16	0.0016	3.80	0.0526
DMDHEU conc. (β_j)	35.47	< 0.0001	6.00	0.0005	152.14	< 0.0001
Interaction ($\tau\beta_{ij}$)	2.97	0.0319	10.93	< 0.0001	6.81	0.0002
Orientation of the 1st layer (ζ_k)	3401.67	< 0.0001	976.49	< 0.0001	15.39	0.0001
n	322		334		224	
R^2	0.920		0.759		0.702	

ture for 5-layer birch plywood. In this work, the plywood modified with DMDHEU presented a modulus of elasticity in bending that was higher or equal to that of the unmodified plywood.

The unmodified samples presented a bending strength of 92.11 Nmm^{-2} and 87.46 Nmm^{-2} for *Betula sp*. and *F. sylvatica*, respectively. The bending strength of 5-layer beech plywood is reported to be 78.65 Nmm^{-2} (Hrazsky and Kral 2005). The modification with DMDHEU presented no significant reduction of bending strength, which is composed by the compression stress and the tensile stress, among other factors such as density and moisture content (Lohmann 1987, Xie et al. 2007). The compression strength is likely to increase due to the increase in surface hardness observed in DMDHEU-treated plywood (Table 2), while on the contrary, the tensile strength is expected to be reduced, as a consequence of the acidic hydrolysis of the hemicelluloses (Xie et al. 2007). Therefore, there was an equilibration of both effects that rendered bending strength barely affected by the modification with DMDHEU. It has been reported that samples of ponderosa pine treated with DMDHEU and $AlCl_3$ as catalyst presented a reduction in bending strength of 38% when compared with unmodified samples, while a similar reduction was observed when the samples were only treated with the catalyst (Nicholas and Williams 1987). Such negative effects on bending strength were not found in this work. Considering the orientation parallel to the grain, the bending strength values of the modified samples were similar to those of the unmodified samples, with the exception of 2.3 M and 1.3 M in *Betula sp*. and *F. sylvatica*, respectively.

Modified wood is more fragile, because the cross-linking between the cell wall and the chemical reduces the mobility of the cell wall components (Xie et al. 2007). As expected, it was observed in this work that the modification with DMDHEU reduces the WMLB, which measures the capacity of wood to absorb energy until its fracture (i.e. shock resistance) (Winandy and Rowell 2005). The reduction of WMLB for the DMDHEU-modified plywood versus the unmodified plywood represented 29–64% and 39–67% for *Betula sp*. and *F. sylvatica*, respectively. The results are presented in Figs. 3 and 4.

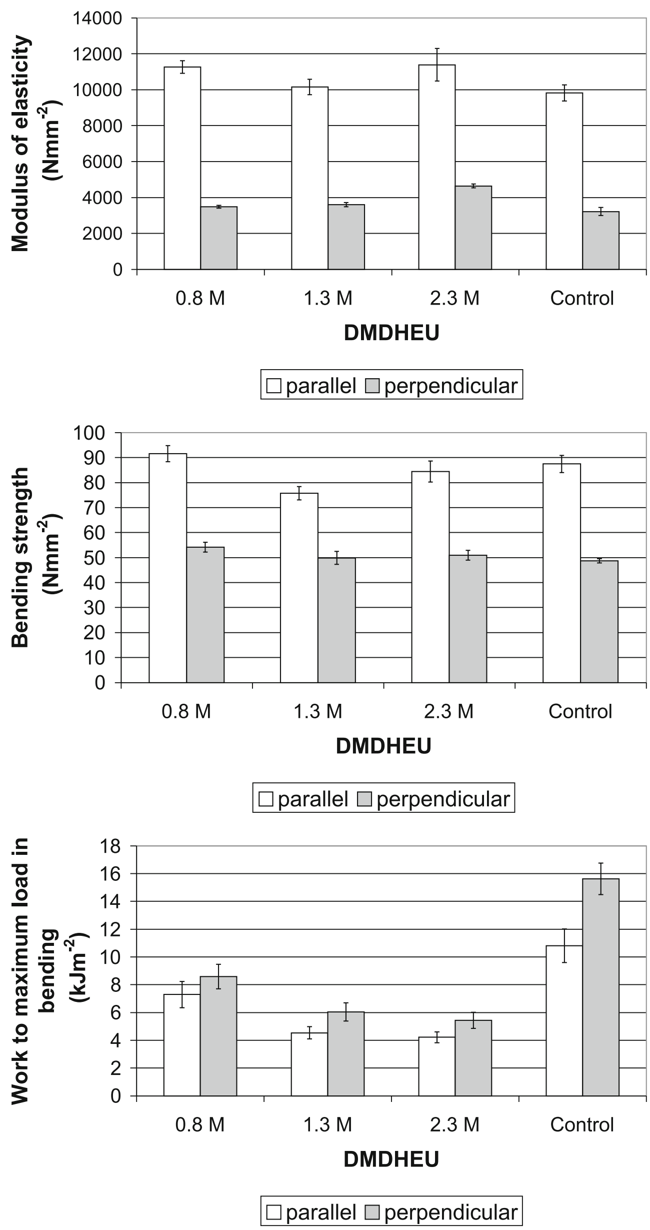

Fig. 4 Determination of modulus of elasticity, bending strength and work to maximum load in bending for DMDHEU-modified plywood of *F. sylvatica* according to the grain orientation of the first layer. The error bars represent the confidence with $\alpha = 0.05$
Abb. 4 Elastizitätsmodul, Biegefestigkeit, Biegearbeit von modifizierten und unmodifizierten Sperrhölzern, hergestellt aus *F. sylvatica*. Der Fehlerindikator zeigt das 95%ige Konfidenzintervall

4 Conclusions

The modification with DMDHEU improved the dimensional stability of plywood produced with *Betula sp.* and *F. sylvatica*. Higher concentrations of DMDHEU rendered higher dimensional stability. The plywood of both species presented a Brinell hardness that was always higher in the modified samples. The modulus of elasticity and the bending strength of *Betula sp*. and *F. sylvatica* plywood were not negatively affected by the modification with DMDHEU. For both species, the WMLB was always higher in the unmodified samples, meaning that unmodified samples were able to absorb more energy than the modified samples. A higher DMDHEU resulted in harder plywood. The modification with DMDHEU rendered plywood with an overall positive effect on dimensional stability and hardness, a neutral or favourable effect on MOE and bending strength, and a negative effect on the ability of the product to absorb impact energy.

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