

PRACE NAUKOWE - RESEARCH PAPERS

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THE INFLUENCE OF DRYING ON THE SORPTION PROPERTIES OF POLYETHYLENE WAX TREATED WOOD

*The importance of the use of waxes in the wood industry has been increasing, particularly in Europe, since consumers, due to their increased environmental awareness, avoid using biocidally treated wood and wood from tropical forests. In this research two water-repellent emulsions in various concentrations were used: polyethylene and oxidised polyethylene wax emulsion. The performance of wax treated Norway spruce (*Picea abies*) and beech (*Fagus sylvatica*) wood was tested in two sorption experiments: conditioning in high relative air humidity (vapour diffusion) and non-continuous dipping (liquid flow). The sorption properties of oven and vacuum dried impregnated specimens were determined. During conditioning, wax treated Norway spruce specimens, vacuum and oven dried, had the same moisture content as control specimens; while treated vacuum dried beech had up to 25% lower moisture content than the parallel control specimens. An even higher difference was evident in volume changes of vacuum and oven dried beech specimens during the sorption test.*

Keywords: beech, Norway spruce, polyethylene wax, sorption, wood preservation, water repellents

Introduction

Wood is the most important biopolymer in the world. It is also the most important natural material used for construction applications. As a natural material, it is exposed to weathering and biotic decay. In order to use wood in outdoor applica-

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tions, it has to be protected somehow. In the past, most preservative solutions had biocidal properties and therefore inhibited pest growth [Richardson 1993]. Future solutions improving durability of wood preservatives are designed differently. They change the structure of wood so that wood pests do not recognise it as a food source [Tjeerdsma et al. 1998]; alternatively, wood moisture content is kept at such a low level that decay processes are no longer possible [Goethals, Stevens 1994]. The treatment of wood with water repellents can affect the long-term properties of this material. Impregnation of wood with resins, waxes, silicones, coatings and other water-repellent formulations greatly reduces the rate of water flow in the capillaries and significantly increases dimensional stability of specimens exposed to wet conditions [Berninghausen et al. 2006; Kurt et al. 2008]. The most important applications of waxes in the wood industry are therefore found in particleboard production. Paraffin emulsions are introduced into particleboards in order to reduce water uptake and improve dimensional stability [Amthor 1972]. However, there are reports that paraffin treatment can also reduce the water capillary uptake in wood [Scholz et al. 2009]. Furthermore, wax treated wood demonstrates increased compression strength and hardness [Rapp et al. 2005]. In addition, wax and oil emulsion additives are incorporated into aqueous wood preservatives to reduce checking and improve the appearance of treated wood used outdoors [Evans et al. 2009]. However, paraffin, montan wax or synthetic waxes do not react with wood. They either form thin films on the surface of wood or on the surface of cell walls, or they fill the cell lumina with waxes and thus limit water penetration into wood. Since they are insoluble in water, they do not leach from wood [Berninghausen et al. 2006].

The importance of the use of waxes in the wood protection industry has been increasing, particularly in Europe, since consumers, due to their increased environmental awareness, avoid biocidally treated wood and wood from non-sustainable tropical forests. The industry is therefore interested in development of alternatives, such as treatment with waxes. There are at least two commercially used wax treatments of wood (Dauerholz in Germany and Natwood in Austria). Nowadays, synthetic waxes, which are used in various coatings, have been becoming more and more important in the wood industry. They have some advantages over natural waxes: they are cheaper and their properties (melting point) can be set during production [Wolfmeier 2003]. Among the most promising waxes for potential applications in the field of wood protection are polyethylene waxes, which considerably slow down fungal degradation of impregnated Norway spruce or beech wood [Lesar, Humar 2010]. They also can be used as water repellents, since they decrease water uptake up to 5 times in a short submersion test. However, it was indicated in previous preliminary research that heating or curing wax treated wood affected the wood sorption properties. The present research was conducted in order to elucidate the effect of heating of wax emulsion treated wood above the melting point on the sorption properties of the treated wood.

Material and methods

Samples (2.0 cm × 2.0 cm × 5 cm) made of Norway spruce (*Picea abies*) and beech (*Fagus sylvatica*) were vacuum/pressure impregnated with various preservative solutions according to the full cell process (20 min -0.9 bar vacuum, 1.5 hour pressure 8 bar, 10 min -0.8 bar vacuum). All specimens had end-sealed (Epoxy coating, EPOLOR, Color) axial surfaces before impregnation. However, sealing needed to be reapplied after the treatment, since some cracks formed. After impregnation, the retention of the preservative solution was determined gravimetrically. The preservative solutions used consisted of aqueous emulsion of polyethylene (WE1) and aqueous emulsion of oxidised polyethylene (WE6) wax. Wax emulsions were purchased from BASF (Germany). The concentrations (dry content) of the wax emulsions are given in table 1. Wax emulsions of two different concentrations were used for impregnation: WE1-A and WE6-A emulsions contained 25% of the original emulsions; while WE1-B and WE6-B emulsions contained 50% of the original emulsions. Control specimens were left un-impregnated and also had end-sealed axial surfaces. After three weeks of conditioning at room temperature, half of the impregnated and half of the un-impregnated specimens were oven dried for 21 hours at $103 \pm 2^\circ\text{C}$ and 3 hours above the melting point of the waxes used ($140 \pm 2^\circ\text{C}$) [Anonymous 2004a; Anonymous 2004b]. Specimens were cured for 3 hours, which ensured that even the centre of the specimens was heated above 135°C for one hour, as determined by a temperature sensor (EL-USB-TC Lascar Electronics, United Kingdom). The other half of the specimens were vacuum dried (-0.75 bar) at 60°C in a vacuum chamber (Kambič, Slovenia) for 24 hours. The samples were then conditioned in room conditions (21°C and 65% RH) for two weeks.

Table 1. Dry content of waxes used and their retention in Norway spruce and beech wood after vacuum pressure impregnation

Tabela 1. Sucha masa zastosowanych wosków i ich retencja w drewnie świerka pospolitego i buka po impregnacji próżniowej

Wax emulsion <i>Emulsja woskowa</i>	Conc. (%) <i>Stężenie (%)</i>	Dry content % <i>Sucha masa %</i>	Wood <i>Drewno</i>	Retention kg/m ³ <i>Retencja kg/m³</i>
WE1-A	25	8.2	Spruce <i>Świerk</i>	197 (67)
WE1-B	50	16.5		138 (56)
WE6-A	25	8.3		189 (35)
WE6-B	50	17.8		135 (27)
WE1-A	25	8.2	Beech <i>Buk</i>	689 (24)
WE1-B	50	16.5		641 (78)
WE6-A	25	8.3		683 (31)
WE6-B	50	17.8		679 (40)

In order to determine the sorption properties, two types of tests were performed: the vapour diffusion test and the liquid penetration test. In order to elucidate vapour diffusion, half of the oven dried and half of the vacuum dried specimens were transferred to a chamber with a relative air humidity (RH) of 87%. The masses and dimensions of the specimens were monitored for 41 days according to predetermined periods (fig. 1, 2).

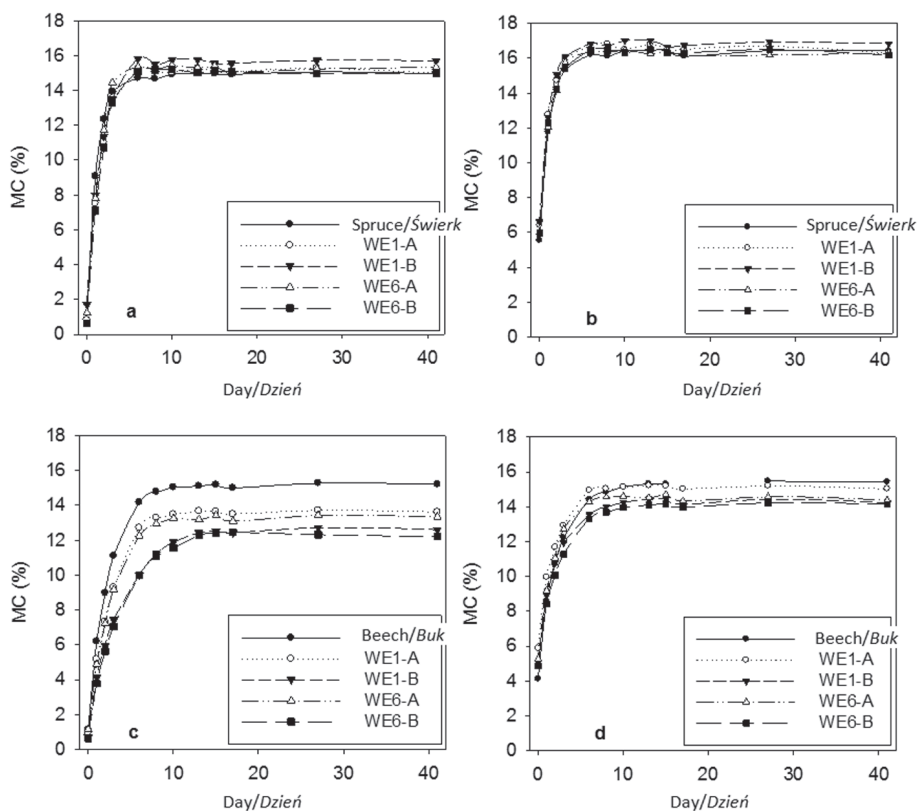


Fig. 1. Moisture content during moisturising in 87 % humidity environment of polyethylene wax emulsion treated: (a) vacuum dried spruce, (b) spruce cured at 140 °C, (c) vacuum dried beech, and (d) beech cured at 140 °C

Rys. 1. Wilgotność podczas nawilżania przy 87% wilgotności otoczenia impregnowanego emulsją wosku polietylenowego: (a) świerka suszonego próżniowo, (b) świerka utwardzanego w temp. 140 °C, (c) buka suszonego próżniowo i (d) buka utwardzanego w temp. 140 °C

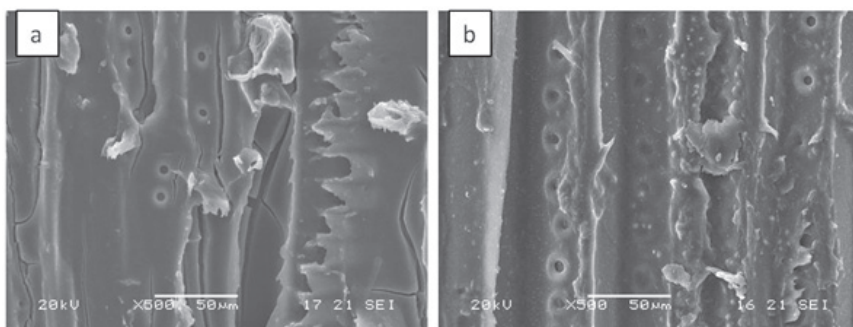


Fig. 2. SEM images of the surface of un-cured (a) and cured (b) WE6-B impregnated Norway spruce specimens

Rys. 2. Obraz SEM powierzchni próbek świerka pospolitego impregnowanych WE6-B nieutwardzonych (a) i utwardzonych (b)

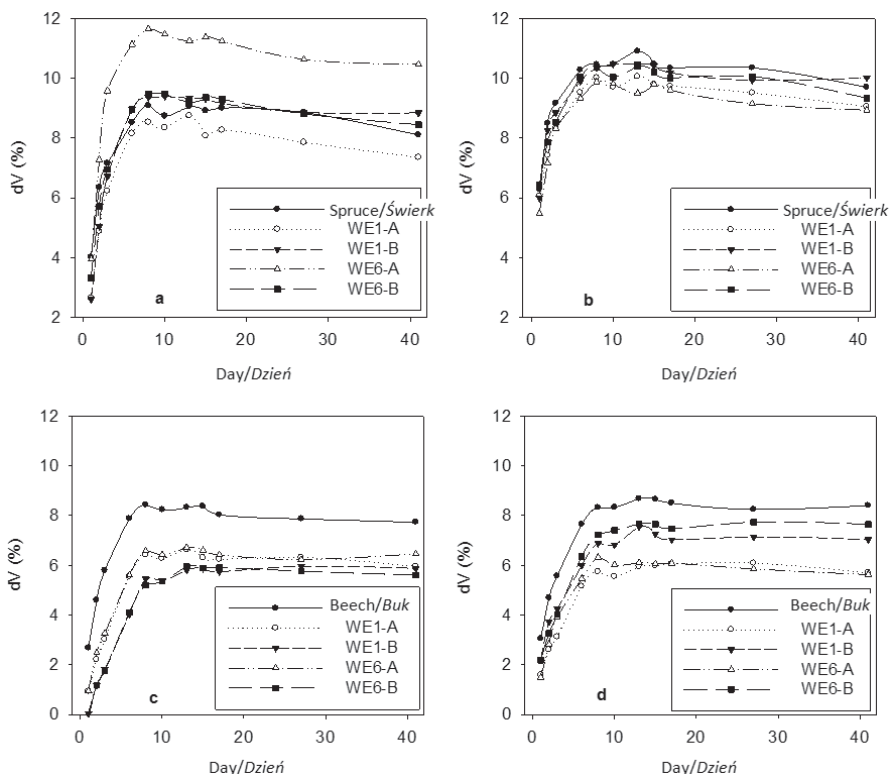


Fig. 3. Volume changes during moisturising in 87% humidity environment of polyethylene wax emulsion treated: (a) vacuum dried spruce, (b) spruce cured at 140 °C, (c) vacuum dried beech, and (d) beech cured at 140 °C

Rys. 3. Zmiany objętości podczas nawilżania przy 87% wilgotności otoczenia impregnowanego emulsją wosku polietylenowego: (a) świerka suszonego próżniowo, (b) świerka utwardzanego w temp. 140 °C, (c) buka suszonego próżniowo i (d) buka utwardzanego w temp. 140 °C

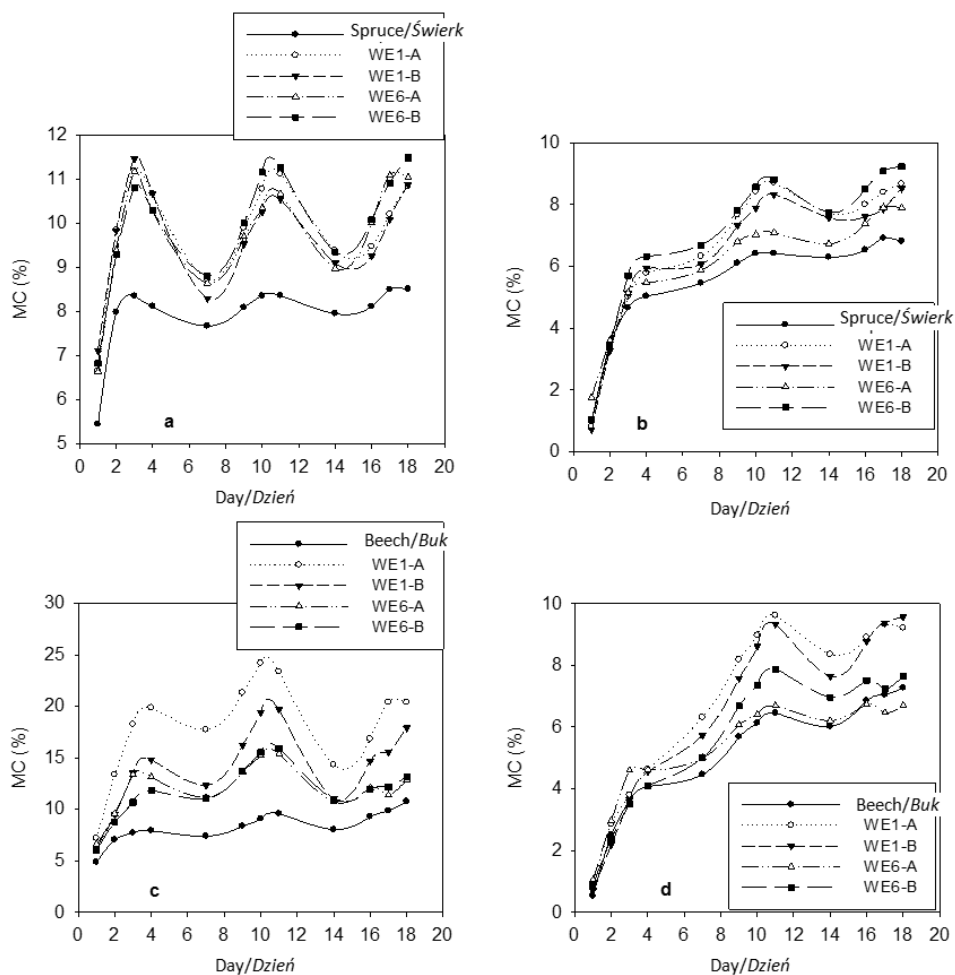


Fig. 4. Moisture content of polyethylene wax emulsion treated: (a) vacuum dried spruce, (b) spruce cured at 140 °C, (c) vacuum dried beech, and (d) beech cured at 140 °C, before 10 minute non-continuous dipping in water

Rys. 4. Wilgotność impregnowanego emulsją wosku polietylenowego: (a) świerka suszonego próżniowo, (b) świerka utwardzanego w temp. 140 °C, (c) buka suszonego próżniowo i (d) buka utwardzanego w temp. 140 °C, przed 10-minutowym przerywanym zanurzeniem w wodzie

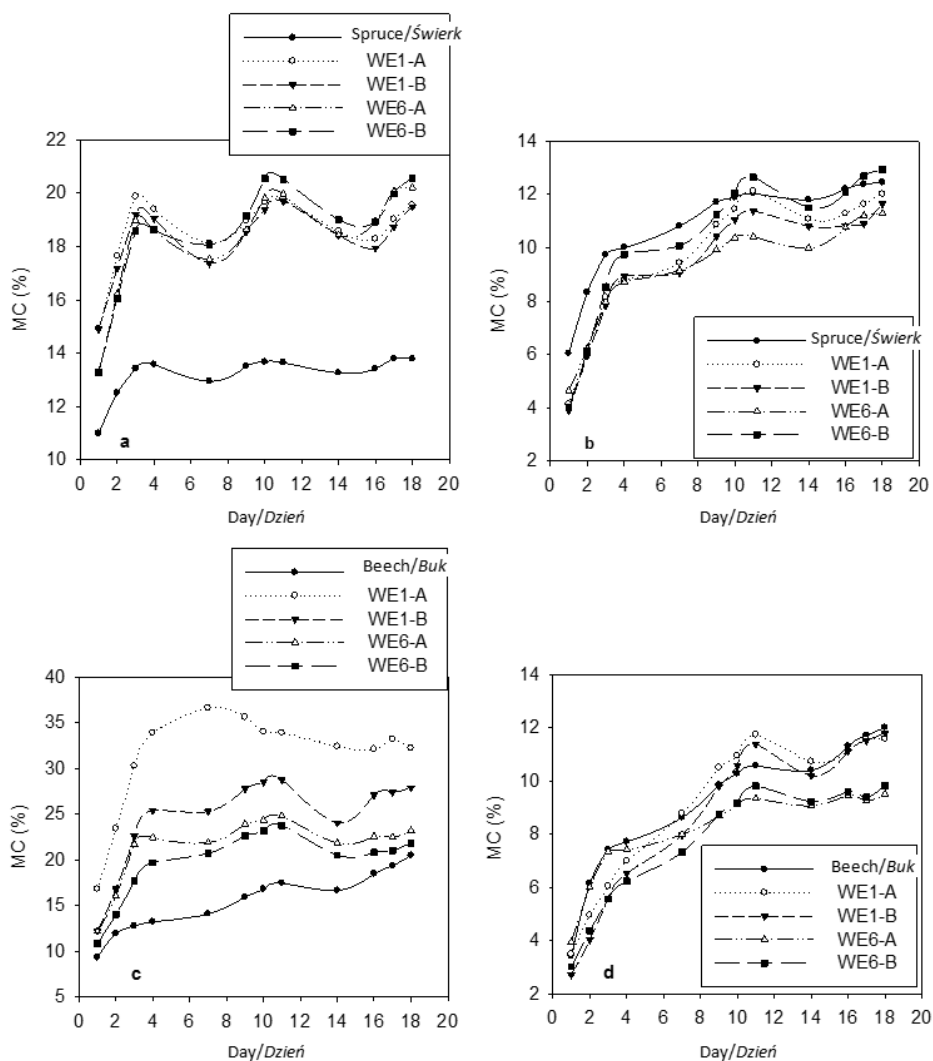


Fig. 5. Moisture content of polyethylene wax emulsion treated: (a) vacuum dried spruce, (b) spruce cured at 140 °C, (c) vacuum dried beech, and (d) beech cured at 140 °C, after 10 minute non-continuous dipping in water

Rys. 5. Wilgotność impregnowanego emulsją wosku polietylenowego: (a) świerka suszonego próżniowo, (b) świerka utwardzanego w temp. 140 °C, (c) buka suszonego próżniowo i (d) buka utwardzanego w temp. 140 °C, po 10-minutowym przerywanym zanurzeniu w wodzie

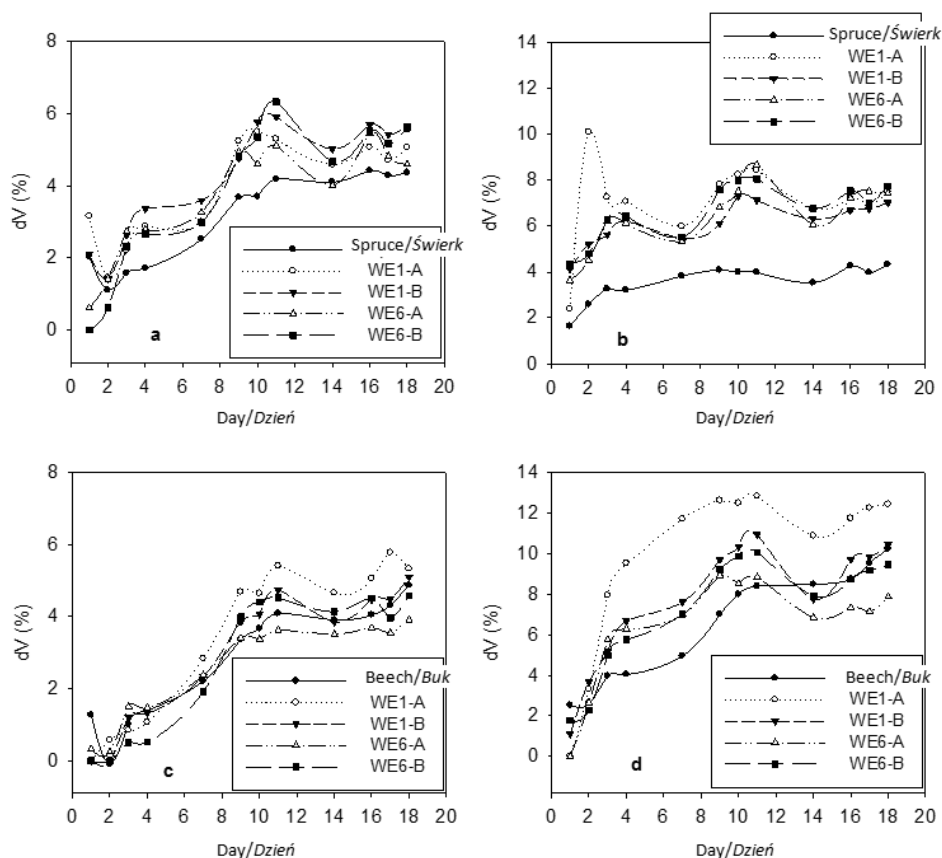


Fig. 6. Volume changes of polyethylene wax emulsion treated: (a) vacuum dried spruce, (b) spruce cured at 140 °C, (c) vacuum dried beech, and (d) beech cured at 140 °C, during non-continuous dipping in water

Rys. 6. Zmiany objętości impregnowanego emulsją wosku polietylenowego: (a) świerka suszonego próżniowo, (b) świerka utwardzanego w temp. 140 °C, (c) buka suszonego próżniowo i (d) buka utwardzanego w temp. 140 °C, podczas przerywanego zanurzenia w wodzie

In the second part of the experiment, the interest was in liquid penetration and drying of impregnated wood. The other half of the specimens intended for water uptake analysis were immersed in distilled water for 10 minutes on the 1st, 2nd, 3th, 4th, 7th, 9th, 10th, 11th, 14th, 16th, 17th and 18th day. The masses and dimensions of the specimens were monitored before and after immersion (fig. 3, 4, 5). Dimensions were measured with laser equipment (Department of Wood Science and Technology, Slovenia). Both sorption experiments were performed on ten identical specimens per emulsion/wood species/drying.

A SEM microscope (JEOL 5500 LV, Japan) was used for surface observation. Wax impregnated and control specimens were coated with a highly conductive

film of gold (Sputter Coater SCD 005, Baltec, Germany). Specimens were scanned in high vacuum and an accelerating voltage of 20 kV was used.

Results and discussion

As a result of the impregnation process, Norway spruce specimens retained between 135 kg/m³ and 197 kg/m³ of the emulsions. In contrast, beech specimens retained considerably higher amounts of the emulsions. In general, they retained between 641 kg/m³ and 689 kg/m³ of wax emulsions (table 1). This result is reasonable as beech wood is significantly more permeable than spruce. It is clearly evident that retention in Norway spruce wood was influenced by the emulsion type and the dry content of the wax. The main reason for this is that the particles in the emulsions are too big (100 nm) [Anonymous 2004a; Anonymous 2004b] to penetrate the cell walls; they even form a barrier on the surface of the cell wall and reduce the penetration of the solvent (water) into the cell walls.

However, the most important focus of the research was to elucidate how polyethylene wax emulsions influence the sorption properties of the impregnated wood. The first set of sorption experiments was performed in a chamber with 87% RH, in which impregnated specimens were conditioned. Half of the specimens were oven dried (140 °C) and the other half were vacuum dried (60 °C). The moisture content (MC) of various control specimens reached equilibrium moisture content (EMC) at approximately 15% after 17 days of conditioning (fig. 1a, 1b, 1c, 1d). Surprisingly, there were no significant differences in the process of moisturising and the final MC after 41 days of conditioning of vacuum and oven dried control and impregnated Norway spruce specimens (fig. 1a, 1b). It is presumed that the reason for this was the low retention of wax emulsions in spruce wood [Lesar, Humar 2010; Lesar et al. 2010]. Furthermore, there were also almost no differences in the MC of oven dried control and wax treated beech specimens (fig. 1d). On the other hand, the most prominent vapour barrier was formed by wax treated, vacuum dried beech. Those specimens demonstrated lower MC than control specimens. The lowest final MC was determined for WE6-B impregnated, vacuum dried beech specimens, whose MC was 25 % lower than the MC of control specimens (fig. 1c). Those results were not in line with the expectations. It had been expected that vacuum dried control and wax treated specimens would have a higher MC than the oven dried. The finding was in contradiction with two things. Firstly, thermal decomposition of wood begins at temperatures above 100°C [LeVan 1989; Esteves, Pereira 2009], resulting in decomposition of hemicelluloses [Fengeland, Wegener 1989; Orfao, Figueiredo 2001], which are the most hygroscopic wood component [Skaar 1972]. Thus, in general heat treated wood has a lower MC in the process of conditioning than non-heated wood. Secondly, curing above the melting point led to the formation of a compact film of wax on the

wood surface, as can be clearly seen in fig. 2. Therefore, there must have been some voids in that wax film which facilitated diffusion of water vapour. However, it must be taken into account that wax itself is not hydroscopic, since the moisture content of the wax at 98% relative air humidity varied between 1.1% and 1.3%, so the sorption properties of wood must be the factor that determines the sorption properties of the wax-wood complex.

The differences in volume changes of wax treated and control specimens were very noticeable. Fig. 3 presents volume changes that occurred during exposure of control and wax treated specimens in a chamber with RH of 87%. The shape of the volume change curve during moisturising is similar to the curve of moisture changes. The differences in volume changes of vacuum and oven dried control and wax treated Norway spruce specimens were insignificant, with the exception of vacuum dried WE6-A impregnated specimens, the swelling of which was considerably more prominent than the swelling of control specimens (fig. 3a, 3b). In contrast, all wax treated beech specimens, regardless of the type of drying applied, had up to 40% improved dimensional stability in comparison to control specimens. Furthermore, a difference was observed in the dimension changes of vacuum dried and oven dried specimens. Surprisingly, the volume changes of impregnated oven dried beech specimens were smaller than dimensional changes of control specimens in terms of moisture content during the test (fig. 1d, 2d). These results indicate that polyethylene wax was to some extent bound in the cell walls of wood [Banks 1973], which prevented swelling of treated wood. The results were in contradiction to those of Rowell and Banks [1985], who reported that water repellents cannot, to any significant extent, interfere with the movement of water into wood by vapour-phase or bound water mechanisms.

In the second set of sorption experiments, control and treated specimens were being dipped in and taken out of distilled water for a period of 10 minutes in order to simulate occasional wetting that occurs in service life. The moisture content of vacuum dried control spruce specimens before immersion varied between 5.4% and 8.5%. The MC of vacuum dried control beech specimens was between 4.8% and 10.8% (fig. 4a, 4c). The MC of wood specimens before immersion depended mostly on the drying time between immersion events. The MC of oven dried Norway spruce and beech control specimens before immersion increased from 1.0% to 7.0% (fig. 4a, 4b, 4c, 4d). Surprisingly, wax treated Norway spruce and beech specimens demonstrated higher MC than control specimens. The type of wax emulsions used for the impregnation of spruce did not have any significant influence on the moisture content of the impregnated wood before immersion. The shape of the curve of MC changes of treated vacuum dried beech specimens before their impregnation with wax was similar to that of vacuum dried Norway spruce specimens, but in general the MC of wax treated beech specimens was higher (fig. 4a, 4c). In all cases oven dried control and wax treated specimens had similar MC at the beginning of the immersion test.

After the immersion events, the MC of all specimens was similar to the reported in the previous chapter, although all values were considerably higher. The MC of vacuum dried Norway spruce control specimens increased by approximately 5 percent on average after immersion. The moisture content of the specimens increased during the tests. For example, the MC of vacuum dried beech control specimens was 9.3% after the first immersion and increased up to 20.5% after the last immersion. Surprisingly, impregnation of spruce wood with waxes did not improve water repellence but, on the contrary, in certain cases even made the wood hydrophilic. The MC of wax treated vacuum dried Norway spruce specimens was nearly 150% higher than that of control specimens. The influence of wax treatment on water uptake during immersion was even more prominent in the case of beech specimens (fig. 5a, 5c). The comparison of the oven dried and vacuum dried wax treated beech specimens clearly indicates that polyethylene waxes (WE1 and WE6 of both concentrations) had to be cured (heated) above the wax melting point in order for the surfaces of the treated specimens to have become hydrophobic. It is presumed that the main reason for this was the morphology of the wax surface on wood. In the case of vacuum dried specimens, there were small cracks on the surfaces of polyethylene wax impregnated wood samples (fig. 2a). The cracks acted as capillaries, which took up water and reduced the contact angles of the surfaces [de Meijer, Militz, 2000]. The contact angle of water on vacuum dried WE6 treated wood was about 10° . This made penetration of water into the wood considerably faster. However, after curing above the melting point polyethylene wax formed a compact thin film which repelled water. This can be clearly seen in the SEM figure (fig. 2b). However, it must be taken into account that oven drying at 140°C has some effect on the material. The MCs of oven dried Norway spruce and beech control specimens increased during the experiment and were approximately 4 percent higher than before immersion. After the last immersion, the MC of spruce specimens was 12.5% and of beech specimens 12.0%. This is reasonable, since heat treatment is a well-established technology for wood modification [Esteves, Pereira 2009]. However, a combination of wax treatment and oven drying was even more effective. WE1-A, WE1-B, WE6-A treated oven dried Norway spruce specimens had lower MC than control specimens (WE6-A up to 15%) (fig. 5b). WE6-B treated specimens had the lowest MC among oven dried beech specimens; MC was on average 18% lower than that determined for parallel control specimens (fig. 5d). The weak hydrophobic efficacy of polyethylene waxes did not reduce the shrinking or swelling of wood. The differences between vacuum dried control and wax treated spruce and beech specimens were insignificant (fig. 5a, 5b, 5c, 5d).

From the results presented in this research, it can be seen that vacuum drying of wax treated wood was not so effective a hydrophobic treatment as oven drying of wax treated wood. As already mentioned, the main reason for the higher MC of vacuum dried specimens was the small cracks visible on the surface of WE6

and WE1 treated wood. The small cracks on the surface of vacuum dried treated wood formed capillaries which increased water flow [de Meijer, Militz 2000]. It is also known that some liquid water flow may occur in water repellent treated specimens due to the phenomenon of preferential wetting of wood surfaces by water [Banks 1973]. A reduction in the MC of polyethylene wax treated Norway spruce and beech specimens was only achieved with oven drying at temperatures above the melting points of waxes. After drying the wax treated wood above the melting point, polyethylene (WE1) and oxidised polyethylene (WE6) wax formed a film which prevented fast water penetration into the treated wood and also facilitated fast drying of that material after dipping (fig. 2b). The lower moisture content of oven dried wax treated Norway spruce and beech specimens during dipping in water did not result in improved dimensional stability. Rowell and Banks [1985] reported that water repellent treated wood exposed to liquid water for a prolonged period, not only would swell to the same extent as similar untreated wood but also accumulate free water in the cell lumina and therefore eventually attain a MC in excess of fibre saturation. This is in line with the findings presented hereinbefore.

Conclusions

The sorption properties of polyethylene (WE1) and oxidised polyethylene wax (WE6) were influenced by its retention. Lower retention of the wax by Norway spruce compared to beech specimens led to smaller differences between the MC of treated and control specimens during conditioning in humid air and moisturising during non-continuous dipping in water. During conditioning in humid air, a positive effect of wax treatment was observed only on vacuum dried treated beech specimens. Improved dimensional stability was found only in the case of oven dried beech. In order to achieve slower water penetration into wax treated wood during dipping in water, the wax treated material had to be oven dried above the melting point of the wax used. Furthermore, the results clearly indicate that polyethylene wax treatment slowed down drying of oven dried Norway spruce and beech specimens.

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WPLYW SUSZENIA NA WŁAŚCIWOŚCI SORPCYJNE DREWNA IMPREGNOWANEGO WOSKIEM POLIETYLENOWYM

Streszczenie

Znaczenie zastosowania wosków w przemyśle drzewnym rośnie, zwłaszcza w Europie, co spowodowane jest wzrostem świadomości ekologicznej, niechęcią konsumentów do używania drewna poddanego obróbce biocydami oraz drewna z lasów tropikalnych. W badaniach wykorzystano dwie emulsje wodoodporne o różnych stężeniach: polietylenową emulsję woskową i jej utlenioną wersję. Właściwości impregnowanego woskiem drewna świerka pospolitego (*Picea abies*) i drewna buka (*Fagus sylvatica*) zostały przebadane w trakcie badań sorpcji: klimatyzowania w warunkach wysokiej wilgotności względnej powietrza (dyfuzja pary) i przerywanego zanurzenia (przepływ cieczy). Określono właściwości sorpcyjne impregnowanych próbek suszonych w suszarce i próżniowo. Podczas klimatyzowania próbki świerka pospolitego impregnowanego woskiem, suszone w suszarce i próżniowo, wykazały tę samą wilgotność, co próbki kontrolne. Impregnowane, wysuszone próżniowo drewno buka osiągnęło wilgotność do 25% niższą niż próbki kontrolne. Jeszcze większa różnica była widoczna w trakcie badania sorpcji w zakresie zmian objętości próbek buka suszonych w suszarce i próżniowo.

Słowa kluczowe: buk, świerk pospolity, wosk polietylenowy, sorpcja, ochrona drewna, środki wodoodporne