WATER AND MOISTURE RELATED PROPERTIES OF BEECH (*Fagus sylvatica* L.) VENEER TREATED WITH SILICONE COMPOUNDS

Trinh Hien Mai

Vietnam National University of Forestry

SUMMARY

Beech (Fagus sylvatica L.) veneers were impregnated with 10% solid content of silicone compounds (amino and epoxy functional polysiloxane) from textile industry. The impregnation process included two steps: vacuum of 60 mbar for 30 min and followed by 2h veneer storing in the solutions at atmospheric pressure. Then, the veneers were pre-dried at 40°C for 24h and cured at 140°C for 2h in a drying-oven before testing water and moisture absorption. The results showed that water repellence effectiveness (WRE) of the treated veneers within each submersion were quite high (up to 70%) in the initial submersion phases of 1 min, 10 min, 1h, 2h, 4h and then lessened gradually in the submersion phases of 24h. After vacuum support and soaking in water overnight (24h), WREs (at water saturation) got the lowest values ranging from micro amino silicone ASE 8130 (3%) to macro amino silicone ASE 8730 (10%). The wetting-drying cycles after each submersion did not result in significant change of WREs in the second, third and fourth submersion although weight loss of each treatment increased from the first cycle to the fourth submersion. After the fourth submersion, silicone SIN treated veneers got the highest weight losses (4%) whereas two silicones ESE 6960 and ASE 8730 revealed the lowest weight loss (1.4 and 1.6%, respectively). When stored in an environment of 30, 65, 90% relative humidity and 20°C, all of the silicone treated veneers get minor lower equilibrium moisture content EMC_R (0 - 1.7%) and radial swelling RS (0 - 0.7%) than the control veneers at different levels. Keywords: Beech, equilibrium moisture content, veneer, water repellence effectiveness, weight loss.

1. INTRODUCTION

Silicones represent a group of compounds consisting of chains of Si-O-Si units in which various organic substitutes are attached to the Si atoms, with reactive groups situated at the chain ends (Hill, 2006). Silicones have been used in many applications e.g building industry (masonry and concrete), textile, plastics and cosmetic industries (Lukowsky *et al.*, 1997; Weigenand, 2006). The similarity of the substrate cotton in textiles and cellulose in wood allows the application of silicone emulsion systems on wood. Depending on the particle size, the silicone emulsions can be divided into micro emulsions (particle size from 10 - 80 nm) and macro emulsions (particle size amounted to 100 nm and more). Due to their size, only micro emulsions are able to penetrate into voids of the wood cell wall (Mai and Militz, 2004).

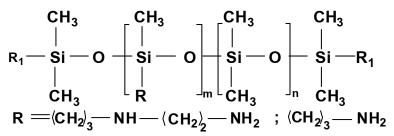


Figure 1. Chemical structure of polysiloxane with amino groups

Weigenand *et al.* (2007) studied the penetration of two amino-silicones in form of a micro and a macro emulsion (Figure 1). A significantly higher amount of silicone from the micro emulsion was found in the cell wall in comparison to that from macro emulsion. Therefore the micro emulsion treatments led to higher cell wall bulking and greater antishrinking efficiency. Treatments with both micro and macro amino silicone emulsions resulted in moderate water repellence and

resistance against decay and blue stain fungi (Ghosh *et al.*, 2008; Weigenand *et al.*, 2008; Weigenand *et al.*, 2007). After exposure to natural weathering for one year according to the EN 927-3, the panels treated with amino silicone showed less colonization by staining fungi and displayed reduced surface roughness than those of the controls (Ghosh *et al.*, 2009).

Beech (*Fagus sylvatica* L.) is one of the most common plantation wood used in veneer industry. It is easily treatable but has low

bioresistance and low dimensional stability which limit area of application. Therefore, wood modification has been employed to improve durability for wood and wood-based products from beech. Beech veneers have been treated with various chemicals eg DMDHEU, acetic anhydride to produce modified plywood with enhanced performance such as high dimensional stability, increased biological resistance, maintained mechanical properties (Wepner and Militz, 2005; Talaei, 2008). In this study, beech (Fagus sylvatica L.) veneers were treated with different amino and epoxy functional polysiloxane from textile industry for testing of water/moisture related properties and weight losses after wetting, freezing and drying processes.

2. RESEARCH METHODOLOGY

2.1. Data base of the chemicals

Siligen SIN (SIN)

Siligen SIN (SIN), delivered by BASF, is a concentrated, very finely dispersed (particle size smaller than 10 nm) micro emulsion of an amino functional polysiloxane. SIN is a whitish emulsion with pH value from 4 - 6.5 at 20°C.

SIN is a slightly hydrophilic softening and smoothing agent which is suitable for woven and knitted fabrics of cellulosic fibers and their blends with synthetic fibers. This kind of agent ensures the stability of shearing, produces a very soft, smooth, supple handle and improves the abrasion resistance, tear strength and sewability. SIN can be diluted well with cold water and applied by padding at room temperature, then pre-drying and curing for 3 min at 150°C or 1 min at 170°C for fabrics.

Siligen SIO (SIO)

Siligen SIO (SIO), delivered by BASF, is an amino functional polysiloxane. SIO is slightly opaque emulsion with pH value from 5.5 - 6.5 at 20°C.

SIO is a smoothing and softening agent for woven and knitted fabrics. It ensures the stability for shearing, increases the resilience, tear strength and abrasion resistance of fabrics. SIO can be diluted with cold water and applied for fabrics by padding at room temperature, then pre-drying and curing for 1 min at 170°C.

Hansa ASE 8130 (ASE 8130)

Hansa ASE 8130 (ASE 8130), delivered by HANSA, is a highly concentrated watery micro emulsion of an amino functional polysiloxane. ASE 8130 is a transparent emulsion with average pH value from 5 - 6 at 20°C.

ASE 8130 is used in the textile industry as long term stable hydrophobic cotton finish for improving washing stability in combination with crease resistance enhancing agents. ASE 8130 can be diluted with cold water and applied by padding at room temperature for fibers, then drying at 130°C.

Hansa ASE 8730 (ASE 8730)

Hansa ASE 8730 (ASE 8730), delivered by HANSA, is a highly concentrated watery macro emulsion of an amino functional polysiloxane. ASE 8730 is a white emulsion with average pH value of 6.5.

ASE 8730 is used in the textile industry as long term stable hydrophobic cotton finishes for improving washing stability. ASE 8730 can be diluted with cold water and applied for fibers by padding at room temperature, then pre-drying and curing for 3 min at 150°C.

Hansa ESE 6960 (ESE 6960)

Hansa ESE 6960 (ESE 6960), delivered by HANSA, is a white emulsion of an epoxy functional polysiloxane with average pH value from 5 - 6 at 20°C.

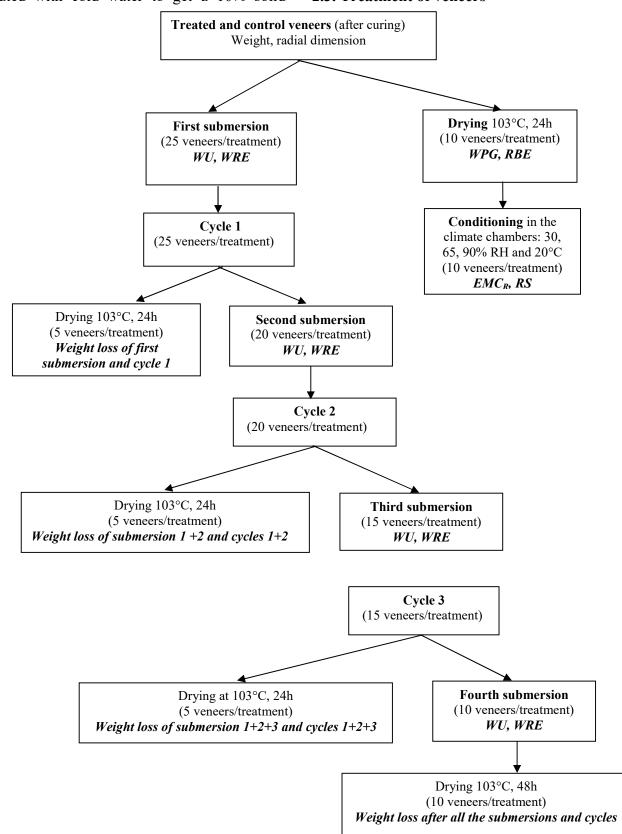
ESE 6960 is particularly suitable for permanent plasticizing of sport and leisure articles from synthetic fibers and their mixtures. ESE 6960 can be diluted with cold water and applied by padding at room temperature for fibers, then drying at 110 -130°C.

2.2. Veneer and chemical preparation

Sliced beech (*Fagus sylvatica* L.) veneers without heartwood were prepared in sizes of $50 \times 0.5 \times 50 \text{ mm}^3$ (rad × tang × long). The numbers of veneer specimens are listed in the table 1.

No	Experiments	Number of veneers/treatment
1	Water uptake and cyclic tests	25
2	Sorption behavior	10
	Total veneers per treatment	35

Table 1. Quantity of veneers for each treatment



Each chemical listed in section 2.1 was diluted with cold water to get a 10% solid **2.3. Treatment of veneers**

Figure 2. Test procedure of the treated veneers

The veneers were oven-dried at $103 \pm 2^{\circ}C$ for 24h, then transferred to a desiccator and

cooled to ambient temperature over silica gel. Prior to impregnation, each oven-dry veneer was weighted on a four-figure balance and measured radial dimension using an electronic micrometer accurate to ± 0.01 mm. A special instrument was used to force veneer to be flat for measuring radial dimension.

After weighting and measuring, the veneers were impregnated with the prepared solutions mentioned in section 2.2 as soon as possible. For comparisons, the beech veneers impregnated with water served as control specimens. The impregnation process included two steps: vacuum of 60 mbar for 30 min and followed by 2h veneer storing in the solutions at atmospheric pressure. Then, the veneers were pre-dried at 40°C for 24h and cured at 140°C for 2 h in a drying-oven. After cooling down in a desiccator, weight and radial dimension of the treated veneers after curing were recorded. The treated veneers were with water/moisture related preceded experiments as described in figure 2.

2.4. Water submersion and cyclic tests

Water submersion

Water repellent characteristic was evaluated through water submersion tests; veneer specimens for these tests were described in sections 2.2, 2.3 and figure 2. For each submersion test, the veneers were submersed one by one in a water bath at room temperature for continuous times: 1 min, 10 min, 1h, 2h, 4h, and 24h. After 24h submersion, water uptake was supported by vacuum (100 mbar, 30 min), then the veneers were stored in water at atmospheric pressure overnight to reach full water uptake (water saturation).

After given times had elapsed, the veneer specimens were removed from the water bath, dabbed off with tissue and weighted immediately. The water uptake was calculated according to equation 1 (Donath, 2005).

$$WU(\%) = \frac{(W_a - W_b)}{W_o} \times 100$$
 (1)

Where:

WU: water uptake;

W_a : veneer weight after water submersion (1 min, 10 min, 1 h, 2 h, etc);

W_b: veneer weight before water submersion;

 W_{o} : oven-dry weight of veneer before impregnation.

For comparison of water uptake between the treated and the control veneers, water repellent effectiveness (WRE) was expressed as in equation 2 (Donath, 2005; Lukowsky *et al.*, 1997; Rowell and Banks, 1985):

$$WRE(\%) = \frac{(WU_{control} - WU_{treated})}{WU_{control}} \times 100$$
(2)

Where:

WRE: water repellent effectiveness; WU_{control} : water uptake of control veneer; WU_{treated} : water uptake of treated veneer. *Cyclic tests*

To evaluate fixation of the chemicals in the treated veneers and the stability of the water repellent effect under influence of wetting-drying process; after each submersion, the veneers were undergone a cyclic test based on the EN 321. Each cycle was carried out by submersing the veneers in water $(20 \pm 2)^{\circ}$ C for (72 ± 1) h, freezing them at between -12°C and -20°C for (24 ± 0.25) h; and then drying them at $(70 \pm 1)^{\circ}$ C for (72 ± 1) h. These cycles might cause cracks on surface of the veneers, which would affect water uptake, swelling/shrinking, and weight loss as well.

After each cycle, all veneers were weighted; 5 veneers from each treatment were ovendried, then weighted again to calculate the weight loss as shown in equation 3:

$$WL(\%) = \frac{(W_1 - W_2)}{W_1} \times 100$$
 (3)

Where:

WL: weight loss after cyclic test;

W₁: oven-dry weight of veneer after curing;

W₂: oven-dry weight of veneer after cyclic test.

2.5. Sorption behavior

Weight percent gain and radial bulking effect of the treated veneers

After curing, 10 veneers from each treatment were oven-dried to determine weight percent gain (WPG) and radial bulking effect (RBE).

The WPG was calculated from the oven-dry weight of veneer before and after treatment as in equation 4:

$$WPG(\%) = \frac{\left(W_1 - W_o\right)}{W_o} \times 100 \tag{4}$$

Where:

WPG: weight percent gain of treated veneer;

W₁: oven-dry weight of veneer after curing; W_o: oven-dry weight of veneer before impregnation.

The RBE was determined by comparison of radial dimension of dry veneer before and after treatment using equation 5:

$$RBE(\%) = \frac{\left(RD_1 - RD_o\right)}{RD_o} \times 100$$
(5)

Where:

RBE: radial bulking effect of treated veneer;

RD₁: radial dimension of oven-dry veneer after curing;

RD_o: radial dimension of oven-dry veneer before impregnation.

Equilibrium moisture content (EMC_R) and radial swelling (RS)

Sorption behavior was evaluated with the veneers described in sections 2.2 and 2.3. Ten veneers from each treatment were conditioned in different climates at 30, 65, 90% relative humidity (RH) and 20°C until the veneers reached equilibrium moisture content (EMC). EMC was considered to be reached when the results of two successive weighting operations within 24h did not differ by more than 0.1% of the weight of veneer. To avoid the reduction in EMC simply due to increased weight of veneer after the treatment, the EMC_R calculation was based upon the oven-dry weight of the wood substance rather than the treated wood. The EMC_R and the radial swelling (RS) are presented in equation 6 - 7 (Hill, 2006):

$$EMC_{R}(\%) = \frac{(W_{3} - W_{1})}{W_{o}} \times 100$$
 (6)

$$RS = \frac{\left(RD_3 - RD_1\right)}{RD_1} \times 100\tag{7}$$

Where:

 EMC_R and RS: equilibrium moisture content and radial swelling of veneer;

W_o: oven-dry weight of veneer before impregnation;

W₁ and RD₁: oven-dry weight and radial dimension of veneer after curing (before conditioning);

W₃ and RD₃: weight and radial dimension of veneer after conditioning.

3. RESULTS AND DISCUSSION

3.1. Water repellent effectiveness

Water repellence is considered to protect wood from water-induced degradation, such as surface deterioration or leaching of preservatives (Donath, 2005). Water repellent effectiveness (WRE) might change due to using condition, thus WRE was evaluated through a submersion test after each cycle. The cycle of wetting, freezing and drying processes (according to EN 321) would lead to the formation of micro cracks on veneer surfaces. These cracks penetrate more or less deeply into the wood and affect water uptake of the wood.

As depicted in figure 2, the first time submersion of the veneers was processed as soon as possible after curing; afterwards the veneers were continued with the first cycle and the second submersion until fulfilled 3 cycles and 4 submersions. The moisture contents of the veneers after curing (before the first submersion) were between 0 and 0.9% whereas the moisture contents of veneers after the cycles (before the second, third and fourth submersion) ranged from 0.3 to 3%. The differences in moisture contents of the veneers before the submersions were negligible and did not cause errors when water repellent effectiveness of different submersion was compared.

As can be seen in figure 3 - 5, WREs of the first submersion were very low (even negative in the case of all functional values) polysiloxane (SIN, SIO, ASE 8130, ASE 8730, ESE 6960) treated veneers. This can be explained by two reasons: 1) The water uptake of the control veneers increased from the first to the last submersion due to the formation of micro cracks on the veneer surfaces caused by wetting-drying cycles (Trinh and Nguyen, Wetting-drying 2017); 2) cycles were supposed to encourage the process of ongoing polymerization which leads the completion of improves water condensation and thus repellent effect (Donath, 2005; Lukowsky et al., 1997; Mai and Militz, 2004).

The cycle after each submersion did not result in significant change of WREs in the

second, third and fourth submersion (Figure 3 - 5) although weight loss of each treatment increased from the cycle 1 to the fourth submersion. Consequently, it can be stated that, the water repellence of the treated veneers was stable after the wetting-drying cycles.

As can be seen in figure 3 - 5, WREs of the treated veneers within each submersion were high (up to 70%) in the initial submersion phases of 1 min, 10 min, 1h, 2h, 4h and then lessened gradually in the submersion phases of 24h. As expected, after vacuum support and soaking in water overnight (24h), WREs (at water saturation) got the lowest values. Within the first 4 h of submersion, water uptake occurred rapidly with the control veneers (Trinh and Nguyen, 2017) because water penetrated easily into empty voids, while water

uptake occurred slowly with the treated veneers (not shown here) because water paths were occluded by the chemicals. Hence, the treatments with these chemicals considerably diminished the water uptake of veneers in the initial submersion phases. The WREs reduced significantly from 4h of immersion to water saturation state (full-water), thus, the treatments mostly inhibited the speed of water uptake. Moreover, with the WREs at water saturation state ranged from micro amino silicone ASE 8130 (3%) to macro amino silicone ASE 8730 (10%), there must be some chemicals deposited in the cell lumen and cell wall of the treated veneers and therefore caused lower full-water uptake in comparison to that of the control veneers. Amino functional polysiloxane treatment

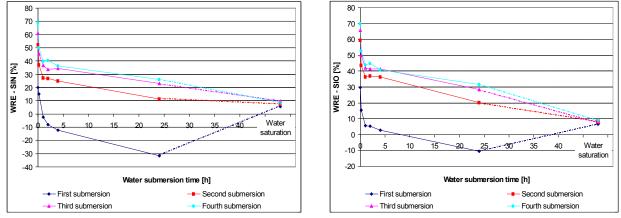


Figure 3. Water repellent effectiveness of amino functional polysiloxane: SIN and SIO treated veneers

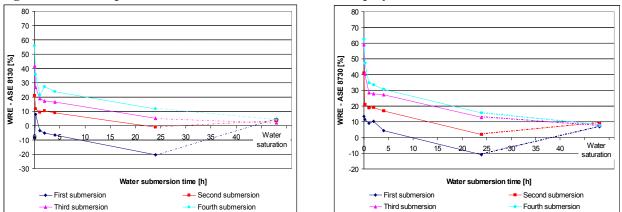


Figure 4. Water repellent effectiveness of amino functional polysiloxane: ASE 8130 and ASE 8730 treated veneers

Water repellent effectiveness of SIN and SIO seems to be higher than those of ASE 8130 and ASE 8730. Three amino silicones SIN, SIO and ASE 8730 revealed quite high WREs at water saturation (around 9%)

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whereas micro amino silicone ASE 8130 showed lower WREs at water saturation (around 3-4%). This trend coincides with the findings of Weigenand *et al.* (2007) who reported macro emulsion ASE 8730 caused the

lower water uptakes along three directions of Scots pine treated specimens compared to micro emulsion ASE 8130. This was elucidated through SEM-EDX studies; the lumens of specimens treated with macro emulsion exhibited higher amounts of silicones than the lumens of micro emulsion treated specimens, thus, there were more silicones deposited in the cell lumen of macro emulsion ASE 8730 treated specimens and water uptake was reduced (Weigenand *et al.*, 2007).

Epoxy functional polysiloxane treatment

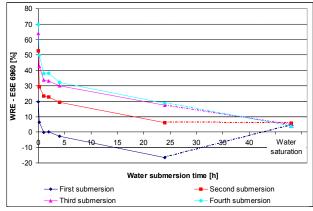


Figure 5. Water repellent effectiveness of epoxy functional polysiloxane ESE 6960 treated veneers

Like the other treatments, the WREs of the second, third and fourth submersions were increased clearly when compared to the WREs of the first one (Figure 5). There may be some hydrophilic compositions removed from surface of ESE 6960 treated veneers during the first water soaking and the first wetting-drying cycle. The WREs reduced from 70% at 1 min of soaking to 5% at the full-water state. The hydrophobation at the initial phases of submersion can be attributed to the deposition of self polimerized chemical in the cell lumen.

3.2. Weight loss through different cycles

When treated veneers undergo cyclic dipping, some compositions such as unfixed chemicals, extractives and volatiles are removed by hot/cold water leaching and thermal effect of drying process, and as a consequence, weight of veneers is reduced. Wood degradation products are also leached out in case the veneers become brittle after the treatment.

Weight losses of the treated veneers increased from the cycle 1 to the fourth submersion (figure 6). These weight losses resulted from the different water submersion and wetting-drying processes as described in section 2.3 and figure 2. After the fourth submersion, silicone SIN treated veneers got the highest weight losses (4%) whereas two silicones ESE 6960 and ASE 8730 revealed the weight lowest loss (1.4)and 1.6%. respectively). These results showed the similar tendency as weight loss of water soxhlet extraction (Trinh, 2010).

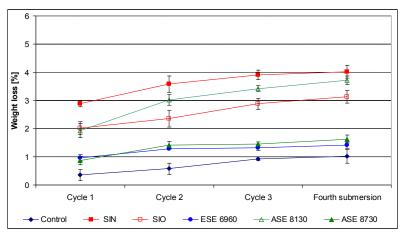


Figure 6. Weight losses after the water submersion and cyclic tests of silicone treated veneers

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The weight loss of the control veneers after the fourth submersion was approx 1%. This should be taken into account when interpretating the results. Hence, the weight losses of unfixed chemicals could be lower than the weight losses depicted here.

3.3. Sorption behavior at different relative humidity

Sorption behavior of Beech veneers treated with different silicones are presented in table 2.

Table 2. Equilibrium moisture content and radial swelling of the control and treated veneers
(10% solid content of the silicones) at different relative humidity and 20°C

No	Treatment	WPG (%)	RBE		um moistur EMC _R (%)	e content	Ra		
			(%)	30% RH	65% RH	90% RH	0% RH	65% RH	90% RH
1	Control	-1.47	-0.99	4.62	13.60	18.42	0.84	2.79	3.74
	STDEV	0.36	0.07	0.13	0.17	0.29	0.04	0.09	0.04
2	SIN	11.41	0.80	3.83	12.24	16.71	0.55	2.19	2.97
	STDEV	0.71	0.08	0.14	0.29	0.36	0.03	0.10	0.15
3	SIO	8.09	-0.29	4.15	12.52	17.41	0.69	2.54	3.61
	STDEV	0.79	0.15	0.13	0.15	0.24	0.03	0.08	0.12
4	ASE 8130	8.94	0.00	3.53	12.17	16.78	0.65	2.44	3.42
	STDEV	0.35	0.09	0.20	0.24	0.36	0.04	0.09	0.12
	ASE 8730	8.87	-0.61	4.17	12.78	17.82	0.69	2.64	3.69
5	STDEV	0.40	0.06	0.24	0.37	0.48	0.05	0.08	0.13
6	ESE 6960	7.80	-0.74	3.98	13.15	18.60	0.78	2.62	3.73
	STDEV	0.86	0.17	0.19	0.12	0.13	0.02	0.06	0.14

All of the silicone treated veneers get minor lower EMC_R (0 - 1.7%) and RS (0 - 0.7%) than the control veneers. However, micro emulsion amino functional polysiloxane (SIN) is assumed to impart better penetration into the cell wall than macro emulsion amino functional polysiloxane (SIO) due to particle size with an evidence of higher radial bulking effect RBE (Table 2), hence, EMC_R and especially RS of SIN treated veneers were lower than those of SIO treated veneers.

As confirmed from the study of Weigenand *et al.* (2007), a higher silicone amount from micro emulsion of amino functional polysiloxane (ASE 8130) could penetrate into the cell wall than from macro emulsions (ASE 8730). As a result, EMC_R and RS of ASE 8130 treated veneers were lower than those of ASE 8730 treated veneers.

Epoxy functional polysiloxane (ESE 6960) impregnated at low weight percent gain (WPG = 7.8%) and radial bulking effect (RBE = -0.7%), did not induce significant changes in EMC_R and RS for the treated veneers in comparison with the control veneers. This is expected to be because ESE 6960 particles probably could not penetrate into the cell wall, there was no deposition or cross linking of the chemical into the cell wall.

4. CONCLUSIONS

Weight losses of the treated veneers through the wetting-drying cycles were quite high, especially with silicone SIN treatments. However, in general, WREs did not decrease after four submersions and 3 cycles, even they were improved significantly after the first submersion. Therefore, the weight losses could be attributed to unreacted (unfixed) chemicals, wood degradation products and extractives, and did not cause detriment to the WREs. High water repellence of the treated veneers resulted from the closing water paths in lumen or blocking micro voids and/or hydroxyl groups of the cell wall as discussed in details.

Sorption behavior is related to the properties of modified cell wall, such as bulking and covalent bonds/cross linking. The treatments which provided higher RBEs or the chemicals which could react with hydroxyl groups or deposit into the cell wall often induced the reduction in EMC_R and RS for the treated veneers in a humid environment (silicone SIN and ASE 8130 treatments).

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TÍNH CHẤT HÚT NƯỚC, HÚT ẨM CỦA VÁN MỎNG GÕ BEECH (*Fagus sylvatica* L.) BIẾN TÍNH VỚI CÁC HỢP CHẤT CỦA SILICONE

Trịnh Hiền Mai

Trường Đại học Lâm nghiệp

TÓM TẮT

Ván mỏng gỗ Beech (Fagus sylvatica L.) được ngâm tẩm với các dung dịch hoá chất có chứa hợp chất silicon ở hàm lượng rắn 10%. Quá trình ngâm tấm gồm 2 bước: 1) sử dụng chân không ở 60 mbar trong 30 phút; 2) tiếp tục ngâm ván mỏng 2h trong điều kiện môi trường áp suất thường. Tiếp theo, ván mỏng được sấy ở 40°C trong 24h và xử lý nhiệt ở 140°C trong 2h trước khi tiến hành các thí nghiệm kiểm tra khả năng hút nước và hút ấm của ván mỏng. Kết quả nghiên cứu cho thấy: Khả năng chống hút nước (WRE) của ván mỏng được xử lý với hợp chất có chứa silicon trong các chu kỳ ngâm nước khá cao (đạt tới 70%) ở giai đoạn đầu: 1 phút, 10 phút, 1h, 2h, 4h rồi sau đó giảm dần khi ngâm trong nước 24h. Sau khi sử dụng chân không và ngâm nước ván mỏng qua 1 ngày đêm (24h), WRE ở trạng thái bão hoà nước nhận giá trị thấp nhất, từ micro amino silicon ASE 8130 (3%) tới macro amino silicon ASE 8730 (10%). Khả năng chống hút nước của ván mỏng khi ngâm nước (từ lần thứ hai đến lần thứ tư) ổn định qua các chu kỳ ngâm nước - sấy khô mặc dù tỷ lệ tổn hao khối lượng của ván được xử lý biến tính tăng dần. Sau lần ngâm nước thứ tự, ván mỏng xử lý biến tính với silicon SIN có tỷ lê tổn hao khối lượng lớn nhất (4%), trong khi đó ván mỏng biến tính với ESE 6960 và ASE 8730 có tỷ lệ tổn hao khối lượng nhỏ nhất (1,4 và 1,6%). Khi đặt trong môi trường không khí ở các độ ẩm 30, 65, 90% và nhiệt độ 20°C, toàn bô ván mỏng biến tính với các hợp chất có chứa silicon có đô ẩm thăng bằng thấp hơn so với ván mỏng đối chứng từ 0 - 1.7% và tỷ lê trương nở theo phương xuyên tâm thấp hơn so với ván mỏng đối chứng từ 0 - 0.7%. Từ khoá: Độ ẩm thăng bằng, gỗ Beech, khả năng chống hút nước, tổn hao khối lượng, ván mỏng.

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