WATER UPTAKE, MOISTURE ABSORPTION AND WETTABILITY OF BEECH VENEER TREATED WITH N-METHYLOL MELAMINE COMPOUNDS AND ALKYL KETENE DIMER

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SUMMARY

With Beech veneers being treated with 10% solid content of the chemicals from paper (*Alkyl ketene dimer,* AKD), wood (*N-methylol melamine*, NMM-1) and textile industry (*fatty acid modified N-methylol melamine*, mNMM-2), the results showed that the treatments with these chemicals considerably increased the water repellence effectiveness (WRE) of veneers in the initial submersion phases; Then, WREs reduced significantly from 4 h of immersion to water saturation state (full-water). Moreover, with the WREs at water saturation state ranged from AKD (5.0%) to mNMM-2 (19.3%), there must be some certain chemical deposited in the cell lumen and cell wall of the treated veneers. The water repellence of the treated veneers was stable after the wetting-drying cycles. The chemicals NMM-1 and mNMM-2 that could react with hydroxyl groups or deposit into the cell wall, may induce the reduction in equilibrium moisture content (EMC_R) and radial swelling (RS) for the treated veneers, while large AKD particles could not penetrate and modify the cell wall therefore did not reduce EMC_R and RS in humid environment for the treated veneers. Wettability of veneers treated with AKD, NMM-1 and mNMM-2 was much lower than the untreated and control veneers.

Keywords: Beech, contact angle, equilibrium moisture content, veneer, water repellence effectiveness, wettability.

I. INTRODUCTION

According to Borgin (1965), a truly water repellent preservative for wood would prevent water from being taken up by capillary system and render cell wall inert to water. This can be accomplished by impregnating the wood with a hydrophobic material which may deposit in the cell lumen, close the main penetration paths of water, and/or penetrate and allocate into the cell wall, resulting in bulking effect or cross-links (Hill 2006; Rowell and Banks, 1985). On the other hand, it is necessary to determine the efficiency of water repellence after several wetting-drying cycles to avoid misleading comparisons.

Wood treated with melamine-based compounds brought about remarkable improvements for solid wood, such as enhanced water repellence and dimensional stability (Inoue et al 1993, Pittman et al 1994, Nguyen et al 2007), increased hardness, modulus of elasticity and bending strength (Inoue et al 1993, Deka and Saikia 2000, Gindl et al 2004). In addition, water-based melamine treated wood indicated the potential to increase resistance against wood destroying fungi (Rapp and Peek 1996, Lukowsky et al 1999) and photochemical wood degradation caused by weathering (Rapp and Peek 1999). Alkyl ketene dimer (AKD) is widely used in the paper industry as an internal sizing agent. The hydrophobic effect of AKD is attributed to an esterification with hydroxyl groups of wood fibers and subsequent orientation of hydrocarbon chains (Isogai and Taniguchi 1992, Hubbe 2006). Thus, the application of AKD to wood and wood-based panels is expected to result in an increased dimensional stability; however, there have been only few studies using AKD for wood modification.

In this paper, veneers treated with N-methylol melamine and AKD chemicals were investigated on water/moisture related properties and their stability after cyclic tests.

Water repellent effectiveness in submersion tests, equilibrium moisture content and radial swelling in different humid climates, contact angle (wettability) are the crucial factors for further consideration.

II. RESEARCH METHODOLOGY

2.1. Data base of the chemicals

2.1.1. Alkyl ketene dimer (AKD)

Basoplast AKD delivered by BASF, is a fatty acid alkyl ketene dimer (AKD) in form of a white dispersion with average pH value from 3.5 - 4.5. AKD is hydrophobization of paper, especially when made under alkaline conditions. AKD is widely used for liquid containers, ink-jet printing paper, and many other grades of paper and paperboard. AKD is especially favored for products that need to resist water over a long period (BASF, 2003).

2.1.2. N-methylol melamine (NMM)

Madurit MW 840/75 WA (NMM-1) delivered by INEOS, is an N-methylol melamine resin dissolved in water. NMM-1 is a colorless and clear liquid with pH value from 10 - 11 at 20°C.

NMM-1 is used for impregnation of solid wood with a solid content between 10 and 40%. The drying process of impregnated wood includes two steps. In the first step, the temperature during the first 24 h must be lower than 50°C to remove the bulk of water and protect the wood against the formation of cracks. In the second step, a sufficient condensation of the resin is needed, if the temperature is not up to 100°C, the time for reaction must last longer (INEOS, 2006).

Phobotex VFN (mNMM-2) delivered by Ciba, is a fatty acid of modified N-methylol melamine (methoxymethylen melamine and paraffin). mNMM-2 is a white dispersion with pH value from 4 - 6 at 20°C.

mNMM-2 is a product for washfast and water repellent finishes which can be used as a finishing agent for textiles. mNMM-2 should be combined with catalyst RB (aluminium salt) to obtain optimal water repellent effect. mNMM-2 can be diluted in cold water and applied by padding at room temperature for cotton fibers, then dried at 120 - 140°C and cured for 2 min at 160°C or 4 - 5 min at 150°C (Ciba, 2002).

2.2. Veneer and chemical preparation

Beech (*Fagus sylvatica* L.) wood with diameter of 60 cm, harvested in Northern part of Germany, was selected for all experiments in this study.

Sliced beech veneers without heartwood were prepared in sizes of 50 × 0.5 × 50 mm^3 (rad × tang × long). The numbers of veneer specimens are listed in the following table 1.

Table 1. Quantity of veneers for each treatment

No	Experiments	Number of veneers per treatment
		50 × 0.5 × 50 (mm^3)
1	Water uptake and cyclic tests	10
2	EMC_R and radial swelling	10
3	Contact angle	10
	Total veneers per treatment	**30**

Each chemical listed in section 2.1 was diluted with water to get a 10% solid content solution for each treatment. mNMM-2 was mixed together with catalyst RB (15% of stock solution of mNMM-2 (w/w).

2.3. Treatment of veneers

The veneers were oven-dried at (103 ± 2)°C for 24 h, then transferred to a desiccator and allowed to cool 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.

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 2 h veneers being stored in the solutions at atmospheric pressure. A complete impregnation of the whole cross section is guaranteed by this impregnation process (Wepner et al., 2007). Excessive solutions on surface of the wet veneers were removed before pre-drying. Then, the veneers were pre-dried at 40°C for 24 h 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 being cured were recorded. The treated veneers were proceeded with water/moisture related experiments as described in Figure 1.

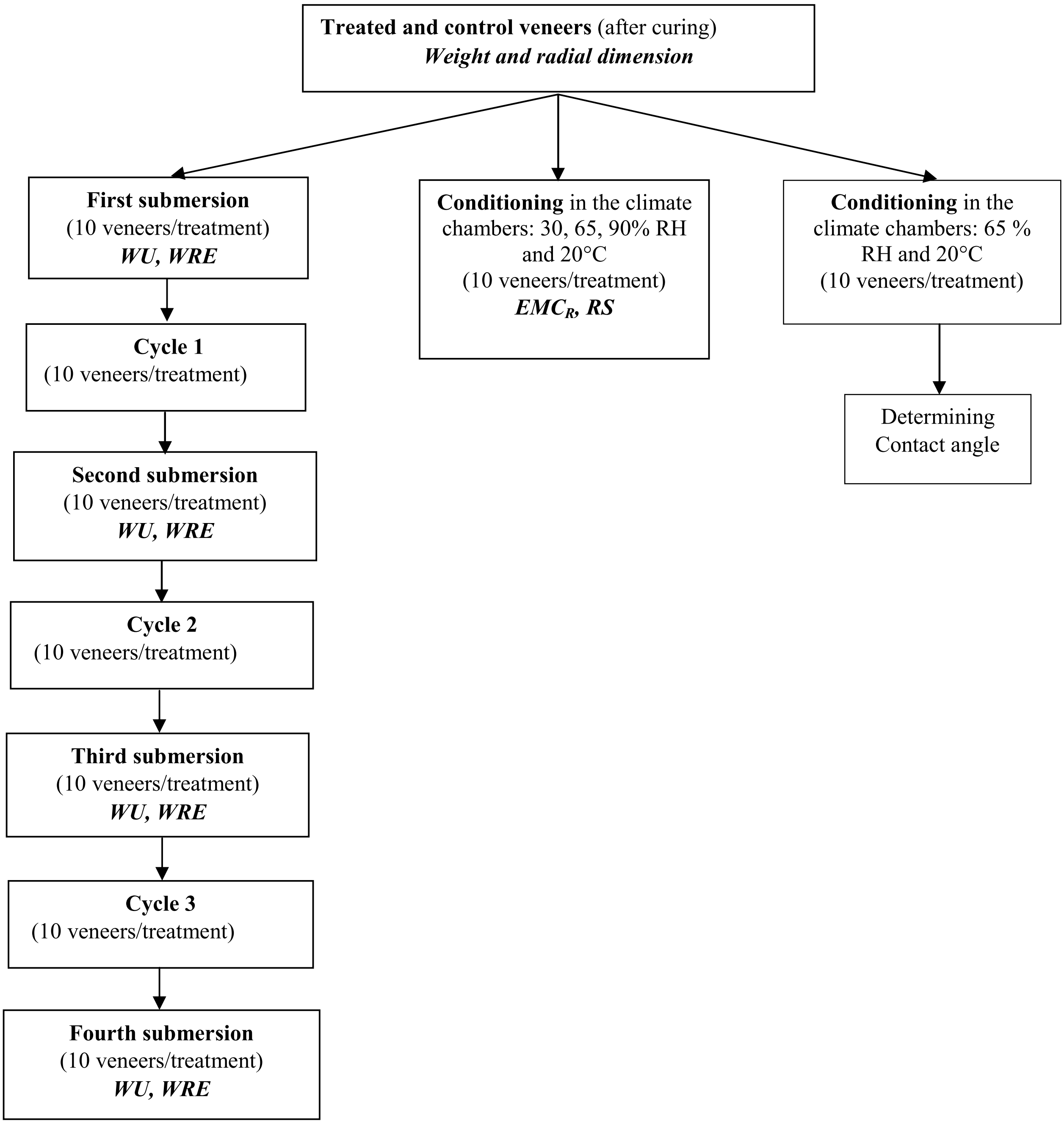


Figure 1. Test procedure of the treated veneers

2.4. Water submersion and cyclic tests

Water submersion

Water repellent characteristic was evaluated through water submersion tests; the veneer specimens for these tests were described in sections 2.2, 2.3 and Figure 1. For each submersion test, 10 veneers per treatment were submersed one by one in a water bath at room temperature for continuous times: 1 min, 10 min, 1 h, 2 h, 4 h, and 24 h. After 24 h 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 elapsde, 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).

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

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 the water uptake between the treated and the control veneers, water repellent effectiveness (WRE) was expressed as in Equation 2 (Donath, 2005).

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

Where:

WRE: water repellent effectiveness;

$WU_{control}$: water uptake of control veneer;

$WU_{treated}$: water uptake of treated veneer.

2.5. Cyclic tests

To evaluate the fixation of 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 EN 321. Each cycle was carried out by submersing the veneers in water (20 ± 2)°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 ± 1)°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.

2.6. Sorption behavior

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 24 h 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 3-4 (Hill, 2006)

$$EMC_R(\%) = \frac{(W_2 - W_1)}{W_o} \times 100 \quad (3)$$

$$RS = \frac{(RD_2 - RD_1)}{RD_1} \times 100 \quad (4)$$

Where:

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

W_o: oven-dry weight of veneer before impregnation;

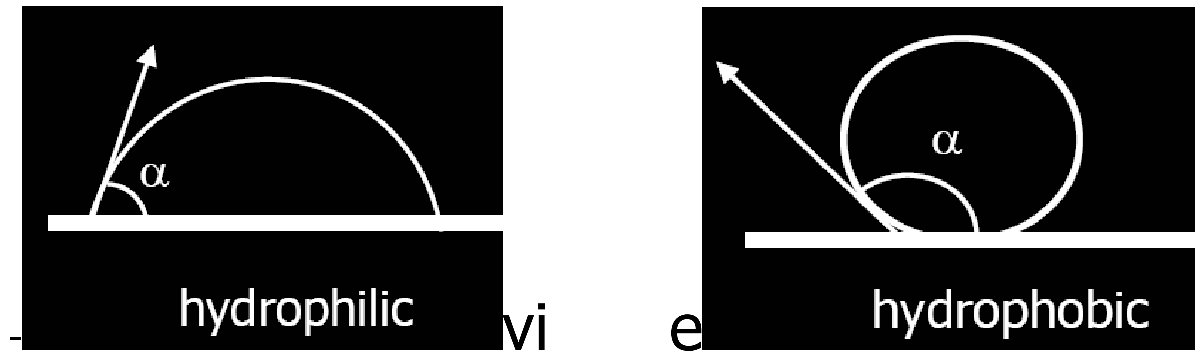
W_1 and RD_1: oven-dry weight and

radial dimension of veneer after curing (before conditioning);

W_2 and RD_2: weight and radial dimension of veneer after conditioning.

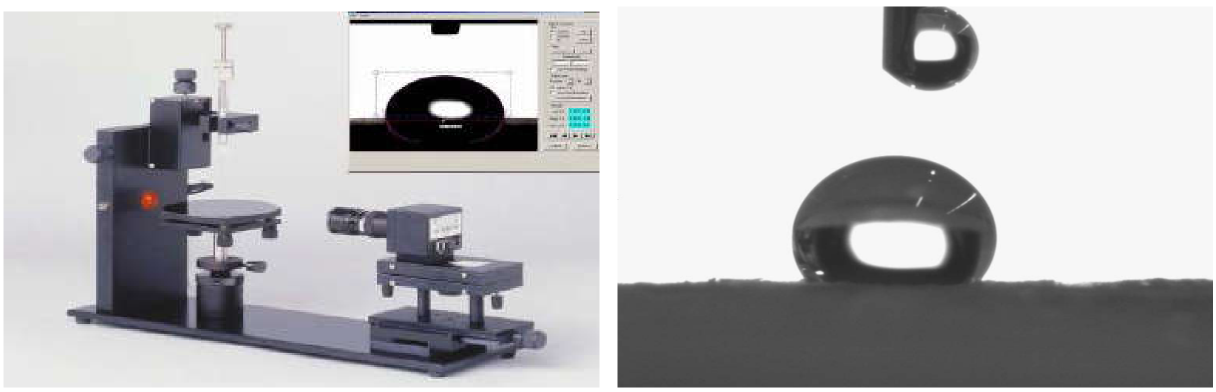
2.7. Contact angle measurement

Wettability of wood surface can be determined through contact angle measurement (Deng and Abazeri, 1998; Jozef K, 2014). In this study, ten veneers from each treatment were conditioned in a climate chamber at 65% RH and 20°C until the veneers reached EMC before measuring contact angle of the control, treated and untreated veneer surface. Contact angle between a distilled water drop and veneer surface was criterion of the measurement, point of view was horizontal. The greater the angle, the more hydrophobic surface and less wettability.



a) *b)*

Figure 2. Contact angle of a surface



a) Contact angle measurement instrument *b) A distilled water drop on a veneer surface*

Figure 3. Distilled water drop on a veneer surface

Drop Shape Analyzer, a high-quality system solution for analysis of wetting and adhesion on solid surfaces was used in this study with the contact angle measurement instrument at HAWK University of Applied Science and Arts in Germany. Three distilled water drops were applied per veneer (drop 0, drop 1 and drop 2). Drop volume: 11µl; rate: 250 µl/min; exposure duration: 20 s; number of pictures per drop: 500; 1 picture/0.04s, contact angle is calculated automatically.

III. RESULTS AND DISCUSSION

3.1. Water repellence effectiveness (WRE)

In general, as can be seen in Figure 5 - 7, WREs of the first submersion were lower than those of the second, third and fourth submersion, especially in cases of dispersion treated veneers. This characteristic can be explained by the following reasons:

Firstly, water uptake of the control veneers increased from the first to the last submersion (Figure 4) due to the formation of micro cracks on the veneer surfaces caused by wetting-drying cycles. Therefore, the relative value WREs became higher with subsequent cycles.

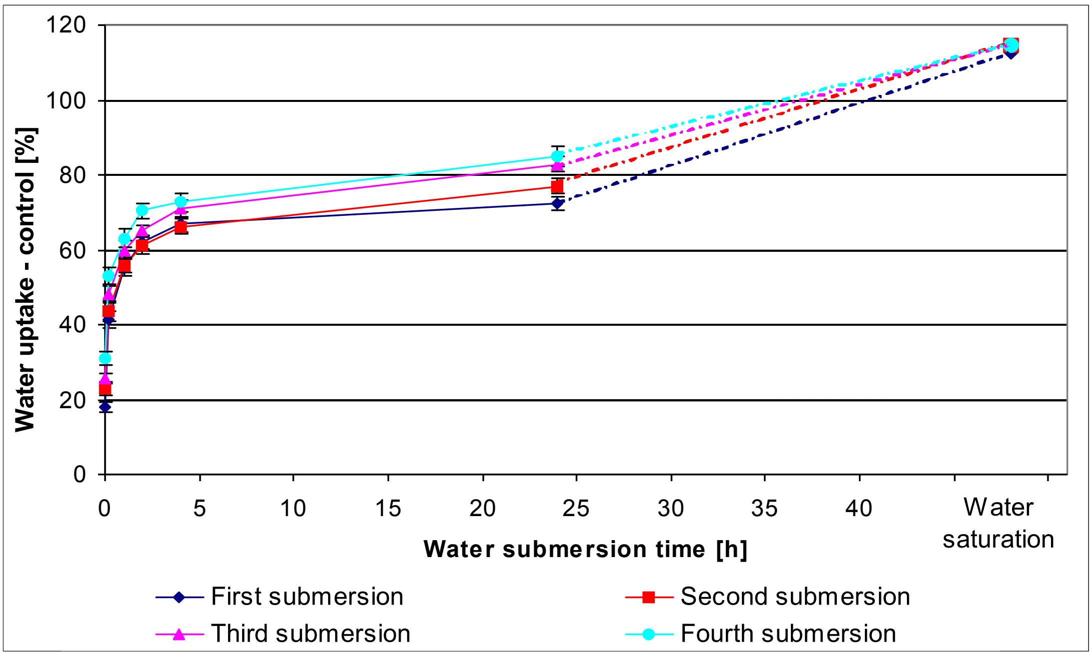


Figure 4. Water uptake of the control veneers

Secondly, wetting-drying cycles are supposed to encourage the process of ongoing polymerization which leads the completion of condensation and thus improves water repellent effect (Donath, 2005; Lukowsky et al., 1997; Mai and Militz, 2004).

As can be seen in Figure 5 - 7, WREs of the treated veneers within each submersion were high (up to 80%) in the initial submersion phases of 1 min, 10 min, 1 h, 2 h, 4 h and then lessened gradually in the submersion phases of 24 h. As expected, after vacuum support and soaking in water overnight (24 h), WREs (at water saturation) got the lowest values. Within the first 4 h of submersion, water uptake occurred rapidly with the control veneers (Figure 4) because water penetrated easily into empty voids, while water uptake occurred slowly with the treated veneers 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 4 h 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 AKD (5.0%) to fatty acid modified N-methylol compound mNMM-2 (19.3%), 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.

Alkyl ketene dimer (AKD treatment)

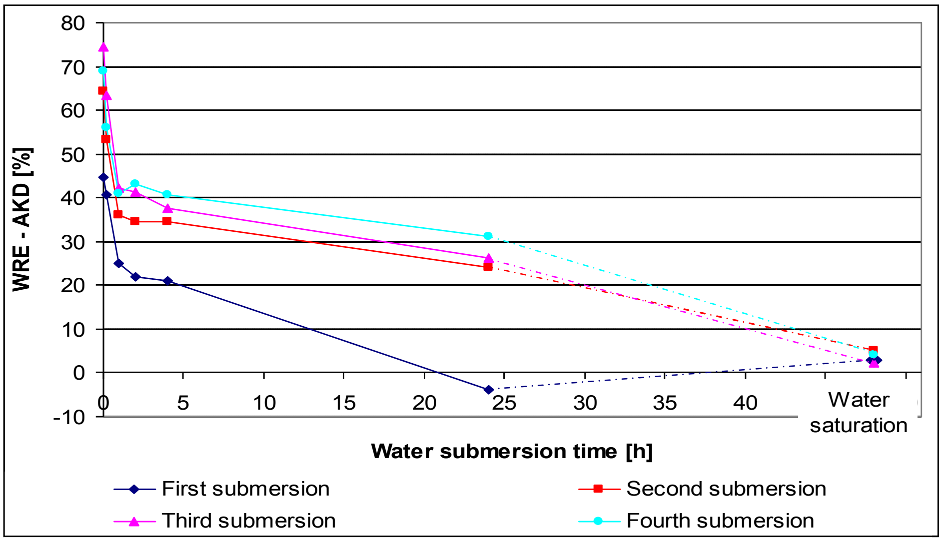


Figure 5. Water repellent effectiveness of AKD treated veneers

Alkyl ketene dimer (AKD) is used in paper industry as an internal sizing agent. Hydrophobic effect of AKD is attributed to an esterification with hydroxyl groups of wood fiber and subsequent orientation of hydrocarbon chains (Hubbe, 2006; Hundhausen et al., 2009). However, AKD is unlikely to penetrate into the cell wall because its dispersion size is about 1 µm (Neimo, 1999) whereas micro void diameter in the cell wall does not exceed 2 nm (Hill and Papadopoulos, 2001). As a result, AKD may only deposit in the cell lumen and modify surfaces of the cell lumen, not modify the cell wall.

The WREs of the first submersion were significantly lower than those of the latter submersions (Figure 5), this can be explained by the leaching of cationic starch which is used as a stabilizer (emulsifier) and has hydrophilic effect. The WREs at water saturation of four times of soaking were almost equal (2.4 - 5%), meaning that there were only small amounts of AKD emulsion permanently deposited in the cell lumen. High values of WRE in the initial phases of the submersions may be due to the fact that AKD particles form a hydrophobic surface coating on the treated veneers as the findings from Suttie et al. (1998).

N-methylol melamine (NMM-1 treatment)

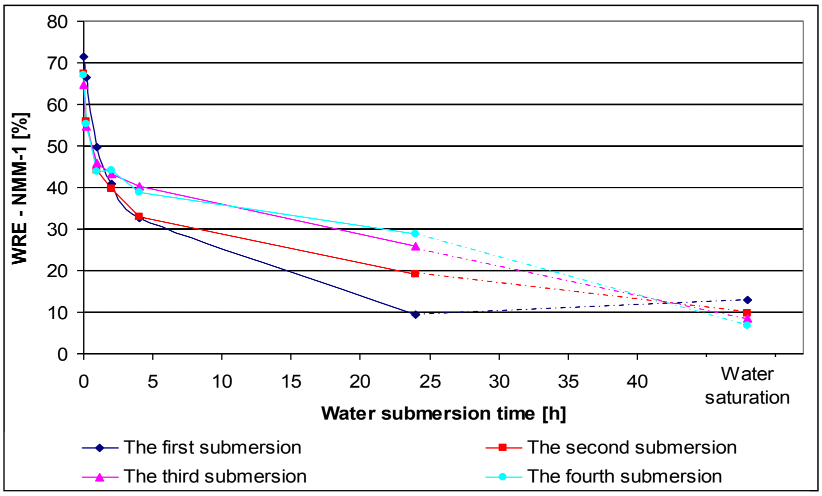


Figure 6. Water repellent effectiveness of NMM-1 treated veneers

As can be seen in Figure 6, unlike the other treatments, the treatments with NMM-1 resins showed WREs of the first submersion not lower than those of the latter times, except at 24 h of soaking. The WREs at water saturation reduced gradually from the first submersion (14%) to the last one (7%), this can be explained by cracks and leaching of NMM-1 in the treated veneers through the wetting-drying cycles.

Penetration and deposition of NMM resin into the cell lumen and cell wall were reported by many scientists (Gindl et al., 2004; Gindl et al., 2003; Rapp et al., 1999). In addition, it is believed that NMM molecules form 3-D dimensional network to provide mechanical fixation in the cell walls (Lukowsky, 1999) although there was an evidence for covalent bonds between melamine resin and wood components given by Troughto and Chow (1968). Because of these reasons, water flow and water vapor diffusion are impeded both in the cell wall and the cell lumen of the treated veneers. WRE and dimensional stability were achieved quite high in the experiments of the wood impregnated with melamine resins (Deka and Saikia, 2000; Inoue et al., 1993).

Fatty acid modified N-methylol melamine/paraffin (mNMM-2 treatment)

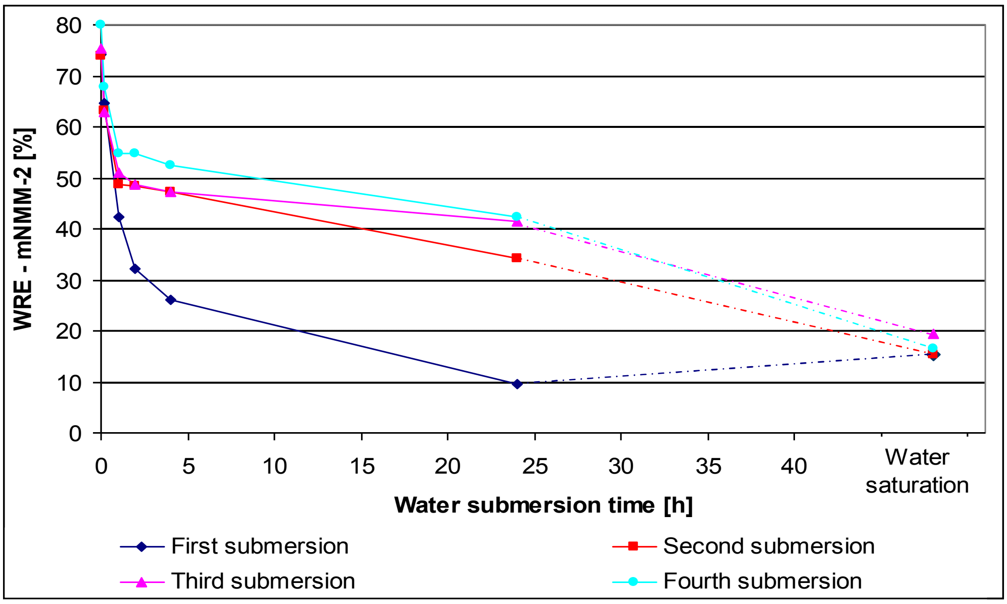


Figure 7. Water repellent effectiveness of mNMM-2 treated veneers (catalyst RB)

As depicted in Figure 7, water repellence of mNMM-2 treated veneers was very high from the second submersion like AKD treatments. The WREs at water saturation state reached approx. 20%, much higher than those of the other treatments.

High WREs of mNMM-2 treatment can be explained by the location in the cell lumen of the polymerized chemicals which cause less free space for water in capillary and close penetration paths of water. Besides, paraffin proportion in these mNMM compounds also resulted in considerable hydrophobic effect. In addition, it is assumed that some active ingredients of mNMM compounds could penetrate into the cell wall and cause micro-pore blocking and undergo chemical reaction with hydroxyl groups of the cell wall (Nguyen et al., 2007).

3.2. Sorption behavior at different relative humidity

Wood is a hygroscopic material due to hydroxyl groups of cell wall polymers, especially cellulose. When dry-wood is exposed to a humid environment, the wood absorbs water vapor until its moisture content becomes sufficient high to be equilibrium with the ambient atmosphere. This moisture content, namely equilibrium moisture content (EMC_R), is approximately proportional to the ambient relative humidity (RH) and temperature (Skaar, C., 1998). As water vapor enters the wood cell wall, the space in the cell wall is occupied, causing increase in dimension of the cell wall, as a result, the wood swells (Hill, 2006).

With regard to wood modification, EMC_R and swelling/shrinking of the treated wood in a specific humid environment depend on cell wall bulking and covalent bonds of the reagent with hydroxyl groups of the wood cell wall (Rowell and Banks, 1985). Moreover, there is a direct relationship between the decrease in EMC_R and the increase in resistance against fungal attack as discussed by Rowell (2005) and Hill (2002). Therefore, the reduction in EMC_R and swelling/shrinking is one of the aims of wood modification.

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

No	Treatment	Equilibrium moisture content EMC_R (%)			Radial swelling RS (%)		
		30% RH	65% RH	90% RH	30% RH	65% RH	90% RH
1	Control	4.62	13.60	18.42	0.84	2.79	3.74
	STDEV	*0.13*	*0.17*	*0.29*	*0.04*	*0.09*	*0.04*
2	AKD	4.13	13.55	18.40	0.80	2.77	3.79
	STDEV	*0.16*	*0.15*	*0.29*	*0.04*	*0.09*	*0.10*
3	NMM-1	4.10	12.37	16.72	0.61	2.27	3.09
	STDEV	*0.20*	*0.30*	*0.33*	*0.05*	*0.10*	*0.15*
4	mNMM-2	3.69	12.64	17.01	0.70	2.52	3.40
	STDEV	*0.30*	*0.48*	*0.46*	*0.03*	*0.12*	*0.13*

At 30, 65, 90% relative humidity (RH) and 20°C, all the treated veneers (except AKD treatments) showed slight reduction in EMC_R and radial swelling (RS) compared to the control veneers (Table 2). Thus, the chemicals more or less penetrated into the cell wall, deposited in micro voids, created covalent bonds or cross-linking with hydroxyl groups of the cell wall, these were reflected by radial bulking effect (RBE) as well.

AKD treated veneers could not impede water sorption in the cell wall after a long exposure in humidity climate. EMC_R and RS of AKD and paraffin treated veneers were equal or slightly higher as compared to those of the control veneers. It is assumed that hydrophobic properties of AKD treated wood results from the esterification of AKD molecules with hydroxyl groups of wood cellulose and the orientation of alkyl chains due to heat effect. However, large AKD particles could not penetrate and modify the cell wall; this was reflected by no cell wall bulking. AKD could only form a hydrophobic layer onto external and internal cell lumen surfaces. Consequently, AKD dispersion could provide good water repellence in initial phases of water submersion but not reduce EMC_R and RS in humid environment for the treated veneers.

N-methylol melamine (NMM) molecules are believed to form three dimension network in the cell wall (Lukowsky, 1999), hence micro voids in the cell wall could be filled with

NMM resins. The quantity of NMM resin in the cell walls mostly depends on molecular weight, degree of methylolation, solution concentration and method of impregnation (Hansmann et al., 2006), the higher amount of NMM in the cell walls, the more reduction in EMC_R and RS.

The NMM-1 treated veneers exhibited around 1.7% lower in EMC_R and 0.6% lower in RS compared to those of the control veneers at 90% RH and 20°C. Due to the deposition of NMM-1 resin into the cell wall (with cell wall bulking), the empty space of micro voids is believed to be smaller; the ability for adsorption of water vapor was lessened, and, as a consequence, the EMC_R and RS were reduced.

The fatty acid modified N-methylol melamine (mNMM-2) treated veneers caused moderate reduction in EMC_R (1 - 1.4%) and RS (0.3 - 0.4%) in comparison to the control veneers. This can be explained with the fact that a part of mNMM dispersions may penetrate into the cell wall; block micro voids and create covalent bonds or cross linking with hydroxyl groups of the wood cell wall.

3.3. Contact angle

The values of contact angle measured at the phase boundary between wood and liquid standard provide an issue point for the study of thermodynamic properties of wood surface. The values of this angle are used to calculate surface free energy and reflect wettability of wood surface (Jozep, 2014).

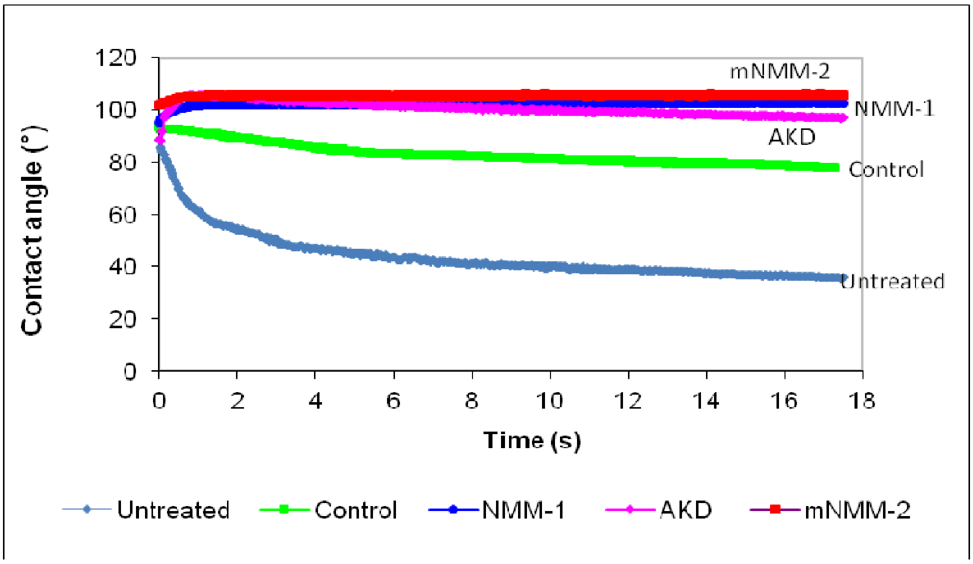


Figure 8. Contact angle of the control, untreated and AKD, NMM treated veneer

As can be seen in the Figure 8, contact angle of the untreated veneers reduced very fast in the first 5 seconds, and then reduced slowly to about 35° after 17 seconds. Congtact angle of the control veneers was much higher than that of untreated veneers because hot press curing made veneers become more hydrophobic. Contact angle values of AKD, NMM-1 and mNMM-2 treated veneers were almost consistant during the first 20 seconds after water drop was applied on veneer surface; and showed insignificant difference. Therefore, wettability of AKD, NMM-1 and mNMM-2 treated veneers presented much lower than the untreated and control veneers.

IV. CONCLUSIONS

Beech veneers were impregnated with 10% solid content of the AKD, NMM-1 and mNMM-2 chemicals. 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. The cycle 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. Consequently, it can be stated that, the water repellence of the treated veneers was stable after the wetting-drying cycles.

Sorption behavior is related to the properties of modified cell wall, such as bulking and covalent bonds/cross linking. The chemicals such as NMM-1, mNMM-2 could react with hydroxyl groups or deposit into the cell wall induced the reduction in EMC_R and RS for the treated veneers, while large AKD particles could not penetrate and modify the cell wall therefore did not reduce EMC_R and RS in humid environment for the treated veneers. The values of contact angle which reflect wettability were in accordance with the water repellence and equilibrium moisture content of the treated veneer.

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TÍNH HÚT NƯỚC, HÚT ẨM VÀ THẤM ƯỚT BỀ MẶT CỦA VÁN MỎNG GỖ BEECH (*Fagus sylvatica* L.) BIẾN TÍNH VỚI CÁC HỢP CHẤT CÓ CHỨA N-METHYLOL MELAMINE VÀ ALKYL KETENE DIMER

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TÓM TẮT

Kết quả xử lý ván mỏng gỗ Beech với các hóa chất từ công nghiệp giấy (*alkyl ketene dimer,* AKD), công nghiệp gỗ (*N-methylol melamine*, NMM-1) và công nghiệp dệt (*fatty acid modified N-methylol melamine*, mNMM-2) ở hàm lượng rắn 10% cho thấy khả năng chống hút nước trong giai đoạn đầu khá cao; Sau đó giảm đáng kể từ 4 giờ sau khi ngâm đến khi ván đạt trạng thái bão hòa nước. Khả năng chống hút nước ở trạng thái bão hòa nước thay đổi từ 5% (AKD) đến 19,3% (mNMM-2), do đó có một lượng hóa chất nhất định tích tụ trong ruột tế bào và vách tế bào của ván mỏng được xử lý với các hóa chất trên. Khả năng chống hút nước của ván mỏng được xử lý hóa chất ổn định qua các chu kỳ ngâm nước – sấy khô. Hóa chất NMM-1 và mNMM-2 có khả năng phản ứng với nhóm hydroxyl hoặc tích tụ trong vách tế bào làm giảm độ ẩm thăng bằng và tỷ lệ trương nở theo phương xuyên tâm của ván mỏng biến tính, trong khi đó các hạt AKD có kích thước lớn không thể di chuyển vào vách tế bào để biến tính do đó không làm giảm độ ẩm thăng bằng và tỷ lệ trương nở theo phương xuyên tâm. Khả năng thấm ướt của ván mỏng xử lý với AKD, NMM-1 và mNMM-2 thấp hơn nhiều so với ván mỏng đối chứng và ván không xử lý.

Từ khoá: Độ ẩm thăng bằng, góc tiếp xúc, gỗ Beech, khả năng chống hút nước, tính thấm ướt, ván mỏng.

Received : 14/9/2017
Revised : 02/10/2017
Accepted : 10/10/2017