

EFFECT OF CATALYSTS ON WATER AND MOISTURE RELATED PROPERTIES OF BEECH VENEER MODIFIED WITH N-METHYLOL MELAMINE COMPOUNDS

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SUMMARY

The treatments with N-methylol compounds often need to be aided by a catalyst to enhance the condensation reaction with wood polymers and reduce time and temperature of curing process. In this study, two fatty acid modified N-methylol melamine compounds combined with magnesium and aluminium salt catalysts were investigated on water/moisture related properties of the treated beech (*Fagus sylvatica*.L) veneers. The result showed that: After water soxhlet extraction, mNMM treated veneers displayed higher water repellence effectiveness (WRE) and anti-shrinkage efficiency (ASE) in radial direction with the presence of the catalyst $MgCl_2$ or RB of at least 1.5% concentration (equal to 5% based on stock solution in 30% stock concentration solution of mNMM compounds). This can be explained by: the catalysts could result in better hydrophobic effect for the treatments with mNMM compounds through locking of hydroxyl groups or incorporation of the chemicals in the wood cell wall and the cell lumen. The combination of mNMM compounds (mNMM-1 or mNMM-2) and catalyst $MgCl_2$ induced an increase in equilibrium moisture content (EMC_R) and radial swelling (RS) of the treated veneers compared to the control veneers. With increasing concentration of $MgCl_2$ from 1.5 to 4.5%, the EMC and RS were increased because the moisture adsorption of $MgCl_2$ enhanced hygroscopic property of the treated veneers. In contrast, EMC_R and RS of modified veneers with a combination of mNMM compounds and catalyst RB were lower than the controls and did not depend on the concentration of catalyst RB.

Keywords: Aluminium salt, magnesium chlorua, modification, N-methylol melamine, radial swelling, veneer, water uptake.

I. INTRODUCTION

N-methylol chemistry is commonly used for cross-linking of cellulose in the textile industry. Fatty acid N-methylol compounds are derivative products of N-methylol compounds connected with long hydro carbon chains. An example of a N-methylol compound is 1,3-dimethylol-4,5-dihydroxy ethylene urea (DMDHEU), with the presence of an acid catalyst, DMDHEU molecules can react with hydroxyl groups of the cellulose to form cross-links; and they may also react with themselves or with reactive $-NH-$ groups (Petersen, 1968). Militz (1993) treated beech wood with DMDHEU and different catalysts; the treatments reduced shrinking and swelling to approx. 50%. Xie (2005) reported that the treatment of wood veneers with DMDHEU and 1,5% catalyst $MgCl_2$ to high WPG (48%) could stabilize lignin to some extents; and at

the condition of natural weathering, this treatment reduced erosion, discoloration and fungal growth on weathered wood surfaces. Tensile strength of pine veneer treated with DMDHEU 30% and catalyst $MgCl_2$ 1,5% was significantly reduced (50-70%) due to hemicellulose degradation in the cell wall (Xie *et al*, 2007). The treatment of solid wood with a fatty acid modified N-methylol melamine/paraffin formulation (mNMM) using an aluminum salt as catalyst to a WPG of 10-14% resulted in significant decrease (40-50%) in water uptake in a submersion test as compared to that of the untreated wood (Nguyen *et al.*, 2007). Trinh.H.M (2012) informed that outside weathering resistance and water/moisture resistance of modified plywood produced from veneers treated with N-methylol melamine and catalyst (aluminum salt) was remarkably improved in comparison

to control plywood. The treatments with N-methylol compounds often need to be aided by a catalyst to enhance the condensation reaction with wood polymers and reduce time and temperature of curing process (Krause et al., 2003; Kullman and Reinhardt, 1978).

In this paper, two fatty acid modified N-methylol melamine compounds combined with magnesium and aluminium catalysts were investigated on water/moisture related properties of the treated veneers, such as water repellence and radial dimensional stability, equilibrium moisture content and radial swelling.

II. MATERIALS AND METHODS

2.1. Materials

2.1.1. Fatty acid modified N-methylol melamine compounds (mNMM) and magnesium and aluminium salt catalysts

Persistol HP (mNMM-1) delivered by BASF, is a fatty acid derivative of N-methylol melamine compound containing paraffin. mNMM-1 is a white dispersion with pH value from 4-6 at 20°C. mNMM-1 is used as a water repellent finishing agent for textiles composed of cellulosic fibers, synthetic fibers and their blends, and gives them a full and soft handle. mNMM-1 can be infinitely diluted with cold water and applied by padding at room temperature, then pre-drying and curing for 4

min at 150°C for fabrics. To improve the water repellence effect, zinc nitrate, zinc chloride or aluminum sulfate should be used as catalysts for the curing process (BASF, 1984; BASF, 2000a).

Phobotex VFN (mNMM-2) delivered by BASF, is a fatty acid of 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 (an 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 drying at 120-140°C and curing for 2 min at 160°C or 4-5 min at 150°C (Ciba, 2002).

Salts of magnesium and aluminium were recognized as particularly effective for cross linking in textiles (Andrews and Frick, 1978; Kullman and Reinhardt, 1978). Thus, MgCl₂ and an aluminum salt (RB) were chosen as catalysts for this study.

2.1.2. Veneers and chemical preparation

Beech (*Fagus sylvatica* L.) sliced veneers without heartwood were cut in sizes of 40 (25) × 0.5 × 50 mm³ (rad × tang × long). The quantity of veneer specimens is listed in table 1:

Table 1. Quantity of veneers for each treatment

No	Experiments	Number of veneers	
		25 × 0.5 × 50 mm ³	40 × 0.5 × 50 mm ³
1	EMC _R , radial swelling	10	0
2	Water extraction	0	10
3	Water submersion	0	10
Total veneers per treatment		10	20

The fatty acid modified N-methylol melamine compounds mNMM-1 or mNMM-2 used at 30% stock concentration were combined with catalysts MgCl₂.6 H₂O or RB applied at

different concentrations: 0; 1.5%; 3.0%; and 4.5% (equal to 0%, 5%, 10% and 15% (w/w) of the mNMM-1 or mNMM-2 stock solutions in their 30% stock concentration solutions).

2.2. Experiments

2.2.1. Veneer treatment

The beech veneers were oven-dried at $103 \pm 2^\circ\text{C}$ 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 a dial 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 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 veneer storing in the solutions at atmospheric pressure. 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 curing were recorded. The treated veneers were proceeded with water/moisture related experiments as described in figure 1.

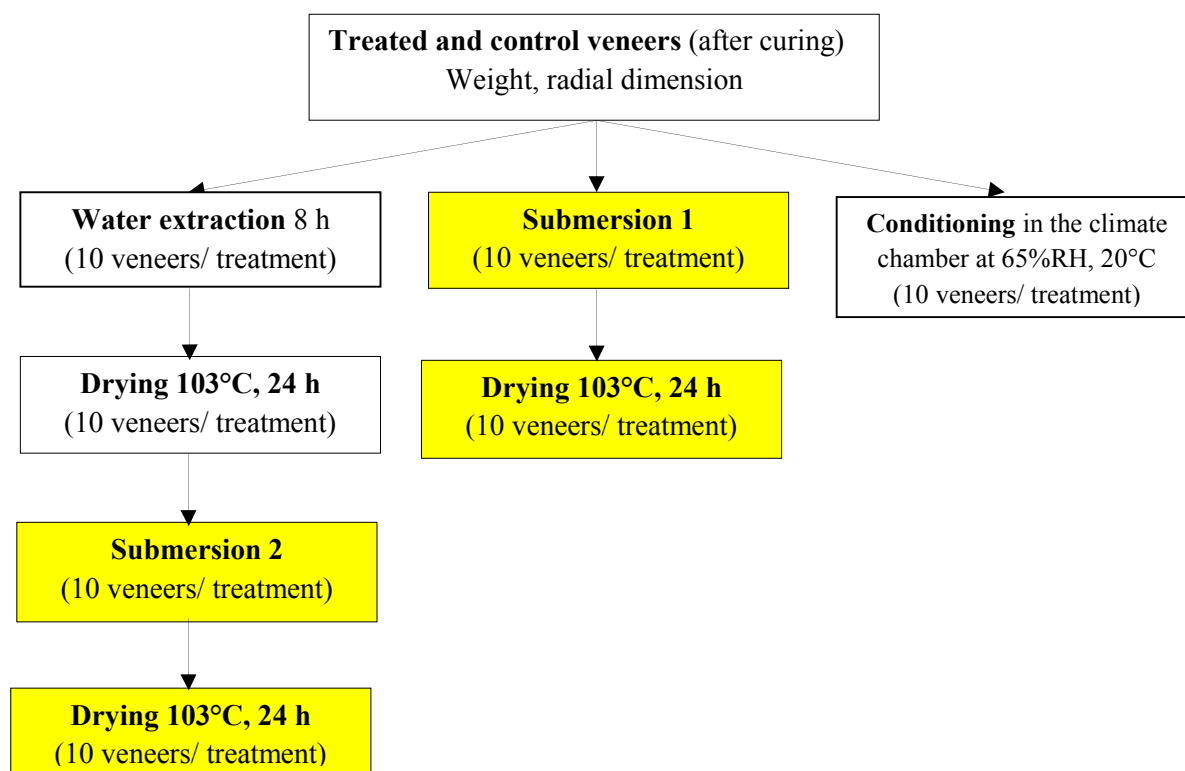


Figure 1. Test procedure of the veneers

2.2.2. Water extraction

Ten veneers ($40 \times 0.5 \times 50 \text{ mm}^3$) from each treatment were placed in a soxhlet apparatus; the veneers were fixed by some small glass marbles during the extraction process. From 1 to 6 soxhlet apparatuses could be operated at the same time. Each round bottom flask was filled with 250 ml distilled water. Few boiling stones were inserted in the round bottom flasks for heating absorption. The time to reaching

the boiling temperature and cooling down was each 30 min. The total running time of the extraction was 8 h constantly. After the water extraction and cooling down the system, the veneers were taken out of the soxhlet apparatuses and oven-dried, then continued with submersion 2 as in figure 2.

2.2.3. Water submersion

Water repellent characteristic was evaluated through water submersion tests. For each

submersion (1 or 2), 10 veneers ($40 \times 0.5 \times 50 \text{ mm}^3$) 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 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:

$$WU(\%) = \frac{(W_2 - W_1)}{W_o} \times 100 \quad (\text{Equation 1})$$

Where: WU: water uptake; W_2 : veneer weight after water submersion (1 min, 10 min, 1 h, 2 h, etc); W_1 : veneer weight before water submersion; W_o : oven-dry weight of veneer before chemical 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; Lukowsky et al., 1997; Rowell and Banks, 1985)

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

Where: WRE: water repellent effectiveness; $WU_{control}$: water uptake of control veneer; $WU_{treated}$: water uptake of treated veneer

Maximum radial swelling (RS_m) and anti-swelling efficiency in radial direction (ASE) were calculated according to equation 3 and Equation 4, respectively.

$$RS_m(\%) = \frac{(RD_2 - RD_1)}{RD_o} \times 100 \quad (\text{Equation 3})$$

Where: RS_m : maximum radial swelling; RD_o : radial dimension of oven-dry veneer before impregnation; RD_1 : radial dimension of oven-dry veneer before submersion; RD_2 :

radial dimension of veneer at water saturation.

$$ASE(\%) = \frac{(RS_{mcontrol} - RS_{mtreated})}{RS_{mcontrol}} \times 100 \quad (\text{Equation 4})$$

Where: ASE: anti-swelling efficiency in radial direction; $RS_{mcontrol}$: maximum radial swelling of control veneer; $RS_{mtreated}$: maximum radial swelling of treated veneer;

The submersion 1 was started with the oven-dry veneers after curing and the submersion 2 was started with the oven-dry veneers after water extraction as in figure 1.

2.2.4. Sorption behavior

Sorption behavior was evaluated with the veneers after curing. Ten veneers ($25 \times 0.5 \times 50 \text{ mm}^3$) 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 5,6 (Hill, 2006):

$$EMC_R(\%) = \frac{(W_3 - W_1)}{W_o} \times 100 \quad (\text{Equation 5})$$

$$RS = \frac{(RD_3 - RD_1)}{RD_o} \times 100 \quad (\text{Equation 6})$$

Where: EMC_R and RS: equilibrium moisture content and radial swelling of veneer; W_o and RD_o : oven-dry weight and radial dimension of veneer before impregnation; W_1 and RD_1 : oven-dry weight and radial dimension of veneer after curing (before conditioning); W_3 and RD_3 : weight and radial dimension of veneer after conditioning.

III. RESULTS AND DISCUSSION

3.1. Water repellent effectiveness and radial dimensional stability

3.1.1. Water repellent effectiveness (WRE)

The veneers treated with mNMM compounds and catalyst MgCl₂ or RB resulted in almost similar WREs in both cases: mNMM compound was mNMM-1 or mNMM-2 (Figure 2, 3, 4, 5). However, there was an obvious difference concerning catalyst MgCl₂ and RB. During the first 24 h of the submersion, the treatments of mNMM

compounds and catalyst MgCl₂ induced very low WRE for the veneers after the curing (the submersion 1, figure 2 and 3 A), but WRE was significantly improved for the veneers after water soxhlet (the submersion 2, Figure 2 and 3 B). The same phenomenon occurred with the treatments of mNMM compounds and catalyst RB but was less pronounced (figure 4 and 5). This can be explained with the typical hygroscopic property of MgCl₂ and leaching of hygroscopic emulsifiers in the treated veneers due to water extraction.

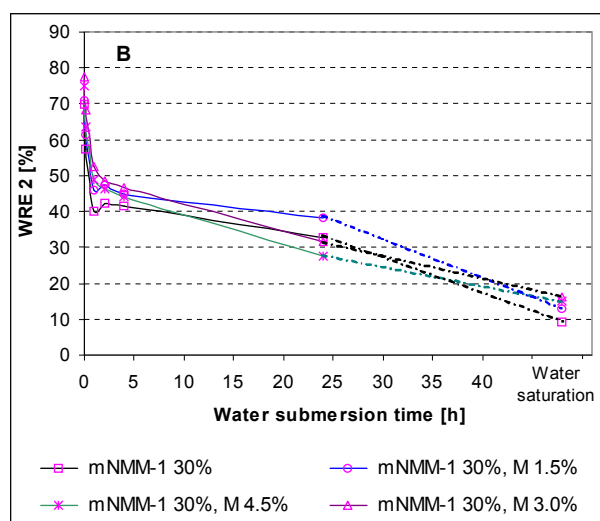
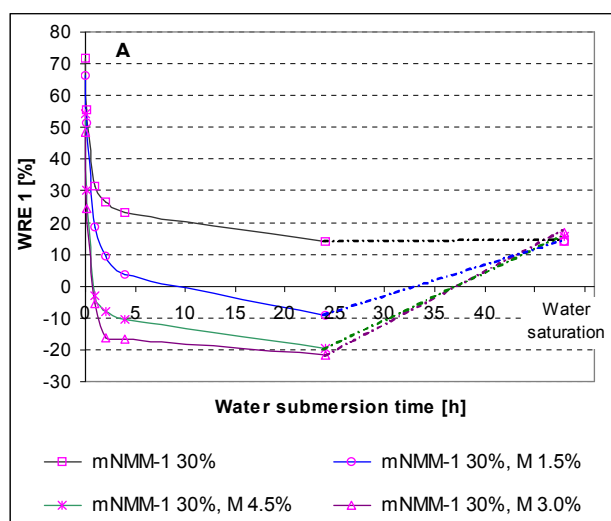


Figure 2. Water repellent effectiveness of the veneers treated with mNMM-1 (30% stock concentration) and catalyst MgCl₂ (0%, 1.5%, 3.0% and 4.5%) A: submersion 1 (after curing); B: submersion 2 (after water soxhlet)

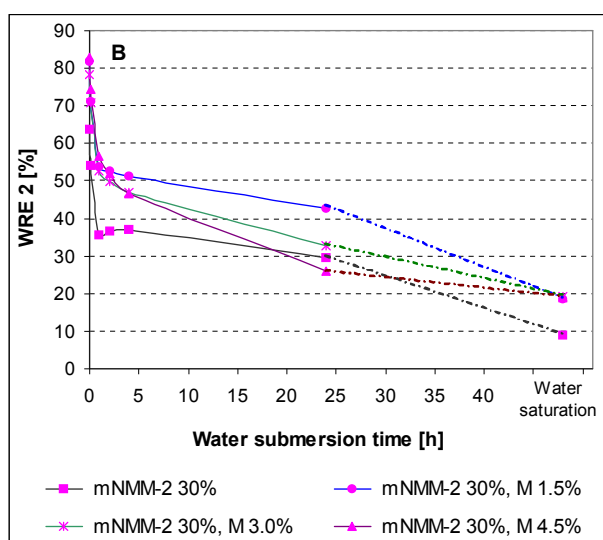
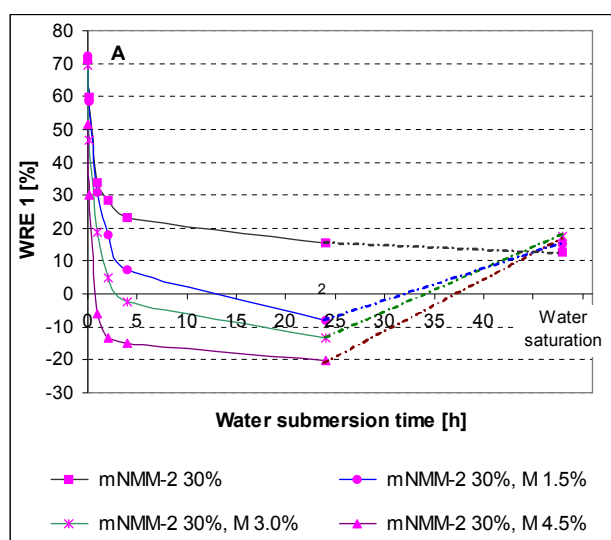


Figure 3. Water repellent effectiveness of the veneers treated with mNMM-2 (30% stock concentration) and catalyst MgCl₂ (0%, 1.5%, 3.0% and 4.5%) A: submersion 1 (after curing); B: submersion 2 (after water soxhlet)

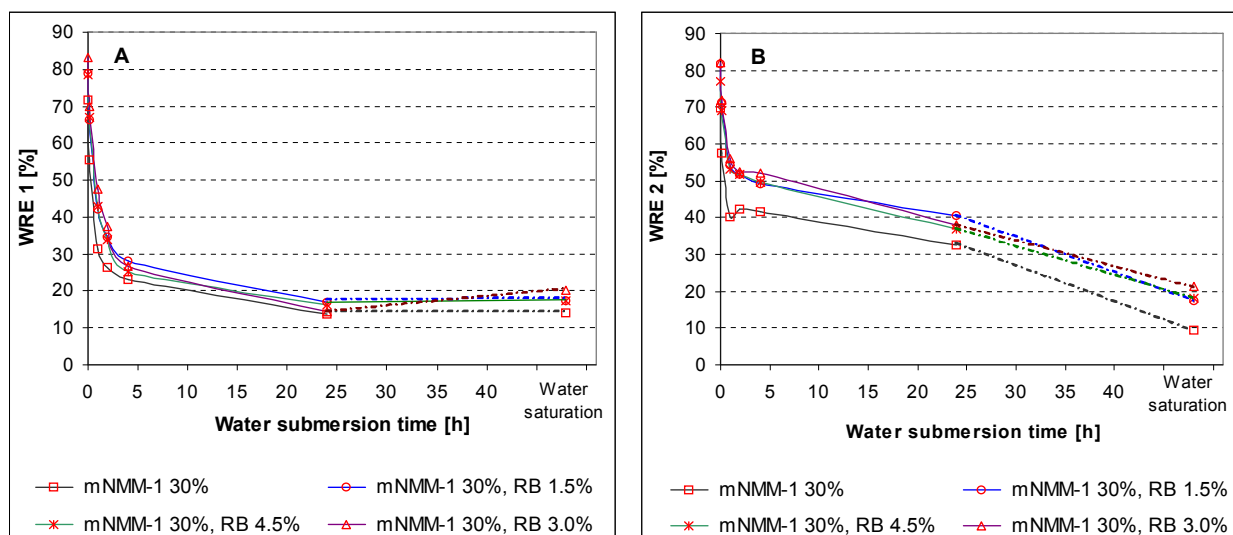


Figure 4. Water repellent effectiveness of the veneers treated with mNMM-1 (30% stock concentration) and catalyst RB (0%, 1.5%, 3.0% and 4.5%) A: submersion 1 (after curing); B: submersion 2 (after water soxhlet)

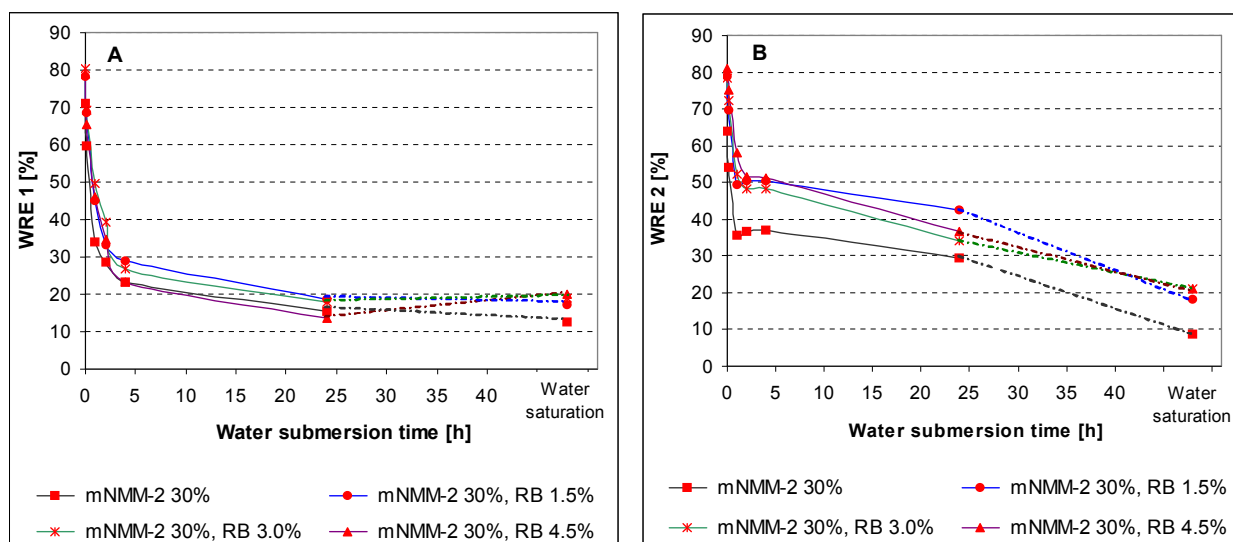


Figure 5. Water repellent effectiveness of the veneers treated with mNMM-2 (30% stock concentration) and catalyst RB (0%, 1.5%, 3.0% and 4.5%); A: submersion 1 (after curing); B: submersion 2 (after water soxhlet)

During initial phases of the submersion 1, catalyst $MgCl_2$ with hygroscopic property caused high water uptakes for the treated veneers, even higher than the control veneers (WRE get negative values). WRE of the treatments with mNMM compounds and catalyst $MgCl_2$ reduced when concentration of $MgCl_2$ increased from 0% to 4.5%, whereas the influence of the RB catalyst concentration on

the WRE was insignificant.

An influence of the concentration (1.5%, 3.0% and 4.5%) of the catalysts $MgCl_2$ or RB on the WRE in initial phases of the submersion 2 is not obvious; however, the water repellence was reduced with an absence of the catalyst. This result suggests that the catalyst concentration of at least 1.5% is required for high water repellence.

Contrary to the first 24 h of submersion, the WREs at water saturation of both submersion 1 and 2 were similar, regardless of the type of mNMM compounds or catalysts. The concentration of the catalysts had slight effect on WRE at water saturation. Especially, WRE at water saturation increased up to 21% with the presence of the catalysts, while it was only 8.7% in the absence of catalyst. Therefore, the catalysts could result in better hydrophobic effect for the treatments with mNMM compounds through locking of hydroxyl groups or incorporation of the chemicals in the wood cell wall and the cell lumen.

3.1.2. Radial dimensional stability (ASE)

Radial dimensional stability of the treated

veneers was evaluated by determining ASE in radial direction through swelling of the veneers from oven-dry moisture content to water saturation state. In general, bulking and cross-linking with hydroxyl groups of chemicals in the cell wall are the main factors which can reduce swelling/ shrinking of the treated wood and increase the ASE. Radial bulking effect (RBE) of all the treatments with a combination of mNMM compounds and catalyst $MgCl_2$ or RB indicated negative values (around -0.3%) in comparison to RBE of the control veneers (-0.9%). The bulking effect is low, therefore the cross linking with hydroxyl groups in the cell wall must be existing for an increasing of the ASE.

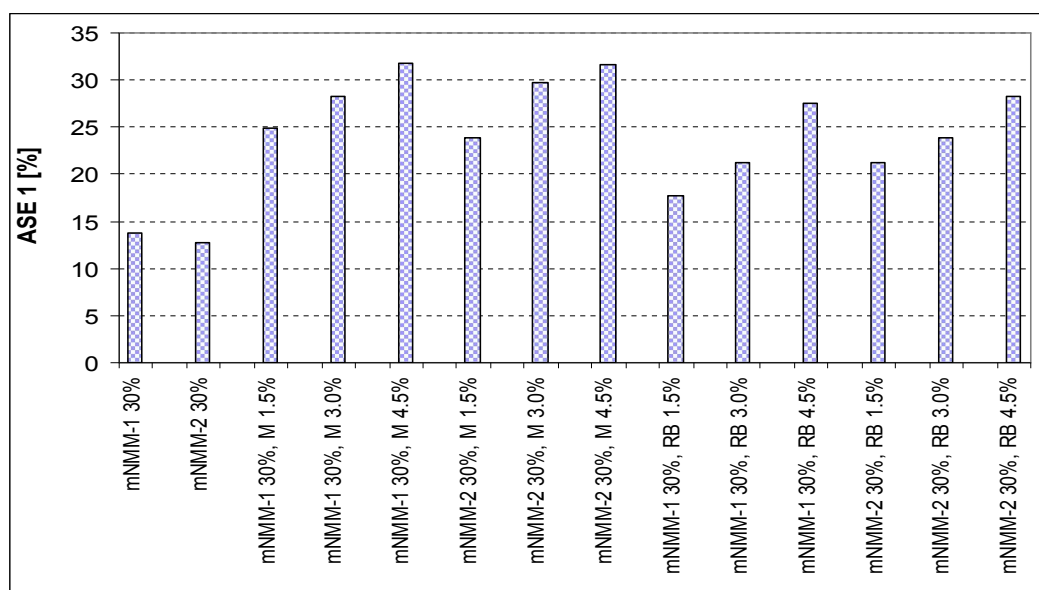


Figure 6. Anti swelling efficiency (ASE) in radial direction of the veneers treated with mNMM compounds (30% stock concentration) and catalyst $MgCl_2$ or RB (0%, 1.5%, 3.0% and 4.5%) in submersion 1

As shown in figure 6 and figure 7, the results showed only minor differences in ASE values between two mNMM compounds (mNMM-1 and mNMM-2).

The ASE in radial direction of the submersion 1 (for veneers after curing) showed moderate values (up to 32%) and depended on

catalyst concentration (figure 6). The ASE increased slightly with increasing concentration of the catalysts. In contrast, the treatments without the presence of catalyst brought about significantly lower ASE. The similar results were observed for pine sapwood treated with 20% DMDHEU and different

concentrations of catalyst $MgCl_2$ (Van der Zee *et al.*, 1998). However, the ASE of mNMM treatments comparable to DMDHEU treated solid wood is rather low. In case of pine sapwood treated with DMDHEU and catalyst $MgCl_2$ to 25% WPG, the maximum ASE in volume can be improved up to 75% (Krause *et al.*, 2003). This can be explained by lower

WPG of mNMM treatments in this study and less penetration ability of mNMM particles into the cell wall which inhibits bulking and cross linking with cell wall polymers.

The treatments of mNMM compounds with catalyst $MgCl_2$ resulted in higher ASE than with catalyst RB (4-7%) in the submersion 1 (figure 6).

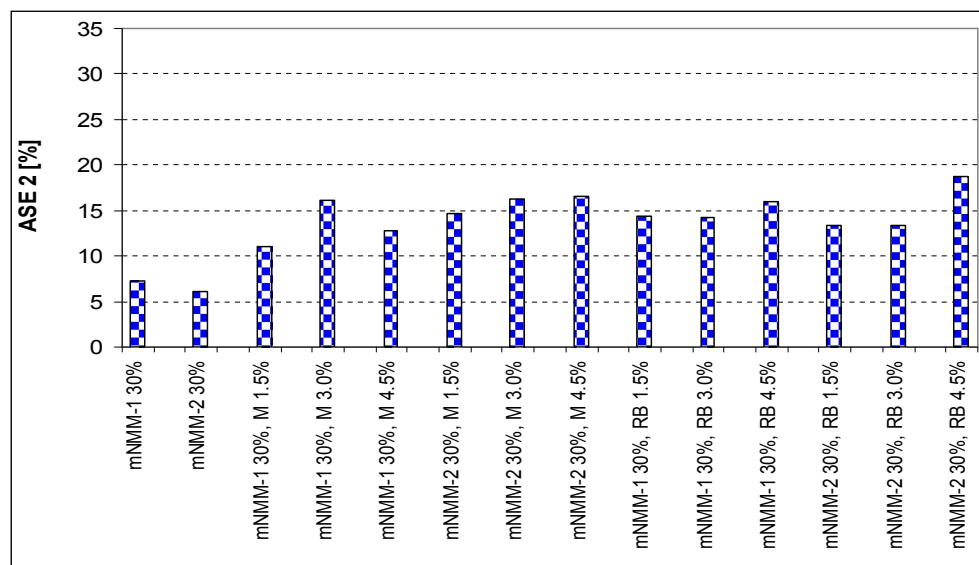


Figure 7. Anti swelling efficiency (ASE) in radial direction of the veneers treated with mNMM compounds (30% stock concentration) and catalyst $MgCl_2$ or RB (0%, 1.5%, 3.0% and 4.5%) in submersion 2

The ASE values in radial direction of the submersion 2 (for the treated veneers after water soxhlet extraction) were much lower than those of the submersion 1 and showed only minor dependence on concentration and type of the catalysts (figure 7). However, the ASE of the treated veneers with the presence of the catalysts was clearly higher than those without catalyst. The veneers treated with 30% stock concentration mNMM-2 and 4.5% catalyst RB resulted in the highest ASE (18%) of the treated veneers after water extraction. Therefore, catalyst RB applied at 4.5% is assumed to enhance the reaction of mNMM-2 and hydroxyl groups in the wood cell wall.

3.2. Equilibrium moisture content and radial swelling at 20°C and 65% RH

The EMC of wood depends on the numbers of hydroxyl groups in the cell wall and the pore sizes in the cell wall (Skaar, 1988). As shown in fig. 8-9, EMC_R and RS of the veneers treated with a combination of mNMM compounds and catalyst $MgCl_2$ were increased when concentration of $MgCl_2$ increased irrespective of mNMM-1 or mNMM-2. The same results of wood treated with DMDHEU and the catalyst $MgCl_2$ were indicated in the studies of Krause *et al.* (2003), Militz (1993) and Xie (2005). It is believed that the moisture adsorption of $MgCl_2$ enhanced hygroscopic property of the treated veneers.

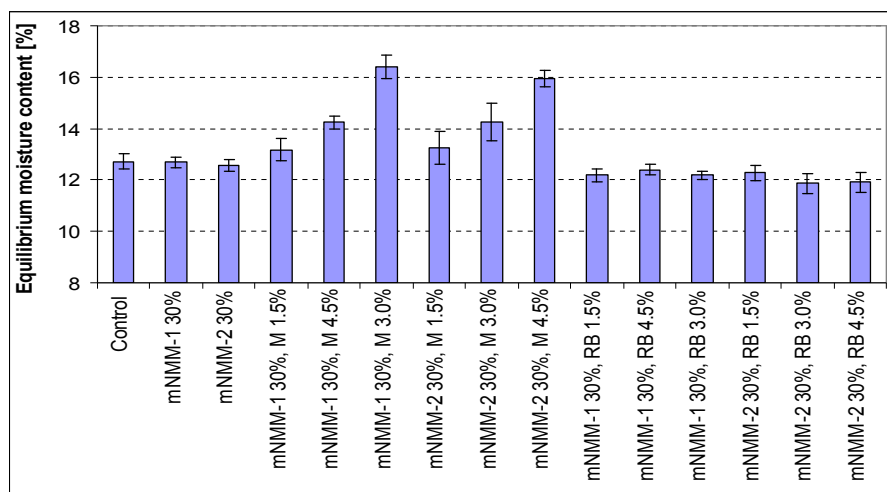


Figure 8. Equilibrium moisture content at 20°C and 65% RH of the veneers treated with mNMM compounds (30% stock concentration) and the catalysts (0%, 1.5%, 3.0% and 4.5%)

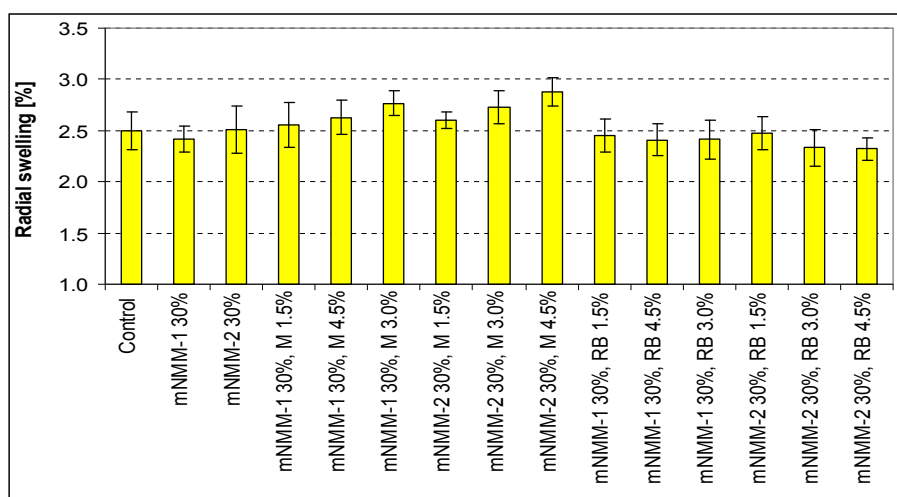


Figure 9. Radial swelling at 20°C and 65% RH of the veneers treated with mNMM compounds (30% stock concentration) and the catalysts (0%, 1.5%, 3.0% and 4.5%)

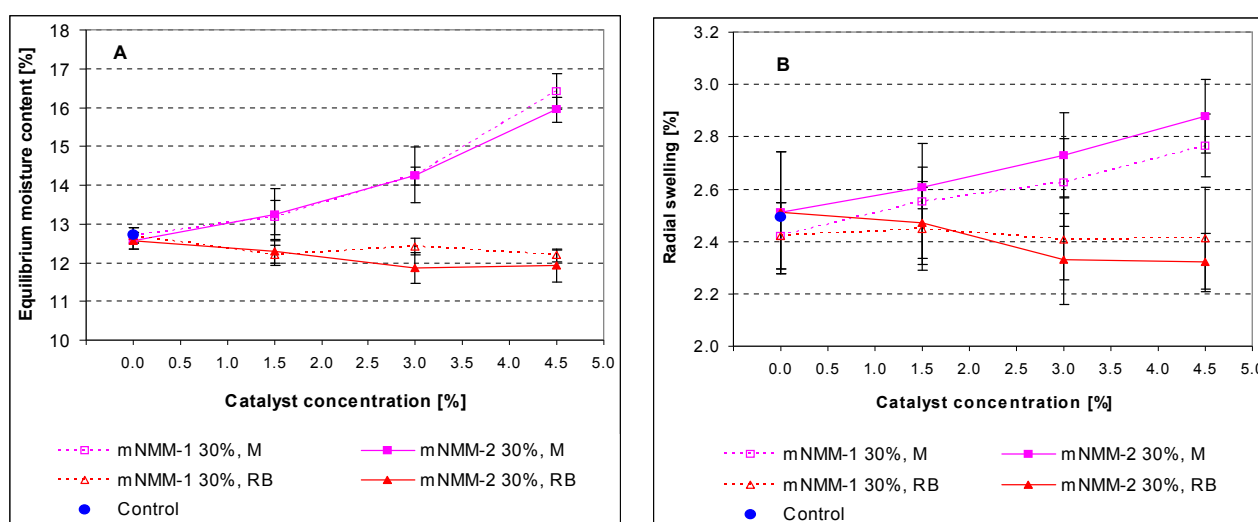


Figure 10. Equilibrium moisture content (A) and radial swelling (B) at 20°C and 65% RH of the veneers treated with mNMM compounds (30% stock concentration) and the catalysts (0%, 1.5%, 3.0% and 4.5%)

With catalyst RB, the influence of catalyst concentration on EMC_R and RS of the treated veneers was minor. However, the veneers impregnated with 30% stock concentration of mNMM-2 and 4.5% of RB showed the best improvement in EMC_R and RS compared to the controls. EMC_R and RS of modified veneers with a combination of mNMM compounds and the catalyst $MgCl_2$ was clearly higher than those of mNMM compounds and catalyst RB (figure 10). This also can be found in the veneers treated with sole catalyst $MgCl_2$ or RB.

IV. CONCLUSIONS

Water repellent effectiveness (WRE) and radial dimension stability (ASE)

After water soxhlet extraction, mNMM treated veneers displayed higher WRE and ASE in radial direction with the presence of the catalyst $MgCl_2$ or RB of at least 1.5% concentration (equal to 5% based on stock solution in 30% stock concentration solution of mNMM compounds). Therefore, the catalysts could result in better hydrophobic effect for the treatments with mNMM compounds through locking of hydroxyl groups or incorporation of the chemicals in the wood cell wall and the cell lumen.

Equilibrium moisture content (EMC_R) and radial swelling (RS) at 20°C and 65% RH

The combination of mNMM compounds (mNMM-1 or mNMM-2) and catalyst $MgCl_2$ induced an increase in EMC_R and RS of the treated veneers compared to the control veneers. With increasing concentration of

$MgCl_2$ from 1.5 to 4.5%, the EMC_R and RS were increased. In contrast, EMC_R and RS of modified veneers with a combination of mNMM compounds and catalyst RB were lower than the controls and did not depend on the concentration of catalyst RB.

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ẢNH HƯỞNG CỦA CHẤT XÚC TÁC ĐẾN TÍNH CHẤT HÚT NƯỚC VÀ HÚT ẨM CỦA VÁN MỎNG GỖ BEECH BIẾN TÍNH VỚI HỢP CHẤT CÓ CHỨA N-METHYLOL-MELAMIN

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Trường Đại học Lâm nghiệp

TÓM TẮT

Chất xúc tác thường được sử dụng khi biến tính gỗ với các hợp chất có chứa N-methylol để tăng tốc độ phản ứng giữa hóa chất và các thành phần polymer trong vách tế bào gỗ, đồng thời giảm nhiệt độ của quá trình xử lý nhiệt gỗ sau khi ngâm tẩm (curing). Trong nghiên cứu này, ván mỏng gỗ Beech (*Fagus sylvatica*.L) biến tính với 2 hóa chất có chứa axit béo của N-methylol melamine (mNMM) và chất xúc tác là muối nhôm và magie đã được kiểm tra các tính chất liên quan đến khả năng hút ẩm và hút nước. Kết quả đã cho thấy: Sau khi ngâm trong nước nóng chảy tuần hoàn 8h (soxhlet) để loại bỏ các thành phần chất chiết suất của gỗ và các hóa chất dư thừa chưa phản ứng hết, khả năng chống hút nước (WRE) và chống trương nở theo phương xuyên tâm (ASE) của ván mỏng gỗ beech biến tính với các hợp chất có chứa mNMM được cải thiện đáng kể khi sử dụng chất xúc tác với nồng độ của muối nhôm (RB) hoặc magie $MgCl_2$ từ 1.5% trở lên. Điều này cho thấy chất xúc tác đã tạo điều kiện cho các phản ứng hóa học xảy ra, làm giảm số lượng nhóm hydroxyl trong vách tế bào gỗ hoặc tăng cường sự tích tụ của hóa chất trong ruột và vách tế bào gỗ. Sự kết hợp của hợp chất có chứa mNMM và chất xúc tác $MgCl_2$ đã làm tăng độ ẩm thẳng bằng và độ trương nở theo phương xuyên tâm của ván mỏng biến tính (không qua soxhlet) so với ván đối chứng. Với nồng độ chất xúc tác $MgCl_2$ từ 1.5-4.5%, độ ẩm thẳng bằng và độ trương nở theo phương xuyên tâm của ván mỏng biến tính tăng do đặc tính hút ẩm của $MgCl_2$ dư thừa trên bề mặt ván đã làm tăng tính hút ẩm của ván mỏng biến tính. Trái lại, độ ẩm thẳng bằng và độ trương nở theo phương xuyên tâm của ván mỏng biến tính với hợp chất có chứa mNMM và chất xúc tác RB thấp hơn so với ván mỏng đối chứng và không phụ thuộc vào tỷ lệ của chất xúc tác RB.

Từ khóa: Biến tính, độ trương nở theo phương xuyên tâm, muối magie, muối nhôm, N-methylol melamine, tỷ lệ hút nước, ván mỏng.

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