WOOD RESEARCH
60 (2): 2015
201-210

DETERMINATION OF SORPTION ISOTHERMS OF SCOTS PINE (*PINUS SYLVESTRIS* L.) WOOD STRANDS LOADED WITH MELAMINE-UREA-PHENOL-FORMALDEHYDE (MUPF) RESIN

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(Received July 2013)

ABSTRACT

The purpose of this study was to determine the sorption isotherms of Scots pine (*Pinus sylvestris* L.) wood strands, taken from a previously prepared strand-adhesive mass, used for the production of a three-layer oriented strand boards (OSB). The study investigated three loading levels of Melamine-Urea-Phenol-Formaldehyde (MUPF) resin used for gluing the OSB, constituting 5, 10, and 15 % of the wood strands dry weight. The obtained experimental sorption data were approximated using a two-parameter Nelson's sorption model and a three-parameter Guggenheim-Anderson-deBoer (GAB) model. It was shown that gluing the strands with 5, 10, and 15 % MUPF resulted in the reduction of equilibrium moisture content (*EMC*) by about 2 % for the adsorption phase, and about 1 % for the desorption phase, as well as a significant increase in sorption hysteresis, as compared to resinless strands. No proportional relationship was found between the gluing degree and equilibrium moisture content reduction. This indicates that MUPF resin applied on the wood strand surface had little effect on reducing hygroscopicity and increasing dimensional stability of OSB.

KEYWORDS: OSB, equilibrium moisture content, sorption, hysteresis, GAB model, Nelson model.

INTRODUCTION

OSB dimensional instability is largely affected by hygroscopic properties of wood used for cutting the strand strands. The measure of OSB dimensional instability is moisture induced deformation, caused by humid air or liquid water. Moisture deformations of OSBs, especially those related to thickness, may cause damage to the board structure (Wu 1998, Wu and Piao

1999, Mirski et al. 2012) and, consequently, damage of the whole construction made of these boards. Moisture-related behaviour of wood-based materials can be partially explained by the presence of adhesives that have different sorption phenomenon than wood (Wimmer et al. 2013). The hygroscopic properties and consequently the dimensional instability of OSB may be limited by using so-called water-resistant adhesives based on phenolic resin (PF), isocyanates (MDI), and melamine urea formaldehyde resin (MUF). The equilibrium moisture content (*EMC*) for wood-based materials such as fibre boards, MDF or OSB is lower than for solid wood of the same species under the same conditions. However, in wood-based materials glued with phenolic resin (PF), the strong hygroscopic properties of alkali result in higher *EMC* than when other adhesives are used (Niemz 2010). Furthermore, the use of adhesives with appropriately flexible joint reduces the risk of damaging the board structure caused by an internal stress resulting from changes in moisture content. *EMC* strongly affects all physical, mechanical and biological properties of wood based materials (i.e. modulus of elasticity, modulus of rupture, internal bond, hardness, thermal conductivity, fungal resistance). Resistance of wood-based materials to short contact with liquid water or humid air can also be achieved by applying waxes – primarily in the form of wax emulsion added as an adhesive solution. Both the increase in loading (pressure, temperature and pressing time) and wax content during manufacture of wood-based materials change the course of sorption isotherms (Halligan and Schniewind 1972, McNatt 1974, Kelly 1977, Wu and Ren 2000). Studies on the sorption phenomena in OSB usually involve industrially produced boards (Wu 1999, Hartley et al. 2007) glued with PF or PF/pMDI resin[1] (Hartley et al. 2007, Neimsuwan et al. 2008). The properties of industrially manufactured OSBs are the result of several factors (e.g. temperature, pressure, and pressing time, wood species, flake dimensions). Therefore, while using these boards for experimental studies, it can be difficult to obtain reliable data on the impact of particular type of adhesive and loading on the course and range of the sorption phenomenon. Therefore, it seems to be necessary to study the hygroscopic properties of the adhesive-coated strands loaded with resin.

Currently utilized OSB production technologies allow for using MUPF resin for gluing the outer layers. The purpose of the study was to determine the sorption isotherms of wood strands with different MUPF gluing degree, and to obtain detailed information, unbiased by other factors (e.g. temperature, pressure, and pressing time), on how the applying of MUPF on the wood strand surface affects the course of sorption in wood.

MATERIAL AND METHODS

The study was performed using pine (*Pinus sylvestris* L.) wood strands, obtained from sapwood and dried under industrial conditions. Average moisture content of the strands was determined by oven-dry method and it amounted to 6.3 %. MUPF with wax emulsion was applied on the strands immediately before the formation of a three-layer OSB with a nominal thickness of 15 mm and a density of 650 $kg.m^{-3}$. Furthermore, the adhesive solution was supplemented with a neutral dye to facilitate identification of uniformly coated wood strands contained in the strand-adhesive mass. Properties of adhesive components used for wood strand loading are presented in Tab. 1.

[1] outer layers - Phenol-Formaldehyde (PF), core layer - polymeric 4, 4′-Methylenediphenyl Isocyanate (pMDI)

Tab. 1: Properties of the adhesive used in the experiments.

Property	Unit	Adhesive components	
		MUPF resin	Wax emulsion
Dry mass	%	64.1	63.2
Viscosity	mPas	891	695
Density	$g.cm^{-3}$	1.295	0.910
pH value	–	9.33	9.34

Immediately after preparation of the strand-adhesive mass (before board formation) 20 wood strands were selected for each MUPF loading option. The selected strands were placed on a carrier plate and heated at 200°C for 30 s, together with previously prepared resinless strands, and strands coated with wax emulsion alone. Pressing and heating of the selected strands were carried out to cure the MUPF adhesive enriched with wax in a way typical for the industrial conditions. All options of wood strands used in the sorption experiments are presented in Tab. 2.

Tab. 2: Options of wood strands used in the experiments.

Wood strands option		Adhesive components*	
No	Symbol	MUPF resin	Wax emulsion
1.	A (control)	–	–
2.	B	–	1.8 %
3.	C	5 %	1.8 %
4.	D	10 %	1.8 %
5.	E	15 %	1.8 %

*in an amount of the wood strands dry weight

The accepted value of a relative error in moisture content determination during experiment was an additional criterion of the wood strand dimensional selection. Considering a characteristic shape and size diversity of the wood strands used for OSB, the selected strands were prepared with the aim of conferring similar dimensions. Minimum mass of the selected wood strands was determined using the approach proposed by Jaros et al. (1994). The dimensions of the prepared wood strands were ca. 0.8 • 30 • 45 mm (thickness • width • length). All wood strands were stored in a desiccator and dried over phosphorus pentoxide (P_2O_5) in order to obtain the oven-dry state. The adsorption and desorption experiments were carried out in the previously prepared test set-up, in which the wood strands were placed in a chamber, while the relative humidity was controlled by salt solutions (Majka and Olek 2007). Air temperature was 22±°C. Air temperature and relative humidity were measured by a thermo-hygrometer, type LB 706, produced by LAB EL. The measurement inaccuracy for temperature was ±0.1°C, and for air relative humidity ±2.0 % (in the range of 10 to 90 %). The use of salt solutions made it possible to achieve nine levels of relative humidity inside the chamber. Salt solutions applied in sorption experiments and registered air relative humidity values are presented in Tab. 3.

Tab. 3: Salt solutions and chemicals applied in sorption experiments and registered air relative humidity values at temperature of 22 ±1°C.

Salt solution	Air relative humidity (%)	
	Adsorption	Desorption
Distilled water (H_2O)	93.4	93.4
Potassium chloride (KCl)	85.0	86.1
Sodium chloride (NaCl)	77.3	77.7
Sodium bromide (NaBr)	60.8	62.2
Potassium carbonate (K_2CO_3)	46.3	47.4
Calcium chloride ($CaCl_2 \cdot 6H_2O$)	35.5	36.9
Potassium acetate (CH_3COOK)	29.0	30.0
Lithium chloride (LiCl)	13.8	14.6
Phosphorus pentoxide (P_2O_5)	2.9*	4.4*

* readings with the higher measurement inaccuracy due to out of range constant work conditions recomended by the thermohygrometer producer.

All wood strands were weighed at least twice after obtaining the equilibrium at each humidity level. The obtained results were stored in a data acquisition system. At the end of the experimental procedure, all the samples were placed in a laboratory drier (at 103±2°C) and their oven-dry mass was determined. Each experimental option, i.e. options of resin and wax loading, consisted of 8 strands. Therefore, each value of the equilibrium moisture content was a mean of 8 measurements. Sorption isotherms were determined at 22±1°C. The obtained sorption data were analyzed by applying two mathematical models based on the laws of physics. The use of these sorption models to analyze the obtained sorption data, allowed us to apply the model coefficient values to interpret the unbiased by other factors sorption phenomena with regard to the effect of the the resin loading on the hygroscopic properties of the produced OSBs.

The modeling of sorption isotherms for wood strands glued with MUPF was performed by means of a two-parameter Nelson's model (Nelson 1983), based on Gibbs free energy used to describe the sorption phenomenon in cellulosic materials. The Nelson's equation was applied by Wu (Wu 1999) for modeling the sorption isotherm in wood-based materials, i.e. commercial OSB, and it is expressed by the following formula:

$$RH = \exp\left\{\left(-\frac{W_w}{R \cdot T}\right)\exp\left[A\left(100 - \frac{EMC}{M_v}\right)\right]\right\} \tag{1}$$

where: RH (%) - air relative humidity,
W_w- (18/mole), molecular weight of water,
R - (8.314 J/mole/K), universal gas constant,
(K)- absolute temperature,
A (J.g^{-1}) - natural logarithm (ln) of Gibbs free energy per gram of sorbed water as RH approaches zero (ΔG_0), i.e., A= ln (ΔG_0); and M_v (%), is the material constant that approximates the fiber saturation point (FSP) for desorption.

The obtained EMC data were fitted into a rewritten form of Eq. 1 (Wu 1999):

$$EMC = M_v\left\{100 - \frac{1}{A}\ln\left[\left(-\frac{R \cdot T}{W_w}\right)\ln RH\right]\right\} \tag{2}$$

Additionally, the modeling of sorption isotherms in wood strands glued with MUPF was based on the three-parameter Guggenheim-Anderson-deBoer (GAB) model. The GAB model was used by Hartley et al. (2007) for sorption isotherm modeling in commercial OSB, based on species groups and resin type. The model is expressed by the following equation:

$$EMC = M_m \frac{K \cdot C \cdot RH}{(100 - K \cdot RH) \cdot (100 - K \cdot RH + C \cdot K \cdot RH)} \tag{3}$$

where: M_m - the monolayer water capacity (%),
C - equilibrium constant related to the monolayer sorption,
K - equilibrium constant related to the multilayer sorption,
RH (%) - air relative humidity.

SigmaPlot 9.0 software with the implemented Levenberg-Marquardt iterative algorithm was applied to identify the coefficients of the investigated sorption isotherm models.

The obtained sorption data for all options of resin and wax loading were compared. The comparison criterion was defined as follows:

$$\Delta EMC = EMC_A - EMC_i \tag{4}$$

where: EMC_A- equilibrium moisture content of control (no resin and no wax loading) wood strands (option A), modeled with GAB equation (Eq. 3),
EMC_i- equilibrium moisture content of the studied options in the resin and wax loaded wood strands (options B-E), respectively.

In order to determine the influence of resin and wax loading on the hystersis phenomenon, the hysteresis was determined and calculated as:

$$EMC_{des} - EMC_{ads} \tag{5}$$

RESULTS

Fig. 1 presents the data from the sorption experiment approximated by GAB model. The obtained isotherm, experimentally determined for resinless wood strand (option A), was significantly different from the sorption curves derived from the data for the other options of MUPF and wax loading (B-E).

It was found that loading the strands with MUPF adhesive resulted in slightly reduced *EMC*. Results of the sorption experiment, summarized in Tab. 4, support the conclusion that the use of MUPF for gluing wood strands may result in reducing the equilibrium moisture content in the oriented strand boards.

The observed differences in *EMC*, calculated according to Eq. 4, were particularly noticeable for the adsorption phase (Fig. 1a). Depending on the degree of MUPF loading the perceived reduction in the *EMC* did not exceed 2 % for the adsorption phase, and about 1 % for the desorption phase. Sorption isotherms presented in Fig. 1 and the summarized data from the sorption experiment shown in Tab. 2 did not unambiguously indicate whether the degree of MUPF loading (options C-E) was connected with proportional *EMC* reduction. Furthermore, it was noticed that *EMC* decrease, being the result of applying only 1.8 % wax emulsion on the surface of the studied strands (option B), brought about a similar effect as in the case of strands loaded with wax-enriched MUPF (options C-E).

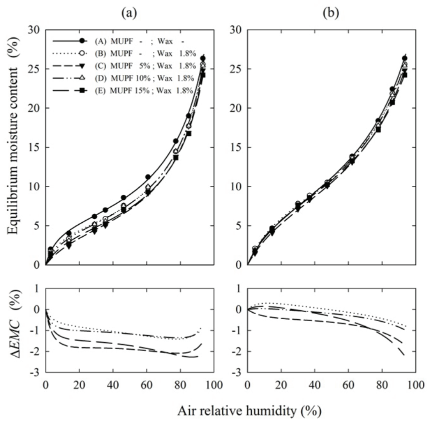

Fig. 1: Sorption isotherms of Scots pine (Pinus sylvestris L.) wood strands at 22±1°C, previously loaded by MUPF. Upper plots - measured data and results of modeling with GAB model (Eq. 3) for a) adsorption, b) desorption. Bottom plots - EMC differences for the studied options of loading with MUPF, calculated using Eq. 5.

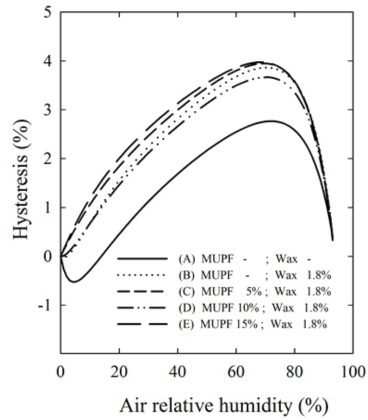

Fig. 2: Hysteresis for EMC data obtained for Scots pine (Pinus sylvestris L.) wood strands at 22±1°C, previously loaded with MUPF.

Tab. 4: Mean values of equilibrium moisture content (EMC) at 22±1°C for Scots pine (Pinus sylvestris L.) wood strands previously loaded with MUPF.

Sorption phase	Air relative humidity (%)	Option of wood strand				
		A	B	C	D	E
		MUPF - Wax -	MUPF - Wax 1.8 %	MUPF 5 % Wax 1.8 %	MUPF 10 % Wax 1.8 %	MUPF 15 % Wax 1.8 %
Adsorption	2.9	2.0 (0.3)	1.7 (0.4)	1.1 (0.5)	1.6 (0.3)	1.4 (0.8)
	13.8	4.0 (0.5)	3.3 (0.4)	2.4 (1.0)	3.1 (0.4)	2.7 (0.5)
	29.0	6.2 (0.5)	5.2 (0.2)	4.3 (0.6)	5.2 (0.6)	4.5 (0.3)
	35.5	7.0 (0.4)	5.9 (0.4)	5.0 (0.5)	5.6 (0.3)	5.2 (0.4)
	46.3	8.6 (0.3)	7.6 (0.7)	6.9 (0.4)	7.5 (0.5)	7.1 (0.6)
	60.8	11.2 (0.5)	9.9 (0.9)	9.1 (0.6)	10.0 (0.4)	9.3 (0.5)
	77.3	15.8 (0.5)	14.5 (0.8)	13.8 (0.7)	14.5 (0.7)	13.7 (0.8)
	85.0	19.0 (0.5)	17.7 (1.1)	16.9 (0.6)	17.8 (0.7)	16.7 (0.8)
	93.4	26.4 (1.0)	25.6 (0.8)	24.8 (1.2)	25.4 (1.2)	24.2 (1.3)
Desorption	86.1	22.4 (0.5)	21.8 (0.7)	20.8 (1.0)	21.6 (0.6)	20.7 (0.9)
	77.7	18.4 (0.4)	18.0 (0.6)	17.4 (0.6)	17.8 (0.7)	17.2 (0.6)
	62.2	13.8 (0.3)	13.7 (0.6)	13.1 (0.5)	13.6 (0.4)	13.3 (0.4)
	47.4	10.5 (0.4)	10.5 (0.3)	10.1 (0.5)	10.4 (0.4)	10.3 (0.4)
	36.9	8.6 (0.4)	8.9 (0.6)	8.3 (0.4)	8.5 (0.5)	8.6 (0.5)
	30.0	7.7 (0.3)	7.8 (0.3)	7.1 (0.3)	7.5 (0.3)	7.6 (0.4)
	14.6	4.5 (0.1)	4.7 (0.2)	4.1 (0.7)	4.5 (0.5)	4.6 (0.4)
	4.4	1.7 (0.2)	2.1 (0.3)	1.5 (0.5)	1.9 (0.4)	1.8 (0.4)

standard deviation in parantheses.

Tabs. 3 and 4 present the values of the estimated coefficients for the Nelson and GAB sorption model, respectively. Coefficient values clearly depended on the sorption phase as well as the parameters of prior resin and wax loading. The values of the standard error of the estimate and determination coefficient of R^2 indicate a very high capacity of the investigated models to approximate the obtained sorption data. However, the GAB model allowed for better fitting of the sorption curve to the experimental data than the Nelson's model. *SE* and R^2 values for the two-parameter Nelson's sorption model remained slightly lower than for the three-parameter GAB model - especially for the adsorption phase.

Tab. 5: Estimated coefficients of the Nelson's model fitted to the sorption data of Scots pine (Pinus sylvestris L.) wood strands at 22±1°C, previously loaded with MUPF.

Sorption phase	Wood strand option			Sorption model coefficients				Gibbs free energy
	Symbol	Resin loading (%)	Wax loading (%)	M_v (%)	A ($J.g^{-1}$)	SE*	R^2	ΔG_0** ($J.g^{-1}$)
Adsorption	A	-	-	29.57	1.137	0.0107	0.9799	3.118
	B	-	1.8	28.17	1.108	0.0151	0.9669	3.029
	C	5	1.8	27.43	1.077	0.0153	0.9662	2.937
	D	10	1.8	28.18	1.104	0.0146	0.9691	3.016
	E	15	1.8	26.80	1.095	0.0143	0.9678	2.988
Desorption	A	-	-	32.18	1.152	0.0044	0.9975	3.166
	B	-	1.8	31.16	1.171	0.0041	0.9977	3.224
	C	5	1.8	30.31	1.150	0.0031	0.9986	3.159
	D	10	1.8	31.02	1.162	0.0040	0.9978	3.196
	E	15	1.8	29.65	1.176	0.0036	0.9997	3.242

* SE – *standard error*, ** $\Delta G_0 = e^A$

Tab. 6: Estimated coefficients of GAB model fitted to the sorption data of Scots pine (Pinus sylvestris L.) wood strands at 22±1°C, previously loaded with MUPF.

Sorption phase	Wood strand option			Sorption model coefficients				
	Symbol	Resin loading (%)	Wax loading (%)	M_m (%)	K	C	SE	R^2
Adsorption	A	-	-	5.60	0.84527	16.658	0.0031	0.9989
	B	-	1.8	4.81	0.87182	14.359	0.0032	0.9987
	C	5	1.8	4.76	0.87096	6.817	0.0037	0.9983
	D	10	1.8	4.94	0.86599	11.262	0.0036	0.9984
	E	15	1.8	4.69	0.86732	9.161	0.0038	0.9981
Desorption	A	-	-	8.67	0.74044	7.220	0.0018	0.9996
	B	-	1.8	8.37	0.73872	8.780	0.0017	0.9997
	C	5	1.8	8.47	0.72916	6.566	0.0010	0.9999
	D	10	1.8	8.46	0.73506	7.702	0.0015	0.9997
	E	15	1.8	8.34	0.72236	8.475	0.0014	0.9997

Comparison of the coefficients of the Nelson's and GAB sorption models (Tabs. 5 and 6) enabled a more detailed analysis of the MUPF effect on the strand sorption phenomenon. Values of M_v coefficient (Tab. 5), which in the Nelson's model is interpreted as fiber saturation point (*FSP*), indicated that *FSP* of wood strands was reduced following the application of MUPF solution on their surface. However, slight differences in the discussed coefficient for the strands glued with 5, 10, and 15 % MUPF (options C-E), did not allow us to ascertain a proportional relationship between the *FSP* and the degree of strands gluing. The values of Gibbs free energy per grams of sorbed water as *RH* approaches zero ($\Delta\ G_o$), calculated as e^A (see Tab. 5) were significantly lower for the strands coated with MUPF (options C-E). However, this correlation was valid only for the adsorption phase. The values of M_m parameter, defined in the GAB model as water content (%) resulting from a chemisorption, varied depending on the studied option. The insignificant reduction of M_m parameter may result from the blockage of hydroxyl groups, mainly in hemicelluloses, being active sorption sites available within the wood cell walls. Similar phenomena have been also reported by other researches (Wimmer et al. 2013). However lowered monolayer water capacity was also recorded for the strands coated with 1.8 % wax alone (option B). Coating the strands with 5, 10 or 15 % MUPF (options C-E) did not result in a significant reduction of this parameter value. In addition, little variation in K and C coefficients (Tab. 6), interpreted in the GAB sorption model as equilibrium constant related to the monolayer sorption, and equilibrium constant related to the multilayer sorption, respectively, did not allow for assessment of the effect of MUPF properties on the strands sorption phenomena. This fact, as well as earlier observations on the level of equilibrium moisture content limitation in strands coated with 1.8 % wax (option B) may indicate, that the application of MUPF resin on the strand surface had little effect on the reduction of the strands hygroscopicity.

Hysteresis calculated according to Eq. 5 are presented in Fig. 2.

It was found that loading the strands with 5, 10 and 15 % MUPF (options C-E) let to a significant increase in the sorption hysteresis, as compared to the sorption in resinless strands (option A). Insignificant differences in the hysteresis were found for wood strand loaded with 5, 10, and 15 % MUPF (options C-E) and with 1.8 % wax (option B). It can be concluded that the investigated MUPF resin loading had negative influence on the hysteresis phenomenon, i.e. it influences higher dimensional instability of OSB due to cyclic sorption.

DISCUSSION

The obtained results showed the minor reduction of *EMC* of strands coated MUPF resin. It was due to the reduction of available surface area for water sorption phenomenon. Moreover, the reduction of *EMC* can be explained by the positive effect of the interaction between wood and adhesive components. It is well known that the adhesives based on MUF and used for OSB manufacturing result in lower *EMC* than for solid wood (Halligan and Schniewind 1972, Kelly 1977). Neimsuwan et al. (2008) observed that the PF resin loading by 2, 4 and 6 % based on oven-dry mass applied to Loblolly pine (*Pinus taeda* L.) wood strands resulted in lower *EMC*. Contrary to that it was found that wood-based materials loading with PF result in higher *EMC* (for higher air relative humidity) as compared to panels loading with other resins (Niemz 2010). The increase of *EMC* may be explained as a result strong hygroscopic properties of alkali resulting in high pH value of the adhesive. However, the MUPF resin used in investigations was characterized by relatively low pH value (see Tab. 1). Therefore, there was observed reduction of *EMC* of strands coated by MUPF resin (max. 2 % for the adsorption phase, and 1 % for the desorption phase).

The lack of correlation between of MUPF loading and the *EMC* reduction could be attributed to the difficulty for uniform application of MUPF resin on strands surface. Tabarsa and Chui (1997) found that *EMC* of strands decreased with platen temperature with considerable reduction in *EMC* from 100 up to 150°C. On this finding Neimsuwan et al. (2008) stated that the reduction in *EMC* is due to hemicelluloses degradation. Wang and Winistorfer (2001) reported that a hot-pressing has also influence on reduction of *EMC* wood strands. Therefore, minor reduction of *EMC* and lack of the correlation between MUPF resin loading and level of *EMC* reduction may be a result of the platen temperature and hot-pressing during strands preparation. However, the influence of temperature on *EMC* was not discussed in this paper, the presented observation suggested that processing factors might have higher influence on sorption phenomenon of strands than resin loading. The significant decrease in the *EMC* of strands coated with 1.8 % wax emulsion (option B), which was observed, can be connected with wax properties such as chemical composition, melting point, viscosity and oil content. Each of these properties is closely related to liquid and vapor repellency of wood strands. What is more, an addition of wax can improve water repellency by reducing surface energy which makes it hydrophobic. Furthermore, Neimsuwan et al. (2008) concluded that wax loading have more influence on sorption phenomenon of strands than resin loading.

CONCLUSIONS

1. The use of MUPF adhesive resulted in about 2 % (adsorption) and 1 % (desorption) reduction in the equilibrium moisture content (*EMC*) of strands used for OSBs. The observed reduction should be considered negligible.
2. The perceived decrease in the equilibrium moisture content of wood strands coated only with a hydrophobizing agent in the form of 1.8 % wax emulsion, was comparable to *EMC* decline observed for the strands loaded with MUPF. This indicates that MUPF resin applied on the wood strand surface had little effect on reducing hygroscopicity and increasing dimensional stability of OSB.
3. Considering limited range of *EMC* reduction and poor confidence limits of *EMC* determination during the sorption experiment, it can not be unambiguously ascertained whether there is any correlation between the degree of MUPF loading (5-15 %) and the range of equilibrium moisture content reduction.
4. It was concluded that the use of MUPF resin for gluing the wood strands intended for OSB production significantly intensified the sorption hysteresis phenomenon. This fact may be important during the initial stage of OSB utilization in variable environmental conditions.

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