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MOISTURE ABSORPTION AND DIMENSIONAL STABILITY OF POPLAR WOOD IMPREGNATED WITH SUCROSE AND SODIUM CHLORIDE

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ABSTRACT

This paper deals with the effect of vacuum-pressure impregnation of Poplar wood (*Populus alba*) by aqueous solutions of sucrose and sodium chloride on its physical properties. Groups of samples with different concentrations of substances in the aqueous solution were compared within each other and also with a reference (non-impregnated) group. The specimens from all groups were tested for density, moisture absorption and dimensional stability. The obtained data were statistically analysed and compared each other. The most satisfying final properties were achieved in impregnation of sucrose with concentration of 6,25 g/100 ml H_2O. The retention was 31 kg m^{-3} (WPG around 8 %). The values of ASE (anti-swelling efficiency) reached to 36 % and MEE (moisture exclusion efficiency) was reduced by 33 %.

Keywords: Wood modification, sodium chloride, sucrose, vacuum-pressure impregnation, swelling, equilibrium moisture content.

INTRODUCTION

Today, wood is a very frequently used material. In comparison to other competitive materials it offers many advantages including the following ones: wood is a renewable material, given its weight it provides a very high strength and elasticity, it has good thermal insulating properties, it can be easily shaped, it is ecologically recyclable and, last but not least, it has its indisputable aesthetic qualities (Gryc *et al.* 2007, Baar and Gryc 2012). But there are also some disadvantages. One of them, hygroscopicity, which induces dimension changes, was examined in this study.

Wood impregnation by natural substances with the aim of modifying its properties has been well known for decades and even centuries. At the break of the industrial revolution mainly synthetic-based substances were preferred – these were artificially produced with frequent negative side effects both on human beings and on the environment. The current trend in many fields of human activity is to return to original harmony with nature and a harmless use of natural renewable resources (Hill 2006). Products made from materials which were modified by natural means can be recycled environmentally friendly. Advantages of the new generation of “eco-modified” products are: first, that the material comes from renewable resources (e.g. wood); and second, that also the impregnation substance is from such resources (e.g. substances obtained from plant processing or, ideally, as waste and by-products). Third, their energy efficiency should be low. One of such technologies is e.g. modification of wood impregnation by sugars (Vigué 2006).

Wood modification by pure sucrose is mainly used for conservation of wooden artwork. Sucrose (cane sugar, beet sugar, chemically α-D-glucopyranosyl-β-D-fructofuranoside, $C_{12}H_{22}O_{11}$), is easily dissolved in water, non-toxic and does not corrode metals. Its chemical structure is similar to cellulose, it has a low molecule weight and it penetrates wood easily and quickly. It has a crystalline structure suitable for wood reinforcement. It dissolves in water at a normal temperature so it does not have to be heated for impregnation. It has a chemical affinity to cellulose and diffuses in wood easily. A raster electron microscopic study found that sucrose penetrates cell walls where it crystallizes. This corresponds to its excellent stabilization of wood dimensions. Modified wood retains its natural appearance; and the price of sucrose is low compared to other substances (Morgós and Imazu 1993).

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Other research (Stamm 1955) compared impregnation by sucrose and impregnation by invert sugar with the aim to decrease water vapour absorption and water absorption. The invert sugar (obtained by acid or enzymatic sucrose hydrolysis) was found to decrease water absorption better than crystalline sucrose. Both ways achieved better results than unmodified specimens. However, the invert sugar does not increase the hardness of wood comparing to sucrose, which improves this property.

Another experiment published by Lesar *et al.* (2009) compared water vapour absorption in specimens impregnated by compounds of boron, sodium chloride and glucose. Impregnation by sodium chloride had a larger effect on the equilibrium moisture content (EMC) than impregnation by boron compounds. An increasing concentration of NaCl in wood was found to be accompanied by increase in EMC. Boric acid, borax, sodium chloride and glucose have all crystalline structures. The researchers assume that these substances are bound in wood mechanically (not chemically) and thus they do not cause any structural changes to wood. The substances are deposited in the form of smaller or larger crystals in cell walls and cell lumens. The crystals dissolve in an extremely humid environment and crystallize again when air humidity decreases. The absorbed and free water in wood works as a dissolvent. This process of crystallization and dissolution is reversible.

The difference between sodium chloride and sucrose is their water solubility – it is six times higher for sucrose than for NaCl. Thus we can assume smaller weight percentage gain (WPG) within NaCl impregnation. An important factor in the case of NaCl will be its hygroscopicity.

Modification of wood with sucrose is commonly used in the preservation of archaeological wood artifacts (Morgós and Imazu 1993).

The use of these materials should be for interior due to increased hygroscopic properties of modified materials. Another alternative would be used for outdoor in small concentrations of sucrose when there is not such a large increase in EMC and adding some biocide compounds.

The main aim of this study was to provide better dimensional stability of the wood of poplar. The authors chose substances that are water soluble and come from natural sources and could replace e.g. synthetic resins. Changes in properties of solid wood in dependence on conditions of use can be huge and need to be eliminated as much as possible (Dejmal *et al.* 2009).

MATERIALS AND METHODS

Samples

Specimens of white poplar (*Populus alba* L.) with dimensions 30 × 20 × 20 mm (L × T × R) were oven dried in a laboratory drying chamber at a temperature of 103 ± 2 °C until constant mass was achieved. Then the specimens were weighed and dimensions were measured.

Vacuum-pressure impregnation

The impregnation solution contained water (dissolvent) and the impregnation substance, either sodium chloride or sucrose. Due to the lower water solubility of NaCl in comparison to sucrose, the impregnation solutions with NaCl were about 6 times less concentrated. In total, five different concentrations of water dilutions were prepared for each substance (Table 1). The mixture was mixed properly in a plastic container and then left standing for 24 hours at a temperature of 20 °C. After 24 hours all NaCl and sucrose were dissolved and homogeneous dilutions were gained.

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Table 1. Groups of samples and concentrations of substance in the solution.

Group	Concentration (NaCl)	Concentration (sucrose)
I	1 g / 100 ml H_2O	6,25 g / 100 ml H_2O
II	2 g / 100 ml H_2O	12,5 g / 100 ml H_2O
III	4 g / 100 ml H_2O	25 g / 100 ml H_2O
IV	8 g / 100 ml H_2O	50 g / 100 ml H_2O
V	16 g / 100 ml H_2O	100 g / 100 ml H_2O
VI	unmod. (reference) specimens	unmod. (reference) specimens

The impregnation was conducted using the vacuum-pressure device JHP 1-072. Specimens from groups I–V were impregnated. For all groups a specific process of impregnation was chosen, based on vacuum phase only, without overpressure. The vacuum based impregnation was carried out in three steps. The pressure was decreased to 20 kPa of absolute pressure in each step and then maintained for chosen time. Breaks at atmospheric pressure followed after each vacuuming step and the impregnation solution was filled up (Table 2).

Table 2. Pressures and times used for phases of vacuum-pressure impregnation.

Phase	Time t (min.)	Absolute pressure (kPa)	Phase description
1	15/60	50/20	slight pressure decrease
relaxation	10	101,3 ± 1	filling up the impregnation solution
2	80	20	
relaxation	10	101,3 ± 1	filling up the impregnation solution
3	60	20	
conditioning	24	101,3 ± 1	removal of the impregnation solution

Testing of physical properties

The specimens were oven dried again after impregnation until the constant mass. Then they were weighed and dimensions were measured again. The weight percentage gain (1), longitudinal swelling due to modification (2) and surface swelling due to modification (3) was calculated as follows:

$$WPG = \frac{m_m - m_n}{m_n} \cdot 100 \quad (1)$$

Where: WPG is the weight percentage gain (%), m_m is the mass of an oven dried modified specimen (g) and m_n is the mass of an unmodified oven dried specimen (g).

$$\alpha_L = \frac{l_m - l_n}{l_n} \cdot 100 \quad (2)$$

Where: α_L is the longitudinal swelling due to modification (%), l_m is the length of an oven dried modified specimen (mm) and l_n is the length of an oven dried unmodified specimen (mm).

$$\alpha_S = \frac{S_m - S_n}{S_n} \cdot 100 \quad (3)$$

Where: α_S is the surface swelling due to modification (%), s_m is the surface dimension of an oven dried modified specimen (mm^2) and s_n is the surface dimension of an oven dried unmodified specimen (mm^2).

The specimens were conditioned in the laboratory (at temperature of 20 °C and a relative air humidity of 20 %) for 60 days until they reached equilibrium moisture content of 4,1 %.

The moisture absorption test was done by placing the specimens into the desiccator above the water level. The lid of the desiccator was hermetically sealed and the specimens were left inside for another 60 days. This time ensures that the equilibrium moisture content of wood was reached. We also checked that the mass of the specimens remained constant after this time. The environment inside the hermetically sealed desiccator became saturated with water vapours (the relative air humidity reached nearly 100 %). The conditioning process was conducted at a temperature of 20 °C. Xylene was used to prevent mildew in the desiccator. The measured values were used to calculate the anti-swelling efficiency (4) and moisture exclusion efficiency (5):

$$ASE = \frac{S_n - S_m}{S_n} \cdot 100 \quad (4)$$

Where: ASE is anti-swelling efficiency (%), S_n is volume swelling of an unmodified specimen (%) and S_m is volume swelling of a modified specimen (%).

$$MEE = \frac{EMC_n - EMC_m}{EMC_n} \cdot 100 \quad (5)$$

Where: MEE is moisture exclusion efficiency (%), EMC_n is equilibrium moisture content of an unmodified specimen (%) and EMC_m is equilibrium moisture content of a modified specimen (%).

Statistical methods

The data were processed in software STATISTICA 9.0. Extreme values were removed using a box-graph (box plot). It was also necessary to investigate the distribution of the data type using a normal probability graph. Shapiro-Wilks test verified the data normality. This information is obtained for further processing. Afterwards the important moment and quantile characteristics (median, arithmetic average, standard deviation, etc.) were calculated. Then we can perform statistical tests (ANOVA, linear regression, etc.), which can reject the null hypothesis or not.

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RESULTS AND DISCUSSION

Treatments

One of the most important factors affecting the changes in wood properties is weight percentage gain (WPG). A close relationship between the concentration of the impregnation solution (content of the substance in water) and the reached values of WPG has been proved. The values of WPG fluctuated between 8 and 75 % after the modification by sucrose and between 0,6 and 30 % after modification by NaCl (Figure 1). The values of WPG achieved with sucrose are higher, which correlates to the higher concentrations of sucrose in water dilutions compared to dilutions concentrations of NaCl. In spite of this, NaCl and sucrose penetrate wood very well and can crystallize in the wood after drying. According to Morgós (2003) sucrose penetrates cell walls easily and creates hydrogen bonds with cellulose molecules. On the other hand, Lesar (2009) assumes that NaCl is bound in wood only mechanically (not chemically) so no structural change of wood occurs. In the table 3 you can see also average retention of NaCl and sucrose.

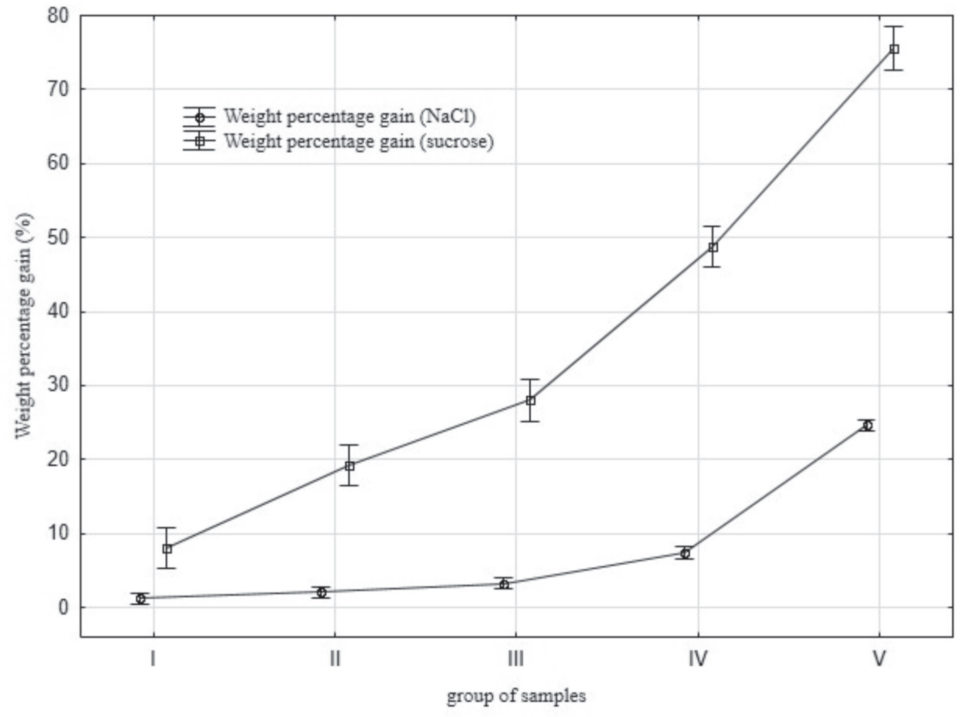

Figure 1. Anova – Weight percentage gain dependence on the concentration of the impregnation solution by group.

Table 3. Average retention of NaCl and sucrose.

Group	Retention ($kg \cdot m^{-3}$)
NaCl I	4,7
NaCl II	8
NaCl III	14
NaCl IV	27
NaCl V	72
Sucrose I	31
Sucrose II	63
Sucrose III	100
Sucrose IV	160
Sucrose V	258

Požgaj *et al.* (1997) states the density ρ_0 of poplar wood to be 390 kg m^{-3}. The mean density (ρ_0) of reference (non-impregnated) specimens was 390 kg·m-3. In dependence on the WPG the density of the specimens of *Populus alba* grew up to 676 kg m^{-3} in the sucrose impregnated specimens and up to 510 kg m^{-3} in the NaCl impregnated specimens.

The differences in density of specimens before and after modification (Figure 2) showed that the NaCl impregnation in group I (1 g / 100 ml) caused a slight decrease in the density, which can be explained by larger swelling of wood and the slight increase in weight after modification with a very small concentration of NaCl in the impregnation solution. Starting from group II the density increased based on the concentration of NaCl in the impregnation solution. Group V reached an average density increase of 63 kgn m^{-3}, which means increase of 17 % compared to density before modification. Regarding sucrose impregnation (Figure 2), the density of the group I increased as well (6,25 g / 100 ml). Group V reached an average density increase of 244 kg m^{-3}, which means increase of 64 % compared to density before modification.

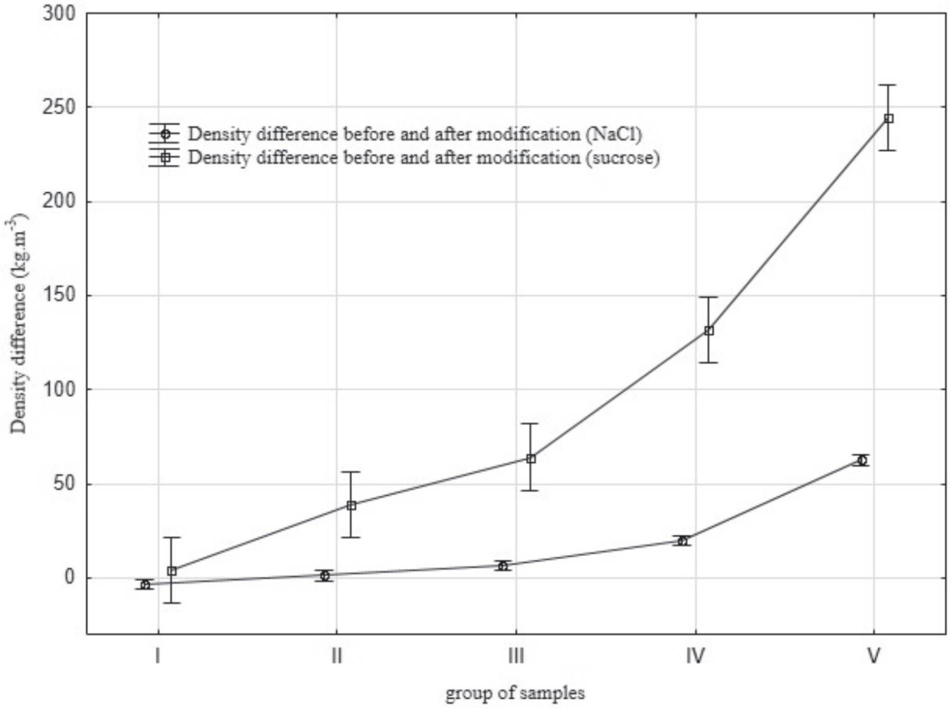

Figure 2. Anova – dependence of density differences before and after modification on the concentration of the impregnation solution by group.

Another indicator was swelling caused by the wood modification. Longitudinal swelling (α_L) due to modification follows the same rules as swelling due to moisture absorption. The average values in the longitudinal direction of group V are 0,6 % (NaCl) and 1,65 % (sucrose). The average values of swelling (αs) in the transverse directions due to modification did not differ much in groups I–IV – they ranged around 1,8 % (NaCl) and 3,6 % (sucrose). Group V (sucrose) also exhibited similar average values. Group V (NaCl) exhibited almost three times higher values – the average value of swelling was 5,2 %. The swelling of samples due to modification confirms the hypothesis that the wood expanded by the NaCl and the sucrose crystallization. This is consistent with that NaCl and sucrose were able to penetrate into cell walls, where they crystallized after drying. Further, we confirmed the influence of swelling due to modification on weight gain using linear regression model (Figure 3 and 4). The dependence of swelling due to modification on WPG is higher for NaCl impregnation than for sucrose impregnation.

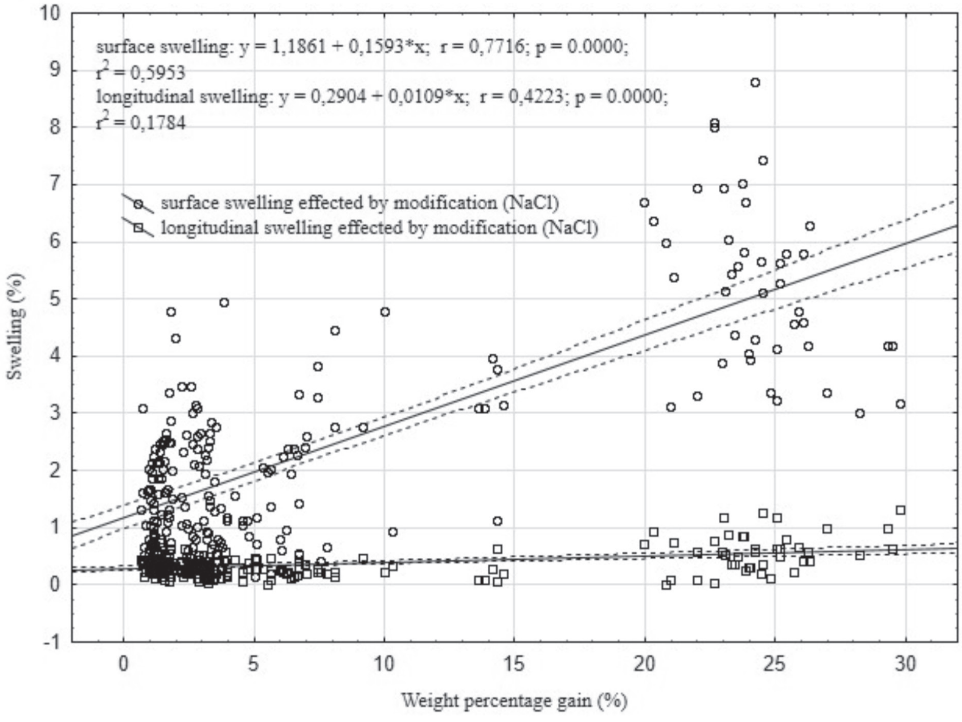

Figure 3. Model of linear regression – dependence of swelling due to modification on weight percentage gain (NaCl).

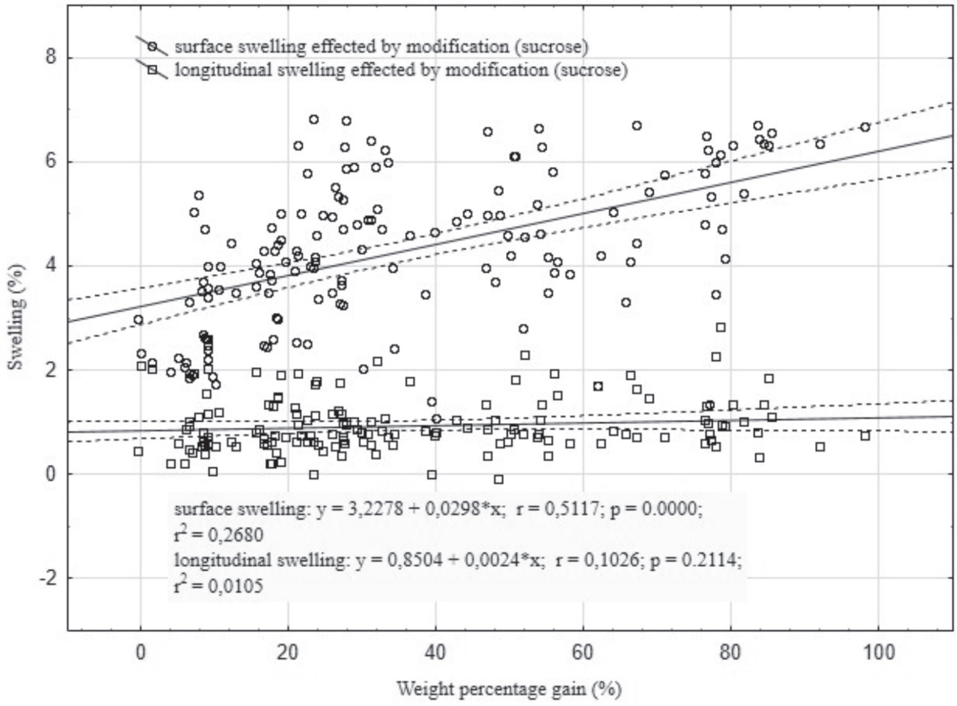

Figure 4. Model of linear regression – dependence of swelling due to modification on weight percentage gain (sucrose).

Moisture absorption experiments

Swelling due to moisture absorption is the main investigated property of this research. In this case, the average values of volume swelling (S_n) were the highest in the reference group: 12,7 % (Figure 5). Groups I (NaCl) and I (sucrose) exhibited average values of volume swelling (S_m) of 11,4 % and 8,1 %, respectively. Volume swelling in groups V (NaCl) and V (sucrose) was 9,6 % and 7,7 %, respectively. We found statistically significant differences when these groups (I and V) were compared with the reference group (VI). Impregnation by NaCl and sucrose manifested a positive effect on the dimensional stability of wood; the effect was higher in sucrose. It is probably caused by the preceding wood swelling caused by modification (NaCl and sucrose crystals in the cell wall) – moisture absorption does not increase the dimensions as much as it would in unmodified wood. The dimensional stability increases due to modification by these substances. Group I and V (sucrose) showed the best indicator ASE, 36 % (group I) and 39 % (group V). Group V showed the best result of ASE in case of NaCl (24 %).

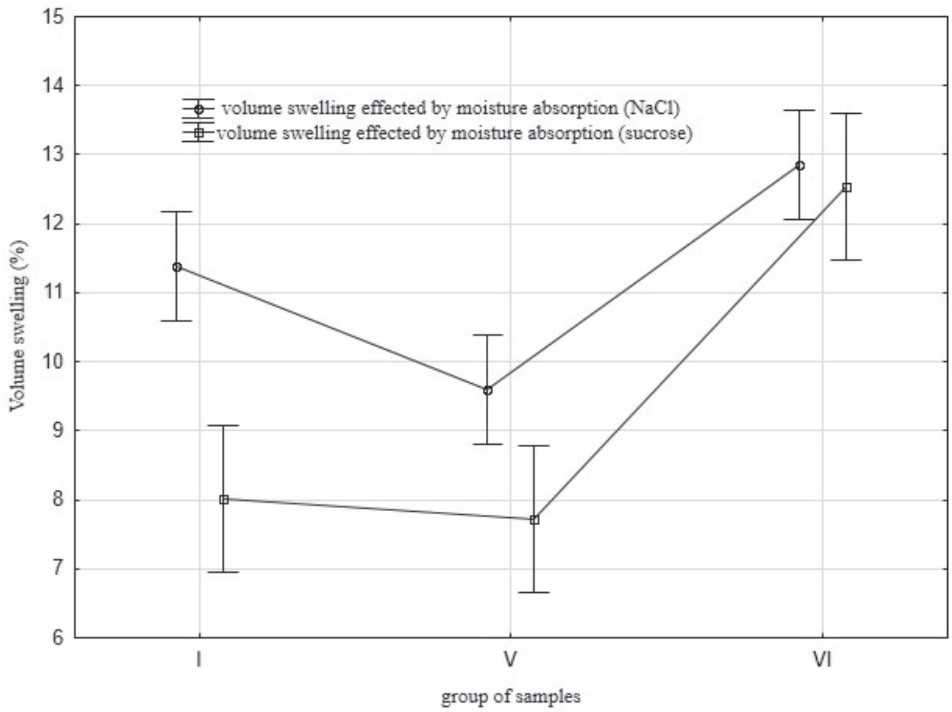

Figure 5. Anova – dependence of volume swelling due to moisture absorption on the concentration of the impregnation solution by group.

Another examined parameter was the average equilibrium moisture content (EMC_n) was about 30 % in the reference group (Figure 6). According to Siau (1995) the fibre saturation point is about 30 %; this means the maximum saturation of cell walls was achieved (wood contained the maximum amount of bound water). Group I (NaCl) reached an average EMC_m of 31,4 %, but no statistically significant differences were proved when compared with the reference group. On the other hand, in group I (sucrose) the average EMC_m was 35,7 % and statistically significant differences were found. The EMC in group V (NaCl) increased considerably – the average value was 66 %. The EMC_m in group V (sucrose) increased to 45 %. The EMC_m increases with an increasing NaCl concentration in the impregnation solution, as has also been confirmed by Lesar *et al.* (2009). These values in group V (NaCl) were achieved with an average WPG of about 22 %, while the value of EMC within sucrose impregnation was only 45 % with WPG of 70 %, which is three times more than in NaCl impregnation. With a relatively low content of NaCl in wood its hygroscopicity is considerably increased in contrast to sucrose impregnation. The model of linear regression (Figure 7 and 8) shows that there is a statistically significant dependence between EMC_m and WPG: the higher WPG, the higher EMC_m. This means that the more NaCl and sucrose there is in wood, the more the wood will absorb moisture, which affects the resulting EMC_m. The MEE (moisture exclusion efficiency) indicator gained negative values. The average value of group I (NaCl) was 4.6 %, group V (NaCl) — 120 %, group I (sucrose) – 33 % and group V (sucrose) – 61 %. According to Morgós (2003), natural sucrose is highly hygroscopic at relative air humidity $\varphi > 85$ %. At lower ambient humidity sucrose manifests the opposite – it decreases wood hygroscopicity.

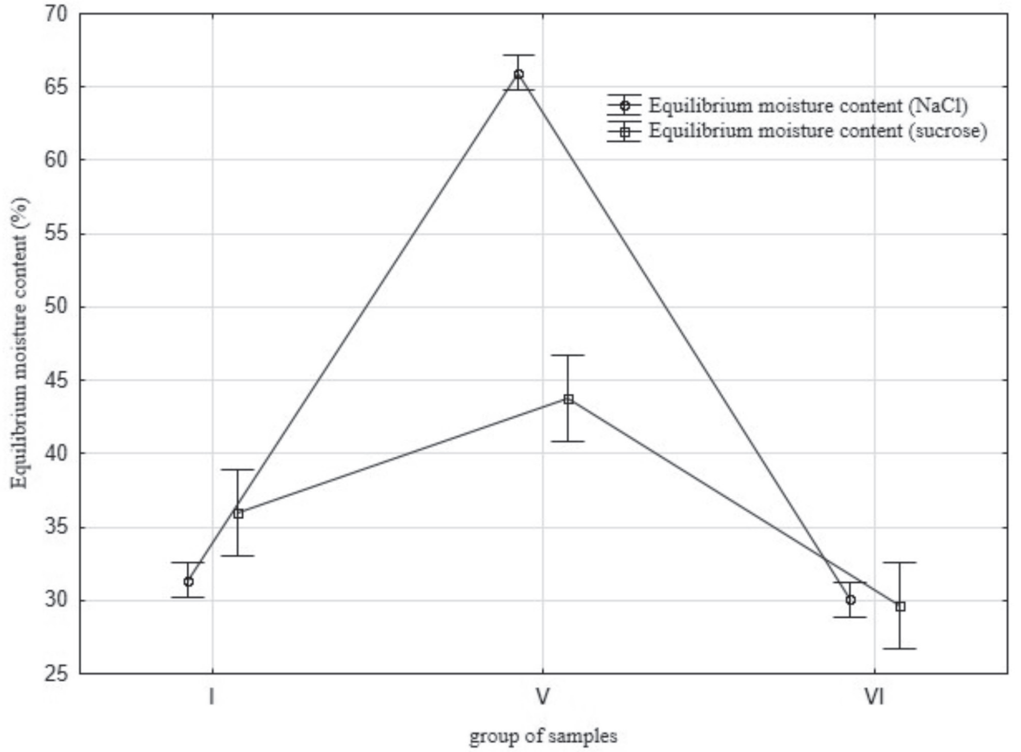

Figure 6. Anova – Equilibrium moisture content dependence on the concentration of the impregnation solution by group.

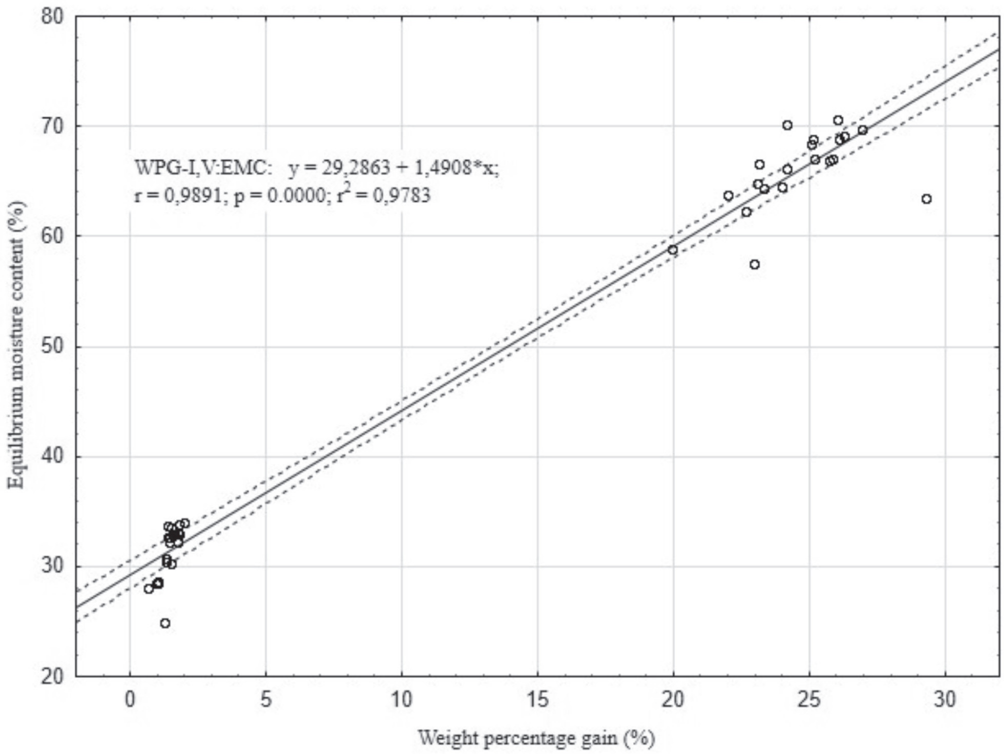

Figure 7. Model of linear regression – Equilibrium moisture content dependence on weight percentage gain (NaCl).

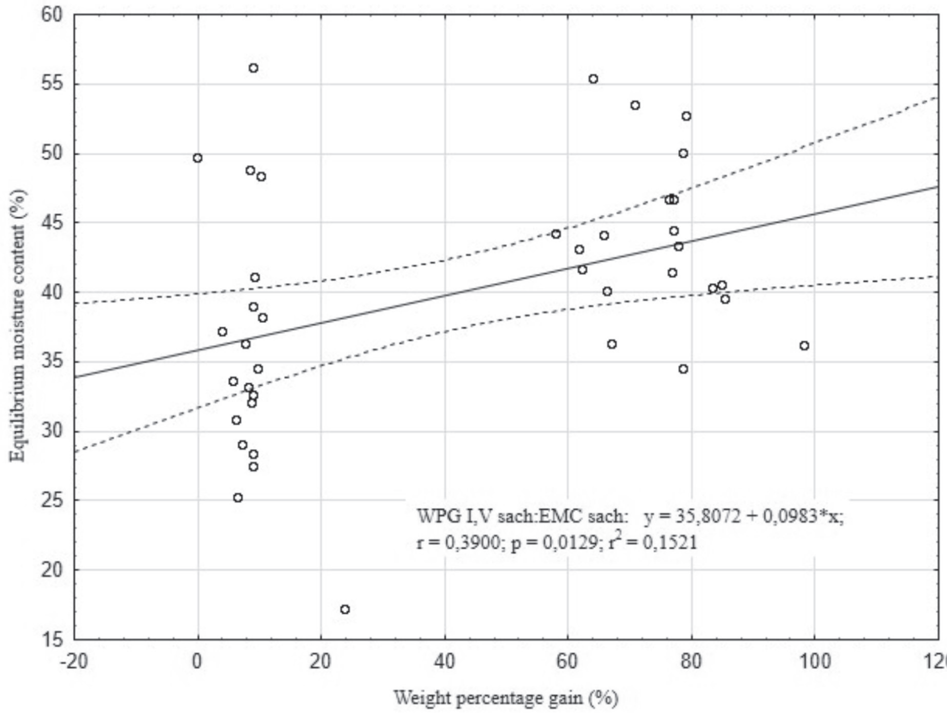

Figure 8. Model of linear regression - Equilibrium moisture content dependence on weight percentage gain (sucrose).

Group V (NaCl) exhibited enhanced properties of ASE but with the rapid increase in EMC, which is negative effect. Group I and V (sucrose) showed the best improvement of dimensional stability but group V provided little additional benefit while greatly increasing EMC_m. Higher EMC could cause increases susceptibility to mold and wood-decaying fungi. Impregnation with the lowest concentration of sucrose (group I) offers the best combination of reduction in swelling and relatively small increases in EMC and WPG.

CONCLUSION

Sucrose and NaCl impregnation are environment-friendly methods for wood modification using natural degradable substances. Poplar wood has a homogeneous structure and is fast-growing; its sapwood can be easily impregnated. The experimental measuring proves that crystallized sucrose and NaCl contained within wood can considerably affect its physical properties. The WPG increases with an increasing concentration of NaCl and sucrose in the impregnation solution and influences other wood properties. Tests also confirmed wood swelling due to modification. This evidences that NaCl and sucrose reached the cell walls where they crystallized after drying. Due to this wood swelled and its weight increased. Density increased in dependence on the concentration of the solution. Moisture absorption tests confirmed a high hygroscopicity of modified samples; the highest hygroscopicity was found in the highest concentration of NaCl. EMC of NaCl impregnation increased by 120 % on average in comparison with the reference group. Swelling caused by moisture absorption was lower in modified groups than in the reference groups, for both of the used substances. Modification reduced volume swelling caused by moisture absorption and thus we can state that it has a positive effect on the dimensional stability of wood. This is caused by wood swelling brought about by the modification itself. The lowest concentration of sucrose showed the best results from the perspective of improved dimensional stability and small increases in equilibrium moisture content.

Usage of the modified materials outdoors or in conditions of increased humidity cannot be recommended except small concentrations of sucrose when there is not such a large increase in EMC and adding some biocide compounds. Although there is still problem with potential leaching from wood. They are more suitable for interiors, e.g. for furniture, panelling and floors. Further research could ascertain how the modified wood would behave in lower relative air humidity. For this EMC in different ambient conditions could be ascertained, concentrations of NaCl and sucrose in the impregnation mixture could be increased and impregnation could be explored in combination of vacuum pressure and overpressure. It is also possible to combine impregnation with other ways of modification (e.g. thermal). Another topic for research could be the examination into the resistance to wood-destroying fungi and insects. Also an exploration should be conducted into fasteners regarding their potential corrosion.

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