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The study of selected properties of black poplar wood (*Populus nigra* L.) subjected to furfurylation and polymerization in lumen

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Abstract: *The study of selected properties of black poplar wood (Populus nigra L.) subjected to furfurylation and polymerization in lumen.* The aim of this work was studying black poplar wood (*Populus nigra* L.) selected properties modified with furfuryl alcohol and styrene. Maleic anhydride and glicydyl methacrylate were used as cross-linking agents. For modified wood samples the index of WPG, density, hardness, water absorbability, colour change and volume swelling were tested. Each test results were compared with values for unmodified black poplar wood. During furfurylation process density of poplar wood samples increased up to 1.3 times. Addition of maleic anhydride to furfurylation process caused increase of WPG index about 5-6 % and at the same time slightly decrease of wood Brinell hardness. Water absorbability and volume swelling of furfurylated poplar wood after 10080 min (168 h) of soaking in water were reduced more than 50% relative to unmodified wood. Polymerization in lumen with using styrene caused increase of density up to 2 times, increase of Brinell hardness up to 1.5 times, while WPG index was in the range from 50.8 to 116.6%. For polymerization in lumen process, water absorbability and volume swelling after 10080 min (168 h) of soaking in water were reduced more than half as well. Furfurylation caused intensive wood darkening when polymerization in lumen changed wood colour slightly. Addition of maleic anhydride to furfuryl alcohol caused an even greater darkening of the wood.

Keywords: furfuryl alcohol, styrene, polymerization in lumen, wood properties, wood modification

INTRODUCTION

Due to constantly increasing timber consumption in recent years and its growing demand because of social and environmental reasons, fast-growing species may play significant role in this area. Black poplar, like representative of this kind of species, is distinguished in poor mechanical properties, low biological resistance and inclination to change dimensions. These properties of poplar wood mainly result from its low density, small amount of extractives and porous structure [Prosiński 1984, Rowell 2005].

Nowadays a various types of wood modifications are known, which try to improve these undesirable features. Lot of them focus on hydrophobisation of wood like thermal modification, acetylation, thermo-hydro-mechanical densification, furfurylation or *in situ* polymerization with organic and non-organic compounds [Rowell 2005, Hill 2006].

Furfurylation is a method with use of furfuryl alcohol (FA). Its relay on furfuryl alcohol impregnation of wood with addition of catalyst in suitable temperature. In this conditions furfuryl alcohol undergoes polycondensation and cross-linking reactions, resulting in the formation of a three-dimensional polymeric network within the cell wall. Moreover FA can react with wood components (probably mainly with guaiacyl structure of lignin) [Hill 2006, Gérardin 2016]. In modification with furfuryl alcohol it is necessary to use a catalyst. Among the catalysts that can be used, zinc chloride was used in the past, and recently the most common are organic acids, e.g. citric, formic, oxalic or tartaric acid [Hill 2006, Li et al. 2015, Sejati et al. 2017]. Also, cyclic carboxylic anhydrides (mainly maleic anhydride - MA) are used as a catalyst [Schneider 1995, Westin 1996, Venås and Rinnan 2008, Bartkowiak et al. 2013, Bartkowiak et al. 2015]. By using cyclic carboxylic anhydrides furfurylated wood gain properties better than those produced by the previous system [Schneider 1995]. Wood after furfurylation improves some mechanical, physical and biological properties, such as dimensional stability, resistance to biological degradation, hardness or bending strength

[Esteves et al. 2011, Li et al. 2015, Sejati et al. 2017]. Additionally, wood after furfurylation changes its colour and became similar to exotic species [Hill 2006, Bartkowiak and Doczekalska 2017, Drywień and Radomski 2018]. Commercialization process is eco-friendly, because FA can be produced from agricultural wastes [Mantanis 2017].

Other often studied modification process is polymerization in lumen, called *in situ* polymerization. It is relay on filling of cell lumen with modification agent which concede to polymerization. This type of modification have important advantage – solvent evaporation is not necessary. Frequently applied modifiers in process of polymerization in lumen include acrylates, vinyl acetate or styrene (S) [Ławniczak 1971, Ermeydan et al. 2014, Żmuda and Radomski 2018, Gnacińska and Radomski 2019]. In this process is possibility to add another substances which bind to polymer chain and give him ability to react with wood components. For such cross-linking substances include maleic anhydride and glycidyl methacrylate (GMA) [Devi and Maji 2002, Hill 2006, Li et al. 2013]. Wood modified above-mentioned method characterized increased fungal and microbial resistance, greater dimensional stability and improved mechanical properties like bending strength and hardness [Rowell 2005, Hill 2006, Li et al. 2013]. Fire resistance of wood could be improved as well [Fan et al. 2013].

The aim of this work was to study of selected properties of *Populus nigra* L. wood subjected to furfurylation and polymerization in lumen. In this paper, for chemically modified wood density (ρ), weight percent gain index (WPG), colour, water absorbability (W), volume swelling (K_v) and Brinell hardness (HB) were determined. Additionally, the effect of adding a cross-linking agent on selected wood properties was investigated.

MATERIALS AND METHODS

The research material was obtained from State Forest District Sokołów Podlaski. To the studies two series of 40-year-old black poplar wood (*Populus nigra* L.) with dimensions of 20 x 20 x 30 mm and moisture content of 7.1% (drying-weight method according to Sluiter et al. 2008) were prepared. The first one was designed for furfurylation and the second one for polymerization in lumen. Additionally, 12 samples were prepared as a control group, which was not modified. Before each modification, samples were dried at 105°C to constant mass.

The series of samples prepared for modification with furfuryl alcohol consisted of 6 groups of 10 samples each. Concentration of FA for groups 1 – 3 was 10, 20 and 40% sequentially. Groups 4 – 6 were modified with FA concentration as above with addition of 1% of maleic anhydride. Citric acid at 1% was added to each group as initiator. Moreover, 96% ethanol at 50% was added to each group according to Venås and Rinnan [2008]. Finally, the prepared solutions were made up with distilled water, so that the total amount of each solution was 400 g.

The series of samples for polymerization in lumen consisted of 4 groups of 12 samples each. The groups were modified with solutions of the following composition:

1. Styrene with maleic anhydride (1%) and methanol (MeOH) (10%).
2. Styrene with glycidyl methacrylate (2%) and methanol (10%).
3. Styrene with methanol (10%).
4. Only styrene.

To each group benzoyl peroxide at 1% was added as initiator.

Furfurylation process consisted of impregnation and curing stages. In the impregnation stage, samples were placed in a glass beakers, loaded with glass stoppers to prevent floating and appropriate modification solutions were added. After that, beakers with samples were placed in a vacuum vessel. Then pressure was lowered using vacuum pump for a period of 30 min. After this time, the pressure has been brought to normal conditions and the samples were maintained under these conditions for a period of 90 min. Each group (1-6) were treated in the same way. In the curing stage, all impregnated samples were wrapped in an aluminum foil

and then placed in a dryer. The curing of samples was done at 120°C for 72 h. After the curing, the aluminum foil from samples was removed and samples were dried at 105°C for 24 h in order to evaporate condensation water and unreacted FA.

Polymerization in lumen was carried out in similar way to furfurylation with one difference. The curing stage was maintained in polyamide capsules, which were hermetically closed and placed in a dryer. Also in this case, the curing of samples was done at 120°C for 72 h. After the curing, the samples were taken from the polyamide capsules and were dried at 105°C for 24 h in order to evaporate unreacted styrene.

After modification process, a density and WPG index were determined. The densities of control samples and samples after modification were calculated using the following formula:

$$\rho = \frac{m}{v}$$

where: m - mass of sample (kg),

v - volume of sample (m^3).

Whereas, the WPG index based on the mass of samples before modification (m_1) and after modification (m_2). The WPG index was calculated using the following formula:

$$WPG = \frac{m_2 - m_1}{m_1} \cdot 100\%$$

Then water absorbability and volume swelling tests were carried out. The tests were done at room temperature. The mass and volume of control and modified samples were measured after 5 min, 10 min, 15 min, 30 min, 60 min (1 h), 1200 min (20 h), 4080 min (68 h) and 10080 min (168 h) of soaking in vessels filled with water. To prevent floating, samples were loaded with glass stopper. Before every mass and dimensions were measured, excess of water was eliminated.

Water absorbability was estimated on basis of the sample mass before soaking (m_1) and mass of the same sample after soaking (m_2) according to the formula below:

$$W = \frac{m_2 - m_1}{m_1} \cdot 100\%$$

Volume swelling was calculated based on volume of samples before soaking (v_1) and volume of the same samples after soaking (v_2) according to the formula below:

$$K_v = \frac{v_2 - v_1}{v_1} \cdot 100\%$$

Hardness of control and modified samples was performed using Brinell method according to the standard EN 1534 (2010). Hardness measurements were done using the multifunctional testing machine HBRV-187.5E produced by Huatec Group Corporation. The machine was equipped with a 10 mm diameter intender. The dwell time was 20 s and the maximum load was 1 kN. The Brinell hardness was calculated using the following formula:

$$HB = \frac{2F}{\pi \cdot D[D - \sqrt{D^2 - d^2}]}$$

where: F - the load applied (N),
d - the diameter of the indentation made by the steel ball on the surface of the test specimens (mm),
D - the diameter of the steel ball (mm).

Changes in colour of the unmodified and modified samples were determined by ERICHSEN SPECTROMASTER 565-D spectrophotometer, using CIELab method [Mokrzycki and Tatol 2011]. The colour in this method is the difference between three coordinates: L, a and b, which are placed in three-dimensional system, where L defines lightness (L = 0 means black and L = 100 means white), while a (a = - 60 means green, when a = 60 means red) and b (b = - 60 means blue, when b = 60 means yellow). The colour changes were calculated according to the formulas below, where coordinates with index 1 come from control samples, while with index 2 come from samples after modification.

Difference in lightness (ΔL):

$$\Delta L = L_2 - L_1$$

Difference in green and red (Δa):

$$\Delta a = a_2 - a_1$$

Difference in yellow and blue (Δb):

$$\Delta b = b_2 - b_1$$

Total colour change (ΔE):

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

All of the analyzed parameters were done in multiple repetitions. In case of furfurylation, density, WPG index and colour tests were done in a ten-fold repetition, but water absorbability, volume swelling and Brinell hardness tests were done in a five-fold repetition. In case of control group or polymerization in lumen, density, WPG index and colour tests were done in a twelve-fold repetition, but water absorbability, volume swelling and Brinell hardness tests were done in a six-fold repetition. The mean value was determined in each case and the standard deviation was calculated.

RESULTS AND DISCUSSION

In this studies *Populus nigra* L. wood was subjected to chemical modification. In table 1 the results of density, weight percent gain index and Brinell hardness obtained for *Populus nigra* L. wood before and after furfurylation and polymerization in lumen were presented.

Table 1. The results of density, weight percent gain and Brinell hardness obtained for *Populus nigra* L. wood before and after furfurylation and polymerization in lumen

Group	Composition of sample	ρ (before modification) [kg/m^3]	ρ (after modification) [kg/m^3]	WPG [%]	HB [N/mm^2]
Furfurylation					
1	10% FA	378 ± 49	408 ± 46	10.6 ± 2.2	30.7 ± 1.3
2	20% FA	370 ± 47	431 ± 43	19.8 ± 3.4	34.0 ± 1.5
3	40% FA	378 ± 49	489 ± 36	30.7 ± 4.6	37.4 ± 1.0
4	10% FA + 1% MA	373 ± 47	431 ± 39	15.6 ± 2.8	30.3 ± 1.4
5	20% FA + 1% MA	369 ± 45	457 ± 42	24.9 ± 3.1	32.0 ± 0.6
6	40% FA + 1% MA	388 ± 72	516 ± 24	36.2 ± 5.5	33.1 ± 1.1
Polymerization in lumen					
1	S + MA + MeOH	359 ± 31	511 ± 47	50.8 ± 20.5	36.2 ± 1.7
2	S + GMA + MeOH	384 ± 42	660 ± 55	85.7 ± 26.1	28.2 ± 1.6
3	S + MeOH	348 ± 30	697 ± 79	116.6 ± 35.8	32.3 ± 1.0
4	S	392 ± 41	712 ± 61	85.9 ± 17.9	34.0 ± 0.9
0	Control	369 ± 52	-	-	23.8 ± 2.3

The density of *Populus nigra* L. wood before modification process was in the range from 359 kg/m^3 to 392 kg/m^3. After chemical modification the density significantly increased and was in the range from 408 kg/m^3 to 712 kg/m^3. Higher densities of poplar wood were obtained for samples subjected to polymerization in lumen than after furfurylation process.
The values of WPG index for poplar wood modified with FA in a concentration of 10%, 20% and 40% were from 10.6% to 30.7%. The addition of maleic anhydride in the amount of 1% to the furfuryl alcohol caused increase of WPG index for each group about 5-6%. Similar correlations were published by Venås and Rinnan [2008], who in this case for Scots pine sapwood observed WPG index increase on the level 4-7%. Moreover, the beneficial effect of maleic anhydride addition on WPG index growth was noted by Bartkowiak et al. [2015] or Bartkowiak and Doczekalska [2017].

In the case of polymerization in lumen a WPG index was higher than for furfurylation process and was from 50.8% to 116.6%. In these studies, a very beneficial effect of methanol on the styrene polymerization process was observed. The use of methanol as a wood swelling agent was to facilitate the penetration of styrene into the wood structure and to increase the contact surface. The addition of methanol in the amount of 10% to styrene resulted in obtaining the highest WPG index (116.6%). In turn, the addition of MA (1%) and GMA (2%) to the mixture of styrene and methanol resulted in a significant reduction of the WPG index to the value of 50.8% and 85.7%, respectively. It is suspected, that this arrangement may lead to side reactions of MA or GMA with methanol, which can hinder efficient process of wood swelling and styrene polymerization. Further research is needed to explain this phenomenon.

Obtained hardness for wood samples of black poplar before modification process was 23.8 N/mm^2, which is similar to values reached by Pelit and Yorulmaz [2019]. Hardness for samples subjected to furfurylation was in the range from 30.3 N/mm^2 to 37.4 N/mm^2 and with the increase of the WPG index, the hardness of furfurylated poplar wood increased. Epmeier et al. [2004] and Li et al. [2016] observed similar tendencies for modification with FA of pine wood. Moreover, it was observed, that the furfurylated wood samples with addition of MA (1%) had slightly lower hardness than wood samples furfurylated without addition of MA.

Whereas in the case of polymerization in lumen, determined hardness was from 28.2 N/mm^2 to 36.2 N/mm^2. The highest hardness (36.2 N/mm^2) was obtained for samples modified with mixture of styrene, maleic anhydride and methanol, but the lowest hardness (28.2 N/mm^2) was reached for wood modified with mixture of styrene, glycidyl methacrylate and methanol. Based on the obtained results, it was observed, that in every case the applied chemical modification increased the hardness of the studied poplar wood.

In order to investigate the dimensional stability and hydrophobic properties of unmodified and chemically modified wood, the volume swelling and water absorbability studies were performed. The results were presented in Figs 1-3. Based on the results presented in Figs 1-3 it was observed, that the worst dimensional stability and hydrophobic properties had unmodified wood (control samples). The volume swelling and water absorbability for unmodified poplar wood were 16.3% and 180.7% after 10080 min (168 h) of soaking in water (Fig. 1). Whereas, poplar samples modified with furfuryl alcohol had significantly lower values of volume swelling and water absorbability. For wood samples, which were treated only with furfuryl alcohol, water absorbability after 10080 min reached about 103.5% (10% FA), 67.7% (20% FA) and 42.1% (40% FA), while volume swelling was 8.1%, 5.1% and 3.8% respectively (Fig. 1). It was observed, that the higher a concentration of FA used, the modified poplar wood exhibited a greater hydrophobicity and a better dimensional stability. Similar correlations were obtained by Szymona et al. [2014] and Drywień and Radomski [2018], who also observed increase hydrophobicity of oil palm trunk and poplar wood after treatment with different concentrations of FA.

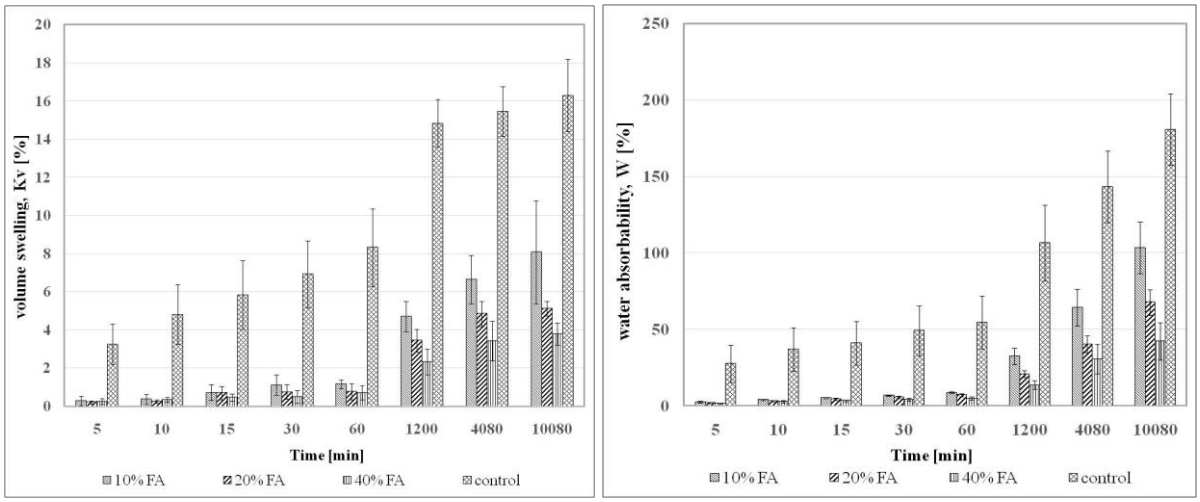

Fig. 1. Volume swelling and water absorbability of unmodified *Populus nigra* L. wood and modified with different concentration of FA

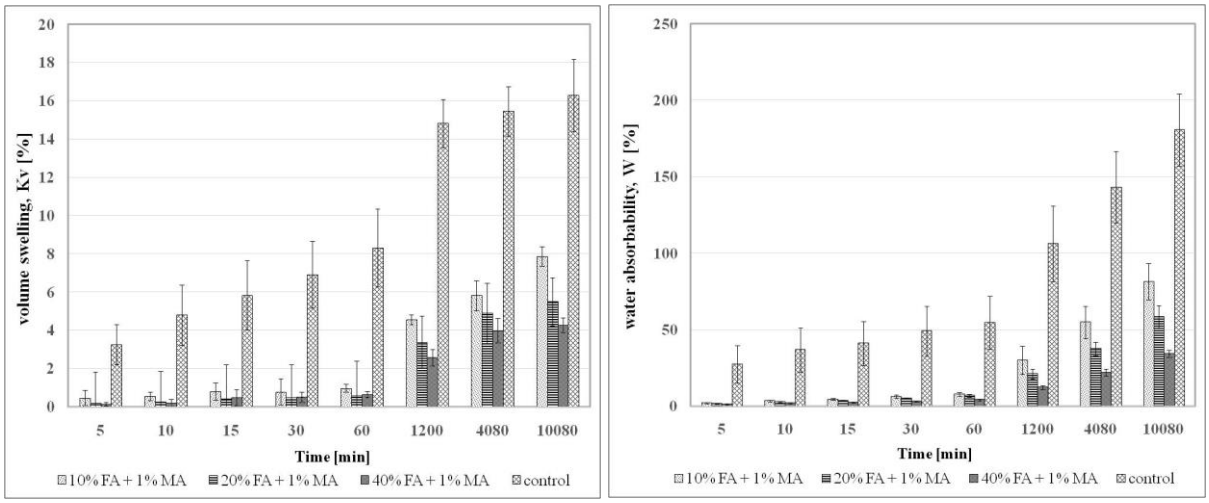

Fig. 2. Volume swelling and water absorbability of unmodified *Populus nigra* L. wood and modified with different concentration of FA and with addition of MA (1%)

Poplar wood samples, which were modified with analogical FA concentrations as earlier, but with addition of MA (1%) distinguished significantly lower volume swelling and water absorbability than control samples (Fig. 2). Values of water absorbability for these samples after 10080 min (168 h) were 81.5% (10% FA + 1% MA), 58.5% (20% FA + 1% MA) and 34.3% (40% FA + 1% MA), while volume swelling were 7.9%, 5.5% and 4.3% respectively. It can be observed that addition of maleic anhydride to furfuryl alcohol significantly increase hydrophobic character of wood, however it dimensional stability was similar to samples in which FA was used only.

For samples modified *in situ* with styrene significantly lower water absorbability and volume swelling were achieved (Fig. 3). Water absorbability after 10080 min (168 h) was 29.6% (S + MA + MeOH), 21.2% (S + GMA + MeOH), 16.9% (S + MeOH) and 26.0% (S) and was significantly lower than for control samples and wood samples subjected to furfurylation. Whereas volume swelling values were 7.4%, 7.0%, 5.9% and 11.7% respectively. For comparison, Xiaoying et al. [2012] published, that volume swelling for poplar wood modified with styrene and methyl methacylate was about 10% after 80 h of soaking in water. On the other hand, Gnacińska and Radomski [2019] reached a higher values of volume swelling for densified poplar wood and thermally treated densified poplar wood modified with styrene and maleic anhydride on the level 16% and 8% respectively, after 100 h of soaking in water.

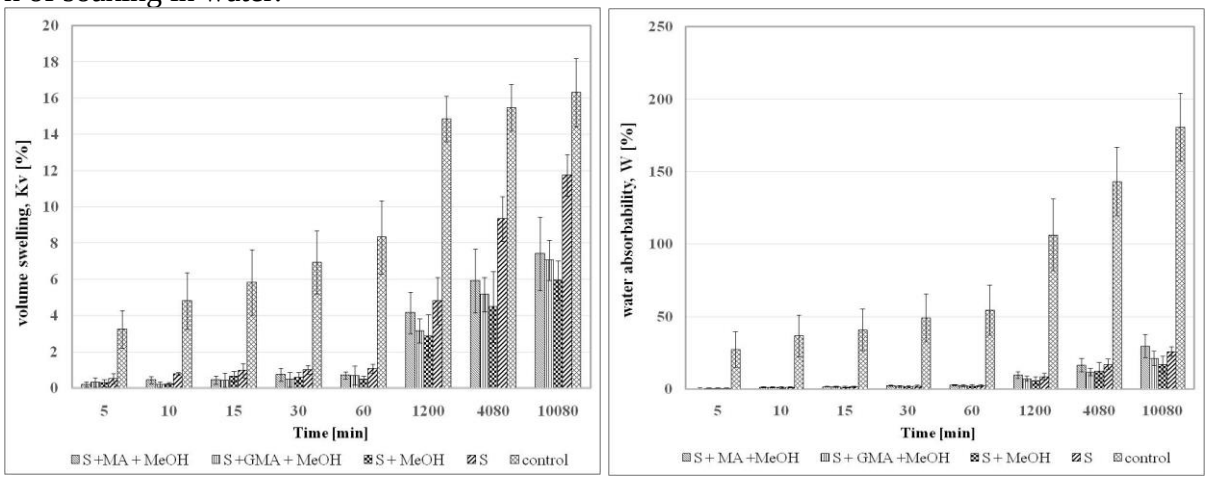

Fig. 3. Volume swelling and water absorbability of unmodified *Populus nigra* L. wood and *Populus nigra* L. wood after styrene polymerization in lumen

In case of polymerization in lumen process, the lowest water absorbability and volume swelling had samples modified with styrene and methanol. On the other hand, among all groups subjected to polymerization in lumen, samples modified only with styrene had the highest value of volume swelling (11.7%). Differences in dimensional stability of *in situ* modified wood can due to limited penetration of styrene (substance of low polarity) in wood cell wall. Hence, the use of methanol caused probably the greater swelling of wood and allowed the styrene to penetrate deeper into the structure of the wood cell wall. Thus, wood after the styrene polymerization process in the presence of methanol resulted in better dimensional stability.

In Fig. 4 colour changes of *Populus nigra* L. wood after furfurylation and polymerization in lumen were presented. Based on the results shown in Fig. 4 it can be observed that furfurylation process caused darkening of the poplar wood samples. The higher a concentration of FA was used, the darker a colour of the wood was obtained. Moreover, an addition of maleic anhydride to FA caused an even greater darkening of the wood. Similar correlations in colour changes of alder wood, pine wood and poplar wood were observed in other studies [Bartkowiak and Doczekalska 2017, Drywień and Radomski 2018].

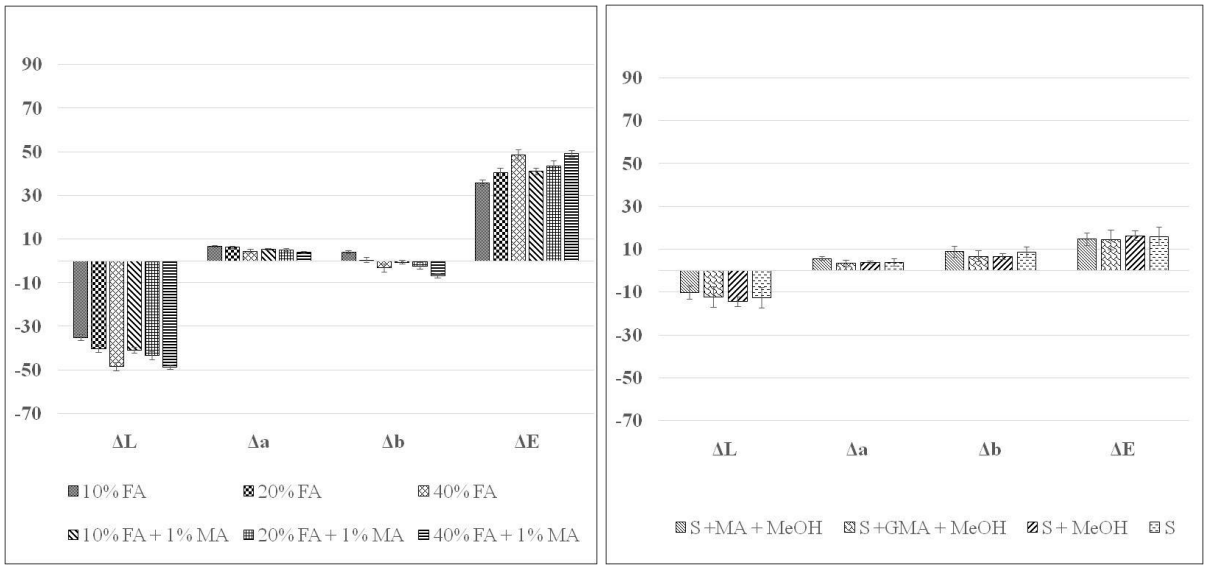

Fig. 4. Colour changes of *Populus nigra* L. wood after furfurylation and styrene polymerization in lumen

Different observations were recorded in case of a polymerization process in lumen, as an obtained colour changes were not significant (Fig. 4). Due to the polymerization process in lumen, a colour of each of a sample became slightly darker and more yellow.

Summarizing, the results obtained in this work showed, that furfurylation and polymerization in lumen had a great influence on wood properties. High WPG index, increased density and hardness of wood, decreased water absorbability and volume swelling proved, that furfuryl alcohol, maleic anhydride, glycidyl methacrylate and styrene were successfully used in wood chemical modification process. Improvement of these properties showed, that modified poplar wood can be applied in new ways.

SUMMARY AND CONCLUSIONS

- Furfurylation and polymerization in lumen processes cause significant increase of poplar wood density.
- Addition of maleic anhydride (1%) in furfurylation process causes increase of density and WPG index but also slightly decrease of wood hardness.
- Addition of methanol (10%) to styrene is very beneficial, because of obtaining the high WPG index. In turn, the addition of MA (1%) and GMA (2%) to the mixture of styrene and methanol results in a significant reduction of the WPG index.
- Under the conditions used, the chemical modification processes cause a significant increase in the Brinell hardness of poplar wood. But the cross-linking agent influences the hardness of the wood in different ways. The highest hardness was obtained when the addition of MA (1%) to the mixture of styrene and methanol was used, but the lowest when the addition of GMA (2%) was used.
- After furfurylation and polymerization in lumen processes, a dimensional stability and hydrophobic properties of poplar wood are significantly improved. The use of cross-linking agent (MA or GMA) is very beneficial in this context.
- Furfurylation process causes darkening of the poplar wood. The colour intensity increases with a concentration of FA. Addition of maleic anhydride to FA causes an even greater darkening of the wood.
- Process of polymerization in lumen does not result significant colour changes.

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Streszczenie: *Badanie wybranych właściwości drewna topoli czarnej (Populus nigra L.) poddanego furfurylowaniu i polimeryzacji w lumenie.* Celem pracy było zbadanie wybranych właściwości drewna topoli czarnej (*Populus nigra* L.) modyfikowanego alkoholem furfurylowym i styrenem. Bezwodnik maleinowy oraz metakrylan glicydylu został użyty do utworzenia kopolimeru szczepionego z drewnem. Dla modyfikowanych próbek został obliczony współczynnik WPG i gęstość oraz przeprowadzone badania: twardości, nasiąkliwości, zmiany barwy i spęcznienia objętościowego. Uzyskane wartości porównano z drewnem niemodyfikowanym topoli czarnej. Gęstość drewna modyfikowanego alkoholem furfurylowym zwiększyła się do 1,3 raza. Dodatek bezwodnika maleinowego do procesu furfurylowania spowodował zwiększenie wartości współczynnika WPG o około 5-6% jednocześnie nieznacznie obniżając twardość drewna. Nasiąkliwość oraz spęcznienie objętościowe po 10080 min (168 h) moczenia w wodzie zostało obniżone o ponad 50% w porównaniu do drewna niemodyfikowanego. Polimeryzacja w lumenie styrenem zwiększyła gęstość drewna do 2 raza, twardość do 1,5 raza natomiast współczynnik WPG był w zakresie od 50,8 do 116,6%. Dla próbek modyfikowanych w ten sposób nasiąkliwość oraz spęcznienie objętościowe po 10080 min (168 h) również zostało obniżone o ponad połowę. Furfurylowanie spowodowało intensywne ciemnienie drewna, podczas gdy polimeryzacja w lumenie spowodowała nieznaczną zmianę barwy próbek. Dodatek bezwodnika maleinowego do procesu furfurylowania spowodował jeszcze większe ciemnienie drewna.

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