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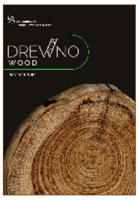

The Effect of Treatment with Fire Retardant on Properties of Birch Veneer and Manufactured Fire-Resistant Plywood

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The production of plywood from impregnated veneers allows a wood-based product protected in the entire cross-section to be obtained. The conducted research was focused on the properties of veneers impregnated with a mixture of potassium carbonate and urea, such as pH, wettability, colour change and surface roughness. In addition, the properties of plywood produced with melamine-urea-formaldehyde resin, such as the shear strength and formaldehyde emission, were the subject of research as well. It was found that the impregnated veneers were characterized by an increased pH, increased wettability, a completely different colour and unchanged surface roughness. Moreover, the impregnation of the veneers caused a decrease in the bonding quality of the plywood and a reduction in formaldehyde emissions from the finished product. It can be concluded that the melamine-urea-formaldehyde adhesive is more suitable for the production of fire-resistant plywood than the urea-formaldehyde adhesive.

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Introduction

Wood combustibility is a consequence of the considerable number of oxygen atoms present in its main components [Grześkowiak et al. 2016]. The presence of unprotected wooden elements can induce both fire growth and the production of carbon monoxide, and thus can increase the overall toxicity of a fire as well [Xu et al. 2015; Thomas et al. 2021]. Due to the fact that fire poses a great threat to people, many technical criteria for the safety of structures are constantly being developed, including the criterion of fire safety of

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the entire construction [Kawalerczyk et al. 2022b]. As a result, studies on the protection of wood-based materials against fire are still a current trend in research [Popescu and Pfriem 2020; Renner et al. 2021; Taghiyari et al. 2020; Yang et al. 2021].

In order to protect wood-based materials against fire, various fire retardants can be applied. They are substances whose purpose is to reduce the flammability of combustible materials [Mazela et al. 2020]. Bryn et al. [2016] studied the effectiveness of a formulation containing ammonium phosphate, ammonium sulphate and ammonium bromide in a weight ratio of 8:5:3, used for the impregnation of birch veneers. The aim of the study was to evaluate the effect of various parameters of diffusive impregnation on the properties and fire protection of plywood. Based on the results, the following parameters were recommended: a 30% concentration of the impregnating solution, temperature of 22°C and duration of the process of 8 min. Bekhta et al. [2016] investigated the possibility to use five different fire retardants for the impregnation of birch veneers. All of the studied chemicals contributed to improvement in the combustible properties of plywood; however, only two formulations did not cause a significant reduction in the bonding quality. The first one was a mixture of sodium dichromate, ferrous sulphate and ammonium chloride (1:1:10), and the second one was a mixture of diammonium phosphate and ammonium sulphate (1:1). Studies have also been carried out to determine the effect of veneer impregnation with borax, zinc chloride, boric acid, diammonium phosphate and a mixture of borax and boric acid on the properties of laminated veneer lumber (LVL) [Özcifci et al. 2007; Özçifçi and Okçu 2008; Kol et al. 2010]. It was found that the applied fire retardant solutions were effective in terms of improving the fire properties of the manufactured panels. Lu et al. [2022] applied the circulating technology of veneer impregnation using a melamine solution (2 wt%) and amino trimethylene phosphonic acid (15 wt%). The results showed that the implemented treatment was effective and the veneer exhibited good hygroscopic and leaching resistance. Hautamäki et al. [2020] compared the effects of veneer impregnation with sodium silicate (SS) and diammonium phosphate (DAP). Both the chemicals improved the fire properties of the wood layers. Moreover, interestingly the DAP-treated veneers demonstrated a significant increase in the glue bond strength.

This paper is a continuation of research focused on the incorporation of a formulation based on urea and potassium carbonate (K_2CO_3) as a fire retardant for plywood. The following features can be listed as the advantages of K_2CO_3: fungicidal properties, a very low harmful effect on human health and low iron corrosiveness [Grześkowiak 2012]. Previously conducted studies show that the impregnation of veneers led to improvement in the fire properties of plywood bonded with UF and PF resins; however, it also affected its bonding quality in a significant way [Kawalerczyk et al. 2019]. This part is focused on determining the effect of impregnation on the properties of veneers (surface roughness, contact angle, pH, colour changes). Additionally, since melamine-urea-formaldehyde (MUF) resins are increasingly replacing UF adhesives in the market due to their increased water resistance and lower formaldehyde emission [Mirski et al. 2020], and according to Zenkteler [1996] their curing process is less pH-sensitive comparing to UF resins, the aim of the study is to determine the suitability of an MUF adhesive for impregnated veneer gluing and the effect on plywood properties.

Materials and methods

Rotary cut birch (*Betula* L.) veneer sheets without visible defects, characterized by a moisture content of 5.3 ± 2.0%, dimensions of 320 × 320 mm and thickness of 1.4 mm were used to conduct the research. Non-modified veneers were employed as a reference and are referred to as REF later in the article. In order to perform the modification, the veneers were impregnated in containers filled with the impregnate by soaking them in the solutions based on potassium carbonate and urea for 1 hour. The applied formulations differed in the concentration and mass ratio of potassium carbonate to urea (Table 1).

Table 1. Compositions of applied fire retardants

Variant designation	Concentration (%)	K_2CO_3:urea mass ratio
A	20	1:1
B		2:1
C	30	1:1
D		2:1

After the assumed impregnation time, the veneers were laid vertically to drain and then dried in a laboratory oven at the temperature of 60°C to reach a moisture content of 5 ± 2%. The weight percentage gain (WPG) was calculated as follows:

$$\text{WPG}\ (\%) = \frac{M_m - M_n}{M_n} \times 100 \qquad (1)$$

where: Mm is the mass of modified veneer; Mn is the mass of non-modified veneer

In order to determine the pH of the veneers, the methodology used by Nemli et al. [2018] and Mirski et al. [2020] was applied. The veneers were ground in a laboratory mill and sieved to obtain a fraction of 0.315 mm. The extract solution was prepared by boiling 3 ± 0.01 g of the obtained powder in 100 ml of distilled water for 30 min. After filtration, the pH was determined with a Testo 206 pH-meter. The measurements were performed three times for each variant.

The wettability of the veneers was investigated by performing contact angle analysis by means of a DSA25 Krüss goniometer by placing a drop of distilled water with a volume of 4 µl on the surface of the loose side of the veneers. They were conditioned for 72 hours in a climatic chamber at 20 ± 1°C and 65 ± 1% relative humidity prior to testing. The measurements were repeated five times for each variant.

The colour change of the veneers was determined with a Datacolor 600 TM spectrophotometer. The following Commission internationale de l'éclairage (CIE) colour coordinates L*, a*, b* were recorded for both the non-impregnated and impregnated samples. Ten measurements were performed for each variant. The total colour change (ΔE) was determined according to Equation 2:

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

where: L* is the achromatic coordinate or luminosity (the axis value ranges from 0 to 100, where 100 means a given colour that is close to white, and 0 means a colour that is close to black); and a* and b* are the chromatic coordinates. Axis a* depicts a green ($a^* < 0$) or red ($a^* > 0$) colour, and axis b* depicts a blue ($b^* < 0$) or yellow ($b^* > 0$) colour; the ΔL* value expresses the luminosity difference; Δa* expresses the difference on the a* axis (red to green); Δb* expresses the difference on the b* axis (yellow to blue).

The colour change in relation to the reference variant was interpreted according to the criteria used by Barański et al. [2017], which are summarized in Table 2.

Table 2. Colour change criteria

ΔE* value	**Colour change**
$\Delta E^* < 0.2$	Invisible colour change
$2.0 > \Delta E^* > 0.2$	Slight change in colour
$3.0 > \Delta E^* > 2.0$	Colour change visible in high filter
$6.0 > \Delta E^* > 3.0$	Colour change visible in average quality filter
$12.0 > \Delta E^* > 6.0$	Large colour change
$\Delta E^* > 12.0$	Different colour

The surface roughness parameters were determined with a Mitutoyo SJ-210 surface roughness tester in accordance with EN ISO 4287 [1997]. Five measurements were performed perpendicularly to the direction of fibres for each side (loose and tight) of the veneer characterized by a moisture content of 10 ± 2%. The average values of the following parameters were used to evaluate the surface roughness: arithmetic mean surface roughness (R_a), total height of the profile (R_t) and maximum height of the profile (R_z). The length of the measuring path was 12.5 mm.

The melamine-urea-formaldehyde adhesive (1247, AkzoNobel, the Netherlands) characterized by the following properties: viscosity 1000 - 1500 mPa·s, pH 9.5 – 10.7, solid content 64 – 69%, gel time 64 s at 100°C , were used to manufacture the plywood. Ammonium nitrate (20 wt%) was introduced as the hardener in the amount of 5% and rye flour was added as the filler in the amount of 10%. All the components were stirred manually until proper homogenization was achieved and the resultant mixture was spread on the surface of the veneers in the amount of 160 g/m² calculated in relation to the weight of the resin solution. The pressing process of three-layer plywood was conducted using the following parameters: pressing temperature 140°C, pressing time 4 min and unit pressure 1.3 MPa. Two panels were manufactured for each variant.

After pressing, the produced plywood was conditioned at a temperature of 20 ± 5°C and relative humidity of 65 ± 2%. Afterwards, it was tested in terms of bonding quality according to EN 314-1 [2004]. The shear strength was determined after 24 hours of soaking in water and after treatment, including boiling in water for 6 hours and cooling in water for 1 hour as required by the standard. Twelve samples from each variant were used for testing. Moreover, the emission of formaldehyde was determined in triplicate utilising the flask method according to EN 717-3 [1996]. The content of formaldehyde in the collected water solution was investigated by means of the commonly used ammonium acetate and acetylacetone method and the measurements were performed on a Biosens UV-5600 spectrophotometer at 412 nm.

The outcomes of the investigations were subjected to the analysis of variance (ANOVA) at a significance level of $\alpha = 0.05$. Furthermore, in order to assess the significance of the observed changes, the HSD Tukey test was carried out.

Results and discussion

Table 3 presents the results of WPG of veneers depending on the formulation of the impregnating solution. Based on the statistical analysis, it was found that the values of WPG depended mainly on the concentration of the impregnate. As the concentration increased, the obtained WPG also grew. The increase in the concentration of the fire retardant solution by 10% led to a rise in the WPG values by approx. 9%. The greater weight gain after impregnation may indicate a better quality of protection against fire [Lin et al. 2020]. Hautamäki et al. [2020] also found that the increase in the concentration of the diammonium phosphate and sodium silicate solutions had a noticeable effect on WPG of birch veneer. On the other hand, it seems like the mass ratio of potassium carbonate to urea had no significant impact on the outcomes.

Table 3. Weight percentage gain depending on variant

	Variant			
	A	**B**	**C**	**D**
WPG (%)	13.81 (1.89)^{a}	13.76 (2.04)^{a}	22.45 (1.56)^{b}	21.88 (1.73)^{b}

Furthermore, the impregnation caused significant growth in the pH of the veneers (Table 4). Both the application of the solution with the concentration of 20% and with the concentration of 30% containing potassium carbonate and urea in a mass ratio of 1:1 resulted in an increment of approx. 22%. Moreover, the increase in the share of alkaline potassium carbonate in the solution (mass ratio of 2:1) with the concentration of 30% caused an even more noticeable rise in the pH. Considering the fact that the resins commonly used in the production of plywood cure in acidic conditions, the observed effect can significantly affect the adhesion strength [Aydin 2004].

Table 4. pH of veneer depending on variant

	Variant				
	REF	**A**	**B**	**C**	**D**
pH	6.64 (0.11)^{a}	9.57 (0.13)^{b}	9.69 (0.17)^{b}	9.61 (0.12)^{b}	10.13 (0.09)^{c}

The results of the contact angle measurements are presented in Table 5. It was found that the applied protection against fire slightly raised the contact angle of the veneers, which is a different result comparing to, for example, the results of research conducted by Yan et al. [2023] on guanidine phosphate or by Mamiński et al. [2013] on sodium chloride and silicon dioxide. Moreover, it was also found that the concentration of the impregnating solution had a significant effect on the wettability of the veneers as well. As the concentration of fire retardant increased, the contact angle of the wood surface also grew. These results are consistent with the observations of Jayamani et al. [2020], who stated that potassium carbonate can considerably decrease the hydrophilicity of wood by reacting with hydroxyl groups, by causing a change in the orientation polarization and a partial degradation of hemicelluloses. Aydin and Colakoglu [2007] stated that low contact angle values are an indication of good adhesion, and therefore the observed effect of the applied protection on the wettability of the veneer could potentially influence the results of the bonding quality observed during previous studies as well [Kawalerczyk et al. 2019].

Table 5. Contact angle of veneer depending on variant

	Variant				
	REF	**A**	**B**	**C**	**D**
Contact angle (°)	85.6 (1.8)[a]	88.4 (1.3)[b]	88.7 (1.2)[b]	91.3 (1.0)[c]	91.7 (1.1)[c]

Figure 4 shows the appearance of the non-impregnated and impregnated samples. As can be seen, the colour change was clearly visible. In addition, no crystalline stains were observed on the surface of the impregnated veneers. Their occurrence is often considered a negative effect of impregnation with the use of salt fire retardants; nonetheless, in this case the concentration of the applied solutions could have been low enough to avoid this type of discoloration [Zenkteler 1996].

Fig. 1. Appearance of veneer sheets after impregnation

Table 6 presents the colour change of the birch veneers caused by the impregnation, determined spectrophotometrically. The total colour change (ΔE*) of the investigated samples ranged from 12.10 to 14.25. According to the adopted criteria, it can be stated that the samples were characterized by a totally different colour owing to the applied modification (ΔE* > 12). The observed differences were mainly caused by the changes in the L* and b* coordinates. The decrease in the value of the L* parameter in relation to the non-impregnated wood indicates that the brightness of the samples was significantly decreased and the veneers darkened. Furthermore, the increase in the values of the a* and b* coordinates indicates that the birch veneers became yellower and redder resulting from the applied protection. No significant effect or trend related to the concentration or composition of the impregnate was observed. Such fluctuations in colour change were most likely related to the inhomogeneities of the colour of the veneers resulting from the natural anatomical structure of the wood.

Table 6. Colour change of veneers

Variant designation	ΔL*	Δa*	Δb*	ΔE*
A	-10.70	2.80	7.66	13.74
B	-9.36	2.55	6.34	12.10
C	-6.16	4.88	11.41	14.25
D	-10.61	2.95	8.02	13.46

As can be seen in Table 7, the results of the performed ANOVA clearly show that both the impregnation in general, and the applied formulation of the impregnating solution had no statistically significant effect on the investigated surface roughness parameters. The result indicating that the impregnation had no effect on the increase in veneer roughness is rather positive. According to Bekhta et al. [2020], the negative effect on the roughness parameters could lead to deterioration of the bonding quality of the produced plywood due to, for example, the formation of a thicker and less continuous glue line. Similar conclusions considering the effect of veneer protection were drawn by Aydin and Colakoglu [2007], who also observed that the impregnation with fire retardant and the composition of the applied formulation containing borax and boric acid did not affect the surface roughness parameters in a significant way. What is more, Table 7 also shows that the only statistically significant difference observed was between the loose and tight side of the veneer.

Table 7. Results of ANOVA for roughness parameters

Roughness parameter	Statistical parameter	Impregnation	Applied formulation	Side of veneer
R_a	*SS*	0.16	1.86	384.93
	Df	1.00	3.00	1.00
	F	0.06	0.23	143.52
	p	0.81	0.87	0.00
R_t	*SS*	1.00	368.00	41224.00
	Df	1.00	3.00	1.00
	F	0.01	0.28	93.99
	p	0.96	0.84	0.00
R_z	*SS*	7.00	169.00	15666.00
	Df	1.00	3.00	1.00
	F	0.06	0.45	126.51
	p	0.81	0.72	0.00

SS means sum of squares; *Df* means degrees of freedom; *F* means Fisher statistic; *p* means p-value

The averaged values of the surface roughness parameters obtained for the loose and tight side are presented in Table 8. The significant effect of the side of the veneer on its surface roughness was previously reported in some studies as well [Laskowska et al. 2018; Jankowska et al. 2021]. According to Bekhta et al. [2018], these differences are caused by the presence of small lathe checks on the loose side, which usually are not present on the tight side of the rotary-cut veneer sheets. Generally, these changes result from the technology of veneer production, not from the applied protection measure.

Table 8. Average roughness parameters depending on side of veneer

Side of veneer	R_a	R_t	R_z
Tight	11.77[a]	85.60[a]	108.93[a]
Loose	14.30[b]	101.76[b]	135.14[b]

The results of the shear strength of the plywood tested after pre-treatment consisting of both soaking in water and boiling in water are summarized in Fig. 2. The results show that the impregnation of the veneers significantly reduced the bonding quality of the resultant plywood, regardless of the pre-treatment method. Additionally, the statistical analysis also indicates that the concentration and the proportions of potassium carbonate to urea did not have any significant influence. According to Ayrilmis et al. [2009] the adverse effect of inorganic fire retardants on adhesive bonds results from the change in pH, reduction of hydroxyl groups available for bonding, mechanical interference by the salts occurring on the wood surface and incompatibility between the resin and fire retardant. In the case of this research, the observed reduction in shear strength was most likely caused by the changes in pH. As shown earlier in Table 4, the pH of the impregnated veneers was approx. 9-10, while the vast majority of the non-impregnated wood species are characterized by an acidic pH. This is particularly important since amino resins generally require an acidic environment to complete the curing process, even when subjected to high temperatures and pressure [Lei and Frazier 2015]. Moreover, the increased contact angle shown in Table 5 could also slightly contribute to the reduction in strength. A previously conducted study reported that the impregnation of veneers with a mixture of potassium carbonate and urea led to a decrease in the wet shear strength of UF resin-bonded plywood by about 89% and the obtained values fell below 1 N/mm^2 required by EN 314-2 [1993] [Kawalerczyk et al. 2019]. The results of this research reveal a much smaller reduction by approx. 25% and 13% in the case of the panels tested after soaking and boiling, respectively. It indicates that the MUF resin does not require such a low pH to cure as previously stated by Zenkteler [1996], and therefore it is more suitable for the gluing of impregnated veneers when compared to UF resin.

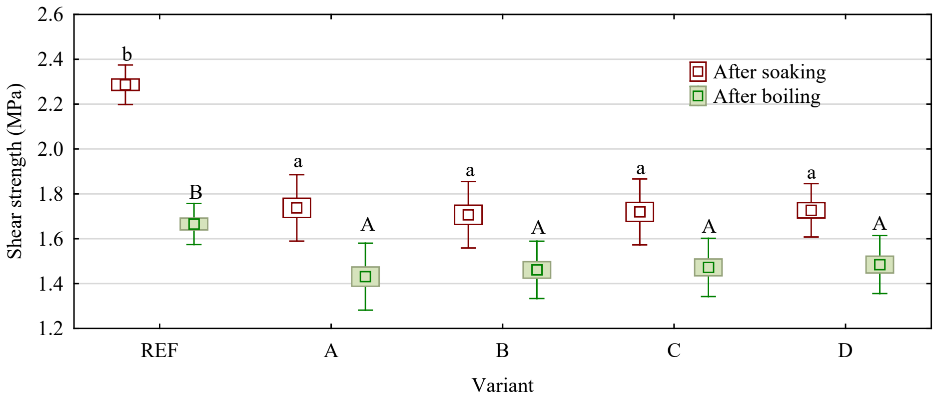

Fig. 2. Shear strength of plywood depending on variant

The results of the formaldehyde emission from the manufactured plywood are presented in Fig. 3. It was found that the impregnation of veneers contributed to a reduction in the formaldehyde emission by approx. 25%. Moreover, the statistical analysis revealed that both the applied concentration of fire retardant and its composition did not have a statistically significant effect on the results. The reason for such a reduction was probably the urea contained in the formulation, which is an amide with two amino groups in its molecule. Amino groups have the ability to react with free formaldehyde in the adhesive and hydrolysed formaldehyde in the resultant wood-based board and they exhibit great effectiveness when working as a formaldehyde scavenger [Kawalerczyk et al. 2022a, 2023]. A similar effect was previously reported by Aydin et al. [2014] in the case of plywood made of veneers impregnated with monoammonium phosphate and ammonium sulphate.

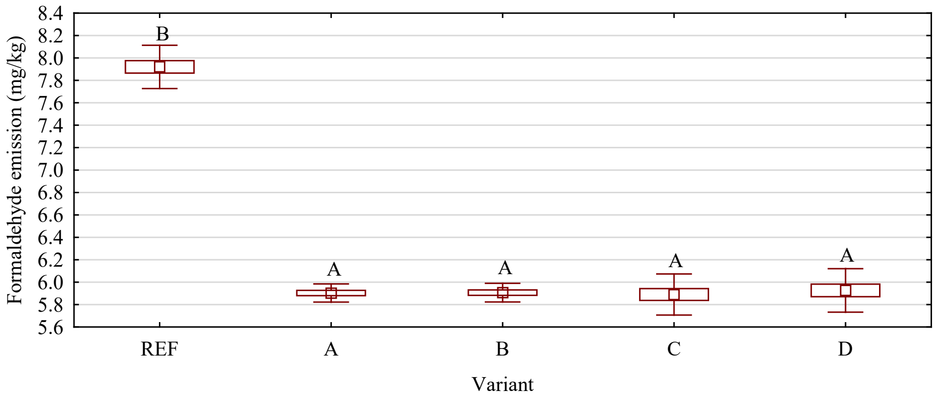

Fig. 3. Formaldehyde emission of plywood depending on variant

Conclusions

Based on the results of the performed research, it was found that the increase in the fire retardant concentration results in a rise in the weight percentage gain of the veneers. The composition of the impregnate does not influence the WPG results. The pH of the veneers significantly grows as a consequence of the applied protection measure. The highest pH was noted for the formulation characterized by the concentration of 30% and the mass ratio of potassium carbonate to urea of 2:1. In addition, it was also found that the impregnation causes a slight increase in the contact angle values and as the concentration of fire retardant rises, the wettability of the veneers also grows. Furthermore, regardless of the concentration and composition of the applied solution, the impregnated veneers have a significantly different colour. They are darker, yellower and redder when compared to the reference ones. Based on the results of the surface roughness measurements, it was found that such parameters as R_a, R_t and R_z are not affected by the impregnation. The plywood panels manufactured from the impregnated veneers are characterized by a decreased bonding quality and reduced formaldehyde emission; nevertheless, the deterioration in shear strength is not as significant as in the case of plywood bonded with urea-formaldehyde adhesive [Kawalerczyk et al. 2019].

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