The Effect of Diffusive Impregnation of Birch Veneers with Fire Retardant on Plywood Properties

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Wood is a natural, organic material that cannot be exposed to high temperatures, fire, or heat, especially when wood or wood-based materials are used as construction elements. The fire-extinguishing composition of di-ammonium phosphate, ammonium sulfate, and ammonium bromide (DAB) was used to increase the fire-resistance of birch plywood. The effects of various parameters of diffusive impregnation of the veneer (temperature and concentration of impregnating solution, duration of impregnation) were investigated. Their dependence was linear for the temperature of impregnating solution, and logarithmic for concentration of impregnating solution and duration of impregnation. Increased retention of fire retardant improved plywood fire resistance. However, considering the quality of impregnation, energy costs, and plywood properties, the following parameters of wet veneer impregnation are recommended: a temperature of impregnating solution of 22 °C, concentration of 30%, and duration of impregnation of 8 min.

Keywords: Birch veneer; Combustible materials; Diffusive impregnation; Fire retardants; Phenol-formaldehyde resin; Plywood; Shear strength

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INTRODUCTION

Birch plywood panels or laminated veneer lumber (LVL) are valuable construction materials that can be widely used in public buildings because they have unique, advantageous strength parameters resulting from their layered structure. However, the flammability of plywood limits its application in many areas. Currently, much attention is being paid to increasing the fire resistance of wood-based materials, including plywood. For example, according to the information of Ukrainian Research Institute of Civil Protection (2015), there were 68,879 fires in Ukraine in 2014, in which 2,246 people died and 1,450 were injured, and material damages amounted to 55 million €. Therefore, improving the capability of fireproof of the plywood becomes one of the important issues. To reduce the flammability and/or fire emissions, wood is treated with fire retardants. Such treatment considerably reduces the rate at which flames spread the wood surface and reduces the amount of potential heat (LeVan and Winandy 1990; Martinka *et al.* 2012). The basic fire performance properties of wood, from the view of wooden construction, have been analyzed by Delichatsios *et al.* (2003) and Zachar *et al.* (2012).

There are some main ways of decreasing the flammability of plywood panels: (a) soaking, diffusion, or impregnation of the individual veneers before gluing (Shim 1982; Grexa *et al.* 1999; Miljković *et al.* 2005; Laufenberg *et al.* 2006; Borysiuk *et al.* 2011;

Bueno *et al.* 2014); (b) incorporation of the fire retardant into the glue used for bonding (Su *et al.* 1998; Grexa *et al.* 1999; Cheng and Wang 2011); (c) impregnating the consolidated plywood with chemicals by vacuum pressure process or other methods (Kim *et al.* 1984; Kim 1987; LeVan *et al.* 1996); and (d) surface treatment by flame-retardant coating (Chou *et al.* 2009; Chou *et al.* 2010; Chuang *et al.* 2010; Chang *et al.* 2011; Yew *et al.* 2015). Some works describe the incorporation of nanomaterials into the polymeric systems to improve their fire behavior, along with analyzing the effects of several treatments based on the use of nanomaterials on the properties of natural wood veneers, primarily on their fire behavior (Bueno *et al.* 2014).

The main method for decreasing the flammability of plywood panels has been diffusive impregnation of wet veneers before gluing (Shim 1982; Miljković *et al.* 2005; Laufenberg *et al.* 2006; Borysiuk *et al.* 2011; Cheng *et al.* 2011; Bekhta *et al.* 2016). The fire protection class of plywood depends on the retention of the fire retardant. The retention of the fire retardant depends on the impregnation parameters, such as temperature and concentration of impregnating solution, along with duration of impregnation (Bekhta *et al.* 2016). In that work it was shown that treatment of birch veneers with DA (di-ammonium phosphate : ammonium sulphate at the ratio 1:1) aqueous solution is the most effective to improve the fire behaviour of plywood. Whereas the DA fire-retardant provides the necessary fire-resistance and shear strength properties of plywood, it is characterized by an increase in corrosive activity (which is caused by the content of ammonium sulphate). Therefore, to reduce corrosion of metals it can be recommended to replace of ammonium sulphate by a less actively corrosive substance.

The aim of this research was to modify of DAB fire-retardant and optimize the regime of diffusive impregnation of rotary-cut birch veneer by modified DAB fire-retardant to obtain fire retardant-treated plywood with the standard corresponding mechanical properties.

EXPERIMENTAL

Materials

Rotary-cut veneer sheets of birch wood (*Betula verrucosa* Ehrh.) free from defects with dimensions of 250 × 250 mm, thickness of 1.5 mm, and moisture content of approximately 100% were used for the experiments.

The modified DAB extinguishing composition was prepared by mixing di-ammonium phosphate, ammonium sulfate, and ammonium bromide in a weight ratio of 8:5:3. Diffusive impregnation process was realized by immersion of wet veneers into the DAB solution in a stainless steel container according to the regime given in Table 1.

Table 1. Impregnation Process

Concentration of Impregnating Solution (%)	10, 30, 50
Duration of Impregnation (min)	5, 40, 75
Temperature of Impregnating Solution (°C)	20, 40, 60

The factors listed in Table 1 determine the DAB retention and impact on the plywood fire-resistance, physical properties, and mechanical properties. The level of the upper temperature was limited because ammonia is released from the fire retardant mixture at temperatures above 70 °C.

Methods

Five-layer experimental plywood panels were laboratory-prepared from impregnated and non-impregnated (control) veneers using commercial phenol-formaldehyde (PF) resin Lignofen G/3/P (Lerg S. A., Pustków, Poland). All veneers were dried to a moisture content of 6% to 7% before resin application. The PF resin (solid content of 42% and viscosity Ford 4/20 of 120 s) was uniformly spread on one side of the veneers. Veneers were then laid up and hot-pressed in a laboratory press using the parameters in Table 2.

Table 2. Plywood Pressing Parameters

Pressing Temperature (°C)	130
Pressure (MPa)	2.0
Pressing Time (min)	10
Glue Spread (g/m^2)	120

The plywood fire-resistance was determined in a combustion chamber according to EN ISO 11925-2 (2011). The testing procedure was based on the exposure of the testing samples to a thermal infrared heater (radiant heat source) in 10-min intervals at a set distance (30, 35, 40, 45, or 50 mm) from the radiating heater surface. During the test, the weight loss was recorded in regular 10-s intervals. The efficiency of the fireproof treatment was assessed as the resistance of the treated plywood to weight loss (*WL*, %), as described by Eq. 1,

$$WL = (W_b - W_a) / W_b \times 100 \quad (1)$$

where W_b is the initial weight before the fire test (g) and W_a is the weight after the fire test (g).

Shear strength of the plywood was determined on standard test pieces according to EN 314-1 (2005) by two methods: (A) – pretreatment for intended use in dry conditions: immersion for 24 h in water at 20 ± 3 °C; and (B) – pretreatment for intended use in humid conditions: boiling for 6 h in water, followed by 1 h of cooling in water at 20 ± 3 °C. The following mechanical and physical properties of fire-resistant plywood were also determined: compression ratio (*CR*), water absorption (*WA*), and thickness swelling (*TS*).

The compression ratio (*CR*, %) is expressed as:

$$CR = (S_1 - S_2) / S_1 \times 100 \quad (2)$$

where S_1 is the sum of the veneer thicknesses (mm) and S_2 is the thickness of the plywood (mm).

Water absorption and swelling in the thickness of the plywood were determined according to EN 317 (1995). Plywood samples (100 × 100 mm) were immersed in distilled water at a temperature of 20 °C. The test pieces were weighed, and the thicknesses were measured at four points before their immersion. The samples were placed on the edge on the grid so that they did not touch each other or the walls of the vessel. The test pieces were regularly weighed, and their dimensions were measured. The first weighing and thickness measurement were performed after 24 h of immersion, and then after 48, 72, and 120 h. The surface of the samples was dried by filter paper before weighing and measuring the thickness. The thickness was measured at the same point after each period. Water absorption (*WA*, %) is expressed as,

$$WA = (m_1 - m) / m \times 100 \quad (3)$$

where m is the mass of the sample before immersion (g) and m_1 is the mass of the sample after immersion (g).

Thickness swelling (*TS*, %) is calculated as,

$$TS = (S_1 - S) / S \times 100 \quad (4)$$

where S is the thickness of the sample before immersion (mm) and S_1 is the thickness of the sample after immersion (mm).

A regression analysis was used to determine correlation between retention of DAB and impregnation parameters (concentration and temperature of the impregnating solution, the duration of impregnation) as well as the correlation between *WL*, σ_{sh}, and *CR*. Reliability of approximation (R^2) of experimental data was carried out by the least squares method.

RESULTS AND DISCUSSION

Our preliminary study (Bekhta *et al.* 2016) showed that increased moisture content has a positive impact on the diffusive impregnation process and can be a factor in reducing the duration of impregnation (Fig. 1). The effect can be explained by the filling of wood cavities by water and therefore by easier diffusion of water inside wood. As can be seen, the increase in moisture content promotes the fire retardant absorption of veneer. The amount of time needed for veneer impregnation with approximately 100% moisture content is 8 min, and the needed time for a moisture content of 70 to 80% is 70 min. That is why the veneer with maximum possible moisture content, which can be achieved in the laboratory, was used in this study.

Increasing temperature and concentration of DAB solution led to higher retention of the fire retardant. This dependence was linear for the temperature of impregnating solution and logarithmic for the concentration of impregnating solution and duration of impregnation. The effect of temperature on the retention of fire retardant is linear, because the increase in temperature causes an increasing the rate of diffusion, which leads to more intense fire retardant absorption. With increasing concentration of solution, the concentration gradient from the outside and inside of veneer is increased, which contributes to saturation of veneer by salts.

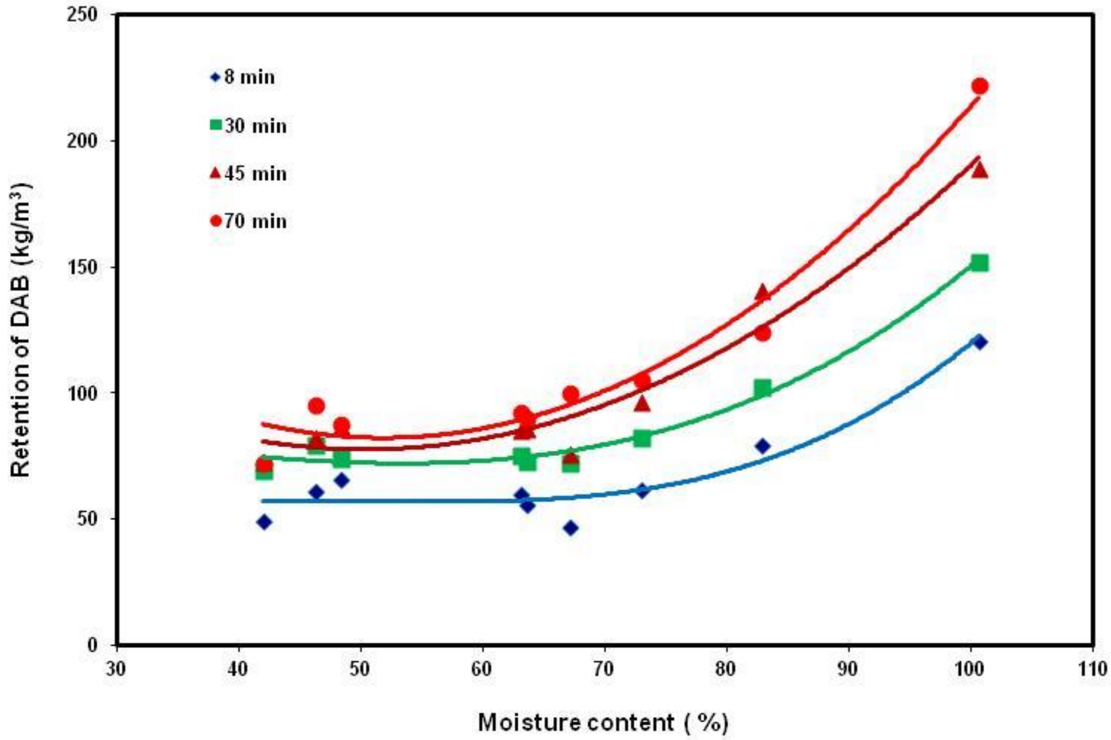

Fig. 1. The retention of DAB *versus* moisture content of veneer at various duration of impregnation

At the beginning of the impregnation process the concentration of fire-retardant within veneer is minimal (around zero), and the concentration of impregnation solution is maximal. That is, at the beginning of impregnation the initial gradient (difference between concentrations in the solution and veneer) is maximal. Then fire-retardant starts to penetrate the veneer. After that, the concentration of fire-retardant increases in the veneer and decreases in the impregnation solution, and therefore the gradient of concentration decreases. The concentration tends to equalize within veneer and in the impregnation solution. The greater the gradient of concentration, the faster is the diffusion of fire-retardant into veneer. This also explains the rapid saturation of fire-retardant into veneer at the beginning of impregnation process and a slight absorption of fire-retardant with increasing duration of impregnation.

The logarithmic dependence of fire retardant absorption on the needed duration of impregnation and concentration of impregnating solution can be explained by the rapid absorption of fire retardant into the veneer at the beginning of the impregnation process and the slower absorption before the veneer became saturated. The results of a previous scanning electron microscope (SEM) analysis (Cheng *et al.* 2011) showed that the fire retardant was mostly confined to the trachea lumen of the wood, the wall of the trachea, and around the pits when using an impregnation method at ambient temperature and pressure. These findings were confirmed by results obtained in this study.

The graphic interpretation shown in Fig. 2 presents the impact of impregnation parameters on the retention of DAB.

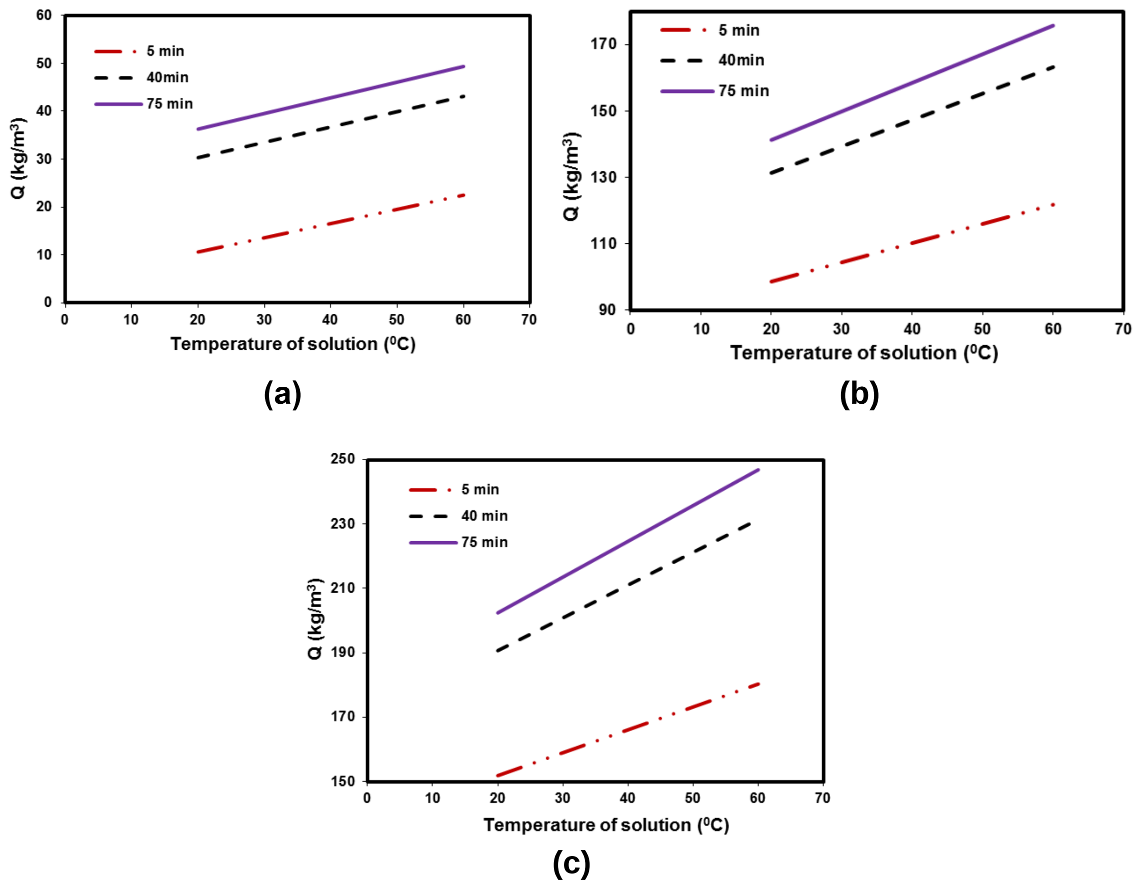

Fig. 2. The retention of DAB *versus* temperature of the solution at various concentrations of impregnating solution and duration of impregnation: (a) g = 10%, (b) g = 30%, (c) g = 50%.

Based on the obtained experimental results, the dependence of the retention (Q, kg/m^3) of DAB on impregnation parameters is described by the following relationship,

$$Q = -64.39 + 8.45 \times \ln(g)^{2,23} - 126.02 \times \ln(t)^{-33,9} + 1463.79 \times T^{-406,7} + \\ + 0.12 \times \ln(g) \times T - 0.18 \times \ln(t) \times T + 4.02 \times \ln(g) \times \ln(t) + 0.08 \times \ln(g) \times \ln(t) \times T \quad (5)$$

where g is the concentration of the impregnating solution (%), t is the duration of impregnation (min), and T is the temperature of the impregnating solution (°C).

The maximum retention of fire retardant 251.6 kg/m^3 was achieved at the temperature of 60 ºC and the concentration of 50% impregnating solution and duration of impregnation 75 min. An increase in impregnation duration is ineffective because the dependence is logarithmic, and further increasing the duration has no effect on the retention of fire retardant.

The graphic interpretation shown in Fig. 3 presents the impact of DAB concentration on the retention.

The increase in retention of DAB reduced weight loss during the plywood fire-resistance test (Fig. 4). When the fire retardant content was 39 kg/m^3, the weight loss of the treated plywood was 18.9%, and the combustion duration was 30 s. No smoldering was observed. This wood-based material corresponded to group “D” (medium combustible materials, with weight loss higher than 10%) according to EN 13501-1 (2010). The plywood with a lower content of fire retardant burned completely.

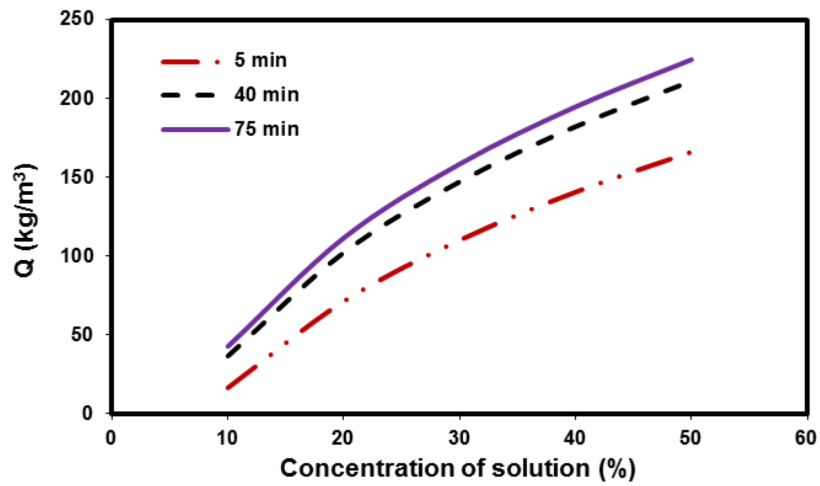

Fig. 3. The retention of DAB *versus* concentration of impregnating solution at various durations of impregnation

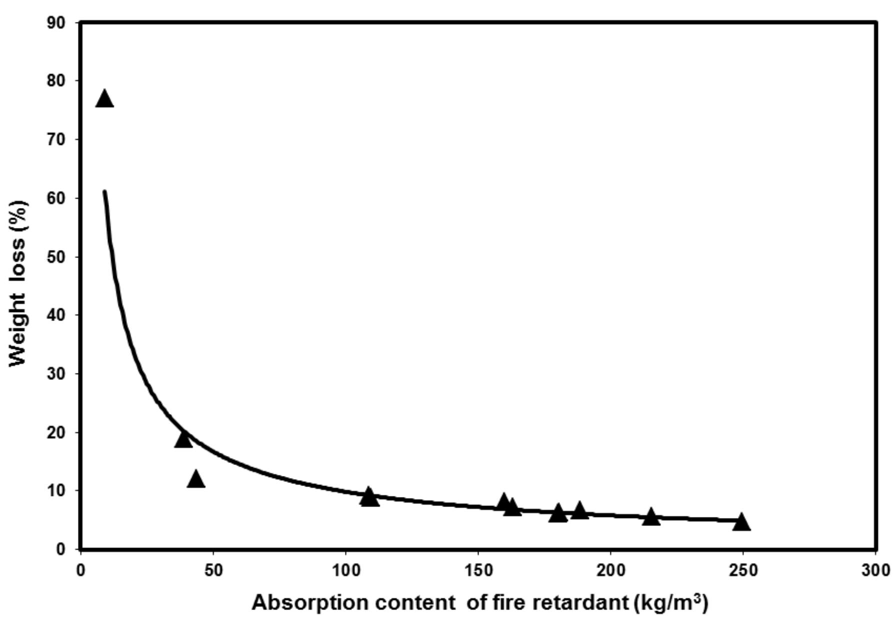

Fig. 4. The fire-resistance of plywood *versus* retention of DAB

The best effect on fire protection was achieved when the content of DAB in the veneer was 105 kg/m^3. The weight loss of the plywood was 8.9%, and there was no self-combustion or smoldering of specimens. The shear strength of plywood with specified fire retardant content was greater than the shear strength of the control specimen (non-impregnated plywood – 3.0 MPa (A) and 2.5 MPa (B)) and corresponded to EN 314-2 (1998) standard requirements, where the value should be over 1 MPa.

The obtained retention level of the DAB after treatment was not much higher than those in commercial applications, where typically used levels are between 32 and 80 kg/m^3 (LeVan and Winandy 1990).

These results are comparable with the results of Terzi *et al.* (2011) from the cone calorimeter tests, who estimated the flame spread index in the Steiner tunnel test (ASTM

E 84-07 (2007)). The heat release rates of the specimens treated with di-ammonium phosphate and ammonium sulfate were lower than those of the untreated control samples.

The water-borne fire retardants decreased the contact angle values of the wood surface because of their hygroscopic characteristics. Crystals tend to accelerate the reaction, which would decrease penetration and mechanical interlocking of the adhesive into the porous structure of the wood (Ayrilmis *et al.* 2009).

The shear strength test is commonly used as a fundamental indicator of the adhesive performance in plywood. Fire retardants have different effects on the shear strength of plywood. As reported by Aydin and Colakoglu (2007), the layer of borax and boric acid crystals on the veneer surface after drying might have been an obstruction to intimate contact between the veneer surface and adhesive molecules. The other reason for poor bonding after treatment may have been the second drying process applied to the veneers. Similar results were found in other studies (Lebow and Winandy 1999; Borysiuk *et al.* 2011).

The dependences of weight loss (WL, %) and plywood shear strength (σ_{sh}, MPa) on the retention of DAB are described by the following functions:

$$WL = 147.93 \times Q^{-0.6018} \qquad (R^2 = 0.92) \qquad (6)$$

(A) Shear strength after immersion for 24 h in water at 20 ± 3 °C:

$$\sigma_{sh} = 0.8151 \cdot \ln(Q) - 0.5543 \qquad (R^2 = 0.84) \qquad (7)$$

(B) Shear strength after immersion for 6 hours in boiling water followed by cooling in water at 20 ± 3 °C:

$$\sigma_{sh} = 0.6598 \times \ln(Q) - 0.4341 \qquad (R^2 = 0.87) \qquad (8)$$

Figure 5 shows the impact of the DAB content on the shear strength of plywood.

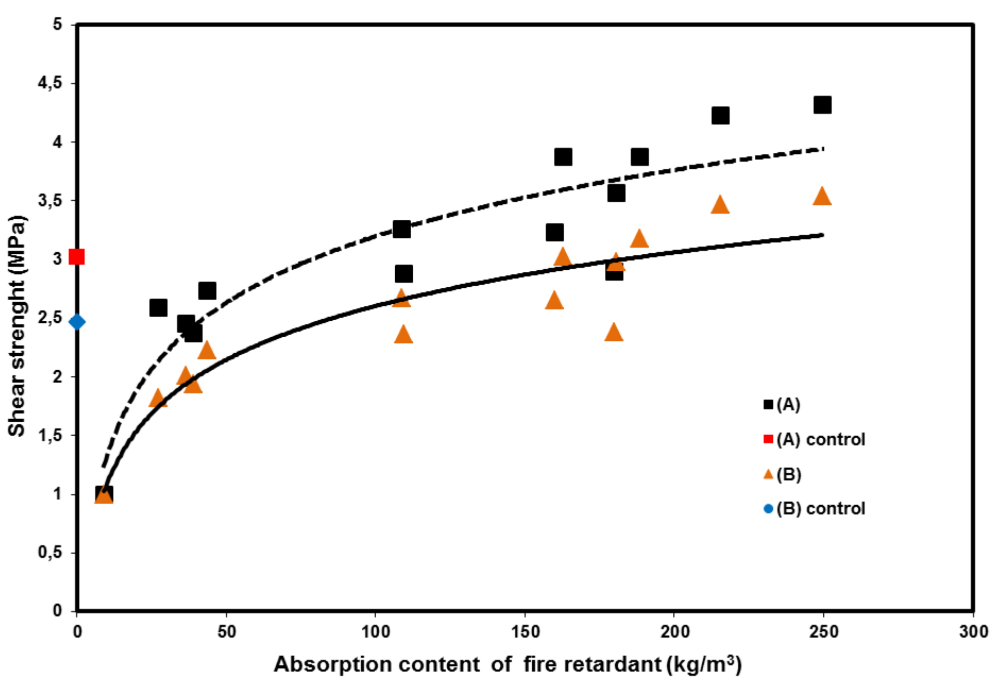

Fig. 5. The shear strength of plywood *versus* retention of DAB

The presence of crystals in the wood cavities also contributed to the reduction of the compression ratio of the plywood (Fig. 6).

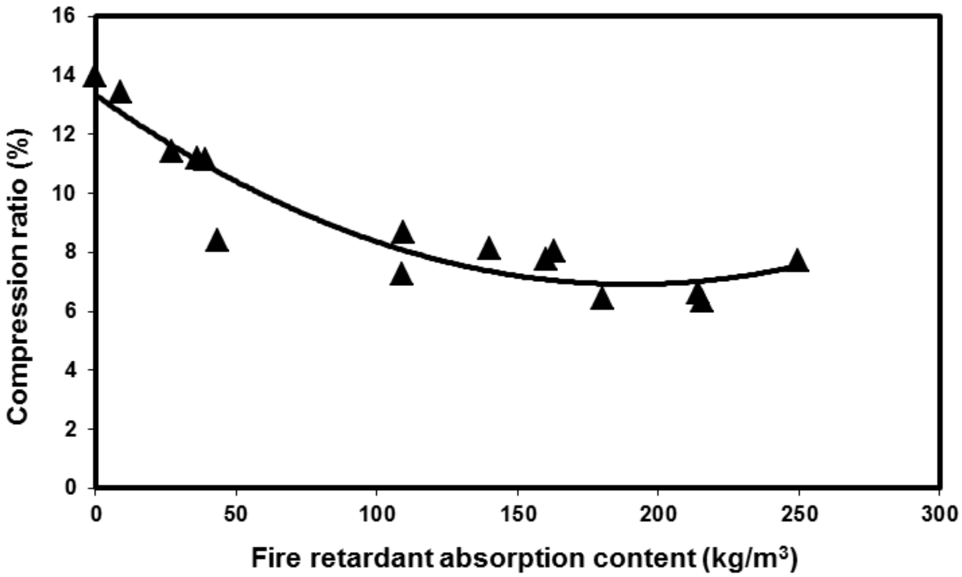

Fig. 6. The compression ratio of plywood *versus* retention of DAB

The dependence of the compression ratio of the plywood (*CR*, %) on the DAB content is described by the function:

$$CR = 13.337 - 0.0674 \times Q + 0.0002 \times Q^2 \qquad (R^2 = 0.88) \qquad (9)$$

Average values of weight loss and shear strength of plywood panels at the different retention of DAB are given in Table 3.

Table 3. Weight Loss and Shear Strength of Plywood Panels

Index	The retention of DAB (kg/m^3)							
	0	39.1	43.6	109.5	163.0	180.5	215.5	249.5
Weight loss (%)	-	18.9	12.1	8.9	7.2	6.4	5.6	4.8
Shear strength (MPa) - A	3.02	2.37	2.73	2.88	3.88	3.57	4.23	4.32
Shear strength (MPa) - B	2.47	1.94	2.23	2.36	3.02	2.98	3.47	3.54

Average thickness swelling and water absorption values of plywood panels at the different retention of DAB and duration of immersion are given in Table 4.

Water absorption decreased when fire retardant content increased. However, it should be noted that the cavities were filled with fire retardant crystals. The fire retardants were water-soluble, so they were dissolved during the experiment, and water took their place in the cavities. Therefore, it is difficult to determine the real value of water absorption that occurred. The influence of plywood water absorption on retention of DAB is shown in Fig. 7.

Table 4. Thickness Swelling and Water Absorption of Plywood Panels

The retention of DAB (kg/m³)		0	9.1	27.4	43.6	109.5	139.9	163.0	180.5	215.5	249.5
Thickness swelling (%)	24 h	16.7 (1.7)	26.0 (1.6)	18.0 (1.1)	12.1 (1.7)	7.8 (2.1)	6.1 (1.3)	6.7 (2.0)	6.7 (1.8)	4.3 (1.4)	5.6 (1.4)
	48 h	17.5 (1.6)	28.1 (1.4)	19.6 (2.6)	13.6 (1.6)	8.5 (2.0)	7.9 (1.0)	7.5 (1.7)	7.2 (1.8)	5.3 (1.1)	7.2 (1.2)
	72 h	17.9 (1.7)	22.1 (0.6)	19.9 (1.6)	14.3 (1.5)	9.1 (2.1)	9.2 (1.3)	8.2 (1.1)	8.0 (1.7)	6.3 (0.9)	8.5 (1.3)
	120 h	18.3 (1.4)	22.9 (0.2)	20.3 (1.4)	14.6 (1.6)	9.8 (1.9)	9.9 (1.4)	9.1 (0.9)	8.4 (1.6)	6.9 (1.0)	9.4 (1.3)
Water absorption (%)	24 h	49.4 (1.9)	42.8 (1.7)	46.3 (4.3)	44.6 (2.3)	30.6 (1.5)	25.0 (1.0)	26.6 (1.3)	26.1 (1.9)	18.0 (1.1)	18.8 (1.2)
	48 h	52.3 (1.9)	45.9 (0.3)	51.0 (4.2)	49.5 (1.8)	34.3 (1.1)	29.0 (0.9)	30.8 (1.2)	30.5 (2.0)	21.5 (1.4)	23.4 (1.2)
	72 h	55.2 (2.3)	50.0 (0.1)	53.5 (4.1)	51.0 (1.4)	35.4 (0.9)	30.2 (0.8)	32.5 (1.2)	31.2 (2.0)	23.2 (1.3)	25.0 (1.2)
	120 h	56.1 (1.9)	52.1 (0.1)	55.8 (4.1)	52.1 (1.2)	36.2 (0.8)	31.1 (1.0)	32.8 (1.3)	32.8 (2.4)	25.0 (1.5)	25.4 (1.1)

Values in parenthesis are standard deviations.

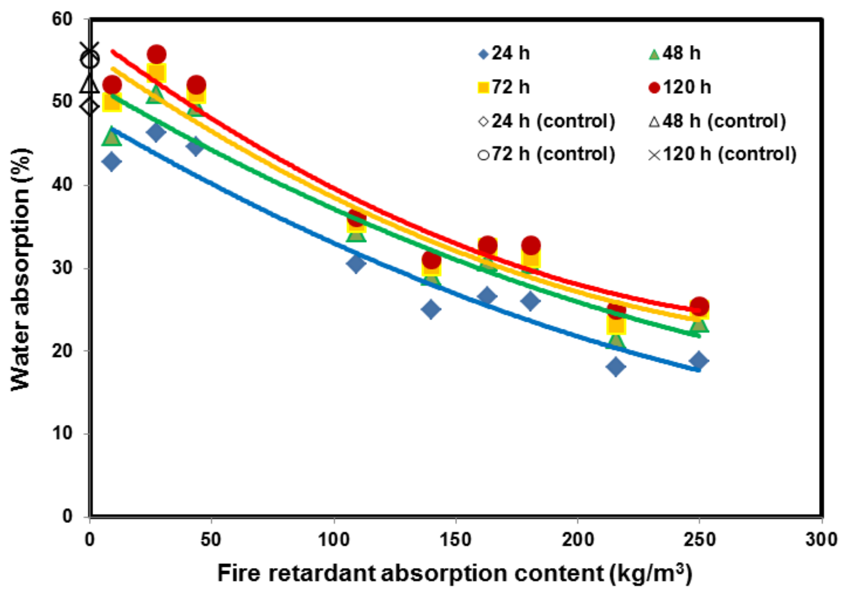

Fig. 7. The water absorption of plywood *versus* retention of DAB

There was a decrease in plywood thickness swelling when fire retardant content increased. This could be explained by a lower compression ratio with increased fire retardant absorption content. The reverse process of thickness swelling would also be less. The influence of fire retardant content on the thickness swelling of plywood during immersion is given in Fig. 8.

The obtained results for *TS* are in compliance with the results of Dundar *et al.* (2009), who indicated that the phosphate compounds did not have a significant negative effect on the dimensional stability of LVL samples, even though the water uptake significantly increased with fire-retardant treatment above 65% relative humidity. These results indicate that phosphate compounds can be used as fire retardants in LVL manufacture.

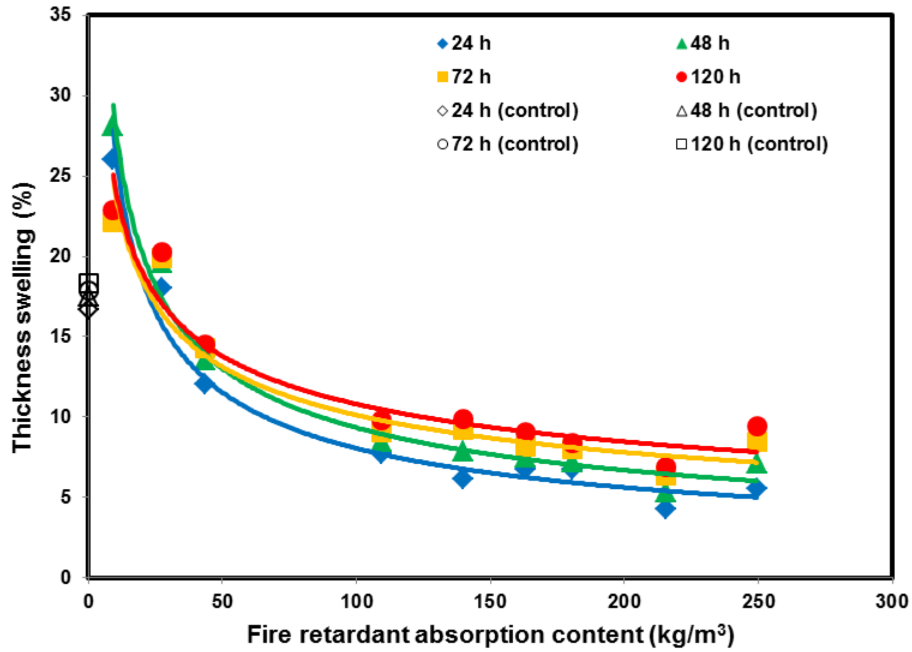

Fig. 8. The thickness swelling of plywood *versus* retention of DAB

CONCLUSIONS

1. It was found that high moisture content of veneer has positive impact on the diffusive impregnation process and can be a factor in reducing the duration of impregnation.
2. The fire protection class of plywood depends on the retention of the fire retardant. Mathematical dependence of the impregnation parameters (temperature and concentration of impregnating solution, duration of impregnation) on the retention of fire retardant was recorded. The obtained model makes it possible to predict the amount of absorbed fire retardant at different parameters of impregnation and to determine a fire protection class of plywood panels. The reliance of fire retardant retention is linear for temperature of impregnating solution and logarithmic for duration of impregnation. An increasing retention of fire retardant improves the fire resistance of plywood.
3. Considering the shear strength and fire-resistance of plywood, and also economic aspects, it is recommended to implement a diffusive method of raw veneer impregnation at a temperature of impregnating solution of 22 °C, concentration of impregnating solution of 30%, and duration of impregnation of 8 min. These parameters provide retention of DAB of approximately 105 kg/m^3 and weight loss of 8.9% after the combustion test. This allows the classification of fire retardant-treated plywood in the group of hard combustible materials (class "C", *WL* < 10%). The mechanical strength of such fire-resistant plywood is 2.6 MPa.
4. Decreases in plywood thickness swelling and water absorption were observed when fire retardant content was increased.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Slovak Research and Development Agency, contracts No. APVV-0200-12 and No. APVV-14-0506. The authors are also grateful for the support of the 7.FP, contract No. PIRSES-GA-2011-295260 "ECONANOSORB".

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Article submitted: May 8, 2016; Peer review completed: June 12, 2016; Revised version received and accepted: August 26, 2016; Published: September 8, 2016.
DOI: 10.15376/biores.11.4.9112-9125