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IMPREGNATION OF SPRUCE WOOD WITH FIRETEX / BORON COMPOUNDS AT VARIOUS CONCENTRATIONS / DIPPING TIMES AND THE EFFECT ON SOME TECHNOLOGICAL PROPERTIES

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ABSTRACT

In this study, according to the ASTM 1413-76 standard oriental spruce (*Picea orientalis* (L.) Link.) was impregnated with Firetex and boron compounds at different concentrations (1%, 2%, 3%) and dipping times (25, 35, 45, 55 min) and changes in technological properties were investigated. The determined technological properties of the samples included % retention, bulk / air dry specific gravity, bending strength, modulus of elasticity in bending and compressive strength parallel to the grain. In the experiment the highest bulk dry specific gravity was recorded in the 25-minute immersion time (0.41 g/cm^3) for a single application of the Firetex preparation, while for air dry specific gravity it was the 25-minute immersion time for a single application of the Firetex preparation (0.39 g/cm^3). In terms of mechanical properties the highest bending resistance was found in the case of the 45-minute immersion in Firetex (70.61 N/mm^2), the highest modulus of elasticity in bending was recorded for 35-minute immersion in the Firetex preparation (8100 N/mm^2), while compressive strength parallel to the grain was highest at 25-minute immersion time in the Firetex preparation (33.56 N/mm^2).

Keywords: Firetex, boron, composites, impregnation, concentration, technological properties

INTRODUCTION

Wood is one of the oldest and most important raw materials used by humans. Trees being renewable resources are commonly found worldwide. Wood as a material is characterised by such properties as hardness, strength, elasticity, colour parameters e.g. lightness L^*, potential for modification and improvement of some properties, screw nail holding capacity as well as adhesion. The natural strength of wood materials with such a wide range of usage is not sufficient. Being an organic substance wood exhibits some undesirable

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characteristics, thus measures need to be taken to prevent dimensional changes due to absorption of water, flammability and damage by insect and fungal pests. It has to be treated with protective chemicals to prevent deterioration and prolong its service life. All these negative properties of wood materials may be reduced by some protective measures and impregnation techniques. In addition to being resistant to external factors (both biotic and abiotic), impregnated wood thanks to its economic and aesthetic properties plays an important role in the construction industry. Railway sleepers, wooden groynes, cooling towers, landscaping, outdoor furniture and construction structures are examples of major applications of water-based impregnants. Water-based impregnants generally modify the porous structure in the treated wood and various surface treatments may be performed after the impregnation treatment. It may be easily carried out on site and during transportation (Kartal, 1998).

Although wood can become resistant thanks to the use of various chemicals with no need for any additional measures, the diversity of risks and prolonged exposure to adverse factors require chemical processes. Because wood is a hygroscopic material, it changes in size and volume depending on ambient temperature and relative humidity at the place of use, it may burn or be destroyed by biotic and abiotic factors (Örs and Keskin, 2008).

Within the scope of this study, spruce wood, which is a major natural resource of Turkey, was immersed applying various dipping times (25, 35, 45, 55 min) and concentrations of the Firetex preparation, boric acid and borax (1, 2, 3%). The aim of the experiment was to provide useful practical data for the furniture / wood industry by determining the technological properties of such treated wood.

MATERIALS AND METHODS

In this study it was decided to use wood of oriental spruce (*Picea orientalis* (L.) Link.) grown in Turkey. Radial sapwood samples were prepared following the TS 2470/2471 standard (TS 2470, 1976; TS 2471, 1976). The Firetex preparation and boron derivatives (boric acid and borax) were applied as impregnating agents. Samples were prepared according to the TS 2470 (1976), 2471 (1976), TS 4176 (1985), TS (53) 1981 standards, so that they were smooth, knotless, crack-free, free from tulle formation and growth defects, colour and density differences, as well as ensure a lack of reaction wood, fungus and insect damage. The tested samples were obtained from the sapwood part of spruce tree trunks. For impregnation treatment air-dried samples of spruce wood were prepared at 20 (radial) × 20 (tangential) × 360 (longitudinal) mm^3 for tests of bending strength and modulus of elasticity in bending, 20 (radial) × 20 (tangential) × 30 (longitudinal) mm^3 for tests of compressive strength parallel to the grain and 20 × 20 × 30 mm for air and bulk dry specific gravity tests. The wood samples were treated with aqueous solutions of boric acid, borax and Firetex according to the ASTM D 1413 (1976) standard test method. The impregnation process was performed by dipping for 25, 35, 45, 55 minutes applying 1%, 2% and 3% solution concentrations. After the impregnation process, the tested specimens were conditioned at 20 ±2°C and 65 ±3% relative humidity in a conditioning room until they reached constant mass. The moisture content of the tested samples was determined according to the TS 2471 (1976) standard. Air and bulk dry specific gravity values were determined according to the TS 2472 (1976) standard. Bending strength, modulus of elasticity in bending and compressive strength parallel to the grain were recorded in

Fig. 1. Universal testing machine
Rys. 1. Uniwersalna maszyna testująca

Table 1. Solution properties
Tabela 1. Właściwości roztworów

Solution Roztwór %	Impregnation agent Czynnik impregnujący	Solvent Rozpuszczalnik	Temperature Temperatura °C	pH		Density Gęstość g/ml	
				IB	IA	IB	IA
1	boric acid kwas borny	distilled water woda destylowana	22	7.83	7.83	0.971	0.971
2	boric acid kwas borny	distilled water woda destylowana	22	7.02	7.02	0.968	0.968
3	boric acid kwas borny	distilled water woda destylowana	22	6.98	6.98	0.964	0.964
1	borax boraks	distilled water woda destylowana	22	7.01	7.01	0.955	0.955
2	borax boraks	distilled water woda destylowana	22	7.05	7.05	0.947	0.947
3	borax boraks	distilled water woda destylowana	22	7.43	7.43	0.965	0.965
100	Firetex	–	22	1.65	1.65	1.215	1.215

IB – before impregnation, IA – after impregnation.
IB – przed impregnacją, IA – po impregnacji.

accordance with the TS 2474 (1977), TS 2478 (1976) and TS 2595 (1977) standards, respectively. The universal testing machine for determining the mechanical properties is shown in Figure 1.

Retention rates (R:%) were calculated from the following equation after the samples were fully dry before and after impregnation.

$$\% R = Moia – Moib / Moib \times 100, \% \text{ retention}$$

where:
Moia – bulk dry weight of the tested sample after impregnation, g,
Moib – bulk dry weight of the tested sample before impregnation, g.

RESULTS AND DISCUSSION

The solution properties are given in Table 1.

No significant changes in pH and concentrations were recorded before and after impregnation. In particular, the basic / acidic structure in the solution showed a positive / negative effect on the physical and mechanical structure.

Retention values are given in Table 2 and the related graph is shown in Figure 2.

The data given in the table and the graph showed the highest % retention value for the 55-minute immersion time (2.25%) for a single use of the Firetex preparation and the 55-minute immersion time (0.55%) for a single use of the lowest boric acid concentration.

Specific gravity changes are given in Table 3.

Based on the data presented in the tables and graphs a partial increase in values was observed in all impregnation variants compared to the control. It was found that the application of the Firetex preparation alone depending on the immersion time causes a significant increase resulting in the highest air dry specific gravity at a 25-minute immersion (0.41 g/cm^3), while the value was lowest at 45- and 55-minute immersion (0.33 g/cm^3). The highest bulk dry specific gravity (0.39 g/cm^3) was also recorded at the use of Firetex at a dipping time of 25 minutes. Gür (2003)

Table 2. % retention values
Tabela 2. Wartości procentowe retencji

Impregnating agents Czynniki impregnujące	Concentration Stężenie %	Retention, %			
		immersion times, min – czas zanurzania, min			
		25	35	45	55
Firetex	100	2.12	1.78	1.36	2.25
Borax Boraks	1	0.86	1.11	1.34	1.09
	2	1.37	0.96	1.62	1.43
	3	1.99	1.16	0.99	1.64
Boric acid Kwas borny	1	0.78	0.85	0.91	0.67
	2	0.90	0.95	0.89	0.73
	3	0.87	0.65	0.72	0.55
1th borax + 2nd Firetex 1. boraks + 2. Firetex 1th boric acid + 2nd Firetex 1. kwas borny + 2. Firetex		In the impregnation process. The first impregnation was made and the second impregnation process was performed. W procesie impregnacji. Pierwsza impregnacja została wykonana i druga impregnacja została wykonana.			

10 samples were used for each group and average values were written in the Table.
Użyto 10 próbek dla każdej grupy; przeciętne wartości zostały zapisane w tabeli.

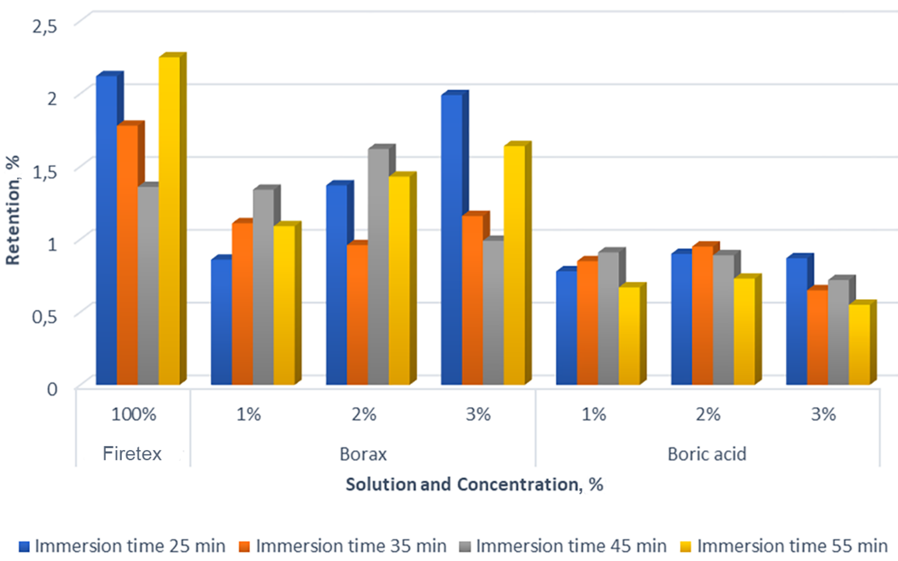

Fig. 2. % retention
Rys. 2. Retencja, %

Table 3. Air and bulk dry specific gravity
Tabela 3. Pozorny ciężar właściwy i gęstość w stanie powietrznosuchym

Impregnation agents Czynniki impregnujące	Control sample – no. and concentration, % Próbka kontrolna – nr i stężenie, %		Air dry specific gravity 0.32 g/cm³ Gęstość w stanie powietrznosuchym 0,32 g/cm³				Bulk dry specific gravity 0.29 g/cm³ Pozorny ciężar właściwy 0,29 g/cm³			
			immersion times, min – czas zanurzania, min							
			25	35	45	55	25	35	45	55
Firetex	1	100	0.41	0.40	0.39	0.38	0.39	0.37	0.38	0.36
Borax Boraks	2	1	0.37	0.36	0.35	0.36	0.35	0.33	0.32	0.34
	3	2	0.34	0.36	0.33	0.33	0.33	0.34	0.30	0.31
	4	3	0.37	0.36	0.35	0.36	0.35	0.33	0.32	0.34
Boric acid Kwas borny	5	1	0.36	0.37	0.34	0.33	0.33	0.34	0.30	0.30
	6	2	0.38	0.35	0.35	0.34	0.34	0.33	0.32	0.31
	7	3	0.39	0.37	0.34	0.35	0.35	0.34	0.32	0.33
Borax + Firetex Boraks + Firetex	8	1	0.35	0.38	0.36	0.34	0.33	0.36	0.35	0.32
	9	2	0.37	0.39	0.35	0.35	0.35	0.38	0.34	0.33
	10	3	0.39	0.38	0.39	0.36	0.38	0.35	0.37	0.34
Boric acid + Firetex Kwas borny + Firetex	11	1	0.38	0.39	0.34	0.36	0.35	0.36	0.32	0.33
	12	2	0.38	0.36	0.35	0.34	0.35	0.33	0.32	0.31
	13	3	0.37	0.35	0.36	0.35	0.34	0.32	0.33	0.31

10 samples were used for each group and average values were written in the Table.
Użyto 10 próbek dla każdej grupy; przeciętne wartości zostały zapisane w tabeli.

tested pine and pine samples impregnated with various impregnation materials and reported that the impregnation process increases specific gravity of the wood material. In turn, Toker (2007) reported that boron compounds (boric acid, borax and sodium perborate) applied in aqueous solutions at various concentrations resulted in higher values of bulk dry specific gravity values compared to the non-impregnated (control) sample.

Differences in compressive strength are given in Table 4 and the related graph is shown in Figure 3.

One-way analysis of variance (ANOVA) was performed to determine differences between the groups in terms of compressive strength and the obtained data are given in Table 5.

The conducted one-way ANOVA showed that the differences between the groups were statistically significant at the 0.05% significance level.

The highest compressive strength parallel to the grain was observed in the case of 25-minute immersion time (33.56 N/mm^2) for the use of the Firetex preparation alone, while it was lowest for 55-minute immersion time (27.44 N/mm^2) for the use of boric acid alone. A slight increase was recorded in the 1st and 2nd impregnation processes. In general very high values are observed compared to the control sample due to the anatomical structure of wood, the applied impregnation agent as well as the adopted dipping method and its duration.

Changes in bending resistance are given in Table 6 and the related graph is shown in Figure 4.

Table 4. Values of compressive strength, N/mm²
Tabela 4. Wartości wytrzymałości na ściskanie, N/mm²

Impregnation agents Czynniki impregnujące	Control sample – no. and concentration, % Próbka kontrolna – nr i stężenie, %		Compressive strength parallel to the grain Wytrzymałość na ściskanie równolegle to włókien N/mm²			
			immersion times, min – czas zanurzania, min			
			25	35	45	55
Firetex	1	–	27.32	–	–	–
	2	100	33.56	32.64	33.49	32.94
Borax – Boraks	3	1	25.67	28.97	30.71	28.45
	4	2	28.11	29.56	28.41	20.11
	5	3	28.94	28.15	27.67	28.01
Boric acid Kwas borny	6	1	26.19	29.76	29.11	28.36
	7	2	28.93	28.62	30.01	29.11
	8	3	27.67	28.03	28.29	27.44
Borax + Firetex Boraks + Firetex	9	1	29.42	28.64	28.17	31.14
	10	2	31.56	31.44	29.18	29.43
	11	3	28.97	29.31	29.07	30.48
Boric acid + Firetex Kwas borny + Firetex	12	1	32.97	31.64	30.99	30.21
	13	2	30.43	29.67	29.56	29.11
	14	3	30.40	29.65	29.40	29.00

10 samples were used for each group and average values were written in the Table.
Użyto 10 próbek dla każdej grupy; przeciętne wartości zostały zapisane w tabeli

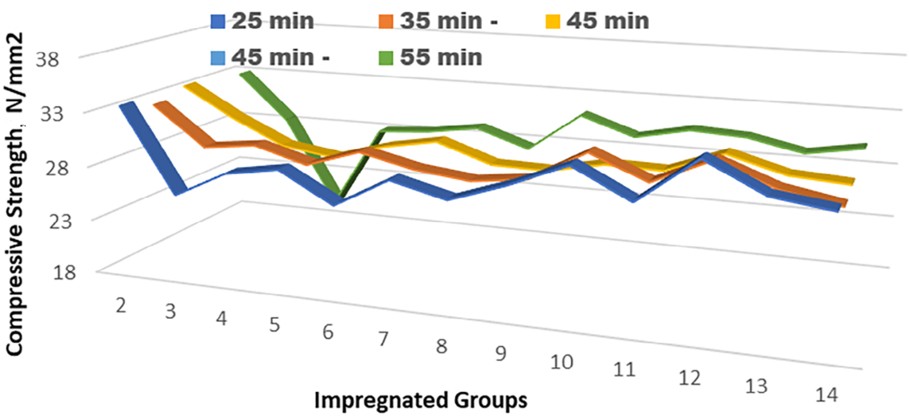

Fig. 3. Changes in compressive strength parallel to the grain
Rys. 3. Zmiany wytrzymałości na ściskanie równolegle do włókien

Table 5. One-way ANOVA for groups
Tabela 5. Jednoczynnikowa analiza ANOVA dla grup

	Sum of squares Suma kwadratów	df	Mean square Średni kwadrat	*F*	Sig.
Between groups Pomiędzy grupami	2951.117	52	56.752	4.019	.000
Within groups Wewnątrz grup	6736.400	477	14.122		
Total Razem	9687.517	529			

Table 6. Bending strength values, N/mm^2
Tabela 6. Wartości wytrzymałości na zginanie, N/mm^2

Impregnation agents Czynniki impregnujące	Control sample – no. and concentration, % Próbka kontrolna – nr i stężenie, %		Bending strength Wytrzymałość na zginanie N/mm^2			
			immersion times, min – czas zanurzania, min			
			25	35	45	55
Firetex	1	–	62.78	–	–	–
	2	100	68.16	69.89	70.61	69.37
Borax – Boraks	3	1	63.68	65.66	63.75	64.49
	4	2	64.73	65.45	65.91	64.71
	5	3	65.14	64.84	64.87	63.95
Boric acid Kwas borny	6	1	63.89	65.17	64.82	64.01
	7	2	65.36	67.12	65.76	64.64
	8	3	63.74	64.43	63.97	63.64
Borax + Firetex Boraks + Firetex	9	1	65.12	65.67	64.57	65.34
	10	2	64.81	65.34	65.06	64.96
	11	3	64.30	63.71	63.84	63.00
Boric acid + Firetex Kwas borny + Firetex	12	1	65.45	65.73	64.94	64.28
	13	2	64.97	65.37	65.14	64.87
	14	3	64.98	64.87	65.04	64.85

10 samples were used for each group and average values were written in the Table.
Użyto 10 próbek dla każdej grupy; przeciętne wartości zostały zapisane w tabeli.

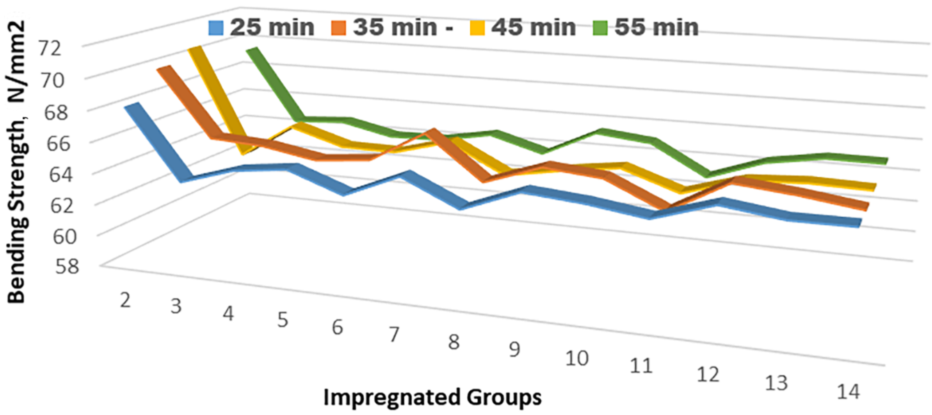

Fig. 4. Changes in bending strength
Rys. 4. Zmiany wytrzymałości na zginanie

Table 7. One-way ANOVA for groups
Tabela 7. Jednoczynnikowa ANOVA dla grup

	Sum of squares Suma kwadratów	df	Mean square Średni kwadrat	*F*	Sig.
Between groups – Pomiędzy grupami	1 186.630	52	22.820	3.053	.000
Within groups – Wewnątrz grup	3 565.900	477	7.476		
Total – Razem	4 752.530	529			

One-way analysis of variance (ANOVA) was performed to determine differences between the groups for bending strength and the obtained data are given in Table 7.

The one-way ANOVA showed that the differences between the groups were statistically significant at the 0.05% significance level.

The use of Firetex and the other impregnation agents caused a slight increase in bending resistance values. The highest bending resistance was found for the use of the Firetex preparation alone (70.61 N/mm^2) at the 45-minute immersion time, while the lowest bending resistance was recorded in the 55-minute immersion time (63.64 N/mm^2) when boric acid was used alone (3%).

Changes in the modulus of elasticity are given in Table 8.

One-way ANOVA was performed to determine differences between the groups for the modulus of elasticity and the obtained data are given in Table 9.

The performed one-way ANOVA showed that the differences between the groups were statistically significant at the 0.05% significance level.

The highest value of the modulus of elasticity in bending was recorded at the use of Firetex alone at a dipping time of 35 minutes (8100 N/mm^2), while the lowest value was determined in the dual treatment of boric acid + Firetex (6330 N/mm^2).

CONCLUSIONS

The highest % retention value for immersion periods was 55-minute immersion (2.25%) for Firetex alone and the lowest % retention for 55 minutes (0.55%) for boric acid alone.

Air dry and bulk dry specific gravity decreased with an increase in immersion time. The highest air dry and bulk dry specific gravity were recorded at 25-minute immersion times.

Table 8. Modulus of elasticity in bending, N /mm^2
Tabela 8. Współczynnik sprężystości przy zginaniu, N/mm^2

Impregnation agents Czynniki impregnujące	Control sample – no. and concentration, % Próbka kontrolna – nr i stężenie, %		Modulus of elasticity in bending, N/mm^2 Współczynnik sprężystości przy zginaniu, N/mm^2 – immersion times, min – czas zanurzania, min			
			25	35	45	55
Firetex	1	–	6 250	–	–	–
	2	100	7 800	8 100	7 850	7 645
Borax – Boraks	3	1	6 850	7 150	6 987	6 500
	4	2	6 980	7 100	6 910	6 357
	5	3	6 720	6 864	6 863	6 761
Boric acid Kwas borny	6	1	6 600	6 873	6 794	6 701
	7	2	6 350	6 558	6 489	6 389
	8	3	6 558	6 647	6 455	6 432
Borax + Firetex Boraks + Firetex	9	1	6 715	6 645	6 702	6 650
	10	2	6 498	6 137	6 358	6 604
	11	3	6 473	6 379	6 395	6 456
Boric acid + Firetex Kwas borny + Firetex	12	1	6 400	6 413	6 517	6 396
	13	2	6 374	6 365	6 355	6 337
	14	3	6 489	6 377	6 360	6 330

10 samples were used for each group and average values were written in the Table.
Użyto 10 próbek dla każdej grupy; przeciętne wartości zostały zapisane w tabeli.

Table 9. One-way ANOVA for groups
Tabela 9. Jednoczynnikowa ANOVA dla grup

	Sum of squares Suma kwadratów	df	Mean square Średni kwadrat	*F*	Sig.
Between groups – Pomiędzy grupami	8.679E7	52	1 668 962.779	678.617	.000
Within groups – Wewnątrz grup	1 173 114.500	477	2459.360		
Total – Razem	8.796E7	529			

Generally Firetex and the other impregnation materials increased the values of mechanical resistance parameters.

A partial increase in specific gravity was observed in all the impregnation variants compared to the control.

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IMPREGNACJA DREWNA ŚWIERKOWEGO PREPARATEM FIRETEX I ZWIĄZKAMI BORU PRZY RÓŻNYCH STĘŻENIACH I CZASIE ZANURZANIA ORAZ ICH WPŁYW NA WYBRANE WŁAŚCIWOŚCI TECHNOLOGICZNE

ABSTRAKT

W badaniach zgodnie z wymaganiami normy ASTM 1413-76 drewno świerka kaukaskiego (*Picea orientalis* (L.) Link) było impregnowane preparatem Firetex i związkami boru z zastosowaniem różnych stężeń (1%, 2%, 3%) i czasów zanurzania (25, 35, 45, 55 min) oraz badano zmiany właściwości technologicznych. Określano następujące właściwości technologiczne próbek: retencja [%], pozorny ciężar właściwy i gęstość drewna w stanie powietrznosuchym, wytrzymałość na zginanie, współczynnik sprężystości przy zginaniu i wytrzymałość na ściskanie równolegle do włókien. W przeprowadzonym doświadczeniu najwyższy pozorny ciężar właściwy stwierdzono przy 25-minutowym czasie zanurzania (0,41 g/cm^3) dla jednokrotnego zastosowania preparatu Firetex, natomiast w przypadku gęstości drewna w stanie powietrznosuchym było to 25-minutowe zanurzenie przy jednokrotnym stosowaniu preparatu Firetex (0,39 g/cm^3). W przypadku właściwości mechanicznych największą wytrzymałość na zginanie stwierdzono w przypadku 45-minutowego zanurzenia w preparacie Firetex (70,61 N/mm^2), najwyższa wartość współczynnika sprężystości przy zginaniu została zarejestrowana przy 35-minutowym zanurzeniu w preparacie Firetex (8100 N/mm^2), a wytrzymałość na ściskanie równolegle do włókien była największa przy 25-minutowym zanurzeniu w preparacie Firetex (33,56 N/mm^2).

Słowa kluczowe: Firetex, bor, materiały kompozytowe, impregnacja, stężenie, właściwości technologiczne