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Original Research Article

COMPARISON OF THE EFFECTIVENESS OF FIRE-PROOF IMPREGNATION METHODS FOR SCOTS PINE WOOD USED IN BUILDINGS CONSTRUCTION

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Abstract

The fight for a healthy and clean climate forces many restrictive changes to European law. Wooden construction fits very well into these changes, as it is able to store carbon dioxide for years. Unfortunately, many regulations, e.g. fire regulations, still hinder the development of this type of structures in Poland. Wooden elements used that have class D must achieve class B of fire resistance. For this purpose, they are modified with flame retardant agents. Three salt flame retardants based on: 1-phosphorus and iron, 2-phosphorus and nitrogen and 3-ogranic componds including benzoates, were used in the tests. The amount of applied fire retardants was compared depending on the impregnation technology used: surface immersion and pressure, as well as the reaction to fire of impregnated wooden elements. As a result of the tests, no impregnation used improved the fire properties, as shown by a small-scale cone calorimeter test. The project results indicate the need to conduct new basic research on the possibility of permanently improving the fire properties of wooden elements, which would allow the widespread use of wood in construction.

Keywords: effectiveness of reaction to fire, fire retardant treated wood, surface impregnation, vacuum impregnation, impregnation rate

1. INTRODUCTION

In 2021, the EU has its first European climate law. It set Europe's targets to reduce CO_2 emissions by 55% by 2030 in its full 1990 version and be climate neutral by 2050. In line with the climate law, the Commission also recommended in 2024 an additional intermediate target to meet emissions by 90% by 2040, the same period intended for Polish activities [1]. The additional European Parliament has approved a zero-energy solution version of the "Net-Zero Industry Act" [2], which aims by 2030 for the overall capacity by EU to produce strategic technologies close to or at least 40% of annual

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implementation needs. This is intended to cause acceleration of heating and energy application, including supported development of technology that can be delivered to store CO_2.

A new study has found that switching to wood as a building material would significantly reduce the negative impact of construction on the environment. If 80% of new constructions in Europe were made of wood, wood was used in the structures, cladding, surfaces and living facilities, these buildings would store 55 million tonnes of carbon dioxide per year. Additionally, the new regulation of May 10, 2023 [3] on the adjustment of customs duties at EU borders (CBAM) imposes additional duties on the cement, iron, steel, aluminum, hydrogen, electricity and fertilizer production sectors. This regulation is intended to establish a fair price for carbon dioxide emitted during the production of goods with high carbon content that enter the EU, as well as to encourage cleaner industrial production in non-EU countries, such as wooden construction. Possibility of constructing new buildings based on a wooden structure is achievable in the long term. The key to the sustainable development of wooden cities, while preserving key biodiversity areas and protected forests, lies in skillful management and careful planning.[4] Unfortunately, wooden construction in Poland still encounters many obstacles and lack of understanding of legal solutions.

2. THE ROLE OF IMPREGNATION IN FIRE PROTECTION OF THE WOODEN STRUCTURES

British Columbia, Canada, was the first province to adopt changes to its building code in 2009, allowing six-story wood-frame residential buildings [5]. Through the Wood First Act and provincial building code updates, it is leading the way in Canada in positioning wood as the building material of choice [6]. In France, the southern mayor's office adopted a resolution ordering the construction of at least 50% of public buildings using wood as a construction material in schools, kindergartens, sports facilities, and municipal apartments. [7]. The result of this regulation is the construction of a state-of-the-art Marengo Multimodal Transport Hub station in Toulouse, which will be largely made of wooden elements.[8] In Europe, in addition to France, Austria, Germany and Japan, Romania, Serbia, Hungary and Ukraine have special regulations regarding wooden structures. Poland, along with Bulgaria, Belarus, Iceland and Russia, does not have additional regulations regarding wooden buildings.[9]

One of the biggest problems is the flammability of wood, which has resulted in restrictions on building regulations in Poland, especially in the case of taller buildings. Wood is a flammable material, and in terms of reaction to fire, according to the PN-EN 13501-1 standard, it most often has class D [10]. Despite this, it is hard to believe, but individual elements of wooden structures with an appropriately selected cross-section do not need to be additionally protected with fire protection due to the fire resistance criteria.[11] However, in Poland, elements of a building structure with more than 4 floors must be classified as fire-retardant and therefore should achieve fire reaction class B. [12] Therefore, in order for the wood to meet these requirements, it must be additionally modified with fire retardants.

The very restrictive requirements of Polish law are controversial due to the fact that the flammability of materials is not the only cause of fires in buildings. More important than the flammability of individual materials used in buildings should be the regulations regarding their fire resistance of the entire structure. This translates into determining how long a building can survive and providing time for evacuation before failure occurs. The fire risk assessment method for historic buildings developed in Malaysia takes into account the percentage impact of fire risk depending on 4 criteria affecting fire safety. The building geometry was rated at 37%, the active protection system at 27%, fire safety management at 18%, and the building parameters at 17% for the impact of fire risk. The share of materials in the "building parameters" as a fire risk factor was estimated at 20%, which can be interpreted as saying that the flammability of building materials accounts for 3% of the fire risk.[13]

2.1. The impregnations

In the case of fireproofing wood, the type of impregnation used and the method of applying it to the elements become important. By definition, a fire retardant for wood should delay the moment of ignition of wood material, reduce the speed of surface spread of flames and reduce the intensity of combustion of flammable materials. The mechanism of action of fire retardants (FR) used on wood is to disrupt the combustion process at a specific stage.[14]

There is a large range of fire-retardant impregnating agents on the European market. The most frequently used flame retardants in fire retardants for wood, belonging to the group of organic compounds, include compounds containing nitrogen atoms, e.g. melamine, urea, guanidine, and organophosphate compounds, e.g. melamine phosphate, phosphorus-nitrogen and phosphorus-halogen compounds.[14] Inorganic compounds include phosphate, ammonium and boron salts. Most agents provide reaction to fire class B-s1, d0. The effectiveness of impregnation depends on the type and amount of the chemical agent introduced and the depth of wood penetration.

The amount of the agent introduced and the depth of penetration of the sawn timber are determined by the impregnation method used as well as the type of used agent.[15] After analyzing the agents available on the market, it was noticed that the agents differ in the amount of agent required by the manufacturer to be provide into the wood. Usually it is 200 g/m^2 - 300 g/m^2 and even over 900 g/m^2 and this affects the number of layers applied: 1-4. Some agents can be applied superficially and deeply, e.g. using a vacuum. Manufacturers also recommend an appropriate drying period after applying the fire retardant. This time also depends on the composition of the impregnation and can range from 0.5 to 8 hours. Some agents have additional capabilities and can provide wood with fire resistance classes of R15, R30, R45 and R60. The methods of applying impregnations depend strictly on the composition and simply on the manufacturer's recommendations. The easiest way to apply the impregnation is to use a brush, spray or simply immerse the element. This method is most often used in sawmills and construction sites.[16]

2.2. Penetration of the impregnation

The literature states that surface methods allow the preparation to penetrate to a depth of 2 to 8 mm.[17,18] In the case of deep penetration, the literature states that the preparation can be determined even to the entire depth of the sapwood of the wood.[19] When comparing wood impregnation using the surface method with deep impregnation using the same fire retardant, a better protective effect is usually obtained for deep impregnation.[14]

Despite the high effectiveness of pressure impregnation, surface methods will remain irreplaceable wherever it is necessary to saturate already built-in wood.[20] According to the EN 351-1 standard, the penetration and retention depth of the preservative assumes 7 classes NP1-NP7, of which the first class has no requirements regarding the penetration depth, while classes 6 and 7 require penetration of at least 6 mm of the exposed heartwood. Classes NP1-NP5 do not require oversaturation of the heartwood. In the NP2 class, the minimum penetration of the impregnation is 3 mm, which is more than the penetration of the impregnation when applied by brushing, spraying or brushing.[21] Therefore, theoretically, applying the impregnation on the surface will not allow meeting the requirements of the NP2 class. The most effective flame-retardant pressure impregnation is associated with advanced technological processes that reduce the mechanical strength of wood, high agent retention and costs, therefore the essence of surface impregnation should be opposite to these features.[22]

2.3. Durability of impregnation

An equally important issue is the durability of such impregnation. This feature is also described in standard 351-1. According to the EN 351-1 standard, the durability requirement is one of the basic elements in the development of a standard for a product subject to maintenance treatments, but the same EN 351-1 standard does not aim to quantify the durability against mechanical damage that could be expected after an impregnation treatment. The standard states that this depends on the geographical location and its associated climate and conditions of use.

European standards determine the durability of impregnations used on surfaces for 5 years.[23] The use of a fire retardant at the prefabrication stage must be covered by European standards for each type of supporting materials. The European harmonized standards for strength-graded structural timber[21], glulam [23] and CLT [24] do not include timber treated with flame retardant products.

There is a significant problem related to the service life of building structures of several dozen years [25] or even protective plaster [26] and the difference in the fire-resistant durability of impregnated wooden elements - 5 years [23]. The lack of a normative method for assessing the service life of flame retardants secured elements causes difficulties in applying this type of fire safety solutions in buildings. Under the conditions of use a wooden structure, fireproof impregnation should be durable. Permanently improved reaction to fire class in this article means a period of time corresponding to the service life of the structure before major renovation. The service life of plasters (not shorter than 10 years) seems to be sufficient for a fire retardant, but it involves significant technical difficulties.[27]

2.4. Reaction to fire class

The reaction to fire classification allows you to determine how a given building material will behave under the influence of fire. In order to assign a given product a specific reaction to fire class, the following are checked: how quickly it ignites; how much heat it releases and at what time, whether it produces smoke or burning drops. In order to test the class of reaction to fire, European and Polish standards (ISO 5660-1) recommend the cone calorimeter method for small samples.[28]

3. RESEARCH METHODOLOGY

For impregnation research were used three water-based fire-retardants agents. All impregnates, according to the technical declarations of manufacturers, lowering the flammability of wood to the class B reaction to fire and are safe for use in residential premises.

Flame retardant no. 1 is based on phosphorus and iron compounds in 10-30% solution, and it binds with structure of the wood. Flame retardant no. 2 is based on phosphorus and nitrogen compounds and can be washed out of the wood structure. For both agents Manufacturer’s require of application in an amount of not less than 300 g/m^2 to the wooden surface. Flame retardant no. 3 is based on organic compounds, including benzoates. To obtain the B reaction to fire class according to the manufacturer's guidelines, it is sufficient to use an 18.5% solution in an amount of not less than 946 g/m^2 what would obtain in amount of 175g/m^2 dry active agent and as agent No.2 it also doesn’t binds with wood structure.

Samples were made from Scots pine. The wood from which the samples were obtained for research came from southwestern Poland. Average density of pine wood 540 kg/m3 and 520 kg/m3 for the heartwood and sapwood parts, respectively. The density of the sapwood and heartwood samples is therefore similar. All samples were provided from planed material without any defects or knots and conditioned to a humidity of 12 ± 1% before impregnation. Humidity was measured using a hammer hygrometer.

Before impregnation, all samples were sealed at both ends with hot paraffin(Fig. 1) to prevent excessive application of flame retardant to the cross-section which would make it much more difficult to assess the penetration of impregnating agents into research material surface.

Fig. 1. Samples sealed in hot paraffin

The weight of the samples was measured before and after impregnation. The second mass measurement was taken immediately after surface dried with filter paper.
Equipment used in the research:
- Vacuum Chamber 3570SSG with a volume of 67 liters and internal dimensions: diameter 350 mm and height 700 mm equipped with an oil-free vacuum pump VP1500 with a capacity of 320 l/min and maximal vacuum 20 mbar (0,0197 atm);
- Laboratory dryer SLW 32 with a capacity of 32 liters, temperature range up to +300°C and forced air circulation;
- Laboratory scale model SBS-LW-2000A with accuracy 0,01g and measurement range 0,05g – 2000g.;
- Resistive wood moisture meter Universal Humid 06036880Z0 with measurement range 6,4-74,7% and accuracy ± 1%;
- Technical thermometer with measurement ranges from - 40 °C to 250°C and accuracy 0,1°C intended for liquids;
- Weather control station Terdens 2123 with thermometer and hygrometer.

3.1. Penetration efficiency of fire-retardant impregnations

Samples were made from heartwood and sapwood of Scots pine with dimensions 350 x 100 x 20mm. For impregnation was prepared 171 samples were divided into three groups according to used impregnate, Samples No.1, Samples No.2, Samples No.3.

Simple method of impregnations is a short-term impregnating by a cold bath. It usually takes place at a temperature of about 20°C and lasts from 30 seconds up to 2 hours.[9]

The impregnation time for each group ranged from 15 seconds to 90 minutes at atmospheric pressure (1 atm). Such selection of impregnation times should allow for the determination of a reliable penetration curve.

3.2. Assessment of the reaction to fire class using the cone calorimeter

Assessment of the reaction to fire class using the cone calorimeter method according to ISO 5660-1. All samples impregnated in agent no. 3 using surface and immersion method from the first stage of research were cut from the 350 x 100 x 20 mm format to 100 x 100 x 20mm. Three samples were selected from one impregnation process.

Climatic conditions in the test room:

-temerature 22°C, humidity 30-34 %;

-impregnating agent temperature: 20°C;

-impregnated wood parameters: 20°C, moisture 7,5-8,5%.

In order to conduct a reaction to fire test with a greater reserve of the amount of flame retardant used in vacuum impregnation. After impregnation, the samples were formatted to dimensions of 100 x 100 x 20mm. Determination of the reaction to fire according to ISO 5660-1:2015/Amd 1:2019 was conducted by Building Research Institute. The distance between the wood specimen surface and the cone was set to 25 mm and the heat flux was set as 50 kW/m^2.

3.3. Multi-cycle surface impregnation with drying between cycles

Samples made of strength graded Scots pine class C24 with dimensions 100 x 100 x 20mm. For impregnation was prepared 30 samples and divided into three groups according to used impregnation agents.

In order to check the effectiveness of multiple surface impregnation in accordance with the recommendations for the application of fire-retardant impregnations developed by the manufacturer and based on literature. It was possible to compare momentary immersion of samples in the impregnation for 15 seconds with four-sided application of the impregnation by painting or spraying on the elements to be protected [9]. Following the recommendations, a drying time of 40 minutes was allowed between subsequent applications. In order to determine the appropriate penetration of the impregnation, the mass of the samples was measured in two stages: the first measurement took place in the first cycle before impregnation, for subsequent applications after the drying process, and the second measurement took place after the application of the impregnation and surface drying of the sample surfaces. Impregnation was carried out until all samples in the test group received the impregnation corresponding to the manufacturer's recommendations.

Results of the previous research phase show variability of required application rate. Even vacuum impregnation did not allow for effective and stable impregnation. To make the process more predictable in further part of work was performed multi-cycle impregnation of all fire retardant tested before. Technological breaks for drying between next cycle of impregnation outcome to be necessary to obtain the required application of fire retardants. Increasing the number of impregnation cycles and extending the drying intervals between them. According to the literature one-time surface application can be compared to 15 seconds of immersing in agent.[9]

3.4. Multi-cycle vacuum impregnation process

Samples made of strength graded Scots pine class C24 with dimensions 100 x 100 x 20mm. For impregnation was prepared 30 samples and divided into three groups according to used impregnating agents. All samples were protected with fire-retardant no. 1 in the vacuum impregnation process in 3 variants. First, a single-cycle impregnation was carried out by immersion for 60 minutes under a vacuum 0,15 atm. The second variant is 2 cycles consist of two steps:10 min immersion impregnation under a vacuum 0,3 atm and 10 min of immersion impregnation without vacuum. The third impregnation variant is 3 cycles consist of two steps:10 min immersion impregnation under a vacuum 0,15 atm and 10 min of immersion impregnation without vacuum. The samples were not dried between impregnation cycles - the cycles took place without technological breaks.

3.5. Multi-cycle vacuum impregnation with drying between cycles

For testing were prepared 15 samples of pine sorted to the KS/C24 strength class according to PN-D-94021:2013. Samples were divided in three groups each for one variant.
Variant of impregnation consist of 7 cycles: 5 min immersion impregnation under a vacuum 0,3 atm, 5 min of immersion impregnation without vacuum and oven-drying in 50°C for 15 min. In this variant of fire retardant, no 1. application was impregnated 15 samples.

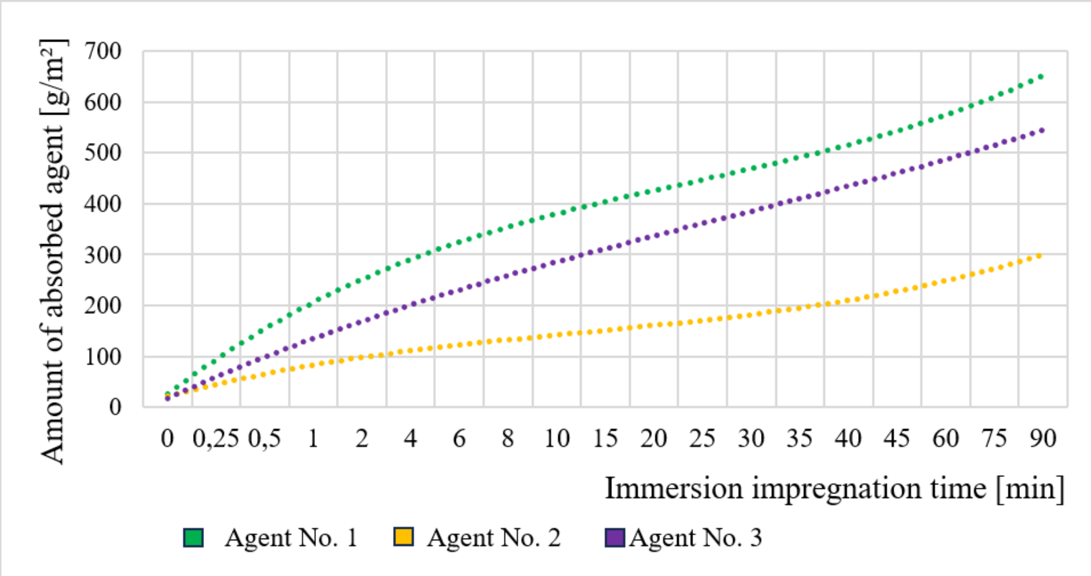
4. RESULTS OF TESTS

4.1. PENETRATION EFFICIENCY OF FIRE RETARDANT IMPREGNATIONS

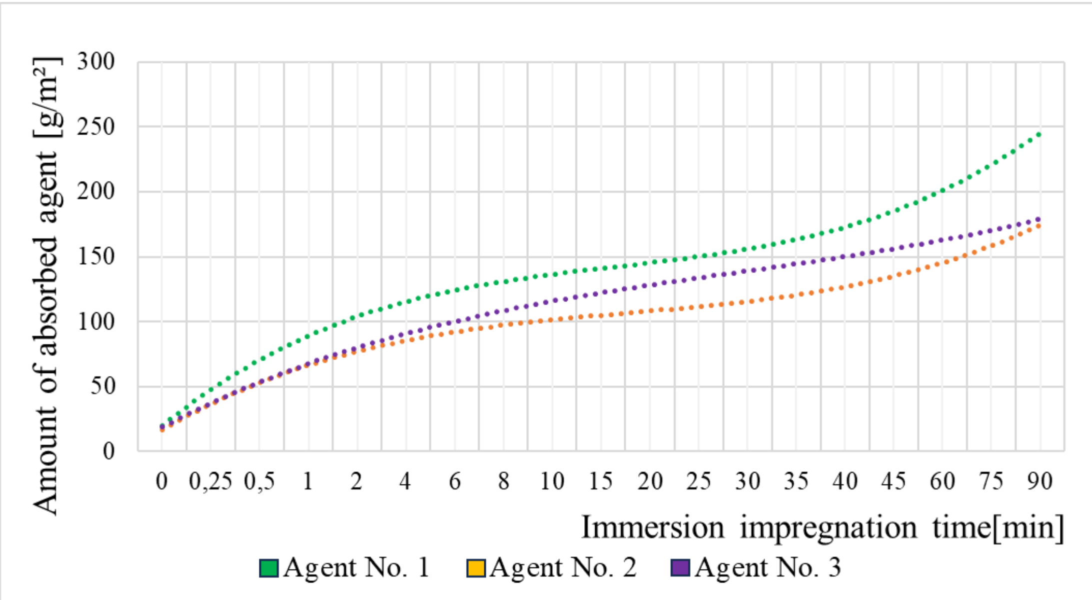
All samples and impregnation process in this phase of research was conducted by employees of Faculty of Forestry and Wood Technology from Poznan University of Live Sciences. The results were presented on graphs 1 and 2.

Graph no. 1 shows the penetration curves of three tested impregnates for immersion impregnation of pine sapwood. The average increase in impregnation penetration for impregnating agents was: for the first 7.3 g/m^2 per minute, for the second 3.2 g/m^2 per minute and 6.14 g/m^2 per minute for the third.

Graph no. 2 shows the penetration curves of three tested impregnates for immersion impregnation of pine heartwood The average increase in impregnation penetration for impregnating agents was: for the first 2.57 g/m^2 per minute, for the second 1.83 g/m^2 per minute and 2.14 g/m^2 per minute for the third.



Graph 1. Immersion impregnation curve for *Pinus sylvestris* sapwood



Graph 2. Immersion impregnation curve for *Pinus sylvestris* heartwood

In both graphs it is easy to see that impregnation no. 1 has the best penetration properties. Impregnation no. 2 shows the lowest penetration values by immersion which is due to its much higher viscosity than the other two impregnations.

The comparison in Table 1 was made for both groups of material and all impregnations used in research. First column presents the impregnation process parameters reflect most effective impregnation time, 4 minutes of immersion impregnation. Second column presents middle time for impregnation research 45 minutes immersion impregnation. For the third column was presented maximum assumed time of immersion impregnation, 90 minutes of immersion impregnation. Fourth column presents in case of negative results of the immersion methods as a comparatives method was used vacuum impregnation, 60 minutes in pressure 0,1-0,2 atm.[28]

Table 1. Amount of fire retardants applied into wood in surface, immersion and vacuum impregnation process

Material	Heartwood pine				Sapwood pine			
Method [t, p]	Immersion [4 min, 1atm]	Immersion [45 min, 1atm]	Immersion [90 min, 1atm]	Vacuum [60 min, 0,1-0,2 atm]	Immersion [4 min, 1atm]	Immersion [45 min, 1atm]	Immersion [90 min, 1atm]	Vacuum [60 min, 0,1-0,2 atm]
Units	[g/m^2]	[g/m^2]	[g/m^2]	[g/m^2]	[g/m^2]	[g/m^2]	[g/m^2]	[g/m^2]
Agent No. 1	128,72	205,65	231,30	693,60	298,44	526,5	657,00	2385,00
Agent No. 2	83,36	148,95	165,50	235,20	106,08	234,45	288,00	571,80
Agent No. 3	83,14	162	192,60	355,80	190,96	445,05	552,60	999,60

None of the flame retardants applied on heartwood pine using the immersion method obtain the required amount of agent per square meter. A flame retardant with average application rate was selected for further research - agent no. 3. In order to determine the influence of the impregnation rate on flame retardancy, samples treated with fire retardant no. 3 were tested using a cone calorimeter.

4.2 Assessment of the reaction to fire class using the cone calorimeter method

The average results for each impregnation method were presented in Tab 2. All parameters below are average from 3 measurements for each process.

Table 2. Results of reaction to fire assessment by cone calorimeter method according to ISO 5660-1

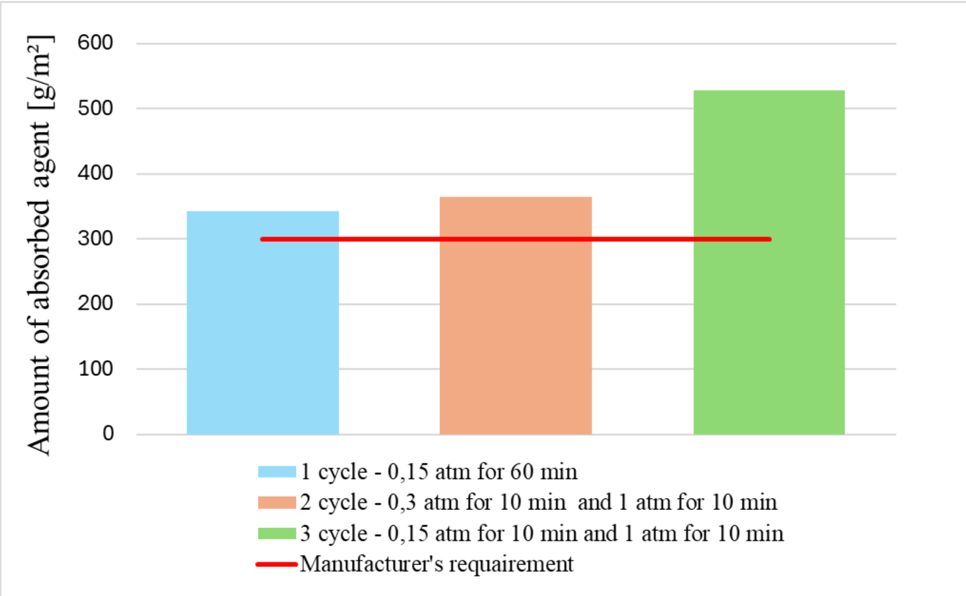
No.	Material	Method of impregnation	Averege application of fire retardant [g/m^2]	Averege FIGRA [W/s]	Averege THR [MJ]	Fulfillment of Figra [%]	Fulfillment of THR [%]	Estimate reaction to fire class
-	Parameters	-	-	120	7,5	-	-	B
1	sapwood pine	-	0	665,59	34,89	18%	21%	D
2		4 min immer. 1 atm	190,96	308,33	15,47	39%	48%	D
3		45 min immer. 1 atm	445,05	242,92	14,83	49%	51%	C
4		vacuum 0,1-0,2 atm	1729,73	135,11	14,11	89%	53%	C
5	heartwood pine	-	0	710,34	37,58	17%	20%	D
6		4 min immer. 1 atm	100,14	342,18	17,53	35%	43%	D
7		45 min immer. 1 atm	205,81	205,83	15,12	58%	50%	C
8		vacuum 0,1-0,2 atm	590,1	185,77	14,74	65%	51%	C

None of the 27 samples achieved the manufacturer's declared B class of reaction to fire. This applies even to samples made using vacuum and attempting to obtain the application recommended by the manufacturer, which turned out to be possible only for pine samples obtained from the sapwood part. Samples from the heartwood obtained the application only in 62% meeting the manufacturer's recommendations for surface application.

Since the required reaction to fire class was not achieved by the agent no. 3 showing the most constant growth rate, next test should be continued for impregnating agent no. 1 according to its best penetration results from first impregnation research.

4.3. Vacuum process modification

Vacuum process is more efficient method than immersion impregnation. That method can be modified by adding several steps like soaking after pressure or adding more cycles to achieved higher absorption of impregnating agent.



Graph 3. Total application of fire retardant no 1. in 3 variants of vacuum impregnation

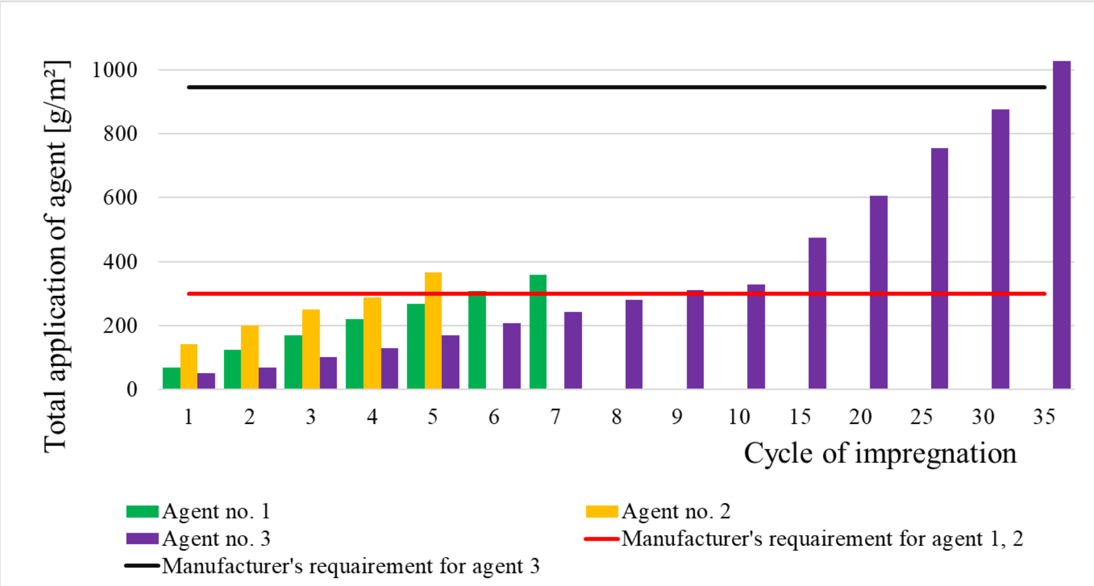
The manufacturer's recommendations were fulfilled in 114% for 1-cycle, 122% for 2-cycle and 175% for 3-cycle impregnated samples selected for testing. The test results showed worse fireproof properties than for fire retardant no. 3 presented in Tab 2.

In performed vacuum impregnation variants samples achieve application rate required by manufacturers and it shows better results for multi cycle variants at the same and shorter time. The research shows that impregnation without breaks between cycles is solution that allows for better control of the process and ensuring the required amount of applied impregnating agent.

4.4. Multi-cycle surface impregnation with drying between cycles

To ensure repeatability of the results an immersion impregnation for 15 second was selected to simulate 1 cycle of surface application of flame retardant in production condition. One test cycle consists of 15 seconds of soaking impregnation and 40 minutes of drying.

For testing were prepared 21 samples (7 for one agents) of pine sorted to the KS strength class according to PN-D-94021:2013. The dimensions of the samples were 100 x 100 x 20mm. Average amount of fire retardants applied in all tested group of samples was presented in Graph 2.

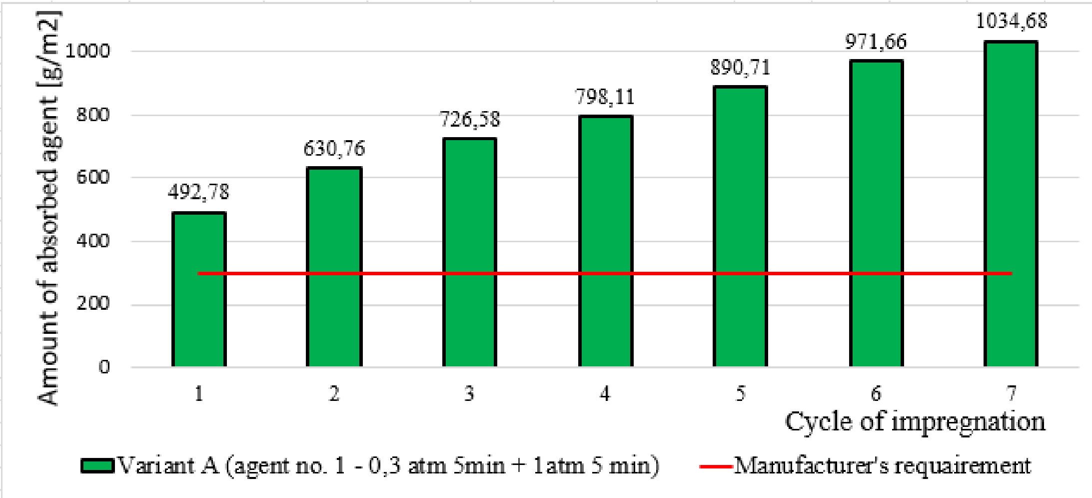


Graph 4. Comparison of surface impregnation efficiency for fire retardants no. 1-3

The best impregnation efficiency presents impregnation agent No. 2 achieving the application required by the manufacturer after 5 cycles. Fire retardant no. 1 achieved required amount of agent for all samples after 7 cycles. The worst efficiency has agent no. 3 (35 cycle to achieve manufacturer’s requirement), which indicates that impregnation no. 3 is not intended for surface application. - the manufacturer's requirements can only be effectively achieved by pressure impregnation is special process. On the surface of samples during the process of impregnation salting out was observed only for third of the impregnating agents.

4.5. Multi-cycle vacuum impregnation with drying between cycles

Multi-cycle vacuum impregnation is a process with noticeably higher efficiency than the single process. Adding an additional drying process to it additionally increases its effectiveness, which allows for obtaining application increments similar to those with surface impregnation.



Graph 5. Multi-cycle vacuum impregnation Agent No. 1

On the Graph 5 are presented levels of impregnation for vacuum impregnation with soaking process and drying in laboratory dryer. It is easily noticeable that even first cycle allows to achieve higher level of impregnation than required by manufacturer by 64%.

Discoloration of agent after drying make visible depth of impregnation and shows the depth of impregnation in Fig. 2, a red frame with a thickness of 2 mm has been drawn in scale the photo.

Fig. 2. Comparison of the impregnation depth in variants A – app. 2 mm, B – app. 1 mm and C – less than 1 mm

Variant A shows depth of penetration sample by impregnating agent No. 1 prepared in vacuum multi cycle impregnation presented on graph 5.

Variant B shows depth of penetration sample by impregnating agent No.1 prepared in multi cycle impregnation presented on graph 4.

Variant C shows depth of penetration sample by impregnating agent No. 2 prepared in multi cycle impregnation presented on graph 4.

5. DISSCUSION

Tests on the penetration of the impregnation show that both the part of the wood (sapwood or heartwood) and the type of method used, and above all, the method of application, are of great importance for obtaining the appropriate fire-retardant properties of the element.

The sapwood of Scots pine is much more susceptible to impregnation than the heartwood, which is confirmed by the research and literature [19, 32]. However, the research also paid attention to the penetration time of the impregnations depending on the part of the wood being tested. When using the surface method for sapwood by immersion in a cold bath, the impregnating agent with iron salts achieved the application recommended by the manufacturer within 4 minutes. In the case of heartwood, the tested impregnating agents showed a much worse index. In the case of surface impregnation of heartwood by immersion, the highest rate of penetration over time was observed when using agents based on orthoformate. After 90 minutes of immersion in a cold bath, it achieved 231 g/cm^2 of application. In the case of urea-based substances, it was 165 g/cm^2. It is worth adding that this agent has the addition of a gelling substance, which may affect its difficulty in penetration, and its viscosity is over 260 mps. The manufacturer recommends applying it with a brush. Unfortunately, none of the variants achieved the application recommended by the manufacturer. Similar impregnation trends are shown in the study conducted for biocides using, among others, Scots pine wood [30]. The research compared methods of applying impregnations by immersion, vacuum impregnation and single-cell impregnation. Immersion impregnation showed a similar tendency as for the tests presented in the article, the compared impregnation times show the validity of immersion impregnation up to about 60 minutes, at which the impregnation penetration was 363 g/m^2 and at 480 minutes the increase in non-transfer was only 10 g/m^2, which shows there is no justification for immersion impregnation for much longer than 1 hour.

Therefore, the application of the impregnation obtained during the work shows a higher effectiveness of the immersion method than in the above-mentioned tests. The effect of vacuum impregnation is also comparable; in the literature, the application obtained as a result of vacuum impregnation was approximately 1500 g/m^2, which is also a lower value than that obtained in the presented study, which amounted to over 1700 g/m^2 [30]. Research clearly confirms that the penetration of sapwood can be almost three times higher than that of heartwood. In addition to the characteristics of the impregnating agents, in this case it is influenced by the structure of the heartwood and sapwood. The heartwood, compared to the sapwood, is denser and contains non-structural compounds, such as resin in this case, which preserves it in a natural way and thus causes a significant difference in the permeability of the sapwood and heartwood structure. According to the literature, the sapwood of pine is easily saturated with impregnating agents, and the heartwood absorbs them in very limited amounts or does not absorb them at all [32]. The penetration time of the impregnation is of great importance in the production process and on the construction site.

In tests carried out with a small-scale cone calorimeter, the test results showed that when immersing impregnation on sapwood for 4 minutes the FIGRA (the fire growth rate index [W/s]) was over 300W/s which definitely does not allow for obtaining class B reaction to fire. After 45 minutes, this index decrises to 200, while with vacuum impregnation in an atmosphere of 0.1-0.2, FIGRA reaches 130 W/s. For reaction to fire class B FIGRA it is 120W/s. Even less satisfactory results were obtained in the case of heartwood, where after impregnation vacuum in an atmosphere of 0.1-0.2, a FIGRA index of 186 W/s was achived. The same relationship applies to the THR index(the total heat released from the sample during the first 600 s of exposure to the main burner flames). For elements with reaction to fire class B, the index should be 7.5. Unfortunately, in the conducted research it was not possible to obtain this indicator below 14 [MJ]. The tested samples obtained class D (as if they had not been impregnated at all) and C. This means that surface fireproof impregnation is not able to protect elements for the appropriate class.

In the presented studies, Class B reaction to fire hasn't achived in the small-scale cone calorimeter test. The European standard EN 16755 provides information on the use of the cone calorimeter method for assessing reaction to fire based on tests of many products, but also that some available data indicate limited correlation of the cone calorimeter method with the base method for building materials described in EN 13823. To obtain certain results should be considered on an average scale, according to the EN 13823 standard.

The reason for the lack of effectiveness of flame retardants is probably a single application of the agents without interrupting their drying. Some studies have shown that surface methods of application often require several layers and drying before applying another one.[21] The presented tests of the impregnation process turned out to be one of many cases in which wood should be dried between applications of the agent in order to obtain satisfactory performance properties for water-based maintenance treatments.[23]

Drying is one of the most energy-intensive and time consuming processes, which contributes to the increase in the cost of treated products even if two applications with drying are necessary.[23] The presented research showed that the multi-cycle drying process requires 5-35 cycles to obtain the amount of agent per square meter in accordance with the manufacturer's guidelines. The technological time of the tested impregnation variants is 4 – 29 hours and is longer than that of pressure impregnation. This calls into question the validity of using surface impregnations on construction sites due to their low effectiveness and the inability to properly control the process.

In order to obtain the application recommended by the manufacturers, the vacuum method was also tested in various cycles. It turned out that for a 1-cycle vacuum method it is possible to apply the recommended amount of impregnation agent. In the case of 2-cycle vacuum impregnation, the application is approximately 10% better. However, 3 cycles allow to obtain almost 600 g/m^2 of application, which is almost twice as much as recommended by the manufacturer. Even better results were achieved when applying the impregnation using the vacuum method with soaking and a break for drying the material. In this case, almost 500 g/m^2 was achieved after the first cycle.

Compared to the impregnation method without drying, in this case, after 7 cycles, the application was more than 3.5 times greater than recommended by the manufacturer. The use of drying also improved the results of surface impregnation. Cyclic impregnation with drying breaks also allowed to obtain the appropriate application of the preparation recommended by the manufacturer. Compared to vacuum impregnation, it required more time and cycles. In the case of immersion impregnation, proper application was achieved only after 7 cycles. In the case of agent No. 3 based on benzoates, this deposition was achieved only after 35 cycles. This is important because it significantly lengthens the entire process of preparing elements for construction. None of the tested measures used in construction conditions is able to bring the desired effect, much less ensure class B reaction to fire. Therefore, it should be noted that the recommendations presented by manufacturers regarding the amount of application and the method of application to obtain the appropriate class may be practically impossible to achieve. These results confirm that only pressure impregnation is able to ensure proper application of the impregnation in a short time.

6. CONCLUSSIONS

The aim of the work undertaken was to obtain appropriate fire protection for elements made of Scots pine wood. During the research, it was possible to obtain the appropriate impregnation all tested agents only agents no.3 presented some difficulties.

1.Impregnation of sapwood brings better results than impregnation of heartwood, regardless of the method of applying the agent.

2.Pressure impregnation is a method with much higher impregnation efficiency.

3.In laboratory conditions, using the manufacturer's recommendations, class B reaction to fire was not achieved for wooden elements.

4.Cyclic impregnation allows for achieving better results than single impregnation in a comparable time.

5.The operation of drying impregnated elements between subsequent applications of the agent is the most effective modification of the process.

6.The highest impregnation efficiency can be achieved by using multi-stage impregnation with drying process.
7.Impregnations available on the market may not achieve adequate protection (class B reaction to fire) for samples tested on cone calorimeter in a small scale.
8.Achieving the application of fire retardants recommended by manufacturers is difficult and time-consuming to achieve using surface impregnation methods.

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