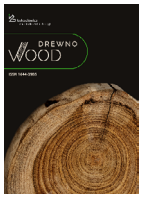
Article citation info:
Janiec M.M., Lisiecka E. 2025. Durability of fire protection impregnation and its impact on selected properties of Scots pine wood. *Drewno. Prace naukowe. Doniesienia. Komunikaty* 68 (216): 00060. https://doi.org/10.53502/wood-205846

Łukasiewicz Poznań Institute of Technology

Drewno. Prace naukowe. Doniesienia. Komunikaty
Wood. Research papers. Reports. Announcements

Journal website: https://drewno-wood.pl



Durability of Fire Protection Impregnation and Its Impact on Selected Properties of Scots Pine Wood

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Article info
Received: 3 January 2025
Accepted: 3 June 2025
Published: 8 July 2025

Keywords
durability of impregnation
Pinus sylvestris
physical properties of wood
hygroscopicity of wood
equilibrium moisture content of wood
impregnation

The durability of fire protection impregnation is of great importance in the protection of wooden structures. At the same time, impregnation itself can affect the physical properties of wood, impairing its characteristics. As part of the project POIR.01.01.01-00-0076/21, research was conducted to test how the hygroscopicity of Scots pine wood obtained from wood of KS strength class changes depending on the impregnation agent used in surface and deep impregnation methods. It was found that wood modified with salt-based fire protection agents is less sensitive to changes in ambient humidity than raw wood.

DOI: 10.53502/wood-205846
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Introduction

Impregnation is a way of increasing a material's resistance to external factors. In wooden construction, impregnation to prevent biocorrosion and to provide fire protection is of great importance. The wood type most commonly used in construction, namely pine or spruce wood, classified as softwood (density below 650 kg/m^3), is assigned to class D in terms of its reaction to fire according to PN-EN 13501-1+A1:2010, meaning a material that is flammable and easily spreads fire. The requirements for fire resistance of structures and the design of products used in construction are regulated by the European design standard Eurocode 5. To enable the use of timber elements in demanding large-scale projects, the timber should meet the requirements of Euroclass B-s1, d0. Fire class B-s1, d0 means that the product contributes to fire to a very limited extent, its smoke production is very limited, and it does not produce flaming droplets. The fire class requirements applicable to construction products depend on the purpose and size of the building and on the location in the building where the products are to be used.

Fire protection will be effective if it is durable. The durability of impregnation is described in EN 351-1. According to this standard, the requirement of durability is one of the basic elements when developing a standard for a product subjected to preservation treatments; however, the same standard does not aim to quantify the durability against mechanical damage that can be expected after impregnation, instead stating that this depends on the geographical location and climate and the conditions of use. European standards define the durability of impregnants applied to surfaces in terms

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of a period of 5 years [PN-EN 14080:2013-07]. Tests conducted by the Wood Technology Institute in Poznań show that the durability of the tested agents is over 3 years when used indoors, but less than 3 years when used outdoors [Ważny and Karyś 2001]. In indoor conditions, the durability of effective fire protection may differ greatly depending on the agent used. Some agents carry a declaration of only 3 years, while for others the declaration is 10, 15, or in extreme cases even 20 years. Some manufacturers of fire retardants claim that in difficult conditions outside the building the durability of the preparations is up to 5 years [Krajewski and Witomski 2003]. The durability of the impregnation of the structure will be closely linked to the durability of the building itself. A problem is posed when the durability of building structures is several decades [EN 1990:2004], and this applies even to protective plasters [EAD 350140-00-1106], while the fire protection durability of impregnated wooden elements may be only 5 years [PN-EN 14080:2013-07]. The lack of a standard method for assessing the durability of elements protected with flame retardants causes difficulties in using this type of fire safety solutions in buildings. In the conditions of use of a wooden structure, fire-resistant impregnation should be durable. In this article, permanent improvement of the reaction to fire refers to a period of time corresponding to the service life of the structure before major renovation. The service life of plasters (not less than 10 years) seems to be sufficient for a fire protection agent, but it is associated with significant technical difficulties [Östman and Tsantaridis 2017]. In the case of plasters, it is possible to replenish a layer on the wall structure. For wooden elements, it is difficult to repeat impregnation using deep methods such as long-term bath or vacuum-pressure processes. Hence, the durability of both the wooden structure and the impregnation will be greatly influenced by the changing ambient conditions: temperature and humidity.

The physical properties of wood include color, pattern, density, moisture, mineralization, gloss, smell, swelling and shrinkage, water absorption, as well as thermal, acoustic and electrical properties. These properties can be modified depending on the need to obtain wood material with specific properties. For this purpose, impregnants are used, among other things. However, fire-retardant impregnants can also negatively affect certain physical properties.

Hygroscopicity is the ability of wood or wood-based materials to exchange water vapor with the surrounding air [Krzysik 1975]. Impregnation should ensure that the moisture content in fire-protected wood is not significantly higher than in unprotected wood, because wooden materials can mold, rot, or corrode their fasteners if their moisture content constantly increases in a highly humid environment. Increased moisture content in the wood material can also affect the durability of the flame retardant. Untreated wood absorbs moisture from the surrounding air until its moisture content reaches equilibrium. However, as reported in the literature, the hygroscopicity of wood treated with inorganic flame retardants is usually greater than that of untreated wood and depends on the size and species of wood, temperature, relative humidity, and the type and amount of chemicals used [U.S. Forest Products Laboratory 1974; Eickner, H. W. J. Mater 1966]. Continuous exposure of wood treated with water-soluble salts to conditions with above 80% relative humidity can result in loss of chemicals and adverse effects on dimensional stability and paint coatings. Phosphate salts affect hygroscopicity mainly when relative humidity exceeds 80% [Brazier and Laidlaw 1974]. Therefore, most commercially available fire retardants are designed for use in conditions not exceeding 80% relative humidity. Modern methods of fire protection of building structures are based on the use of intumescent coatings, which are complex systems of organic and inorganic components. Many studies have been conducted on the effect of various treatments on the mechanical properties of wood. Researchers have concluded that treatment with flame retardants and subsequent drying reduces the initial strength properties of wood. In addition, it has been reported that pressure treatment caused a decrease in the bending strength of various types of wood by 8–10% [Su and Ahmed 2006]. Other studies showed that salt-based impregnation materials increased the compressive strength by 4.6–9.6% but reduced the bending strength by 2.9–16% [Keskin 2009; Wang et al. 2005]. This reduction in strength occurs when wood products are exposed to elevated temperatures, which often occur as a result of solar loading in roofing applications. Some studies have investigated to what extent and why flame retardants affect the properties of treated wood [Winandy 2001; Bao et al. 1999].

This article focuses on research on the influence of humidity and temperature on impregnated wooden elements and the influence of impregnation on the equivalent moisture content of wood.

Materials and methods

1. Material

For the tests, samples of Scots pine (*Pinus sylvestris* L.) wood were used, with dimensions of 350×100×22 mm, oriented with the longer side along the direction of the grain, cut without defects and assigned to the KS strength class according to PN-D-94021:2013. The density of the samples was in the range 450–530 kg/m^3, and their moisture content was 10–12%. The sapwood content

in the samples was negligible. Each group contained 10 samples, the number of samples being the maximum that could be placed in the vacuum chamber. Before impregnation, the ends of each sample were immersed in paraffin to a height of 15 mm to protect the cross-sections from impregnant penetration, as described in the literature [Angelis 2022]. This procedure serves to ensure proper penetration of the impregnant, as is appropriate for surface impregnation.

Two water-based flame retardants were used for impregnation. These agents reduce the flammability of the wood to fire reaction class B. Agent I is an agent based on a iron orthophosphate compound, concentration 10–30%. The manufacturer's recommended rate of surface application is not less than 300 g/m^2. Its density is 1.1 g/cm^3 at 20 °C. Agent II is based on phosphorus and nitrogen compounds; the manufacturer does not provide information on the concentration of the impregnating agent in the technical data sheet. The application rate required by the manufacturer to achieve fire reaction class B is 300 g/m^2. Its density is 1.06 g/cm^3. After impregnation, the samples were shortened (i.e., the ends of the samples soaked in paraffin were cut off) to give them dimensions of 250×100×22 mm.

2. Methods

The samples were impregnated to obtain the amount of fire retardant recommended by the manufacturer. The samples were impregnated using multi-cycle vacuum impregnation with a break for drying – naturally or using a climatic chamber. The conditions of impregnation are listed in Table 1.

Impregnation variant A was carried out for 7 cycles, where one cycle consisted of 5 minutes of immersion impregnation at a pressure of 0.3 bar, then 5 minutes of immersion impregnation without vacuum (approx. 1 bar), and at the end of the cycle drying in an oven at 50 °C for 15 minutes. Variant B was carried out for 8 cycles, where one cycle consisted of 15 seconds of immersion impregnation (without vacuum – approx. 1 bar) and then 40 minutes of air drying. Variant C was carried out for 7 cycles, where one cycle consisted of 15 seconds of immersion impregnation (without vacuum – approx. 1 bar) and 40 minutes of air drying. The amount of impregnant applied was tested by weighing samples to an accuracy of 0.01 g after impregnation, following prior removal of excess impregnant from the surface.

In the second stage of the tests, for each impregnation variant, three samples (measuring 250×100×22 mm) were sent to the Building Research Institute in Warsaw for tests of resistance to high humidity conditions. Samples were selected from the range corresponding to the mean value +/– standard deviation. The samples were conditioned (climatic chamber type C-20/350, manufacturer CTS GmbH) at a relative humidity of 50 ± 3% and a temperature of 23 ± 2 °C until a constant mass (± 2%) was obtained for 10 days. In the next stage, all samples were exposed to constant humidity of 90 ± 5% at a temperature of 27 ± 2 °C until a constant mass was obtained for 15 days. In the last part of the procedure, each sample was dried in a WAMED incubator (with a working range of 30–250 °C) at a temperature of 103 ± 2 °C until a constant mass was obtained for 26 hours. The mass of the samples was determined using a type E1200S electronic scale manufactured by Sartorius.

Results and discussion

1. Impregnant application rates

Different rates of application of impregnant were obtained for the individual impregnation variants. In the case of variant A (Agent I), where a vacuum chamber was used, the application rate was almost 900 g/m^2, with an average sample density of 497 kg/m^3. Variant B (Agent I), using immersion impregnation, led to a rate of approximately 320 g/m^2, with an average sample density of 512 kg/m^3. In the case of variant C (Agent II), where immersion impregnation was also

Table 1. Summary of the impregnation conditions for each of the tested variants

Variant	Number of cycles	Immersion impregnation I		Immersion impregnation II		Drying III	
		Time	Pressure	Time	Pressure	Time	Temperature
A (Agent I)	7	5 min	0.3 bar	5 min	1 bar	15 min	50 °C
B (Agent I)	8	15 s	1 bar	-	-	40 min	20 °C
C (Agent II)	7	15 s	1 bar	-	-	40 min	20 °C

used, the deposition rate was about 330 g/m^2, with an average sample density of 504 kg/m^3. For comparison, the control group consisted of unimpregnated pine samples with an average density of 492 kg/m^3. Samples subjected to pressure impregnation obtained the highest deposition rates, which is consistent with literature reports [Krajewksi and Witomski 2003; Babiński 1992].

2. Effect of moisture on impregnated wood

Tests were conducted to determine the change in the mass of impregnated samples after exposure to conditions of 90% humidity and a temperature of –27 °C, and after drying to a constant mass at 104 °C (Figure 7). In accordance with literature data on the hygroscopicity of wood, we observe an increase in the mass of samples for all variants exposed to harsher environmental climatic conditions [Kozakiewicz 2013]. For variant A, we observed an average increase in the sample mass by 7.5% compared to the mass of conditioned samples. After drying, the average sample mass dropped about 16.03% (Figure 1). For variants B and C (surface immersion), the average increase in the sample mass was 6.95% and 7.43% compared to the mass of conditioned samples. As a result of drying, the average mass decrease for both variants was over 15%. The unimpregnated samples in the control group showed the greatest changes in mass compared to the impregnated samples. The control samples of unimpregnated material underwent an average mass increase of 11.06%. The average mass loss after the drying process for the control samples was 17.01%. This represents large differences of 4 percentage points

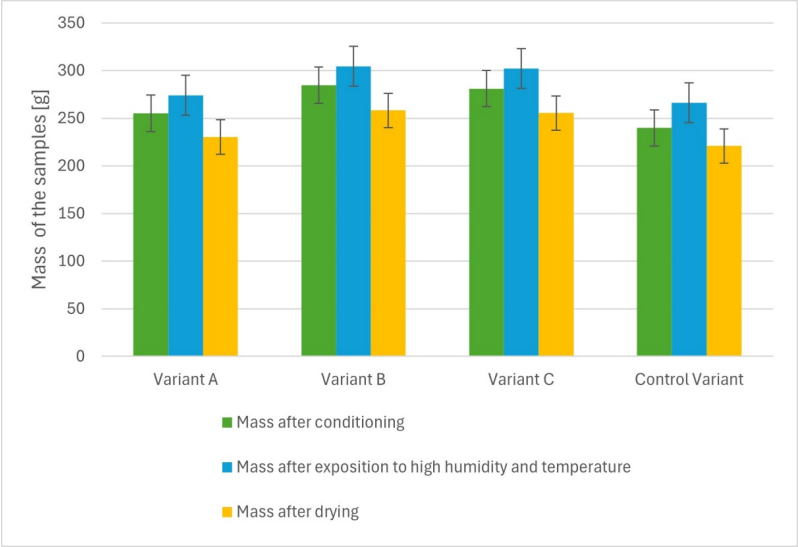


Fig. 1. Mass change during durability test according to Annex A of the EN 16755 standard

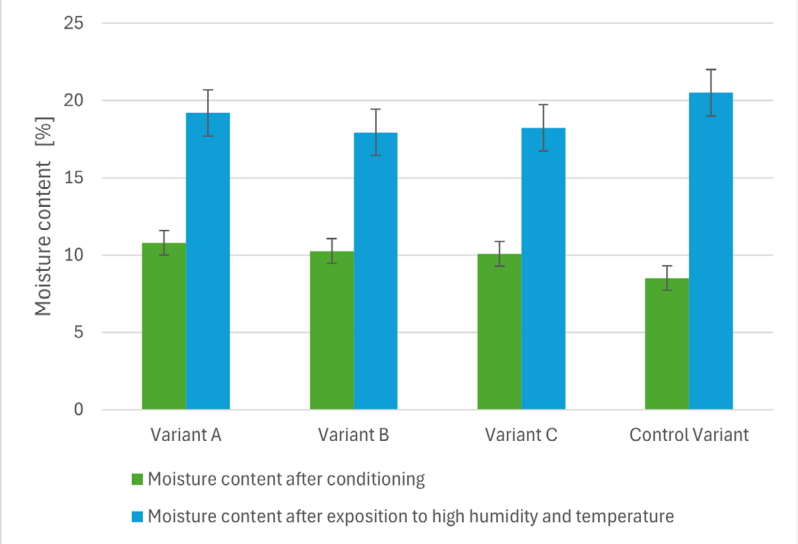


Fig. 2. Moisture content change during durability test according to Annex A of the EN 16755 standard

for the average mass increase and 1.8 percentage points for the mass loss. It appears that impregnation with salt-based fire retardants reduces the sensitivity of wood to ambient climatic conditions [Brazier and Laidlaw 1974]. This would be a desirable feature, but it is not consistent with the majority of literature data. In most tests of salt impregnation, a greater sensitivity to changes in humidity is observed. However, an exception is found in the case of preparations based on phosphates, which within a certain range of ambient humidity lead to a decrease in the equivalent humidity of impregnated pine wood [Bendtsen 2009].

Comparing variants B and C, where the impregnation application and test methods were similar, it is observed that Agent I (based on phosphorus and iron) had a greater effect on the hygroscopic properties of the tested material. However, when the same Agent I was used with different methods (variants A and B), which led to different impregnant application rates, variant A gave worse results. A larger amount of impregnant caused a deterioration in the moisture stability of the modified material.

In the case of changes in the percentage of water content in wood, the greatest changes were recorded for the control group (Figure 2). In laboratory conditions, the equivalent moisture content of the samples was 8.5%, and the moisture content for the conditions described under the EN 16755 standard was 20.5%; the observed increase in moisture content was 12 percentage points.

For the impregnated material, the equivalent moisture content of the material in laboratory conditions was within the range 10–11%. The lowest moisture content of the tested impregnated materials was observed for samples from variant C, where Agent II was used. These samples obtained 10.07% moisture content. With Agent I, the values were 10.79% for variant A and 10.25% for variant B.

The smallest variation in material moisture content was found for variant B; the difference was 7.67 percentage points, more than 4 percentage points less than for the control group. Variant C also led to a small change in moisture content; the difference was 8.15 percentage points, only 6.25% higher than the value for variant B. Of the variants tested, the largest changes in humidity resulted from variant A (where pressure impregnation was used), for which the difference was 8.4 percentage points. This is 9.5% larger than the difference recorded for the variant in which the same impregnating agent was applied using immersion impregnation (variant B).

It is interesting that all groups of impregnated material (variants A, B, C) showed significantly smaller humidity fluctuations than the control group.

3. Influence of conditions on impregnated samples

Material impregnated with Agent I changed color to more orange-red after the impregnation process. Material impregnated with Agent II did not show any color change. However, for both impregnants used, a large color change occurred after the drying process at 104 °C. The material darkened significantly irrespective of the protective agent used, while additionally for variant C, dark brown streaks appeared on the wood surface (Figs. 3–8).



Fig. 3. Impregnated sample before exposure to humidity (variant A)



Fig. 4. Impregnated sample before exposure to humidity (variant B)



Fig. 5. Impregnated sample before exposure to humidity (variant C)



Fig. 6. Impregnated sample after exposure to humidity (variant A)



Fig. 7. Impregnated sample after exposure to humidity (variant B)



Fig. 8. Impregnated sample after exposure to humidity (variant C)

Conclusions

Based on tests of pine wood consisting of 80–95% heartwood subjected to impregnation in three different variants, the following conclusions were drawn:

1. Wood modified with salt-based flame retardants is less sensitive to changes in ambient humidity than raw wood.
2. Vacuum impregnation, which produced a three times higher rate of salt agent deposition, does not lead to decreased sensitivity to change in ambient humidity.
3. Equivalent moisture content in laboratory conditions is much higher for impregnated wood.
4. Phosphorus and iron-based impregnation may lead to less change in the appearance and color of wood under conditions of 104 °C than phosphorus and nitrogen-based impregnation.

Conflict of interest

The author(s) declare(s) that there is no conflict of interest concerning the publication of this article.

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