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# INVESTIGATION OF THE INFLUENCE OF IMPREGNATION ON THE PINE TIMBER COMBUSTION USING FLOW THROUGH TESTS

# Badanie wpływu impregnacji ogniochronnej na skład i ilość produktów toksycznych powstałych podczas spalania drewna sosnowego

#### Summary

Millions of people have lost their lives during fire in the recent decades, the majority died from inhalation of toxic fire effluents. Toxic fire effluents cause death in fire, as any incapacitation is likely to impede escape, and increase the chance of becoming trapped. Better understanding of this problem will contribute to the reduction in the number of such deaths in the future. This paper analyses emissions of carbon monoxide (CO), hydrogen chloride (HCl), hydrogen cyanide (HCN) and ammonia (NH<sub>3</sub>) during the combustion of non-impregnated and impregnated pine timber with fire retardants in research equipment for toxic combustion products emitted from solid materials after the impact of the heat source (e.g. flow through test). The toxicity of pine timber specimens was investigated at two powers of external heat source of 8 kW/m<sup>2</sup> and 10 kW/m<sup>2</sup>.

#### Streszczenie

Co roku wielu ludzi traci życie w czasie pożaru. Większość ginie z powodu wdychania toksycznych produktów rozkładu termicznego i spalania. Produkty te stanowią nie tylko bezpośrednią przyczynę śmierci ale także mogą

utrudniać skuteczną ewakuację. Lepsze zrozumienie problematyki toksyczności produktów spalania przyczyni się do zmniejszenia liczby tych zgonów w przyszłości. Artykuł ten przedstawia wyniki badań emisji tlenku węgla (CO), chlorowodoru (HCl), cyjanowodoru (HCN) i amoniaku (NH<sub>3</sub>) podczas spalania bezpłomieniowego drewna naturalnego sosnowego, jak i impregnowanego przeciwogniowo dwoma komercyjnymi środkami ogniochronnymi produkowanym na Litwie. Do badań eksperymentalnych wykorzystano nie normatywną technikę pomiarową. Próbki poddano oddziaływaniu strumienia promieniowania cieplnego o gęstości 8 i 10 kW/m<sup>2</sup>.

**Keywords:** pine timber, fire retardants, treatment, fire, toxicity, heat source, combustion products, carbon monoxide;

**Słowa kluczowe:** drewno sosnowe, środek ogniochronny, pożar, toksyczność, produkty spalania, tlenek węgla, chlorowodór, amoniak, cyjanowodór;

# 1. Introduction

There are factors to be encountered that endanger not only people inside the building but also those performing rescue works during the fire occurance. The factors include: (Buhanan 2002; Kolbrecki 2000; Seńczuk 1998; Hartzell 1993; Иванников and Клюс 1987):

- toxic combustion products,
- flame and high temperature (heat),
- reduction of oxygen concentration,
- instability of constructions,
- reduced visibility (smoke).

Each of these factors and their effect on the escape behaviour are briefly discussed below. The time available for escape is a period between the time of ignition and the time after which conditions prevent occupants from safe evacuation. In Great Britain and the United States of America more than 50 % of all deaths in fires have been caused by toxic combustion products constituting smoke (Stec and Hull 2010; Brushlinsky *et al.* 2009; Drysdale 1998).

The consequences of fire may be reduced by performing effective preventing actions during the emergence of fire, such as extinguishing it before spreading (Buhanan 2002). One of the possibilities for implementing effective prevention is usage of materials protecting from the impact of fire, i. e. fire retardants (Ozkaya *et al.* 2007; Jun-wei Gu *et al.* 2007; Koo *et al.* 

2001; Nassar 1999). For building materials, i. e. timber constructions that are impregnated with fire retardants, time to flame combustion is longer and flame spread rate is lower (Karpovič 2009; Gu *et al.* 2007).

Fire science is multidisciplinary, it includes chemistry, physics, engineering as well as computer modelling. The conditions in real fires are difficult to recreate in a laboratory scale. Physical and chemical methods used for analysis of combustion of different flammable materials can be divided into small, medium and full scale.

This paper is the beginning of three-part project. Further research on the toxicity of non-impregnated and impregnated pine timber with fire retardants are to be performed in a small and full scales (second part) and with application of fire hybrid modelling (third part).

The aim of the further studies is to analyse the toxicity and compare the results obtained from small and full scales methods as well as from fire hybrid modelling (Galaj 2009, 2010).

Most small scale methods used for toxicity tests are developed to simulate a single fire source or the conditions, where real scale fires simultaneously involve different fire phases in different places. These methods can be grouped as those with constant combustion conditions, often achieving a prolonged steady state period, and those with unstable combustion conditions. The latter ones are the most suitable data for comparison and modelling, for example German tube furnace method DIN 53436, and its derivative ISO 19700. Most other small scale methods have changeable combustion conditions, such as those in closed or semi closed chambers exposed to a constant source of heat, for example NBS smoke chamber ISO 5659, cone calorimeter ISO 5660-1, stationary tube furnace test NFX 70-100 and fire propagation apparatus ASTM E 2058. The methods can be grouped according to their physical arrangement: open tests (cone calorimeter), closed tests (smoke chamber) and flow through tests (stationary and steady state tube furnaces, controlled atmosphere cone calorimeter and the fire propagation apparatus) (Stec and Hull 2010).

Fire zone during the open tests is well ventilated. The cone calorimeter has been developed specifically to determine the rate of heat release and effective heat of combustion of building materials. It has been subsequently modified to determine smoke generation (ISO 5660-2) and later applied to furnitures. The cone calorimeter is probably the most widely used apparatus for measurement of flammability properties (Schartel and Hull 2007).

Closed chamber tests attempt to direct the transition through the fire stages by enclosing the sample in a fixed volume of air, heating it, with or without ignition, and monitoring the formation of toxic gases, as the oxygen concentration falls, and the fire condition changes from well ventilated to under ventilated. The methods can be subdivided into two broad categories, where decomposition or combustion occurs in the main chamber, or in a side chamber (Schartel and Hull 2007).

In flow through methods the specimen is thermally decomposed, with or without flame, in a furnace over a known volume of flowing air, which drives the effluent to the sampling system or gas measurement devices.

The field of fire protection examines resistance to fire of ceramics (Abraitis and Stankevičius 2007; Žurauskienė and Nagrockienė 2007), concrete (Chung Kyung-soo *et al.* 2007; Abramowicz and Kowalski 2007; Jonaitis and Papinigis 2005), ferro concrete (Bednarek and Ogrodnik 2007) and steel (Bednarek and Kamocka 2006), application of the zone model to investigate the combustion of different flammable materials (Galaj 2007), combustion of polymeric materials (Konecki and Półka 2009) as well as the impact of isolating materials to timber strength (Bednarek and Kaliszuk-Wietecka 2007), combustion of timber treated with fire retardants, the effectiveness of fire retardants (Karpovič 2009; Półka 2008), the hazardousness of pine timber and cork-oak while fuming (Karpovič and Šukys 2009) and reaction-to-fire of nine different wood species having different density and thickness (Harada 2001). Lewin 2005 emphasized the need for research on timber treated with fire retardants. There are many questions unanswered concerning the protection of timber against fire. One of such questions is the toxicity of timber impregnated with fire retardants was partly analysed in Šukis and Karpovič (2010) work.

The aim of this work is to analyse the toxicity of non-treated and treated pine timber with fire retardants, to determine alternation of emission of carbon monoxide (CO), hydrogen chloride (HCl), hydrogen cyanide (HCN) and ammonia (NH<sub>3</sub>) subjected to the different heat source intensity using flow through method.

### 2. Materials tested and test method

Tests were performed in the Main School of Fire Service by using research equipment for toxic combustion products emitted from solid materials after the impact of the heat source (flow through test). To investigate the influence of impregnation on combustion properties, three types of samples were tested: pure pine timber without impregnation and two pine timbers impregnated with fire retardants A and B:

1. The primary ingredient of fire retardant A is potassium carbonate. Used as an aqueous solution of inorganic salts. While the impact of thermal radiation. 8-10 mm is formed

coating, protective wooden structures. Recommended use - 500 ml/m<sup>2</sup>. Fire classification according to LST EN 13501 is B-s 2, d 0.

The primary ingredient of fire retardant B is potassium carbonate. Used as an aqueous solution of inorganic salts. While the impact of thermal radiation. 6-8 mm is formed coating, protective wooden structures. Recommended use - 450 ml/m<sup>2</sup>. Fire classification according to LST EN 13501 is B-s 1, d 0.

The research equipment for toxic combustion products shown in Fig. 1 enables to determine concentrations of emitted toxic combustion products while impacting specimen with different heat sources. The possible range of the heat sources measured on the surface of specimens varies from 2 to 80 kW/m<sup>2</sup>. During the test two heat sources were used, i. e.  $8 \text{ kW/m^2}$  an10 kW/m<sup>2</sup>. By impacting specimens with such heat sources the conditions were established for the emission of the main amount of combustion products. At the heat sources of less than  $8 \text{ kW/m^2}$  the temperature on the specimens does not reach 160° C, while at the heat sources higher than 10 kW/m<sup>2</sup> the specimens inflame (the research equipment cannot be applied for tests when flammable combustion occurs). The computer connected to the research equipment ensured accurate control of the test and automatically recorded the results. Five specimens in every test group were tested. The timescale for one test course was 1 hour. The dimensions of the specimens were 200 mm x 200 mm x 20 mm.

Two tests were performed for the following three main groups of the specimens:

- non-treated pine timber specimens,
- pine timber specimens treated with the fire retardant A,
- pine timber specimens treated with the fire retardant B.

The fire retardants used for the treatment of pine timber specimens have been produced and used in Lithuania.



Fig 1. A view of the research equipment for the measurement of the concentration of toxic combustions products

## 3. Flow through test results and analysis

The results have been processed with "Statistica" programme. Correlation coefficients of the emission of CO, HCN, HCl and NH<sub>3</sub> are given in Tab. 1.

Table 1.

Correlation coefficient of the emission of CO, HCN, HCI, NH3					
Heat source, specimen	Correlation coefficient of the emission of				
	combustion products on the same parameters				
	СО	HCN	HCl	NH <sub>3</sub>	
$8 \text{ kW/m}^2$ , pine	0.993	0.980	0.903	0.992	
$8 \text{ kW/m}^2$ , pine with A	0.943	0.993	0.985	0.969	
$8 \text{ kW/m}^2$ , pine with B	0.974	0.996	0.909	0.917	
$10 \text{ kW/m}^2$ . pine	0.985	0.981	0.912	0.985	
$10 \text{ kW/m}^2$ , pine with A	0.921	0.979	0.934	0.992	
$10 \text{ kW/m}^2$ , pine with B	0.914	0.965	0.942	0.998	

Correlation coefficient of the emission of CO, HCN, HCl, NH<sub>3</sub>

The variation of temperature on the surface of the specimens for two values of heat source power of 8 kW/m<sup>2</sup> and 10 kW/m<sup>2</sup> and different specimens is shown in Fig. 2. In turn, the changes in concentrations of the emission of CO, HCN, HCl and NH3 are presented in Fig. 3-6.

Analysing flow through tests results, the following observations with reference to temperature can be pointed out:

the value of temperature rapidly increases during first period of combustion (from 0 to approx. 600 s) for two powers of heat source 8 kW/m<sup>2</sup> and 10 kW/m<sup>2</sup>. Afterwards the changes of temperature are significantly slower not exceeding 0.2° C/s (only between 1700 and 2000 s this value is bigger in case of pine timber without retardant) (Fig. 2);

- the average temperature of the specimens non-treated with fire retardants was about 21.1° C (8 kW/m<sup>2</sup>) and 14° C (10 kW/m<sup>2</sup>) higher than the average temperature on the pine timber specimens treated with the A fire retardant (Fig. 2);
- after influencing non-treated specimens with the heat source of 8 kW/m<sup>2</sup> the temperature observed on their surface was approx. 9.4° C higher than on the pine timber specimens treated with the B fire retardant, while when impacting pine timber specimens treated with B fire retardant with the heat source of 10 kW/m<sup>2</sup> the temperature on their surface was about 9.5° C higher compared to the non-treated specimens (Fig. 2).

The differences between the average temperature on the surface of treated specimens compared with to the average surface temperature of non-treated specimens during steady phase of combustion are given in Tab. 2.

#### Table 2.

The differences of the average of temperature on the surface of treated specimens compared with the average of temperature on the surface of non-treated specimens during steady phase of combustion

Heat source	Specimens, temperature		
neat source	pine with B	pine with A	
$8 \text{ kW/m}^2$	-9.4° C	-21.1° C	
$10 \text{ kW/m}^2$	+9.5° C	-14° C	



Fig 2. Temperatures on the surface of tested specimens: pine, pine treated with A and pine treated with B for heat sources of 8 kW/m<sup>2</sup> and 10 kW/m<sup>2</sup>



Fig 3. Emission intensity of carbon monoxide for heat source of 8 kW/m<sup>2</sup> and 10 kW/m<sup>2</sup> for all tested specimens



Fig 4. Emission intensity of hydrogen cyanide for two power of heat source 8 kW/m<sup>2</sup> and  $10 \text{ kW/m}^2$  and all tested specimens



Fig 5. Emission intensity of hydrogen chloride for two power of heat source 8 kW/m<sup>2</sup> and  $10 \text{ kW/m^2}$  and all tested specimens



Fig 6. Emission intensity of ammonia for two power of heat source 8 kW/m<sup>2</sup> and 10  $kW/m^2$  and all tested specimens

In the following tables such parameters obtained during tests are given:

1. The average values of time  $\tau_1$  and temperature  $t_r$  corresponding to the beginning of registration of measured toxic products (CO, HCN, HCl and NH<sub>3</sub>) for heat source 8 kW/m<sup>2</sup> in table 3 and 10 kW/m<sup>2</sup> in table 4.

- 2. The values of characteristic times  $\tau_2$  and  $\tau_3$  as well as steady value *SV* corresponding to the parameters of the emission intensity of measured toxic products (CO, HCN, HCl and NH<sub>3</sub>) for heat source 8 kW/m<sup>2</sup> in table 5 and 10 kW/m<sup>2</sup> in table 6.
- 3. The total quantities of the toxic gases, such as CO, HCN, HCl and NH<sub>3</sub>, emitted during the tests in table 7.

Table 3.

# The average values of time $\tau_1$ and temperature $t_r$ corresponding to the beginning of the registration of CO, HCN, HCl and NH<sub>3</sub> emissions during combustion of all tested specimens at power of heat source of 8 kW/m<sup>2</sup>

Gas	Type of specimen	$ au_1$ s	t <sub>r</sub> °C
СО	non-treated	300	140
	with A	420	165
	with B	365	156
HCN	non-treated	955	200
	with A	880	198
	with B	900	200
HCl	non-treated	430	167
	with A	1015	207
	with B	920	203
NH <sub>3</sub>	non-treated	670	197
	with A	730	199
	with B	765	202

Table 4.

The values of time  $\tau_1$  and temperature  $t_r$  corresponding to the beginning of the registration of CO, HCN, HCl and NH<sub>3</sub> emissions during combustion of all tested specimens at power of heat source of 10 kW/m<sup>2</sup>

~	Type of	τ1	tr
Gas	Specimen	S	°C
СО	non-treated	170	135
	with A	270	170
	with B	300	178
HCN	non-treated	570	220
	with A	550	217
	with B	500	219
НСІ	non-treated	275	169
	with A	460	215
	with B	485	221
NH <sub>3</sub>	non-treated	365	201
	with A	475	217
	with B	565	223

#### Table 5.

#### The values of times $\tau_2$ , $\tau_3$ and steady value *SV* corresponding to the characteristic parameters of the emission intensity of CO, HCN, HCl and NH<sub>3</sub> during combustion of all tested specimens at power of heat source of 8 kW/m<sup>2</sup>

Gas	Type of	$ au_2$	$ au_3$	SV
	specimen	S	S	mg/s
СО	А	2600	2700	50
	В	2200	2780	60
HCN	А	1800	2600	1.8
	В	1750	2700	2.3
НСІ	А	2600	*	4.5
	В	2600	*	7.0
NH <sub>3</sub>	А	1000	2000	0.45
	В	1100	2100	0.67

\*- emission intensity of HCl from non-impregnated sample is always greater than from impregnated samples

Table 6.

The values of times  $\tau_2$ ,  $\tau_3$  and steady value *SV* corresponding to the characteristic parameters of the emission intensity of CO, HCN, HCl and NH<sub>3</sub> during combustion of all tested specimens at power of heat source of 10 kW/m<sup>2</sup>

Cas	Type of	$ au_2$	$ au_3$	SV
Gas	Specimen	S	S	mg/s
СО	A	1100	2020	130
	В	1050	1950	120
HCN	A	1100	2200	5.0
	В	1100	2010	4.2
	A	1180	**	6.3
нсі	В	1100	**	11.0
NH <sub>3</sub>	А	1050	1990	1.1
	В	1100	2080	1.3

\* - emission intensity is only steady until t=2000 s and then it systematically grows

\*\* - emission intensity of HCl from non-impregnated sample is always greater than from impregnated samples

Table 7.

# Toxic combustibility products and their quantities emitted during tests with different heat sources and type of the specimens

Heat source, specimen	Total quantity of toxic combustibility products [mg]				
	СО	HCN	HCl	NH <sub>3</sub>	
$8 \text{ kW/m}^2$ , pine	31539.9	1146.23	8769.05	476.27	
$8 \text{ kW/m}^2$ , pine with A	23228.6	834.96	1233.2	195.95	
$8 \text{ kW/m}^2$ , pine with B	27833.1	975.09	2987.9	211.29	
$10 \text{ kW/m}^2$ , pine	94828.5	3149.22	11722.3	932.54	
$10 \text{ kW/m}^2$ , pine with A	61299.6	2514.04	4379.94	489.87	
$10 \text{ kW/m}^2$ , pine with B	56889.8	2317.76	6014.64	633.89	

The views of non-treated and treated specimens after tests with heat source of  $8 \text{ kW/m}^2$  and  $10 \text{ kW/m}^2$  are presented in Figs. 7–10.



Fig 7. Views of non-treated pine timber specimen after tests with heat source of 8  $kW/m^2$ 



Fig 8. Views of non-treated pine timber specimen after tests with heat source of  $10 \text{ kW/m}^2$ 



Fig 9. Views of treated pine timber specimen after tests with heat sources of 8  $kW/m^2$ 



Fig 10. Views of treated pine timber specimen after tests with heat sources of  $10 \text{ kW/m}^2$ 

# **5.** Conclusions

Based on the analysis of the measured emissions of combustion products the following conclusions can be formulated:

1. The treated pine timber specimens affected with the heat source of 8 kW/m<sup>2</sup> emit approximately

1.46 times less of toxic combustion products than the non-treated pine timber specimens. The treated pine timber specimens affected with the heat source of 10 kW/m<sup>2</sup> emit approximately 1.65 times less of toxic combustion products than non-treated pine timber specimens. The power of heat source influence on the emission of toxic combustibility products. The treated pine timber specimens affected with the heat source of 8 kW/m<sup>2</sup> emit 2.34 times less of toxic combustion products than pine timber specimens treated with the same fire retardants affected with the heat source of 10 kW/m<sup>2</sup>. Pine timber specimens without fire retardants affected with the heat source of 8 kW/m<sup>2</sup> emit 2.64 times less of the toxic gases than the same pine timber specimens affected with the heat source of 10 kW/m<sup>2</sup> (see Fig. 3-6).

- 2. Time denoted by  $\tau_1$  and temperature denoted by  $t_r$  as the characteristic parameters of the beginning of the registration of CO, HCN, HCl and NH<sub>3</sub> depend on either type of gas or power of heat source. The comparison showed longer times  $\tau_1$  (over 1.5 times) and higher temperatures (except for HCN) for impregnated specimens than for non-impregnated ones. Generally, better results for B at 10 kW/m<sup>2</sup> and for Flamesapas-2 at 8 kW/m<sup>2</sup> were obtained. For HCN these times are comparable or shorter in case of non-impregnated sample (see Tab. 3 and 4).
- 3. Emissions of all measured gases from all specimens were higher at 10 kW/m<sup>2</sup> than at  $8 \text{ kW/m}^2$  (see Tab. 3 and 4).
- 4. Emission of CO, HCN and NH<sub>3</sub> from non-treated pine timber is of the increasing character during the whole test. It's value is lower than emission from treated timber up to time denoted by  $\tau_3$  (see Tab. 5 and 6).
- 5. The increases in emissions of all considered toxic products of combustion from impregnated specimens can be noticed in the initial phase of the process, which are faster for experiments with higher power of heat source (10 kW/m<sup>2</sup>). The emissions begin to oscillate around steady values *SV* after the time  $\tau_2$ . Some difference in emission of HCl from specimen impregnated with A, consisting in the repeated increase, can be observed in the second period of process (see Tab. 5 and 6).
- 6. The steady values of emissions SV of all measured products of combustion were greater (about 1.5 times on average) for pine timber impregnated with B in case of lower power of heat source 8 kW/m<sup>2</sup> (see Tab. 5). A little higher values of SV of CO and HCN emissions were obtained for pine timber impregnated with Flamesapas-2 than BAK-1 in

the case of power of heat source equal to  $10 \text{ kW/m}^2$ . The tendency was opposite in case of remaining gases e. g. it was almost two times lower for specimen with A (see Tab. 6).

- After termination of combustion process the surface of treated pine timber specimens is smoother, the layer of carbon is solid and un-cracked. For non-treated surfaces – the layer of carbon is cracked, while deeper layers of timber are affected by the process of thermal destruction (see Figs. 7–10).
- 8. Non-treated pine timber specimens generate larger quantity of these products compared to the pine timber specimens treated with fire retardants (see tab. 7).

Taking into account the results obtained from both experiments with combustion of non-treated pine timber specimens and treated with two different fire retardants, A and B, it can be concluded that the impregnation influences the combustion process decreasing the rate and the level of emission of toxic products. Generally, lower concentrations have been observed for specimens impregnated with A. Impregnation increase emission of toxic products (except for HCl) in the initial phase of the combustion. But total quantities of all tested toxic combustibility products during complete period of experiment were significantly lower in the case of impregnated pine timber.

Considering the above, it can be concluded that in order to choose a suitable fire retardant, reducing unfavourable impact of the combustion on human during the potential internal fire, each situation should be analysed independently, based on the smokiness as well as generation of toxic combustion products. Analyzed problem requires further studies based on the results obtained during small scale tests using cone calorimeter method and full scale fire experiments.

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