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TOXICITY RESEARCH OF SMOULDERING AND FLAMING PINE TIMBER TREATED WITH FIRE RETARDANT SOLUTIONS

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Abstract. The emission of toxic gaseous combustion products from timber constructions influences on the time required for evacuation of people from a building during a fire. In order to prolong the time interval until inflammation of timber constructions, fire retardant solutions are used. It is relevant and very important to determine how the emission of toxic gaseous combustion products from pine timber non-treated and treated with fire retardant solutions used in Lithuania differs during thermal destruction. Measuring carbon monoxide (CO) emissions, the paper focuses on toxicity analysis determined by nonstandard and standard research methods of smouldering and flaming pine timber, both non-treated and treated with fire retardant solutions. The description comprises specimens used in research and their preparation, non-standard and standard research equipment and methods determining toxicity of smouldering and flaming pine timber, both non-treated and treated with fire retardant solutions. The article presents the analysis of experimental results processed by statistical methods.

Keywords: pine timber, smouldering, flaming, toxicity, carbon monoxide.

1. Introduction

Timber is one of the main building materials, long-time used in construction as well as most widely applied. There are many building materials produced from raw materials however many villagers live in wooden buildings. Many household buildings are wooden (Nagrodzka, Maloziec 2011; Teischinger 2010).

Use of timber in construction is limited due to easy flammability and quick spreading of fire. Even at the temperature of 300 ºC timber under thermal destruction emits a sufficient amount of flammable gas causing inflammation and combustion (Drysdale 1998).

Timber combustion is a complicated process. Thermal destruction of timber has been analysed and presented thoroughly by a number of authors (Jeguirim, Trouvé 2009; Windeisen, Wegener 2008; Hosoya *et al.* 2007; Frey *et al.* 2009). Two types of timber combustion may be distinguished: smouldering and flaming. The first one is considered to be more dangerous as it remains invisible for a period of time. Besides, smouldering results in greater emissions of toxic gaseous combustibility products. The intensity of combustibility, the quality and quantity of the emitted toxic gaseous combustibility products depend on the type of timber, its moisture, and circumstances of combustion (Stec, Hull 2010).

Combustion produces smoke – the mix of gas, fumes and soot. Researches prove that toxic gaseous combustibility products that are found in smoke impede breathing, reduce range of visibility and prolong time needed for evacuation of people (Papinigis *et al.* 2010; Tserng *et al.* 2011). It poses 60–80% of all deaths in fires. During the last five years, on an average 262 people died in fires in Lithuania annually (Brushlinsky *et al.* 2012). In 2011, 8 people per 100 thousand people died in fires in Lithuania. This indicator is one of the highest among the European Union countries. In 2008 and 2011, in terms of this indicator Lithuania overtook Latvia and Estonia (Fire and Rescue Analysis 2012).

According to the fire safety requirements, the combustibility of timber constructions in a building must be reduced. Usually, the combustibility of timber constructions is reduced by treating them with fire retardant solutions (Wang *et al.* 2008). During combustion, the temperature of timber treated with fire retardant solutions is lower, the layer of carbon is thicker, the rapidity of heat emission and weight reduction is decreased (Jiang *et al.* 2010; Hagen *et al.* 2009).

Timber (Šaučiuvėnas *et al.* 2011; Kängsepp *et al.* 2011; Mačiulaitis, Praniauskas 2010; Bednarek *et al.* 2009; Juodeikienė 2009; Bednarek, Kaliszuk-Wietecka 2007), fire retardants (Glenn *et al.* 2012; Babrauskas *et al.* 2011; Gałaj *et al.* 2011a; Grigonis *et al.* 2011; Karpovič *et al.* 2010; Karpovič 2009; Vobolis, Albrektas 2009; Pereyra, Giudice 2009; Półka 2008), timber treated with fire retardant solutions in connection with its toxicity during combustion (Gałaj *et al.* 2011b; Šukys, Kar-

povič 2010; Karpovič, Šukys 2009; Paul *et al.* 2008; Lestari *et al.* 2006) and computer modelling for combustion (Capote *et al.* 2012; Keshavarz *et al.* 2012; Fouladgar *et al.* 2012; Vaidogas *et al.* 2012; Cheng, Hadjisophocleous 2011; Gałaj 2009) have been already analysed in the field of fire safety. However the toxicity of smouldering and flaming pine timber which is non-treated and treated with fire retardant solutions has not yet been studied within an integrated approach. This issue is very important in Lithuania and globally. Toxicity of construction products and (or) interior decoration products during fire is regulated in few countries (Gann *et al.* 2011). The toxicity of construction products in Lithuania is not regulated either. Such research and analysis could open up opportunities for making required decisions in order to reduce number of victims in fires.

The aim of the work: using nonstandard and standard research equipment and methods, to determine the toxicity by assessing CO of smouldering and flaming pine timber, both non-treated and treated with fire retardant solutions.

2. Specimens, research equipment and research methods

Pine timber specimens non-treated and treated with fire retardant solutions used in tests were cut from defect-free (i.e. crack-free) pine timber boards of 0.2 m in width, 0.02 m in thickness and 530 kg/m^3 of average density. Pine timber boards were naturally dried to humidity of less than 15%. It was treated with fire retardant solutions Flamasepas-2 and BAK-1 (with K_2CO_3 as the main component) according to the recommendations of the producers, i.e. brushing the surface with not less than 500 ml/m² of the fire retardant solution. To ensure fire retardant solutions do not evaporate and penetrate the treated timber as deeply as possible, surfaces of the pine timber boards were covered with foil for 24 hours. The pine timber boards treated with fire retardant solutions were naturally dried to humidity of less than 15%.

The fire retardant solutions Flamasepas-2 and BAK-1 (hereinafter – A and B) used for the treatment of timber have been certified and used in Lithuania.

This research was performed for three groups of specimens:

- pine timber specimens non-treated with fire retardant solutions;
- pine timber specimens treated with the fire retardant solution A;
- pine timber specimen treated with the fire retardant solution B.

The nonstandard research on toxicity of smouldering pine timber – both non-treated and treated with fire retardant solutions – was performed in the Main School of Fire Service in Warsaw. The research equipment for toxic combustions products emitted from solid materials after the impact of a heat flux was used (Fig. 1).

The aforementioned equipment can be used to determine toxic combustibility products and their quantity emitted while affecting a specimen with different heat fluxes. The possible range of a heat flux was from 2 to 80 kW/m^2. The necessary condition for the research was set: thermal destruction had to proceed without breaking into flames, i.e. the tested specimens had to be smouldering. Two heat fluxes – one of 8 kW/m^2 and another of 10 kW/m^2 – were used in this research. By affecting the specimen with heat fluxes of 8 kW/m^2 and 10 kW/m^2, the conditions were established for the emission of the main amount of toxic combustion products. At the heat fluxes of less than 8 kW/m^2, the temperature on the specimens did not reach 160 ºC; while at the heat fluxes higher than 10 kW/m^2, the specimens inflamed.

Fig. 1. Research equipment for toxic combustion products emitted from solid materials affected by a heat flux

5 specimens in every group were tested. The time-scale for one test course was up to 55 min. The dimensions of the specimens were 0.2×0.2×0.02 m.

The standard research on toxicity of smouldering pine timber – both non-treated and treated with fire retardant solutions – was performed in the Main School of Fire Service in Warsaw using the cone calorimeter that corresponds to the requirements of the ISO 5660-1:2002 standard. The cone calorimeter is depicted in Fig. 2.

Before each of the tests, a specimen of 0.1×0.1×0.02 m dimensions was weighted and folded in foil except for the surface exposed to a heat flux. The prepared specimen was placed in the special frame, which was laid onto the scale under the heating cone.

Fig. 2. A view of the cone calorimeter

5 specimens in every group were tested. During the tests, the specimens were affected by the heat flux of 30 kW/m^2. No piloted ignition was used. Affecting the specimens with the given heat flux and with no piloted ignition, the conditions for smouldering were established and the temperature on the surface of the specimens did not exceed 270 ºC. The duration of each toxicity test of the smouldering pine timber non-treated and treated with fire retardant solutions amounted to 15 min.

The nonstandard research on toxicity of flaming pine timber – both non-treated and treated with fire retardant solutions – was performed in the Main School of Fire Service in Warsaw. The confined cabin containing the research equipment was used. The interior of the confined cabin is depicted in Fig. 3.
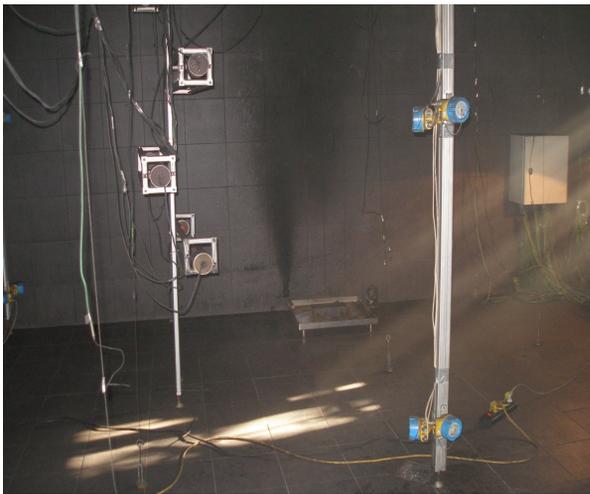

Fig. 3. Interior of the research equipment

The two walls of the cabin of 5×5×2.8 m were made of aluminium and glass. The other two brick walls were covered with ceramic tiles. The measuring equipment for CO concentration was located in the cabin. During the research, the concentration of CO was measured by nine electrochemical sensors of "ALTER SA MG 72" type, accurate to 1 ppm. The sensors were mounted on three columns at three heights in the research cabin: 0.35 m, 1.4 m and 2.5 m. The scheme of the vertical and horizontal positioning of the sensors measuring CO concentration in the research cabin is depicted in Fig. 4.

During each test, a steel tray with three specimens was placed inside the unventilated research cabin and combusted so that the flame could affect the surface of the specimens treated with fire retardant solutions. The specimens were combusted by pouring and firing up 0.4 *l* of denatured alcohol in the steel tray. During every test, the steel tray with specimens was placed in the same place of the test cabin near the back wall.

9 specimens in every group were tested. The dimensions of the specimens were 0.2×0.2×0.02 m. After inflammation of a specimen, the doors of the cabin were closed. During the tests, the changes in CO concentration level were measured and saved in different points of the test cabin every 5 s. When fire parameters stopped altering, the ventilation system was started and the test was closed.
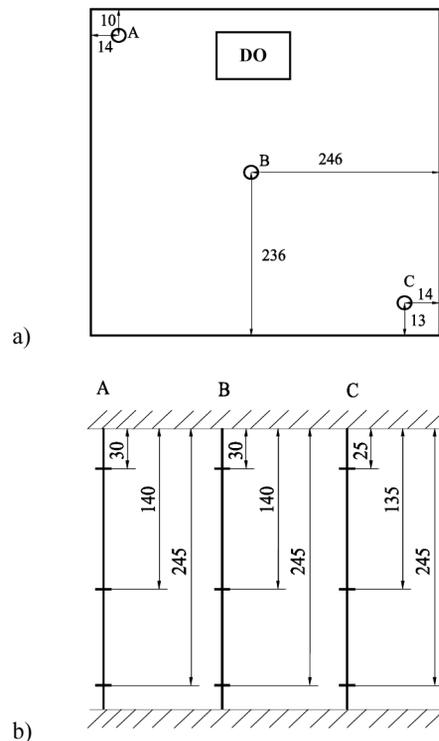

Fig. 4. Scheme of vertical (a) and horizontal (b) positioning for the sensors measuring CO concentration, installed on A, B and C columns (numbers – in cm; DO – burning object)

The standard research on toxicity of flaming pine timber non-treated and treated with fire retardant solutions was performed in the Main School of Fire Service in Warsaw. The cone calorimeter that corresponds to the requirements of ISO 5660-1:2002 standard was used. The description of the cone calorimeter and research methods for the determination of toxicity of flaming pine timber – both non-treated and treated with fire retardant solutions – which was described above was the same as the standard research methods used for testing smouldering pine timber, both treated and non-treated with fire retardant solutions.

During the tests of flaming timber, the specimens were affected by the heat flux of 30 kW/m^2 together with piloted ignition of 10 kV. Affecting the specimens with the given heat flux together with piloted ignition, the conditions for flaming were established.

The piloted ignition source was formed from two parallel electrodes located at the height of 1 cm above the surface of a specimen. The electrodes were attached to the mechanism of piloted ignition, which regulates the electrodes to approach the surface of the specimens and to distance from it. When approaching the surface of the specimens, the electric current was transmitted through the electrodes at a fixed periodicity. It created a spark at the ends of the electrodes.

Subjected to time, the obtained results of toxicity of smouldering and flaming pine timber – both non-treated and treated with fire retardant solutions – determined by nonstandard and standard research equipment were analysed statistically. The arithmetic averages were developed to the statistical selections of the results $x_1, x_2, ... x_n$ (Sakalauskas 2003).

The arithmetic averages of the statistical selections of the results were processed using the programme "Statistika 8". The negative exponential function high-reflecting test results was applied. The correlation coefficients r and linear and non-linear curve regression equations formed by the programmes "Statistika 8" and "TableCurve 2D" are also presented (Sakalauskas 2003; Kleiza 2003).

3. Experimental results and discussion

The results of the nonstandard research on toxicity of smouldering timber

The average emission of CO depending of the heat flux and the specimens is shown in Figs 5–6.

Affecting pine timber specimens with the heat flux of 8 kW/m^2 in 300 s on the average, after the surface temperature of the specimen reached the average of 140 °C, the sensor started registering CO (Fig. 5). The temperature was reached at which pine timber specimens emitted CO during thermal destruction.

Affecting pine timber specimens treated with the fire retardant solution A with the heat flux of 8 kW/m^2 in 420 s on the average, after the surface of the specimen reached the mean temperature of 165 °C, the sensor started registering CO (Fig. 4). Affecting pine timber specimens treated with the fire retardant solution B by the heat flux of 8 kW/m^2 in 365 s on the average, after the surface of the specimen reached the mean temperature of 156 °C, the sensor started registering CO (Fig. 5). Due to the protective features of fire retardants to impede gas emission during thermal destruction, the emission of CO from pine timber specimens treated with the fire retardant solutions A and B started after a longer period of time and at a higher surface temperature as compared to the emission of CO from the non-treated pine timber specimens.

Affecting the non-treated pine timber specimens with the heat flux of 10 kW/m^2 in 170 s on the average, after the surface of the specimens reached the mean temperature of 135 °C, the sensor started registering CO (Fig. 6).

Affecting pine timber specimens treated with the fire retardant solution A with the heat flux of 10 kW/m^2 in 270 s on the average, after the surface of the specimen reached the mean temperature of 170 °C, the sensor started registering CO (Fig. 6).

Affecting pine timber specimens treated with the fire retardant solution B by the heat flux of 10 kW/m^2 in 300 s on the average, after the surface of the specimen reached the mean temperature of 178 °C, the sensor started registering CO (Fig. 6).

Increasing the heat flux and the speed of temperature rise, the emission of CO during thermal destruction from pine timber specimens non-treated and treated with fire retardant solutions started after a shorter period of time.

At the beginning of the tests, pine timber specimens treated with the fire retardant solutions A and B emitted CO more intensely as compared to the non-treated pine timber specimens in the period up to 2700 s (8 kW/m^2) and up to 1900 s (10 kW/m^2). However, this emission altered insignificantly after 1500 s at 8 kW/m^2, when the surface of the specimens reached the mean temperature of 211 °C and after 1000 s at 10 kW/m^2 when the surface of the specimens reached the mean temperature of 260 °C. The emission of CO from non-treated pine timber was growing during the entire research (Figs 5–6).

In the course of the tests during the initial 2700 s, the non-treated pine timber specimens affected by the heat flux of 8 kW/m^2 emitted 2.8 times less CO; and during the initial 1900 s, the non-treated pine timber specimens affected by the heat flux of 10 kW/m^2 emitted 3.1 times less CO as compared to the pine timber specimens treated with the fire retardant solutions A and B (Figs 5–6).

Equations shown on figure:

(1) $\ln y = 1.075 + 3.88 \times 10^{-7} \times x^2$; r = 0.995
(2) $y = -1.652 + 0.019 \times x$; r = 0.9529
(3) $y = 0.742 + 0.022 \times x$; r = 0.9186
(4) $y^2 = 484.625 + 1284.32 \times x^{0.5}$; r = 0.953
(5) $y^{-1} = 0.0033 + 0.056/x^{0.5}$; r = 0.911
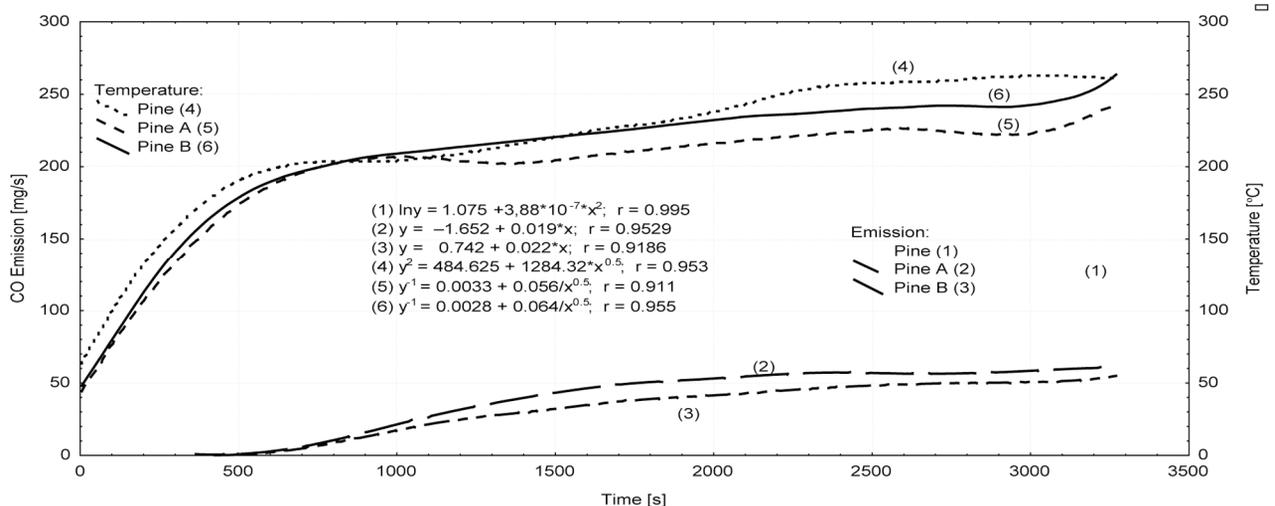(6) $y^{-1} = 0.0028 + 0.064/x^{0.5}$; r = 0.955

Fig. 5. Average emissions of CO from tested specimens and alternation of average temperature on the surface of tested specimens affected by heat sources of 8 kW/m^2 subjected to time: (1) – non-treated pine timber specimens; (2) – pine timber specimens treated with A; (3) – pine timber specimens treated with B; (4) – non-treated pine timber specimens; (5) – pine timber specimens treated with A; (6) – pine timber specimens treated with B
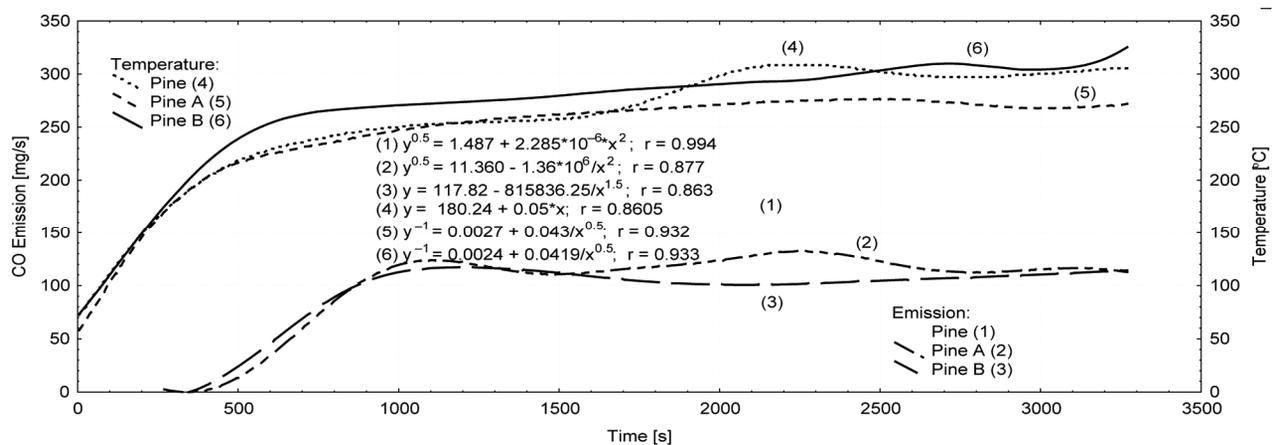
Fig. 6. Average emissions of CO from tested specimens and alternation of average temperature on the surface of tested specimens affected by heat sources of 10 kW/m^2 subjected to time: (1) – non-treated pine timber specimens; (2) – pine timber specimens treated with A; (6) – pine timber specimens treated with B

The fire retardants reduced the temperature on the surface of the specimens and the delivery of oxygen to the pyrolisis zone. It increased the emission of CO – thermal destruction product of partial oxidation – at the beginning of the tests. In the course of the tests, the emission of CO from the pine timber specimens treated with the fire retardant solutions A and B as compared to the non-treated pine timber specimens altered insignificantly. The protective features of fire retardants impeded the penetration of temperature to the deeper layers of timber and stopped the emission of thermal destruction gas.

Comparing pine timber specimens treated with the fire retardant solution A with specimens treated with the fire retardant solution B, pine timber specimens treated with the fire retardant solution A affected with the heat flux of 8 kW/m^2 emitted on average 1.2 times more CO than pine timber specimens treated with the fire retardant solution B. Pine timber specimens treated with the fire retardant solution A affected by the heat flux of 10 kW/m^2 emitted on average 1.1 times more CO than pine timber specimens treated with the fire retardant solution B (Figs 5–6). The difference in the emission of CO depended on the unequal microscopic structure, inequality in wood tar content and in the composition of fire retardants.

The results of the standard research on toxicity of smouldering timber

The average alternation of CO concentration depending on the specimens is depicted in Fig. 7.

At the beginning of the research concentration of CO for pine timber specimens treated with the fire retardant solutions A and B in the period up to 300 s was growing intensely. After 300 s the concentration of the CO for pine timber specimens treated with the fire retardants A and B was changing insignificantly and did not exceed the value of 0.13 kg/kg (kg/kg – the fraction of CO mass to the mass of air). In the period up to 350 s, pine timber specimens treated with the fire retardant solutions A and B as compared to the non-treated pine timber specimens obtained the highest concentration of CO due to the same reason as mentioned above. The concentration of CO for non-treated pine timber specimens was growing during the entire research and reached the value of 0.16 kg/kg (Fig. 7).

The highest concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was about 20% lower than the highest concentration of CO for the non-treated pine timber specimens. During the initial 350 s the concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was about 20% higher than the concentration of CO for the non-treated pine timber specimens (Fig. 7).

Comparing pine timber specimens treated with the fire retardant solution A with specimens treated with the fire retardant solution B, the concentration of CO for pine timber specimens treated with the fire retardant solution A was on average 1.1 times lower than the concentration of CO for pine timber specimens treated with the fire retardant solution B (Fig. 7).

The results of the toxicity of the nonstandard research on toxicity of flaming timber

The average concentration of CO near the burning object, measured by the sensor mounted on the B column in the centre of the cabin at the height of 1.4, depending on the specimens is depicted in Fig. 8.

At the beginning of the research, in the period up to 260 s, the concentration of CO for all groups of pine timber specimens grew intensely. After 260 s, the concentration of the CO for pine timber specimens treated with the fire retardant solutions A and B altered insignificantly and did not exceed the value of 6x10^{-5} kg/kg. The concentration of CO for the non-treated pine timber specimens grew during the entire research and reached the value of 7.2x10^{-5} kg/kg (Fig. 8).

During flaming of the non-treated pine timber specimens, the sensor started registering CO during initial seconds of the tests. In the case of the flame combustion of pine timber specimens treated with the fire retardant solutions A and B, the sensor started registering CO after 62 s on the average (Fig. 8). Due to the protective features of fire retardants, the emission of CO from pine timber specimens treated with the fire retardant solutions A and B started after a longer period of time comparing with the emission of CO from the non-treated pine timber specimens.
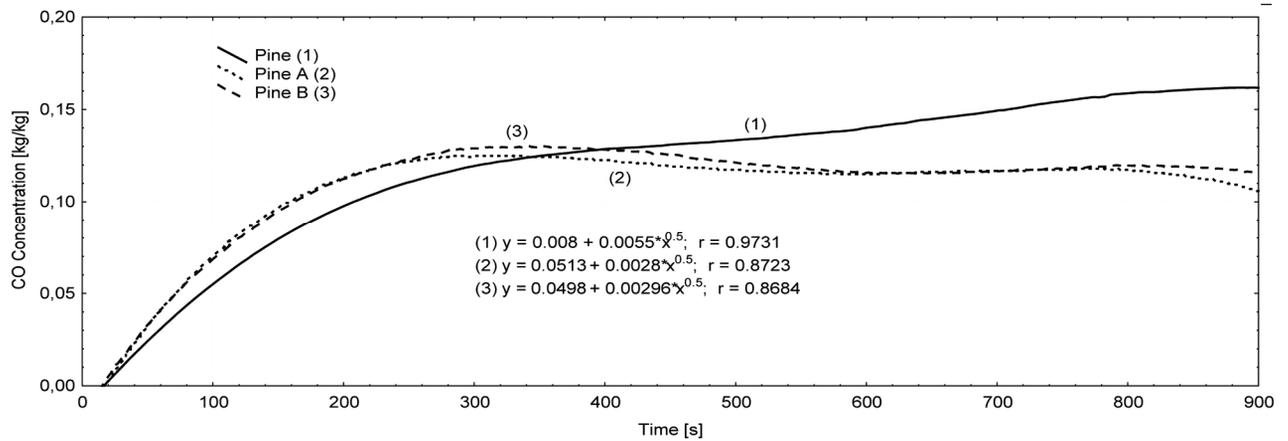
Fig. 7. Alternation of average CO concentration obtained during smouldering of tested specimens subjected to time: (1) – non-treated pine timber specimens; (2) – pine timber specimens treated with A; (3) – pine timber specimens treated with B

Fig. 8. Alternation of average CO concentration measured by the sensor in the centre of the compartment at the height of 1.4 m obtained during flame combustion of tested specimens subjected to time: (1) – non-treated pine timber specimens; (2) – pine timber specimens treated with A; (3) – pine timber specimens treated with B
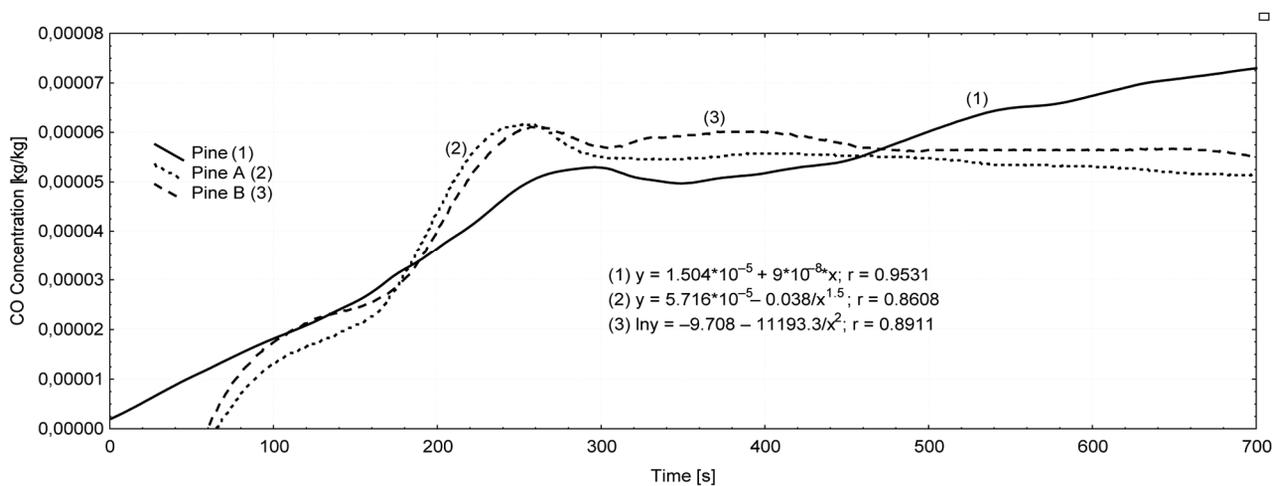
The highest concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was on average 1.2 times lower than the highest concentration of CO for the non-treated pine timber specimens. During the initial 180 s the concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was averagely 1.4 times lower than the concentration of CO for the non-treated pine timber specimens. At the time interval of 180–460 s the obtained concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was 1.2 times higher than the concentration of CO for the non-treated pine timber specimens (Fig. 8).

Comparing pine timber specimens treated with the fire retardant solution A with specimens treated with the fire retardant solution B, the concentration of CO of pine timber specimens treated with the fire retardant solution A was on average 1.2 times lower than the concentration of CO for pine timber specimens treated with the fire retardant solution B (Fig. 8).

The results of the standard research on toxicity of flaming timber

The average alternation of CO concentration depending on the specimens is depicted in Fig. 9.

At the beginning of the tests in the period up to 200 s, the concentration of CO for pine timber specimens treated with the fire retardant solutions A and B grew intensely and reached 0.019 kg/kg and 0.022 kg/kg respectively. After 200 s, the average concentration of CO for pine timber specimens treated with the fire retardant solutions A and B began reducing. The concentration of CO for the non-treated pine timber specimens was growing up to 400 s on the average and did not exceed the value of 0.005 kg/kg (Fig. 9).

The highest concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was on average 4.4 times higher than the highest concentration of CO for the non-treated pine timber specimens. During the research, the concentration of CO for pine timber specimens treated with the fire retardant solutions A and B was on average 1.8 times higher than the concentration of CO for the non-treated pine timber specimens (Fig. 9). The fire retardants reduced the delivery of oxygen to the pyrolisis zone by increasing the emission of thermal destruction product of partial oxidation – CO.
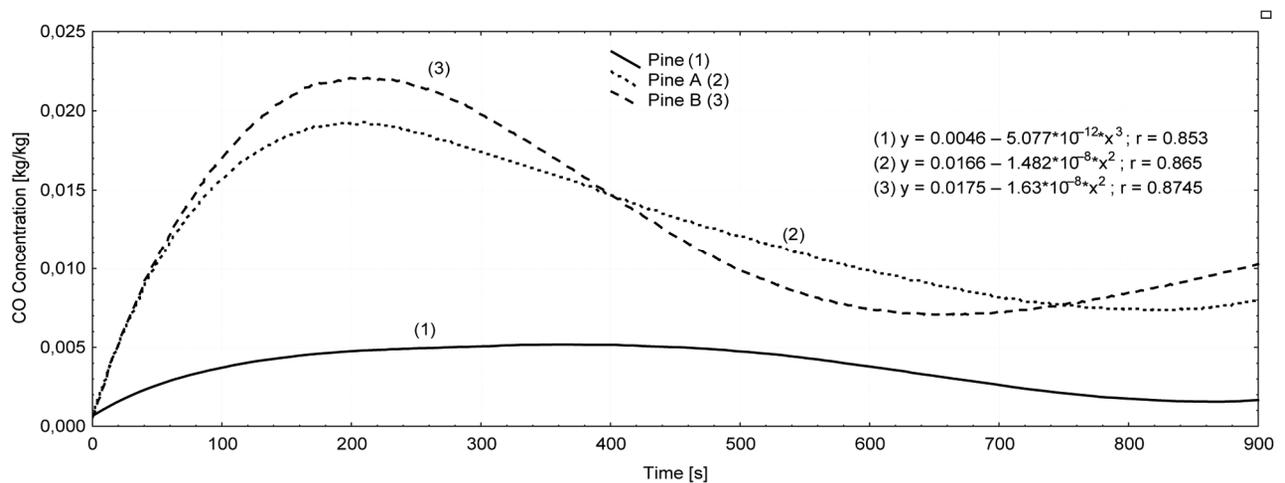
Fig. 9. Alternation of average CO concentration obtained during flame combustion of tested specimens subjected to time: (1) – non-treated pine timber specimens; (2) – pine timber specimens treated with A; (3) – pine timber specimens treated with B

Comparing pine timber specimens treated with the fire retardant solution A with specimens treated with the fire retardant solution B, the concentration of CO for pine timber specimens treated with the fire retardant solution A was on average 1.1 times lower than the concentration of CO for pine timber specimens treated with the fire retardant solution B (Fig. 9). The difference in the emission of CO depended on the unequal microscopic structure, inequality in wood tar content and in the composition of fire retardants.

4. Conclusions

1. The beginning of the emission of CO from smouldering pine timber non-treated and treated with fire retardant solutions depends on the temperature and time at which it starts emitting. On average, the emission of CO from pine timber treated with fire retardant solutions starts after a 1.5 times longer time period after the beginning of the test and at the temperature is on average 1.2 times higher as compared to the non-treated pine timber. This is explained by the protective features of fire retardants to stop the emission of thermal destruction gas from pine timber treated with fire retardant solutions.

2. During smouldering of pine timber non-treated and treated with fire retardant solutions:
   - fire retardants reduce the temperature on the surface of timber and the delivery of oxygen to the pyrolisis zone by increasing the emission of CO;
   - the emission of CO from treated smouldering pine timber is higher during the initial seconds of the test than the emission of CO from the non-treated smouldering pine timber;
   - during the test the emission of CO from pine timber treated with fire retardant solutions alters insignificantly while the emission of CO from the non-treated pine timber intensifies;
   - on average, pine timber treated with the fire retardant solution A emitted 1.2 times more CO than pine timber treated with the fire retardant solution B (nonstandard research equipment);

   - on average, pine timber treated with the fire retardant solution A emitted 1.1 times less CO than pine timber treated with the fire retardant solution B (standard research equipment).

3. The results of toxicity research obtained using nonstandard research equipment on flaming pine timber non-treated and treated with fire retardant solutions showed that:
   - on average, the emission of CO from the non-treated pine timber starts at the beginning of the test while from pine timber treated with fire retardant solutions starts after 62 s from the beginning of the test;
   - during the initial 180 s the emission of CO from pine timber treated with fire retardant solutions is 1.4 times lower than from non-treated pine timber;
   - at the time interval of 180–460 s the emission of CO from pine timber treated with fire retardant solutions is 1.2 times higher than from the non-treated pine timber;
   - the concentration of CO for flaming non-treated pine timber grows during the entire test while the concentration of CO for pine timber treated with fire retardant solutions alters insignificantly after 260 s.

4. The results of toxicity determined by the standard research equipment on flaming pine timber non-treated and treated with fire retardant solutions prove that the emission of CO from pine timber treated with fire retardant solutions is 4.4 times higher than from non-treated pine timber.

5. Flaming pine timber treated with the fire retardant solution A emitted on average 1.15 times less CO than pine timber treated with the fire retardant solution B.

6. While performing tests using the standard and non-standard research equipment, at the beginning of the tests the emission of CO from smouldering and flaming pine timber treated with fire retardant solutions is higher than from the non-treated pine timber. This is subjected to the reason that fire retardants reduce temperature on

the surface of the specimens and the delivery of oxygen to the pyrolisis zone.

7. The correlation coefficient r used for data analysis and defining the strength of dependence between curves and regression equations has shown that correlation link is strong enough. The lowest value of the correlation coefficient r is 0.853.

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