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Cone calorimeter study of nano silica as flame retardant in selected woods

WALDEMAR JASKÓŁOWSKI[1)], DARIUSZ BILSKI[1)], MARIUSZ MAMIŃSKI[2)]

1) Faculty of Fire Safety Engineering, The Main School of Fire Service (SGSP)
2) Faculty of Wood Technology, Warsaw University of Life Sciences (SGGW)

Abstract: *Cone calorimeter study of nano silica as flame retardant in selected woods.* The aim of study is determine the fire properties for selected woods before and after treating with silica (SiO_2) as fire retardant. The fire retardancy of SiO_2, wood was tested by cone calorimeter method. The selected wood samples: pine (*Pinus sylvestris)* and birch *(Fagus sylvatica)* were treated with 0,1% aqueous solution of nano silica. Several parameters are examined from a cone calorimeter test, such as average and peak heat release rate (av HRR and pk HRR, kW/m^2), total heat release (THR, MJ/m^2), effective heat of combustion (EHC, MJ/kg), total smoke production (TSP, m^2), yield CO and CO_2 ($Y_{CO,}$ Y_{CO2}, kg/kg), specific extinction area (SEA, m^2/kg). The results of research are summarized. Conclusions are provided at the end of the paper.

Keywords: wood, fire properties, silicon dioxide, silica, fire retardant

INTRODUCTION

The wood is one most important natural materials and for centuries is being used nowadays in buildings, furniture, industries because of its versatile properties such as easy processability, accessibility, high weight to strength ratio, good insulating properties favoured appearance (Pries and Mai, 2013, Mahr at al., 2012, Rosenthal and Beaus, 2010). Despite of its excellent engineering properties, it has some serious drawbacks, these include, inter alia, that the flammability. Fire safety is an important concern in all types of construction and building materials. The new buildings and the existing ones must comply with the conditions as provided by fire protection regulations. As far as fire safety is concerned, the materials used for building industry should be characterized by low combustion heat, low smoking intensity in fire conditions and toxicity of combustion products should be leas possible. Producers of wood derived building elements do their utmost to ensure full safety by realization of the goals as mentioned above. (Jaskółowski and Borysiuk, 2010). Therefore protective treatments for the material have been known for almost as long as it has been in use becomes one of important action, which affects ensure an appropriate level of fire safety. Most of fire retardants used for wood protection are based on compounds of mono/diammonium phosphate, ammonium sulfate, zinc chloride, sodium tetraborate, boric acid phosphorus, bromine, boron and nitrogen (Liodakis et al., 2006; White and Dietenberger 2010; Grześkowiak, 2012). Flame retardants can be classified into two generally types: (1) those impregnated into the wood or incorporated into wood composite products, and (2) those applies as paint or surface coatings.

Depending of their nature, fire retardants can act chemically and/or physically in the solid or gas phase (Liodakis et al, 2006). In general, conventional fire retardants effectively protect wood from fire in the first stage their growth. Due to less toxic impact, cost effective processing and easy handling makes nano mineral fillers derived materials as one of the potential candidate for wood modification and preservation at industrial scale.

In this work two low-cost inorganic compounds silica (SiO_2) were used for wood impregnation and the effect of the treatment on fire properties were described.

MATERIALS AND METHOD

The experiments were carried out using pine *(Pinus silvestris)* and birch *(Fagus sylvatica)* samples, with density 460 kg/m^3 (pine) and 580 kg/m^3 (birch). Nano silica impregnation was performed using 0,1 % water solution by an empty cell process under vacuum conditions. After the treatment, samples were air conditioned for 7 days at 23 and a relative humidity of 65 %, until they reached a moisture content of 10%. Control samples have the same moisture content during the tests. A 0,1% aqueous dispersion of silica was using an electromechanical stirrer. The size range of the silica nanoparticles was measured to be 60 - 80 µm. The pH of the suspension was 6-7. Prior to the test, the samples were placed into a conditioning box, until they reached moisture content 10 ± 2%. To evaluate the fire properties characteristics of the woods quantitatively, a cone calorimeter testing ISO 5660 was done (fig.1). The testing involves a constant radiant heat being irradiated onto the surface of the sample, and then the sample would ignite a spark igniter First, each specimen of dimensions 100 x 100 x 10 mm is wrapped in aluminum foil and exposed horizontally to an external heat flux of 30, 50 and 70 kW/m^2. Several parameters are examined from a cone calorimeter test, such as average and peak heat release rate (av HRR and pk HRR, kW/m^2), total heat release (THR, MJ/m^2), effective heat of combustion (EHC, MJ/kg), total smoke production (TSP, m^2), yield CO and CO_2 (Y_{CO}, Y_{CO2}, kg/kg), specific extinction area (SEA, m^2/kg)

a) b)

Fig.1. Experimental set-up of the cone calorimeter. a) Cone calorimeter, b) cone heater

RESULTS AND DISCUSSION

Results of experimental research are presented in table 1 and figures 2-7.

Table 1. Fire properties of the untreated and treated pine wood species from cone calorimeter test

No.	Parmeter	External heat flux		
		30 kW/m^2	50 kW/m^2	70 kW/m^2
Untreated Pine *(Pinus sylvestris)*				
1.	Maximum heat release rate [kW/m^2]	240,74	275,93	332,58
2.	Average heat release rate [kW/m^2]	129,86	157,42	196,90
3.	Total heat release [MJ/m^2]	69,11	68,68	68,53
4.	Effective heat of combustion, [MJ/kg]	15,07	14,15	14,50
5.	Total smoke production [m^2]	4,19	4,46	5,99
6.	Yield CO, [kg/kg]	0,0151	0,0093	0,0094
7.	Yield CO_2, [kg/kg]	1,3929	1,3570	1,3546
8.	Specific extinction area, [m^2/kg]	100,42	102,87	142,85
Treated Pine *(Pinus sylvestris)*				
1.	Maximum heat release rate [kW/m^2]	228,00	312,22	328,93
2.	Average heat release rate [kW/m^2]	120,57	159,89	199,67
3.	Total heat release [MJ/m^2]	72,97	70,90	70,95
4.	Effective heat of combustion, [MJ/kg]	14,97	14,59	14,75
5.	Total smoke release [m^2]	2,85	4,51	6,04
6.	Yield CO, [kg/kg]	0,0157	0,0104	0,0097
7.	Yield CO_2, [kg/kg]	1,3927	1,3772	1,3894
8.	Specific extinction area, [m^2/kg]	65,30	104,14	142,17

Table 2. Fire properties of the untreated and treated birch species from cone calorimeter test

No.	Parmeter	External heat flux		
		30 kW/m^2	50 kW/m^2	70 kW/m^2
Untreated birch *(Fagus sylvatica)*				
1.	Maximum heat release rate [kW/m^2]	330,05	430,64	511,10
2.	Average heat release rate [kW/m^2]	137,54	177,80	205,31

3.	Total heat release [MJ/m^2]	72,39	73,80	78,02
4.	Effective heat of combustion, [MJ/kg]	13,20	13,25	13,63
5.	Total smoke release [m^2]	2,52	3,43	4,22
6.	Yield CO, [kg/kg]	0,0142	0,0106	0,0110
7.	Yield CO_2, [kg/kg]	1,3305	1,3136	1,3555
8.	Specific extinction area, [m^2/kg]	51,07	69,14	83,38
Treated birch *(Fagus sylvatica)*				
1.	Maximum heat release rate [kW/m^2]	320,18	479,61	408,58
2.	Average heat release rate [kW/m^2]	153,63	181,09	205,52
3.	Total heat release [MJ/m^2]	81,14	79,08	80,17
4.	Effective heat of combustion, [MJ/kg]	14,08	13,40	13,94
5.	Total smoke release [m^2]	2,44	3,51	4,5360
6.	Yield CO, [kg/kg]	0,0119	0,0081	0,0078
7.	Yield CO_2, [kg/kg]	1,3641	1,3428	1,3848
8.	Specific extinction area, [m^2/kg]	47,17	66,87	89,10

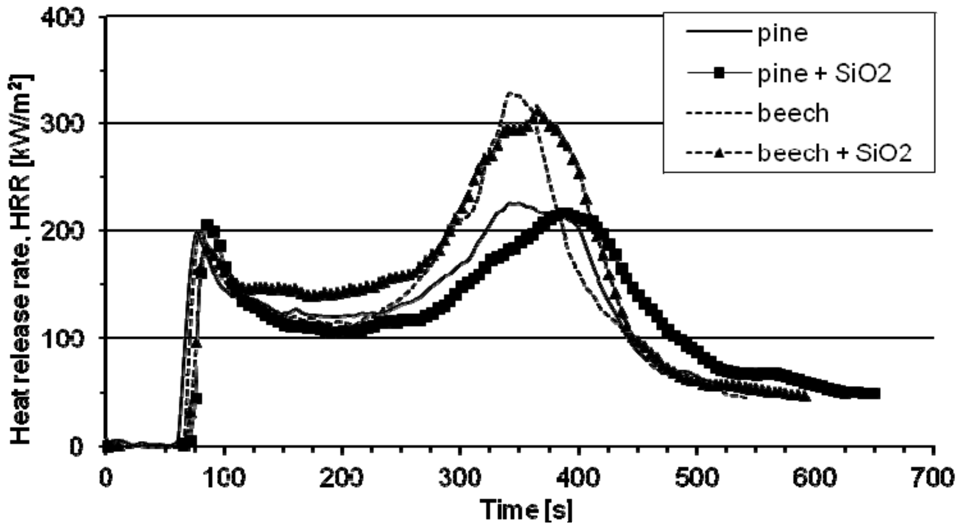

Fig.2. Heat release rate (HRR) curves of the untreated and treated woods at an external heat flux 30 kW/m^2

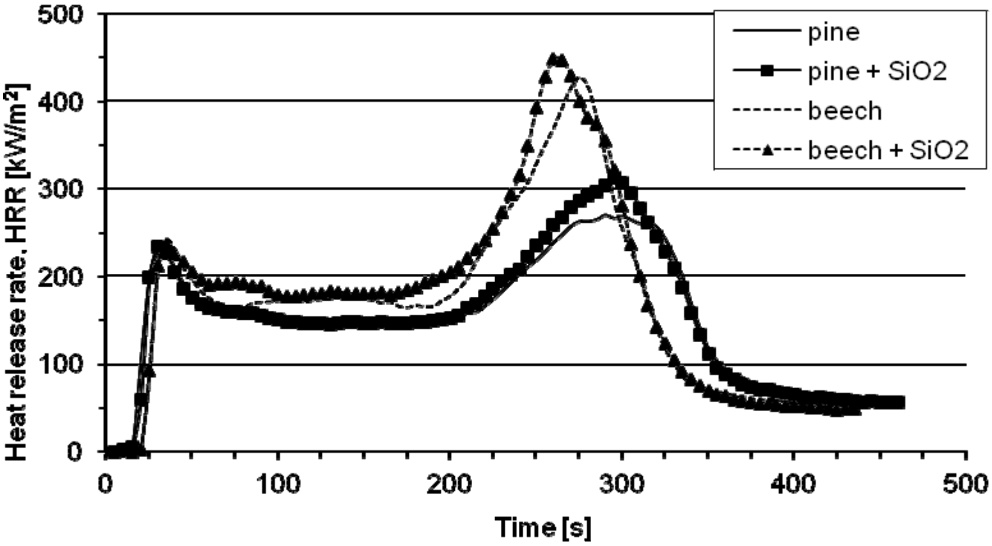

Fig.3. Heat release rate (HRR) curves of the untreated and treated woods at an external heat flux 50 kW/m^2

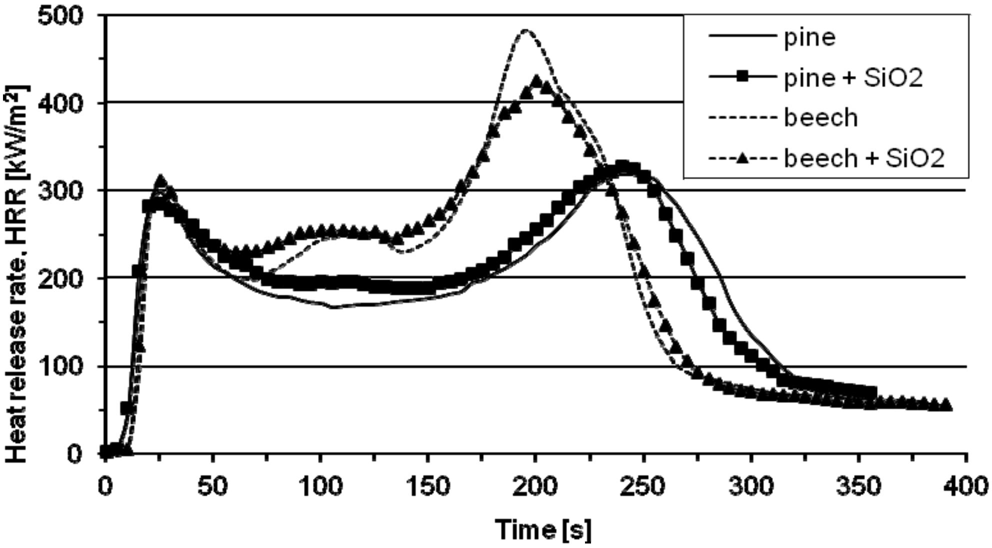

Fig.4. Heat release rate (HRR) curves of the untreated and treated woods at an external heat flux 70 kW/m^2

All HRR curves showed a two profile characteristic for tested materials as a special kind of thick char forming substances. The first peak occurred in HRR, when the char build up an efficient protection layer; the further the composition and cracking of the char towards the end of burning goes along with the second in HRR. After flame out all samples showed after glowing due the thermo oxidative decomposition of char. This afterglow is typical for wood materials and turns the black carbonaceous char into grey white ash over the time. The differences between the untreated and treated wood samples were not significant for the HRR within the same kind of wood.

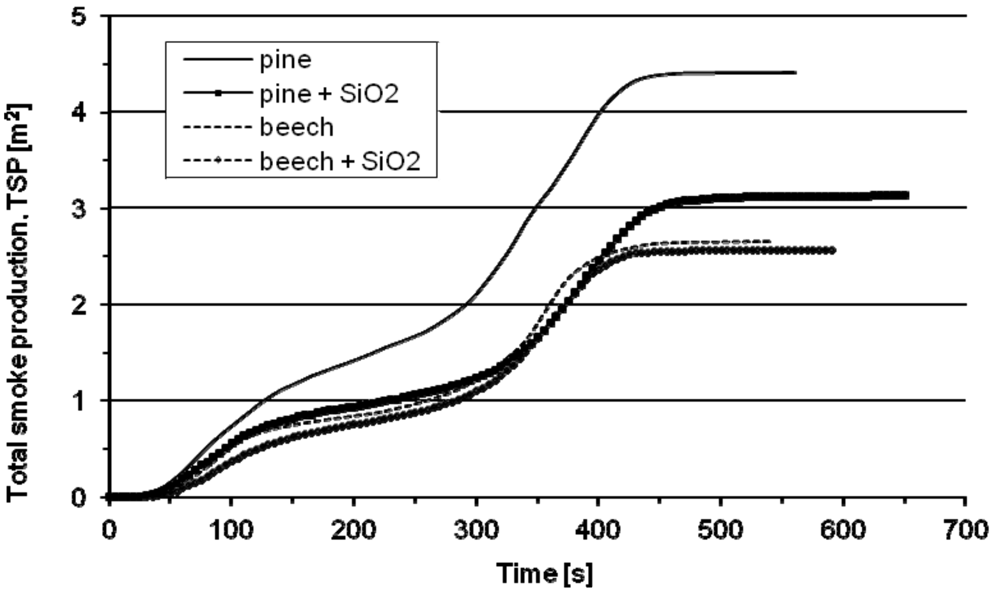

Fig.5. Total smoke production (TSP) curves of the untreated and treated woods at an external heat flux 30 kW/m^2

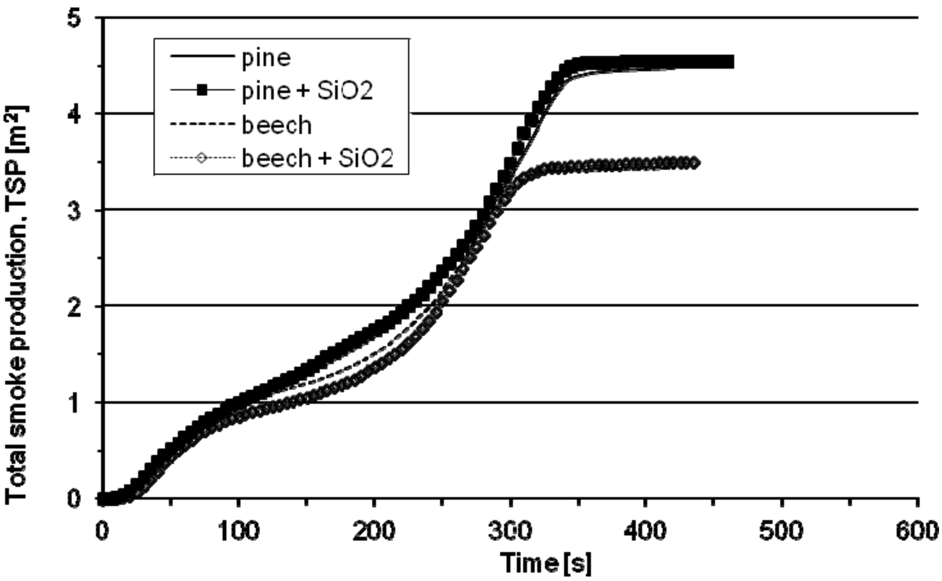

Fig. 6. Total smoke production (TSP) curves of the untreated and treated woods at an external heat flux 50 kW/m^2

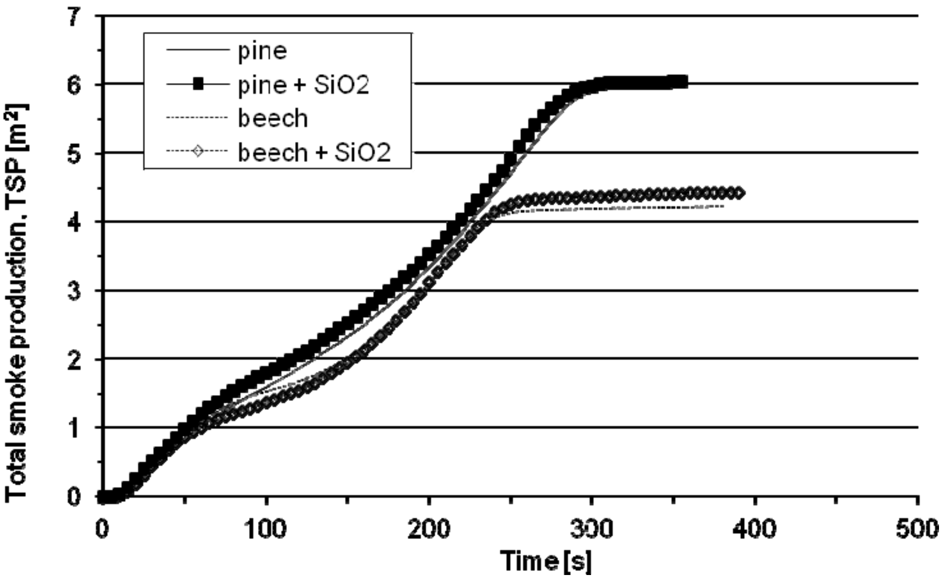

Fig. 7. Total smoke production (TSP) curves of the untreated and treated woods at an external heat flux 70 kW/m^2

Woods release dense smoke that limits visibility and can cause disorientation for people attempting to escape from a fire. The smoke consists of fine soot particles produced by thermal decomposition and flaming combustion of the wood. Fig.2-7 presents a plots of total smoke production for tested materials. Analysis of plots shows that the impregnation of the silica in the concentration used did not affect on the amount of smoke evolved.

CONCLUSIONS

Heat release rate, specific extinction area and others fire properties are often used to define fire hazard. These properties are important because they influence on the temperature and flame spread rate. Of the many fire properties, it is generally recognised that heat release rate is the single most controlling fire hazard. As far as fire retardancy activity of silicon dioxide (silica) are concerned, no significant effect was found. However, using SiO_2 solutions of higher concentration might occur effective in fire safety.

Streszczenie: *Badanie wybranych gatunków drewna impregnowanych nano krzemionką (SiO_2) z wykorzystaniem kalorymetru stożkowego.* Celem badań była ocena wpływu impregnacji nano krzemionką drewna sosnowego i bukowego na ich właściwości pożarowe. Do badań wykorzystano kalorymetr stożkowy. Próbki materiałów były impregnowane wodnym roztworem SiO_2 o stężeniu 0,1%. Podczas badań rejestrowano wybrane parametry, takie jak: szybkość wydzielania ciepła, całkowita ilość wydzielonego ciepła, całkowita ilość wydzielonego dymu. Wyniki badań przedstawiono na rysunkach i tabelach. Wnioski z badań zwarto na końcu artykułu.

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Corresponding author:

Waldemar Jaskółowski,
The Main School of Fire Service
Faculty of Fire Safety Engineering
Department of Combustion and Fire Theory,
52/54 Słowackiego St.,
01-629 Warsaw,
Poland
e-mail: wjaskolowski@sgsp.edu.pl