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Evaluation of plywood fire behaviour by ISO tests

Qiang Xu[a] · Martin Zachar[b] · Andrea Majlingová[b] · Cong Jin[a] · Yong Jiang[c]

[a]School of Mechanical Engineering, Nanjing University of Science and Technology, 210014 Nanjing, China
[b]Technical University in Zvolen, Faculty of Wood Science and Technology, T.G. Masaryka 24, 960 53 Zvolen, Slovakia
[c]State Key Laboratory of Fire Science, University of Science and Technology of China, 230026 Hefei, China

ABSTRACT

Fire behaviour of plywood was studied by bench scale ISO5660 tests and room scale ISO 9705 tests. Samples were tested with three incident irradiance heat flux from 25kW/m^2 to 50kW/m^2 to get the minimum heat flux required for ignition of the samples from fitting line. Petrella's arbitrary scales evaluation, Östman/Tsantaridis' empirical linear regression model, Hansen/Hovde's multiple discriminant function analysis (MDA) and Kokkala's model were also used in predicting flashover time and classifying the plywood from the results of 50kW/m^2 tests. The FO-categories classification of the plywood was obtained, and the results were compared with the room scale ISO 9705 tests.

Keywords: Fire behaviour • Plywood • Cone calorimetry • ISO 9705 • Heat release rate

1. Introduction

Plywood is a type of manufactured timber made from thin sheets of wood and it is one of the most widely used wood products. It is used instead of plain wood because of its resistance to cracking, shrinkage, and twisting or warping, and its general high degree of strength. Plywood could be made to ready-to-paint surfaces for constructions and ready-to-install component for buildings. Most common end use of plywood is floor, wall and roof in house construction, as well as plywood-based furniture. Many kinds of wood could be used in manufacturing plywood. Elm and pine are most widely planted trees in China, both as ornamental trees and economic wood source. Elm wood was valued for its interlocking grain, and consequent resistance to splitting, with significant uses in wagon wheel hubs, and chair seats. The elm's wood bends well and distort easily making it quite pliant. Pines are among the most commercially important of tree species, valued for their timber and wood pulp throughout the world. In China pine wood is widely used in high-value carpentry items such as furniture, window frames, panelling, floors and roofing. The fire behaviour of two kinds of plywoods based on elm and pine was analysed in this paper.

Fire development is generally characterized in terms of heat release rate (HRR) vs. time. Thus, determining the HRR is an essential aspect of a fire behaviour analysis. The HRR is not a fundamental property of a fuel and, therefore cannot be calculated from the basic material properties [1]. It is usually determined from testing. The most common method to measure HRR is known as Oxygen Consumption Calorimetry, and the most commonly used bench scale HRR apparatus is cone calorimeter which has been standardized by ISO5660 and ASTM E1354.

To study the fire behaviour of material used inside rooms, large scale fire test in ISO 9705 room is a good method to classify the material according to its flashover time, and a classification method is developed based on the ISO room tests, which is called FO-category to predict the flashover time for materials. But the room tests are costly to perform and take a relatively long time to prepare. Bench scale cone test could also be involved in quantifying the degree of fire behaviour [2]. Ignitability, flammability, heat release and smoke emission can be evaluated [3].

Researchers have tried to find rational model to relate cone test data to ISO room classification [4-6]. Petrella's [7] arbitrary scale parameters are based on 50kW/m^2 cone test results and evaluate the fire risk level from peak heat release rate, ignition time and total heat release from the cone tests. Janssens [8] developed the method to estimate the minimum heat flux for ignition from ignition time in cone tests under different incident heat flux.

Kokkala et al. [9] developed classification indexes by applying dimensional analysis and fire growth modelling to

Prof. Qiang Xu, PhD. (corresponding author)
School of Mechanical Engineering, Nanjing University of Science and Technology
Nanjing 210014, China, e-mail: kentqxu@hotmail.com

the ISO room fire test based on the test results in the Cone Calorimeter at 50kW/m^2 incident heat flux. This model connects the bench scale test with room scale test. The indexes can be used to group materials based on their predicted time to flashover. Östman and Tsantaridis [10] presented a very simple empirical linear regression model for prediction of time to flashover in the room corner test also based on cone calorimeter results from tests under 50kW/m^2 incident heat flux. Hansen and Hovde [11] evaluated the application of multiple discriminant function analysis (MDA) also on cone test data to predict the FO-category in the room corner test, which made up for the deficiency of Östman/Tsantaridis' model performing poorer for time to flashover above 10 min. We verified Östman/Tsantaridis' and Hansen/Hovde's models on GRP fire behaviour by cone and ISO room tests [12] in previous research.

Several other empirical methods [13] were also developed in predicting flashover, such as method of Babrauskas, method of McCaffrey, Quintiere and Harkleroad and method of Thomas. The minimum energy release rate of fire required for flashover in a compartment is defined in these methods. It is related to the size and ventilation factor of the compartment, effective heat transfer coefficient, fire spread speed and heat release rate of lining material. Kokkala's and those models which are based on the minimum energy release rate are relatively difficult to use.

In this paper, fire safety behaviour of elm and pine-based plywood was studied by both bench scale cone calorimetry (ISO 5660) and room scale ISO 9705 tests. Cone test data were analysed by Petrella's arbitrary scale parameters method, Janssens' minimum heat flux for ignition method, Östman/Tsantaridis' empirical linear regression model, Hansen/Hovde's multiple discriminant function analysis (MDA), and Kokkala's classification index model, the fire behaviour of both plywoods were evaluated and classified. The results are compared with the ISO room scale tests.

This study will be helpful to fire hazard calculations and fire risk analysis for fire protection design, especially to the fire design used in developing building codes based on performance criteria rather than prescriptive requirements, for example fire protection engineer must estimate the heat release rate history for a particular fuel based on fire behaviour tests.

2. Material and methods

The bench scale plywood tests were performed with the cone calorimeter in State Key Laboratory of Fire Science in University of Science and Technology of China. This calorimeter is a Standard Cone Calorimeter from Fire Testing Technology Limited, U.K. based on "the oxygen consumption method", and meets ISO5660 standard.

The specimens of 4mm thick plywood were cut as 100 by 100 mm square samples. Three tests were carried out with one incident heat flux for each kind of plywood, the incident heat flux was set as 25kW/m^2, 35kW/m^2, and 50kW/m^2. Totally nine tests were performed for one kind of plywood, and the samples were labelled as E25-1 to E25-3, E35-1 to E35-3, and E50-1 to E50-3 corresponding to incident heat flux level and elm plywood, while P25-1 to P25-3, P35-1 to P35-3, and P50-1 to P50-3 are for the pine samples. The average density of elm plywood is 583kg/m^3, and pine plywood is 566kg/m^3.

The samples were prepared according to cone test requirements. To avoid the edge effect, each sample was positioned on a sample plate with edge and bottom was covered by an aluminium foil. The effective test surface area of each sample was about 0.008836m^2. All test specimens were exposed in the horizontal orientation with the standard pilot operating. The nominal exhaust system flow rate for all tests was about 0.24m^3/s. Before testing all materials were conditioned under room temperature 23±2℃ and a relative humidity of 50±5% for 1 week.

3. Results and discussion

The peak-HRRs of all tests are illustrated in Fig.1. Elm-plywood has higher HRR peak than that of pine-plywood of different incident heat flux. In conjunction with HRR and mass loss rate measurements, the effective heat of combustion (EHC) (which is a measure of the amount of heat released from a burning material per unit of sample burned) as a function of time is calculated. Fig.2 illustrates the EHC under different incident heat fluxes during flame stage of plywood burning procedure. The average EHC is 13.83MJ/kg for elm plywood and 12.96MJ/kg for pine plywood.

Plywood sample in cone calorimeter undergoes a piloted ignition procedure, during which the plywood is heated sufficiently to vaporize and form a flammable pre-mixed system, then ignited by a spark. The ignition time could be a function of incident heat flux. Material ignition properties were derived by the method of Janssens [8] by plotting the irradiance heat flux against the reciprocal ignition time (see Fig.2). The tests reveal that the minimum heat fluxes required for ignition elm-plywood is about 14.53kW/m^2 which is lower than so-called the flashover heat flux at floor level of 20kW/m^2, and for pine-plywood is about 21.95kW/m^2 which is beyond 20kW/m^2. It is observed that the elm-plywood is of higher fire risk than pine-plywood under 20kW/m^2.

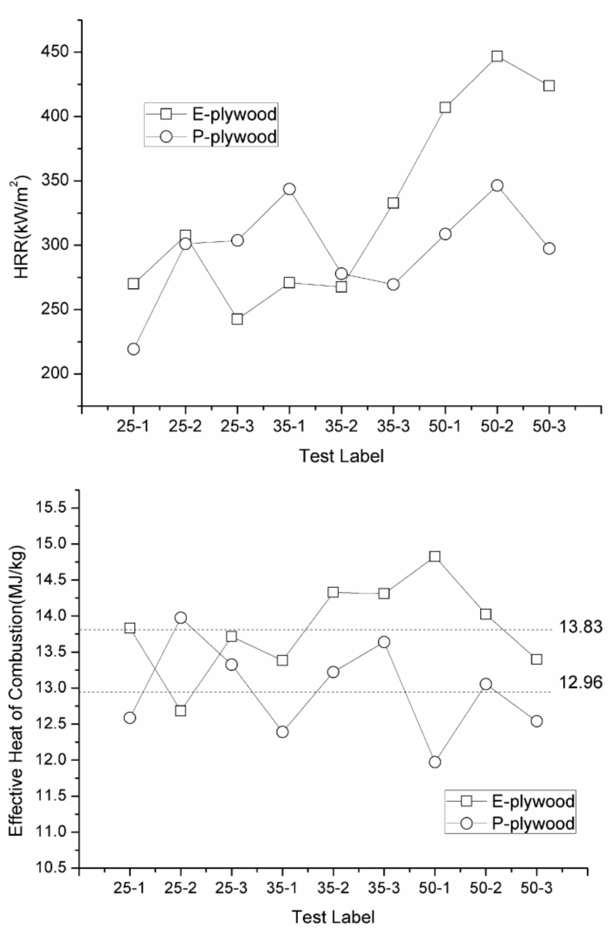

Figure 1 Peak HRR and effective heat of combustion of different incident heat flux levels

3.1. Arbitrary scales evaluation

Two parameters, the flashover propensity x (in kW/m^{2}s) and y on THR (in MJ/m^2) were proposed by Petrella [7] (Eq. 1 and 2) for studying the contribution of the materials to flashover and thermal contribution:

$$x = \frac{\text{peak(HRR)}}{t_{ig}} \tag{1}$$

$$y = \text{THR} = \int_0^\infty \text{HRR}(t)dt \tag{2}$$

Arbitrary scales suggested [7] for *x* are:

Low risk: 0.1 to 1.0
Intermediate risk: 1.0 to 10
High risk: 10 to 100

Similarly, arbitrary scales [7] for *y* are:

Very low risk : 0.1 to 1.0
Low risk : 1.0 to 10
Intermediate risk : 10 to 100
High risk : 100 to 1000

Calculated results of investigated materials are shown in Tab. 1 and Tab. 2.

Table 1 Calculated results of Eq. 1 and Eq. 2 for elm-plywood

	E50-1	E50-2	E50-3
x(kW/m^{2}s)	19.40	17.87	13.71
x-Risk level	High risk	High risk	High risk
y (MJ/m^2)	21.73	22.56	28.42
y-Risk level	Intermediate risk	Intermediate risk	Intermediate risk

Table 2 Calculated results of Eq. 1 and Eq. 2 for pine-plywood

	P50-1	P50-2	P50-3
x(kW/m^{2}s)	18.16	21.56	15.67
x-Risk level	High risk	High risk	High risk
y(MJ/m^2)	19.30	22.96	18.13
y-Risk level	Intermediate risk	Intermediate risk	Intermediate risk

3.2. FO-category

Östman and Tsantaridis [10] presented a relatively simple empirical linear regression model for prediction of time to flashover in the room corner test. The model is based on empirical data, and was found to predict time to flashover with good accuracy for several products. Cone calorimeter results from tests at 50kW/m^2 are used as input data to this model, which also requires information about mean density of the tested product. The regression model is expressed in the following Eq. (3):

$$t_{\text{FO}} = 0.07\frac{t_{\text{ig}}^{0.25}\rho^{1.7}}{\text{THR}_{300}^{1.3}} + 60 \tag{3}$$

where t_{FO} is the time to flashover in the room corner test, t_{ig} is the time to ignition in the cone calorimeter at 50kW/m^2, THR_{300} is the total heat release during 300s after ignition at 50kW/m^2 and ρ is the mean density. The model was applied to our set of test data. In the calculations we replaced the observed time to ignition with the apparent time to ignition, as Östman and Tsantaridis [10] did in their calculation, take the ignition time as the definition in Kokkala's model [9]. THR_{300} is then calculated as total heat release during 300 s after apparent time to ignition.

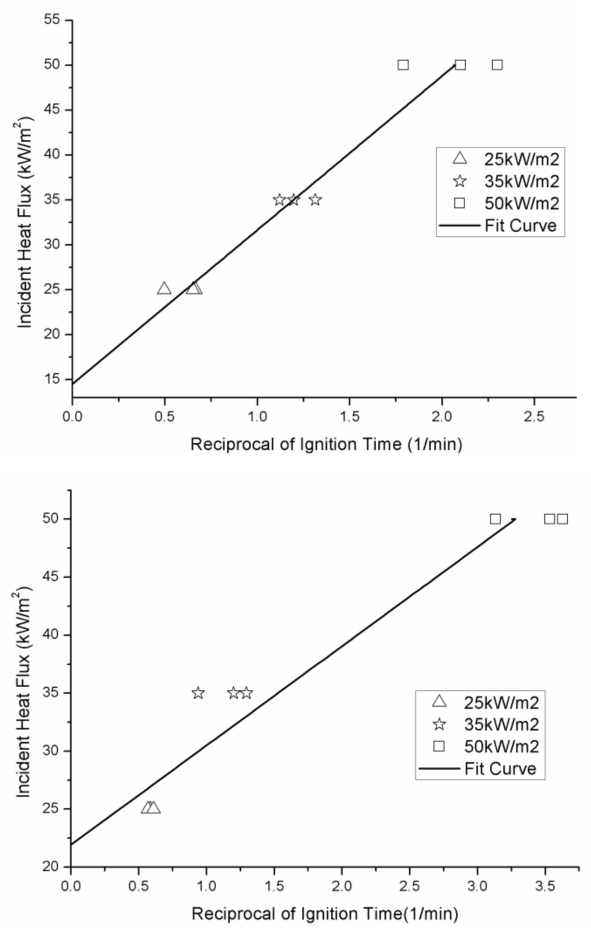

Figure 2 Effect of incident heat flux on the time to ignition (t_{ig})

Determining surface material belongs to which FO-categories can help to predict the time to flashover. The FO-categories grouping is based on ISO room tests. The ISO room corner test is used for classification of surface materials. A propane burner placed in a corner exposes the test material to a heat release rate of 100 kW for 10 min and then 300 kW for the next 10 min. The test is terminated if flashover has been reached; otherwise the total testing time is 20 min. A set of separation criteria for grouping products according to the time to flashover (t_{FO}) based on above ISO room test. These criteria divide the tested products into four groups, the so-called FO-categories [11] 1 to 4.

Surface material belongs to which category is determined by application of the following set of rules:

- FO-category 1: products not reaching flashover during 1200 seconds of testing time,
- FO-category 2: 600 seconds ≤ t_{FO} <1200 seconds,
- FO-category 3: 120 seconds ≤ t_{FO}<600 seconds,
- FO-category 4: t_{FO} <120 seconds.

For the plywood tests addressed in this paper, the duration of flame stage are less than 100 seconds because the plywood is thin, little fuel to support a long time burning. Thus, THR_{300} can not be calculated from the test data. To use the Eq. (3), a revised form is adopted as Eq. (4):

$$t_{FO} = 0.07\frac{t_{ig}^{0.25}\rho^{1.7}}{THR_{Exp300}^{P}} + 60 \qquad (4)$$

where THR_{exp300} is product of average heat release during flame combustion stage and 300 second, P is calculate as $1+0.3.(t_{if}-t_{ig})/300$, t_{if} is the end time of flame combustion and t_{ig} is the ignition time.

Calculated t_{FO} are of elm-plywood listed in Tab. 3 for the three samples under 50kW/m^2, and Tab. 4 for pine-plywood. All t_{FO} are less than 600 seconds and more than 120 seconds, thus both of these two plywoods should be classified to FO-category 3.

Table 3 Calculated t_{FO} for elm-plywood

	E50-1	E50-2	E50-3
t_{FO}(s)	149	147	139
FO-category	3	3	3

Table 4 Calculated t_{FO} for pine-plywood

	P50-1	P50-2	P50-3
t_{FO}(s)	152	144	157
FO-category	3	3	3

Surface material can also be determined to belong to which FO-category based on statistical information from cone calorimeter [11], which is called multivariate statistical method. This method may find links among different variables that are recorded in cone calorimeter tests, such as time to ignition, smoke gas concentrations, heat release rate, specimen mass loss, optical smoke density, density and thickness of samples.

Hansen and Hovde [11] evaluated the application of multiple discriminant function analysis (MDA) to deal with cone calorimeter data, which could be used to predict the FO-category in the room corner test with satisfactory accuracy. MDA is a multivariate statistical method used to classify cases into groups. The groups are determined based on a categorical dependent variable. By using Fisher's linear discriminant function for classification of cases, the result of this analysis is a set of four linear functions, one for each of the four FO-categories. A new case will be assigned to the FO-category for which the classification function obtains the highest value. Three out of about 20 variables, which give information concerning smoke production, production of CO, HRR, time to ignition, time to extinction etc, were found to be able to distinguish between the four FO-categories were. The selected parameters were:

- $z_1 = \rho_{mean}$ (kg/m^3) = mean density,
- z_2 = THR$_{300}$ (MJ/m^2) = total heat release during 300 seconds after apparent time to ignition,
- z_3 = ln(FIGRA$_{cc}$) where FIGRA$_{cc}$ is the maximum value of the ratio between HRR and time when HRR was measured.

Hansen and Hovde [11] gave the four classification functions that are expressed as follows (Eq. 5 to Eq. 8):

$$F_{FO1} = 0.01789z_1 - 0.06057z_2 + 0.971z_3 - 7.910 \qquad (5)$$

$$F_{FO2} = 0.01492z_1 + 0.03354z_2 + 1.877z_3 - 7.418 \qquad (6)$$

$$F_{FO3} = 0.008589z_1 + 0.409z_2 + 2.721z_3 - 13.406 \qquad (7)$$

$$F_{FO4} = 0.0000256z_1 + 0.347z_2 + 3.621z_3 - 9.215 \qquad (8)$$

THR$_{exp300}$ is used instead of THR$_{300}$. Ln (FIGRA$_{cc}$) is 1.564, 2.545, 1.261 for E50-1, E50-2, and E50-3, while 1.176, 1.405, and 1.1963 for P50-1 P50-2, and P50-3. Substitute these data into Eq. 5 to Eq. 8, and the results are listed in Tab. 5 and Tab. 6.

Table 5 Calculation results of MDA for elm-plywood

	E50-1	**E50-2**	**E50-3**
F_{F01}	0.5445	1.79	0.1976
F_{F02}	6.0763	7.27	5.67
$\mathbf{F_{F03}}$	**19.0625**	**21.7931**	**18.5938**
F_{F04}	16.1727	19.7769	15.3774
t_{FO}(s)	$120s \le t_{FO}<600s$	$120s \le t_{FO}<600s$	$120s \le t_{FO}<600s$
FO-category	3	3	3

For both elm-plywood and pine-plywood samples under 50kW/m^2 heat flux, all F_{FO3} give the largest value of the four Fisher’s liner discriminate functions. Thus, these two plywoods should be determined as members of FO-category 3, which would reach flashover in ISO room test from 120 to 600 seconds. The calculated results from Östman/Tsantaridis’ empirical linear regression model and Hansen/Hovde’s multiple discriminant function analysis (MDA) are the same in predicting flashover time and classifying the plywoods. It is the same as Hansen and Hovde [6-7, 9-11] mentioned that the model by Östman and Tsantaridis is excellent at predicting time to flashover before 10 min of testing time, i.e. predicting membership of FO-categories 3 and 4.

Table 6 Calculation results of MDA for pine-plywood

	P50-1	**P50-2**	**P50-3**
F_{F01}	0.5684	0.6012	0.7341
F_{F02}	4.58	4.06	4.31
$\mathbf{F_{F03}}$	**11.75**	**13.7108**	**10.70**
F_{F04}	9.43	11.96	8.16
t_{FO}(s)	$120s \le t_{FO}<600s$	$120s \le t_{FO}<600s$	$120s \le t_{FO}<600s$
FO-category	3	3	3

3.3. Kokkala's classification indexes method

The classification indexes were developed by Kokkala, Thomas and Karlsson by applying dimensional analysis and fire growth modelling to the ISO Room Fire Test [9] based on the test results in the Cone Calorimeter at 50kW/m^2 irradiance. The indexes can be used to group materials based on their predicted time to flashover. The following steps describe the procedure used to determine the material grouping. The material classification assumes boundaries between classes at 2, 10, 12 and 20 minutes.

1. The specimen is tested in the Cone Calorimeter at 50kW/m^2 irradiance.
2. The calculates the ignition time (t_{ig}) which is defined as the time for the heat release rate to reach 50kW/m^2, and the Ignition Index (I_{IG}) which is the reciprocal of the ignition time.
3. The calculates rate of heat release index (I_Q) from the following Eq. (9):

$$I_Q = \int_{t_{ig}}^{t_f} \left[\frac{\dot{q}''(t)}{\left(t - t_{ig}\right)^m} \right] dt \qquad (9)$$

where: q,.” is rate of heat release per unit area and m is an exponent determined empirically.

4. Values of I_Q are determined for two values of m, 0.34 and 0.93. These values are compared with criteria established empirically for determining whether flashover occurs:
   (a) either before 10 minutes or before 20 minutes,
   (b) either before 2 minutes or before 12 minutes.

This enables materials to be divided into four groups:

(i) materials that go to flashover before 2 minutes (before 2/12 and before 10/20 min.),
(ii) materials that go to flashover in 2 to 10 minutes (after 2/12 and before 10/20 min.),
(iii) materials that go to flashover in 10 to 12 minutes (before 2/12 and after 10/20 min.),
(iv) materials that do not go to flashover (after 2/12 and after 10/20 min.).

The values for the limits are:

- $I_Q = 6800 - 540\, I_{IG}$ for the 10 or 20 minute criterion,
- $I_Q = 1650 - 165\, I_{IG}$ for the 12 minute criterion,
- $I_Q = 2475 - 165\, I_{IG}$ for the 2 minute criterion.

For these calculations: $m = 0.34$ is used to compare to the 10 or 20 minute criterion, and $m = 0.93$ is used to compare to the 2 and 12 minute criteria.

If:

$I_{Q(m=0.34)} > 6800 - 540\, I_{IG}$
then flashover is expected before 10 minutes or before 20 minutes,
$I_{Q(M=0.93)} > 1650 - 165\, I_{IG}$
then flashover is expected after 10 minutes and before 12 minutes,
$I_{Q(M=0.93)} > 2475 - 165\, I_{IG}$
then flashover is expected before 2 minutes. Otherwise flashover is not expected.

The calculated results of classification indexes for the two kinds of plywood are listed in Tab. 7, and the same results are obtained as those with MDA method and revised empirical linear regression model.

Table 7 Calculated classification indexes

Test label	Ignition Index	Heat Release Index		Estimated Flashover time
	(1/min)	m=0.34	m=0.93	(min)
E50-1	2.86	5681	1623	2 to 10
E50-2	2.40	5714	1659	2 to 10
E50-3	1.94	6537	1703	2 to 10
P50-1	3.53	5159	1397	2 to 10
P50-2	3.75	5315	1419	2 to 10
P50-3	3.16	5613	1514	2 to 10

One test was performed in ISO room for each kind of plywood in State Key Laboratory of Fire Science in University of Science and Technology of China (results are shown on Fig. 3). The room tests were conducted in ISO 9705 room. The room, smoke collected hood, exhaust duct, and all instrumentations met the specifications of ISO 9705. The burn room has internal dimensions 3.6 m long, 2.4 m wide and 2.4 m high, and the wall and ceiling was covered with tested plywood. A calibrated propane gas burner was used as fire source. The test results are illustrated in Fig.3, and the flashover time 135 seconds for elm-plywood and 180 seconds for pine-plywood. Both of the plywoods could be classified to FO-category 3.

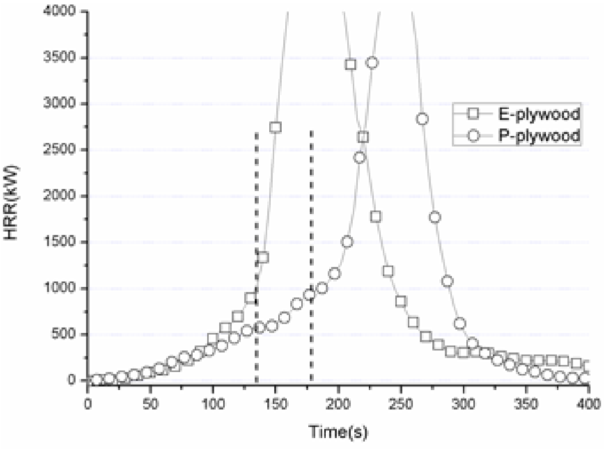

Figure 3 HRR of ISO 9705 tests for plywood

4. Conclusion

In this paper, several methods or models are used to evaluate the fire behaviour (flammability levels) of plywood, and the FO-category classification results are verified by room scale ISO tests. Among these methods, only Janssens' minimum heat flux method uses all the data from different incident heat flux tests. The minimum heat flux required for ignition obtained from ignition time could be more useful if the surface temperature of test material is also measured. The Petrella's arbitrary scale parameters method is simple, but the results are relatively difficult to compare with other results. Others are all based 50kW/m^2 cone test data, and the classification results could be compared with each other, and could be used in round robin exchange. When the amount of test material is limited resulting a shorter burning stage, Östman's empirical linear regression model and Hansen's MDA can also be effective with revised form of total heat release and corresponding exponent. How to use the data from the tests of different incident heat fluxes other than 50kW/m^2 sufficiently to predict the flammability of material should be taken into consideration to make the prediction more rational. Cone calorimeter data can be used to derive useful information on studying of fire behaviour, while room tests could not be ignored because some kinds of burning procedure are really different in bench and large scales.

Acknowledgements

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References

[1] Iqbal N, Salley MH, Weerakkody S. Fire dynamics tools (FDT^s). Wasinghton: U.S. Nuclear Regulatory Commission; 2004. 1016 p.

[2] Underwriters Laboratories of Canada. CAN/ULC-S114:2005. Standard method of test for determination of non-combustibility in building materials. Ottawa: ULC; 2005.

[3] Rybinski P, Janowska G, Helwig M, Dabrowski W, Majewski K. Flammablility of butadiene-acrylonitrile eubbers. *J Therm Anal Calorim* 2004;75(1):249-256.

[4] Martinka J, Hroncová E, Chrebet T, Balog K. A comparison of the behaviour of spruce wood and polyolefins during the test on the cone calorimeter. *Adv Mater Res* 2013;726-731(1):4280-4287.

[5] Chow WK, Han SS. Superposition of heat release rate curves for combustibles with bench-scale tests. *Polym Test* 2006;25(1):75-82.

[6] Grenier AT, Dembsey NA, Barnett JR. Fire characteristics of cored composite materials for marine use. *Fire Saf J* 1998;30(2):137-159.

[7] Petrella RV. The assessment of full-scale fire hazards from cone calorimeter data. *J Fire Sci* 1994;12(1):14-43.

[8] Janssens ML. Improved method of analysis for the LIFT apparatus. Part I: ignition. In: *Proceedings of Fire and Materials*; 1993. London: Interscience Communications; 1993. p. 37-46.

[9] Kokkala MA, Thomas PH, Karlsson B. Rate of heat release and ignitability indices for surface linings. *Fire Mater* 1993;17(5):209-216.

[10] Östman BAL, Tsantaridis LD. Correlation between cone calorimeter data and time to flashover in the room fire test. *Fire Mater* 1994;18(4):205-209.

[11] Hansen AH, Hovde PJ. Prediction of time to flashover in the ISO 9705 room corner test based on cone calorimeter test results. *Fire Mater* 2002;26(2):77-86.

[12] Xu Q, Griffin GJ, Burch I, Jiang Y, Preston C, Bicknel AD, Bradbury GP, White NJ. Predicting the time to flashover for GRP panels based on cone calorimeter test results. *J Therm Anal Calorim* 2008;91(3):759-762.

[13] DiNenno PJ. SFPE Handbook of fire protection engineering. 3rd ed. Quincy: National Fire Protection Association; 2002. 1606 p.

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Modification of procedure to initiate the solids according to EN 60695-2-10 for materials used in historic buildings

Andrea Majlingová[a] · Milan Oravec[b] · Marek Šolc[c] · Štefan Galla[d]

[a] Technical University in Zvolen, Faculty of Wood Sciences and Technology, T.G. Masaryka 24, 960 53 Zvolen, Slovakia
[b] Technical University of Košice, Faculty of Mechanical Engineering, Letná 9, 042 00 Košice, Slvoakia
[c] Technical University of Košice, Faculty of Metallurgy, Letná 9, 042 00 Košice, Slvoakia
[d] Fire Research Institute of the Ministry of Interior SR, Rožňavská 11, 831 04 Bratislava, Slvoakia

ABSTRACT

The effect of a reduced oxygen concentration during constant air pressure on the fire behaviour of the most used types of wood in the historical museum buildings, such as spruce, pine and larch and of materials used as insulation, such as cotton and linen is experimentally investigated in this paper. The experimental set-up is in accordance with STN EN 60695-2-10 and uses the exactly reducible heat source. Experimental tests in the area of 192 m^3 pointed out the correlation with measurements according to EN 60695-2-10, which allows measurements in small volumes ($0.5m^3$). After identifying the effect of different heating time on the fire behaviour of selected materials in atmospheric oxygen concentration, the effect of a reduced oxygen concentration on the relative mass loss and the ignition temperatures is defined. By experimental results was shown that oxygen concentration has a significant impact mainly on the duration of combustion and ignition temperatures. The dependency of the duration of combustion on the reciprocal value of oxygen concentration was shown by experimental tests. Oxygen concentration in the area of fire does not affect the rate of initiation of wooden materials. The burning rate of wood samples in the oxygen concentrations from 7 to 20 volume percentage is in small measuring device almost linear.

Keywords: Combustion · Oxygen concentration · Solid matters initiation · Historic buildings

1. Introduction

Application of heat to combustible materials leads to physical and chemical reactions. Burning of solids is complicated, redox process. During the burning, the solid material is pyrolytically decomposed and the degradation associated with weight loss and the formation of gas products occurs. Flammable gas products together with oxygen create the flammable set. Carbon enriched residues may then be oxidized in flameless combustion. A necessary condition for combustion is the presence of combustible materials, oxidizers, ignition sources [1-2].

As a result of fires in historic buildings, there are significant financial losses. During the fire in the Anna Amalia Library in the German city of Weimar in 2004, there burned 50,000 historical books, another 62 000 were damaged, some of them seriously. Property damage was about € 80 million.

Wood and textile fabric are the most common building materials applied in the interior of historic buildings. Therefore, the selection and dimensioning of fire extinguishers and their adaptation to the specific requirements for use, is important prerequisite. In addition to the water, there are also applied extinguishing agents based on inert gases in historic buildings. Major supplements of extinguishing systems are devices which reduce the ratio of oxygen and combustible gases in a protected area so that the danger of fires is substantially reduced.

The aim of this paper is to verify the modified procedure of solids initiation in the context of EN 60695-2-10:2001 [3] in a large scale test and application for historic buildings and museums.

From the experiments of various authors [4-13] have drawn the following conclusions:

- Vol% of oxygen in the fire area does not affect the rate of initiation,

Ing. Andrea Majlingová, PhD. (corresponding author)
Technical University in Zvolen, Faculty of Wood Sciences and Technology, Department of Fire Protection
T. G. Masaryka 24, 960 53 Zvolen, Slovakia, e-mail: majlingova@tuzvo.sk

- Time to initiation of organic polymer samples is linear in case of oxygen concentration from 13 to 20 vol% in experimental measuring devices with capacity up to 0.5 m^3,
- Parameters of the atmosphere affect the pyrolytic decomposition of wood decisively only when the formation of combustion gases is reduced.

In the paper, those statements are verified with respect to quantification of parameters for selected materials used in museums and historic buildings, regarding the modified procedure of initiation of solids according to EN 60695-2-10:2001 in large scale measuring device.

Within this objective were identified:

- Time to initiation of samples of spruce, pine, larch, cotton and linen at oxygen concentrations of 13 vol% to 20.9 vol%,
- Dependencies between the ignition temperatures of spruce, pine, larch, cotton and linen and the oxygen concentrations of 13 vol% to 20.9 vol%.

2. Material and methods

To determine the effect of oxygen concentration on the behaviour of solids in the fire, there was proposed a procedure to monitor and measure the continuous weight loss of test samples. The lower marginal value, the value of 13 vol% was set. This is a value, which affects humans and can result in their physical injury [14]. Reduction of oxygen was performed by the saturation of space with nitrogen.

The test programme consisted of a sequence of subsequent experiment steps:

1. *Creation of test samples* (spruce, pine, larch, cotton, linen),
2. *Drying* - the value of 12 vol% [15] corresponds with heated area in museums,
3. *Measurements of sample parameters* (weight, density, moisture),
4. *Experiment* - monitoring of dependencies (temperature, pressure, relative humidity, oxygen concentration) when exposed to heat loading:
    - Analytical scales - measuring the mass loss of samples at a particular oxygen vol%,
    - Thermocouple - determination of initiation temperatures at a particular vol% of oxygen,
5. *Evaluation:*
    - Evaluation of dependencies (temperature, pressure, relative humidity, vol% of oxygen) when exposed to heat loading,
    - Determination of dependency between the time to initiation and vol% of oxygen for particular samples,
    - Determination of dependency between the initiation temperature and vol% of oxygen.

2.1. Samples description

The test range was limited to ten samples of each type of wood (spruce, pine and larch) and textiles (cotton, linen). The most important material properties are briefly summarized in Tab. 1 and 2.

Table 1 Materials properties of investigated wood species

Sample	Spruce *Picea abies*	Pine *Pinu sylvestris*	Larch *Larix decidua*
Density ($kg.m^{-3}$)	450	500	550
Cellulose (%)	41	45	34
Polyose (%)	25	21	13
Lignin (%)	30	29	30

Table 2 Materials properties of investigated textiles

Sample	Cotton *Gossypium hirsutum*	Linen *Pinu sylvestris*
Density ($kg.m^{-3}$)	20 - 60	20 - 80
Cellulose (%)	91	71

2.2. Experiment description

The experiment was conducted in the experimental area shown in Fig. 1, with the deployment of particular sensors that are introduced in Tab. 3. For defined concentration of oxygen, in the interval from 13.0 to 20.9 vol%, there were monitored the dependencies of the weight loss of the sample ($mg.min^{-1}$).

The experiments were performed at a heating rate of 5 $K.min^{-1}$, 10 $K.min^{-1}$, 20 $K.min^{-1}$ and 50 $K.min^{-1}$. The proposed modified procedure according to EN 60695-2-10:2001 was constructed (modified) the source of ignition (Fig. 2).

The position of flameless ignition source, incandescent wire, in measuring device is defined in the standard [3]. This fact was decisive in process of selecting a heat source, because use of the heat sources with open flame cannot guarantee the reproducibility of experiment arrangement. Experimental assembly deployment [14] is shown in Fig. 3.

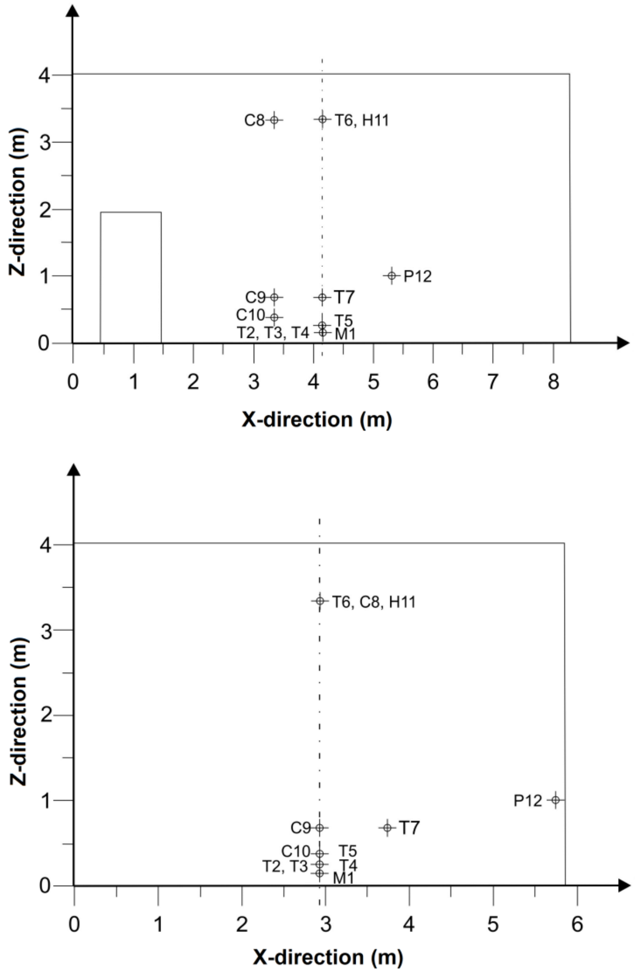

Figure 1 Experimental area with the deployment of measuring (sensors) positions

Table 3 Sensors position

Labelling	*x* (m)	*y* (m)	*z* (m)	Measured quantity
M1	4.15	2.93	0.15	Sample weight
T2	4.15	2.93	0.25	Initiation source temp.
T3	4.15	2.92	0.25	Sample temperature
T4	4.15	2.94	0.25	Sample temperature
T5	4.15	2.93	0.27	Sample temperature
T6	4.15	2.93	3.34	Ambient temperature
T7	4.15	3.73	0.68	Ambient temperature
C8	3.35	2.93	3.34	Vol% of oxygen
C9	3.35	2.93	0.68	Vol% of oxygen
C10	3.35	2.93	0.38	Vol% of oxygen
H11	4.15	2.93	3.34	Relative humidity
P12	5.30	5.75	1.0	Pressure

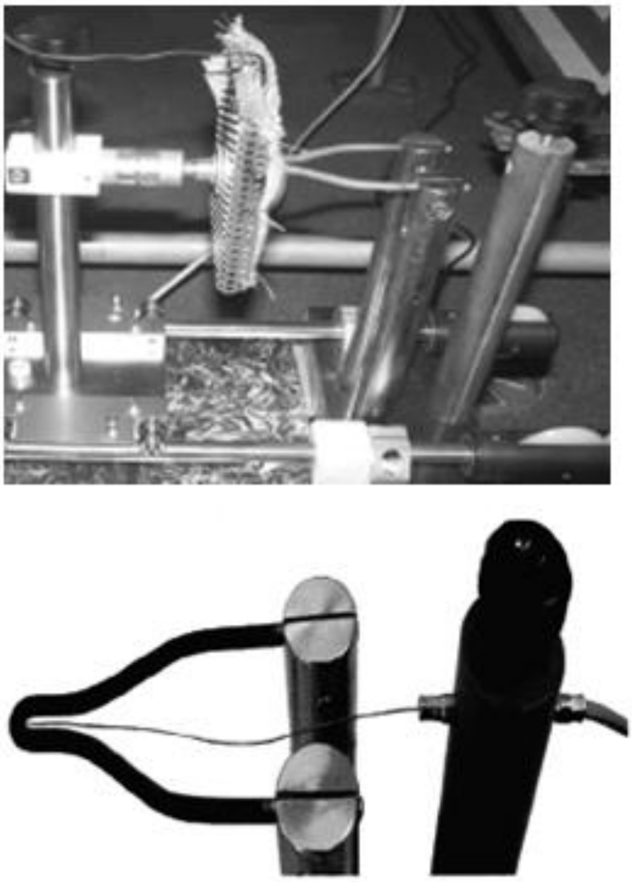

Figure 2 Incandescence source with measuring system

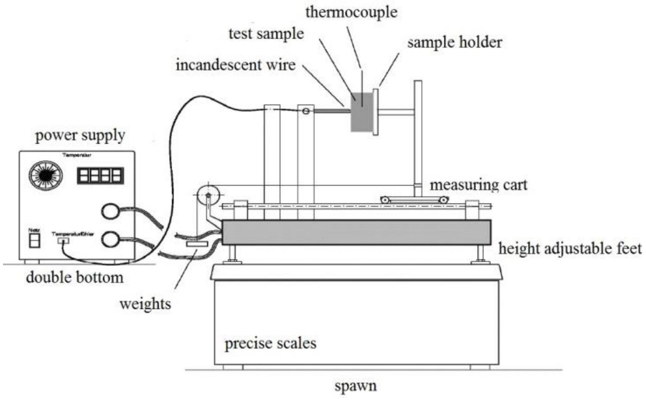

Figure 3 Experimental assembly deployment

3. Results and discussion

Course of weight loss of spruce test samples and weight loss time curve at a heating rate of 5 K.min^{-1} for each of the oxygen concentrations are shown in Fig. 4. A similar course was registered in case of the other samples (spruce, pine and larch), too.

Results in other experiments (pine, larch) showed that the effect of different concentrations of oxygen is manifested by an increase of overall time to initiation. Reducing the oxygen concentration from 20.9 vol% to 13.0 vol%, at heating rate of 5 K.min^{-1}, the time of burning is increased by 14 % in case of spruce, 17% in case of pine 17% and by 16% in case of larch.

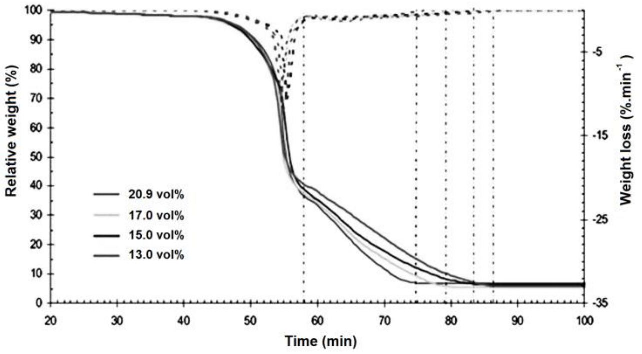

Figure 4 Dependence of the spruce sample weight loss on the time at various oxygen concentrations by the heating rate of 5 K.min^{-1}

Results in other experiments (pine, larch) showed that the effect of different concentrations of oxygen is manifested by an increase of overall time to initiation. Reducing the oxygen concentration from 20.9 vol% to 13.0 vol%, at heating rate of 5 K.min^{-1}, the time of burning is increased by 14 % in case of spruce, 17% in case of pine 17%, and by 16% in case of larch.

The initial phase of combustion was almost not affected by reducing the oxygen concentration. For samples of spruce the initiation phase was changed by 0.3% and phase of gas products formation by 0.5%. For samples of pine and larch, the drying phase was elongated by nearly 4%. With the reduction of oxygen from 20.9 vol% to 13.0 vol %, the burning time significantly increased, in case of spruce samples by 66%, pine samples by 67% and in case of larch samples by 69%.

Similarly, in experiments with textiles (cotton, linen), by reducing the oxygen concentration from 20.9 vol% to 13.0 vol %, the time to initiation of cotton and linen samples increased by about 3%. Similarly as in case of wood samples, the drying stage and phase of gas products formation, was almost not affected by reduction of oxygen concentration. With the reducion in oxygen concentration from 20.9 vol% to 13.0 vol%, the time to the initiation of cotton samples increased by 19% and linen samples by 10%.

With the reduction in oxygen concentration at a heating rate of 50 K.min^{-1} increased also the initiation temperature, for spruce by 19 %, for pine by 16 %, for larch by 14 %, cotton by 14 % and for linen by 15 %.

The experiments with the test samples showed:

- Oxygen concentration has a significant effect on time to initiation,
- Drying phase, phase of gas products formation and the residues after the burning were almost not affected in the process of oxygen reduction.

The evaluation of the experimental data with the wood samples showed that the time to ignition and oxygen concentration do not show a linear dependency (Fig. 5).

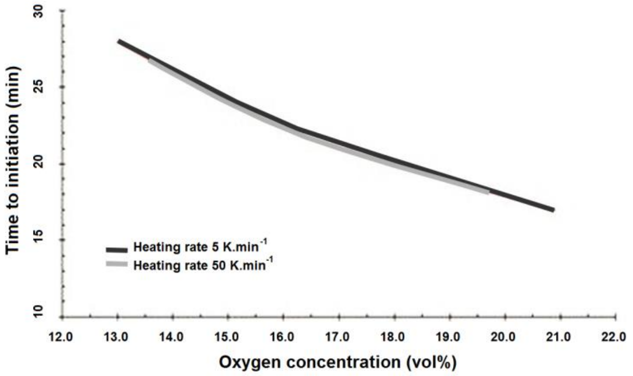

Figure 5 Dependence of the spruce wood time to initiation on the oxygen concentration

For experiments with the textile test samples was found that the oxygen concentration has a significant effect on the sample initiation. The evaluation of the data obtained from experiments showed the strong dependency between initiation temperature and oxygen concentration. From Fig. 6 is evident that the increasing initiation temperature and oxygen concentration reduction of wood samples show a linear dependency. This dependency is valid for all experimental samples and all defined heating rates, from 5 K.min^{-1} up to 50 K.min^{-1}.

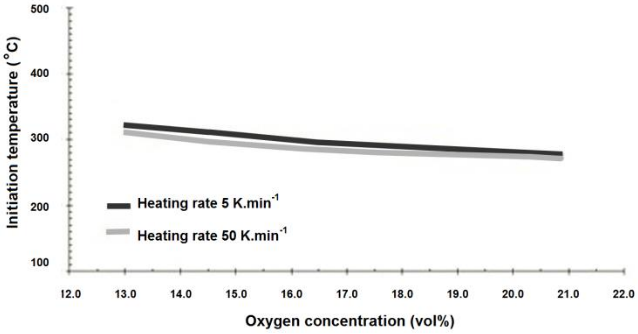

Figure 6 Dependence of the spruce wood initiation temperature on the oxygen concentration

The results of dependency analysis between oxygen concentration and initiation temperature of cotton samples are introduced in Fig. 7.

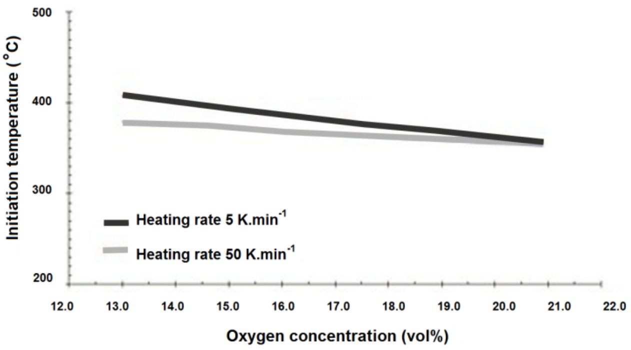

Figure 7 Dependence of the cotton initiation temperature on the of oxygen concentration

4. Conclusions

Experiments in small measuring area with capacity of 192 m^3, Fig. 1, pointed out the context of measurements according to STN EN 60695-2-10, which allows measurement of small volumes (up to 0.5 m^3). The larger the volume, the stronger nonlinearities occur in the range up to 17 vol% (Fig. 5). Prolonged is also the time to initiation. For objects of historic buildings is therefore appropriate to take these corrections into account in the design of fire protection systems. The proposed modified procedure using the specific device (Fig. 3), allows specify timeframes necessary to minimize losses in museums respectively historic buildings areas.

The statement that the oxygen volume percentage in the fire area does not affect the rate of initiation is true only for wood materials, not for textile fabrics. This argument correlates well with the results of the measurements [10-12, 16-17].

The statement, that the dependency between the time to initiation of wood samples at oxygen concentrations of 7 to 20 vol% is linear, is not accurate. It shows an almost linear dependency (Fig. 5).

The statement that the parameters of the burning atmosphere decisively affect the pyrolytic decomposition of wood only when the formation of combustion gases is reduced has been also shown in the measurements [10, 17].

Acknowledgements

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References

[1] Babrauskas V. *Ignition handbook*. Issaquah: Fire Science Publisher; 2003. 1116 p.

[2] Lees FP. *Loss prevention in the process industries*. 2nd ed. Vol. 2, Hazard identification assessment and control. Oxford: Elsevier; 1996. 1302 p.

[3] European Committee for Electrotechnical Standardization. EN 60695-2-10:2001. Fire hazard testing. Part 2-10: Glowing/hot-wire based test methods - Glow-wire apparatus and common test procedure. Brussels: CENELEC; 2001.

[4] Cote AE, editor. *Fire protection handbook*. 20th ed. Vol. 2, Fire protection handbook. Quincy: National Fire Protection Association; 2008. 1886 p.

[5] Jopp RK, Lehmann KD, Schweigler P. *Change of library technology during operation*. Munster: WWU Munster; 1989. 278 p.

[6] Lache M. *Investigation of burning rate and fire resistance of solid wood and glued beams*. Munich: LMU Munich; 1992. 120 p.

[7] White RH, Nordheim EV. Charing rate of wood for ASTM E 119 exposure. *Fire Technol* 1992;28(1):5-30.

[8] Patzak W. *Wood combustion theory*. Düsseldorf: VDI Verlag; 1972. 52 p.

[9] Kabat S. *Fire safety of historical buildings*. Berlin: Kohlhammer Verlag; 1996. 182 p.

[10] Martinka J, Kačíková D, Hroncová E, Ladomerský J. Experimental determination of the effect of temperature and oxygen concentration on the production of birch wood main fire emissions. *J Therm Anal Calorim* 2012;110(1):193-198.

[11] Martinka J, Balog K, Chrebet T, Hroncová E, Dibdiaková J. Effect of oxygen concentration and temperature on ignition time of polypropylene. *J Therm Anal Calorim* 2012;110(1):485-487.

[12] Tang C, Man X, Wei L, Pan L, Huang Z. Further study on the ignition delay times of propane–hydrogen–oxygen–argon mixtures: Effect of equivalence ratio. *Combust Flame* 2013;160(11):2283-2290.

[13] Fuentes A, Henríquez R, Nmira F, Liu F, Consalvi JL. Experimental and numerical study of the effects of the oxygen index on the radiation characteristics of laminar coflow diffusion flames. *Combust Flame* 2013;160(4):786-795.

[14] Sepp R, Balog K, Oravec M. *Oxygen reduction during museum fire*. Berlin: Equilibria; 2009. 138 p.

[15] Deutsches Institut für Normung. DIN 1052-1:1998. Methods of test for masonry. Part 1: Determination of compressive strength. Berlin: DIN; 1998.

[16] Tureková I. *Study of high-temperature degradation by lignocelulose materials*. Dresden: Forschungszentrum Dresden - Rossendorf; 2009. 130 p.

[17] Tureková I. *High-temperature degradation of wood based materials and determination od selected fire-technical characteristics*. Trnava: AlumniPress 2007. 123 p.

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European Science and Research Institute (Original Research Paper)

Evaluation of thermal oxidation of vegetable oils by the means of safety calorimeter SEDEX

Ivan Hrušovský[a] · Jozef Martinka[a] · Tomáš Chrebet[a]

[a]Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava, Paulínska 16, 917 24 Trnava, Slovakia

ABSTRACT

Evaluation of self-heating propensity of vegetable oils can be useful for determination of critical parameters of the system (porous material – vegetable oil), which must be fulfilled to achieve spontaneous ignition. However, the results strongly depend on the preparation of the sample and the type of method used for the evaluation of self-heating propensity of the oil. The safety calorimeter SEDEX was used to determine the selfheating propensity of three different vegetable oils tested under dynamic and adiabatic heating conditions. The calorimeter allows the use of large samples up to 15 ml, which was necessary to provide enough space for porous carrier which was wetted by oil. Three edible vegetable oils (linseed, sesame and sunflower oil) soaked in cotton were examined by the means of sequential DTA in the safety calorimeter SEDEX. First, the samples were heated from room temperature to 350°C, at heating rate 45 K/h, and then from room temperature to 200°C, at heating rate 10 K/h, to determinate the onset temperature of first exothermic reaction. Based on these results, the samples were investigated by the means of Accelerating Rate Calorimetry.

Keywords: Spontaneous ignition · Self-heating · Vegetable oils · Accelerating Rate Calorimetry · Fire investigation · Safety calorimeter SEDEX

1. Introduction

There are many different methods that serve for determination of the tendency of substances to self-heat or self-ignite. Mostly, when dealing with substances that have the tendency to self-heat, it is dealt with pure solids or pure liquids. Another option is the porous solid material soaked with liquid. Many drying oils used as a wood coating and eatable vegetable oils have the tendency to self-heat due to an exothermic oxidation. When they are spread on sufficiently large surface and suitable conditions are present, then the heat generated by the oxidative self-heating can be greater than the heat transferred to the surrounding and a self-ignition can occur [1-5].

Vegetable oils and fats are triesters derived from glycerol, originating from the condensation of a glycerol molecule with three molecules of fatty acid, where the fatty acids contain long chains of carbon (8–24 units long) [6-8]. Vegetable oils containing unsaturated fatty acids such as oleic, linoleic and linolenic acids are easily susceptible to auto-oxidation, which may lead to auto-ignition. The amount of unsaturated bonds in vegetable oils is characterized by iodine value. Unfortunately, it has been erroneously used to estimate the susceptibility of an oil to undergo self-heating based on the wrong premise that the higher the iodine value, the more likely the oil is to oxidize [9-10]. Because the iodine value does not take into account the position of the double bonds on the fatty acids, it may not be directly correlated to the self-heating capability of the oil [11]. Moreover, the iodine value does not take into account the presence of any additives [10]. Therefore the best way to determine the propensity of oil to self-heating is experimental, under elevated temperatures under isothermal, dynamical or adiabatical conditions. The most common, and probably the oldest method is the Mackey test.

The Mackey test consists of taking a given amount of the questioned oil and mixing it with a combustible material, such as cotton gauze, in order to create a quasi-isolated medium. The material is then heated, and the temperature at the core is measured. Based on the temperature reached within a given timeframe, the material’s self-heating propensity is qualified. This test is neither practical nor does it

Ing. Ivan Hrušovský, PhD. (corresponding author)
Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava
Paulínska 16, 917 24 Trnava, Slovakia, e-mail: ivan.hrusovsky@stuba.sk

allow for a detailed study of the kinetics of the different phenomena of the auto-oxidation reaction [10].

More modern thermo-analytical techniques are often used to study the kinetics of endothermic and exothermic reactions. Among them are differential scanning calorimetry DSC, differential thermal analysis (DTA), and thermogravimetry (TG). These have all been applied to the analysis of vegetable oils, mostly from a food chemistry perspective [10].

2. Material and methods

In this presented paper, safety calorimeter SEDEX from SYSTAG Switzerland was used for thermal analysis of samples. The SEDEX Safety calorimeter was developed for determination of risk parameters of materials stressed under lower temperatures (from room temperature to up to 400 °C). Different measurement cells and different control systems allow many combinations, which can be used particularly for routine operations, but can also be used for scientific purposes in research. Functions such as DTA (but sequential; comparison test is first measured as calibration run), ARC, DPSC, all with or without pressure measurement are possible under different background conditions and with different test quantities [12]. The standard pressure vessel (Fig. 1) is suitable for experiments with samples of larger volumes, which an advantage in the case of investigation of porous materials soaked with drying oils.

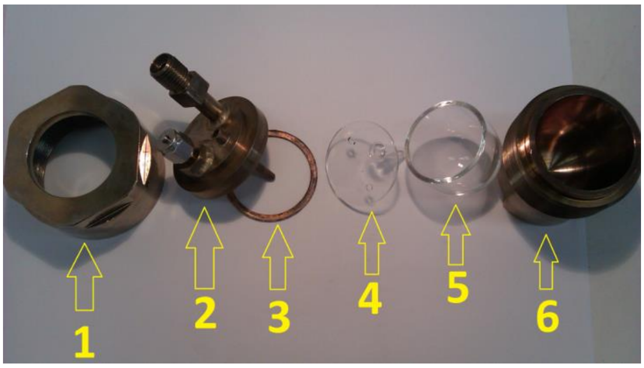

Figure 1 SEDEX stainless steel vessel assembly 1: Top part 2: Lid 3: Copper o-ring 4: Glass liner top 5: Glass liner bottom 6: Bottom part

Three vegetable oils were chosen for analysis. The specification of these oils can be seen in table (Tab. 1) as stated by manufacturer.

Shredded 100% Cotton was used as a porous carrier for the vegetable oils. Total amount of 0.5 g of cotton was put in to the glass liner of the stainless steel pressure vessel, and 1g of the oil vas dropped in the middle of the porous carries to form a small spot (Fig. 2). The whole vessel was assembled and put to the oven of the safety calorimeter SEDEX. Under normal conditions, the vessel is pressure tight closed and allows the study of pressure rise during experiment. For this set of experiment we left the pressure valve opened to assure air supply for the oxidation of vegetable oil.

Table 1 Specification of investigated vegetable oils

Parameter	Value
Name	Linseed oil
Manufacturer	Rapunzel Germany
Saturated fatty acids (%)	9.7
Simple unsaturated fatty acids (%)	20.9
Polyunsaturated fatty acids (%)	69.4
Linoleic acid (%)	16.1
Linolenic acid (%)	53.3
Iodine value (-)	165-204
Name	Sesame oil (Sesamol)
Manufacturer	Rapunzel Germany
Saturated fatty acids (%)	14
Simple unsaturated fatty acids (%)	42
Polyunsaturated fatty acids (%)	44
Linoleic acid (%)	43
Iodine value (-)	175-195
Name	Sunflower oil (Gaya)
Manufacturer	BušlakOil Slovakia
Saturated fatty acids (%)	7.49
Simple unsaturated fatty acids (%)	82.07
Polyunsaturated fatty acids (%)	10.2
Iodine value (-)	113-145

3. Results and discussion

The samples were first heated from room temperature up to 350 °C at a rate 45 K/h. After requiring the basic characterization of the auto-oxidation exothermic processes, the samples were heated at much slower rate 10 K/h to gain a better resolution. The maximal temperature was 200 °C, since we were interested in the onset temperatures of the first exothermic events. The samples were investigated by the means of Accelerating rate Calorimetry, after the determination of the onset temperatures for the first significant exothermic event. The heating step was set to 5 K and the hold time to 2 hours.

The first scanning at heating rate 45 K/h showed (Fig. 2), that the firs exothermic event for Linseed oil showed up at approximate temperature 100 °C and the maximal temperature difference between the sample and the oven temperature was 1,48 K. After that the first exothermic events of sesame and sunflower oils occurred approximately at temperature 140 °C, with peak temperatures 1.26 K for sesame oil and 1.38 K for sunflower oil. After the peaks, the temperature differences slowly decreased and start to increase again at 300 °C.

The second scanning at heating rate 10 K/h revealed several small exothermic events (Fig. 3) just before the significant events that occurred during the first scanning. The thermogram shows, that the first small and insignificant exothermic reaction occurs already approximately at 30 °C for the Linseed oil and Sesame oil, and approximately at 70 °C for the sunflower oil. However these events were considered insignificant for the purpose of Accelerating Rate Calorimetry, but they can play an initiation role in the process of self-heating of the oils.

The results from accelerated rate calorimetry (Fig. 4) show, that the oil with lowest onset temperature was linseed oil. This was also shown by the first two scanning experiments. The onset temperature for the linseed oil was approximately 100 °C. The second onset was at approximately at 114 °C for sesame oil, and the least at approximately 119 °C foe sunflower oil. The peak temperatures under adiabatic conditions corresponded with the onset temperatures of the oils with approximately 132 °C for linseed oil, 155 °C for sesame oil and 203 °C for sunflower oil.

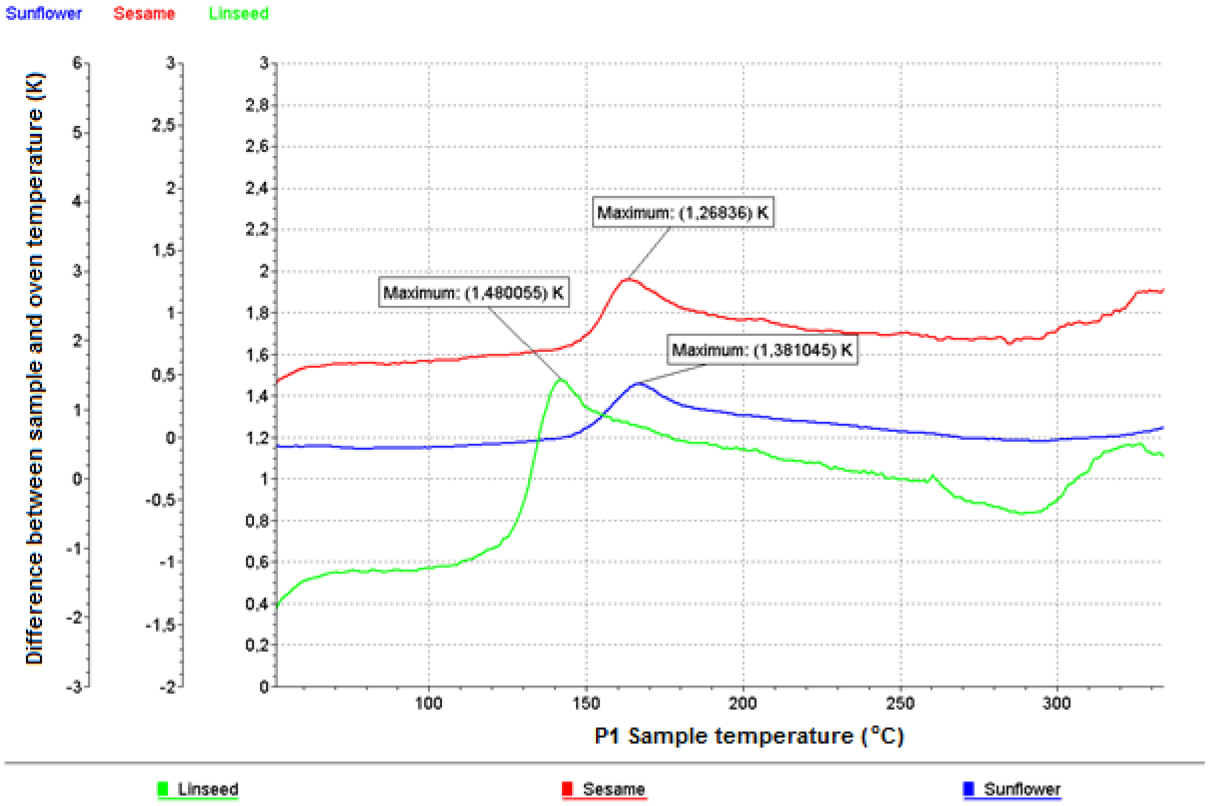

Figure 2 Sequential DTA results of vegetable oils dynamically heated at heating rate 45 K/h

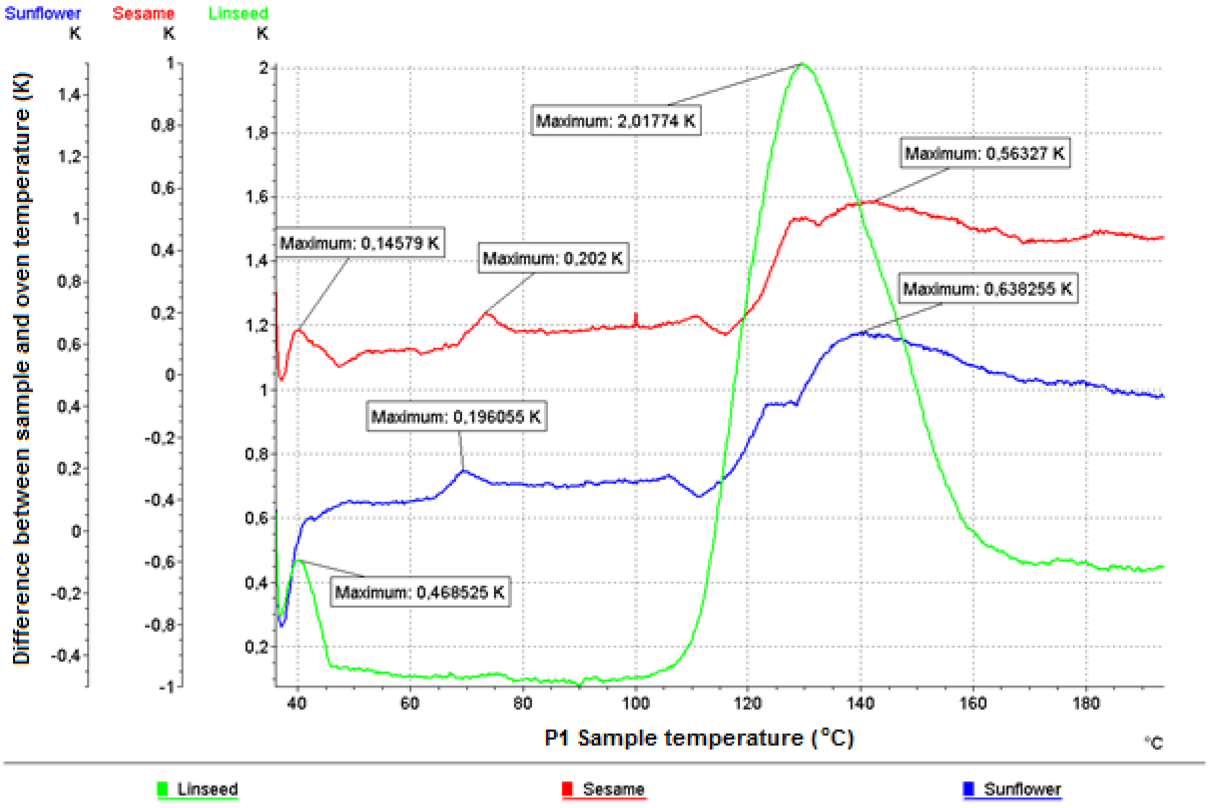

Figure 3 Sequential DTA results of vegetable oils dynamically heated at heating rate 10 K/h

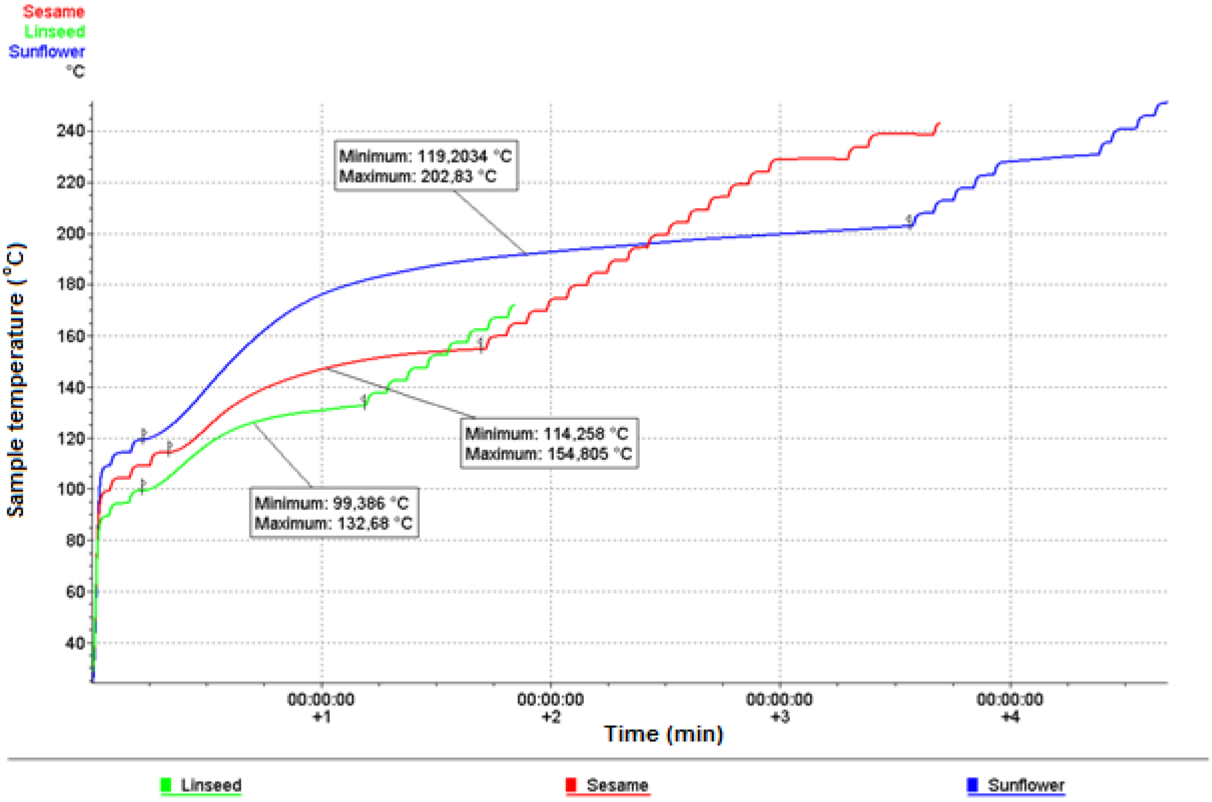

Figure 4 Results of sample temperatures investigated by the means of Accelerating Rate Calorimetry

4. Conclusion

The results of experiments confirmed that the level of propensity of vegetable oils to self-heating strongly depends on the amount of unsaturated acids in vegetable oils. The higher the content, lower the onset temperature of the initial exothermic reaction. However, even oils with lower content of unsaturated acids can result in auto-ignition, under right circumstances. As the results of accelerated rate calorimetry showed, even thou sunflower oil contain low amount of unsaturated oils, the maximal temperature reached under accelerated rate calorimetry was much higher than that of a linseed oil. This means, that the reaction was slower, but reached its maximal rate at much more higher temperature than linseed oil. There were also recorded another two small accelerations of the reaction for sesame oil approximately at 230 °C and at 240 °C, and one smaller acceleration for sunflower oil approximately at 230 °C.

Acknowledgements

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References

[1] Hrušovský I, Balog K, Martinka J, Chrebet T. Self-heating of porous substances soaked with unsaturated oils. In: Osvald A, editor. *Proceedings of the 7th International Scientific Conference on Wood and Fire Safety*; 2012 May 13 – 16; High Tatras Štrbské Pleso, Slovakia. Ostrava: Šmíra – print; 2012. p. 73-78.

[2] Blijderveen M, Bramer EA, Brem G. Modelling spontaneous ignition of wood, char and RDF in a lab-scale packed bed. *Fuel* 2013;108(1):190-196.

[3] Juita, Dlugogorski BZ, Kennedy EM, Mackie JC. Roles of peroxides and unsaturation in spontaneous heating of linseed oil. *Fire Saf J* 2013;61(1):108-115.

[4] Misra RD, Murthy MS. Straight vegetable oils usage in a compression ignition engine – A review. *Renew Sustain Energy Rew* 2010;14(9):3005-3013.

[5] Medeiros EJL, Queiroga RCRE, Souza AG, Cordeiro AMTM, Medeiros AN, Souza DL, Madruga MS. Thermal and quality evaluation of vegetable oils used in ruminant feed. *J Therm Anal Calorim* 2013;112(3):1515-1521.

[6] Conceicao MM, Fernandes VJ, Bezerra AF, Silva MCD, Santos IMG, Silva FC, Souza AG. Dynamic kinetic calculation of castor oil biodiesel. *J Therm Anal Calorim* 2007;87(3):865-869.

[7] Vecchio S, Campanella L, Nuccilli A, Tomassetti A. Kinetic study of thermal breakdown of triglycerides contained in extra-virgin olive oil. *J Therm Anal Calorim* 2008;91(1):51-56.

[8] Pardauil JJR, Souza LKC, Molfetta FA, Zamian JR, Filho GNR, Costa CEF. Determination of the oxidative stability by DSC of vegetable oils from the Amazonian area. *Bioresour Technol* 2011;102(10):5873-5877.

[9] Abraham CJ. A solution to spontaneous combustion in linseed oil formulations. *Polym Degrad Stab* 1996;54(2-3):157-166.

[10] Baylon A, Stauffer E, Delmont O. Evaluation of the self-heating tendency of vegetable oils by differential scanning calorimetry. *J Forensic Sci* 2008;53(6):1334-1343.

[11] Stauffer E. A review of the analysis of vegetable oil residues from fire debris samples: spontaneous ignition, vegetable oils, and the forensic approach. *J Forensic Sci* 2005;50(5):1091-1110.

[12] Meier P. *Technical vade mecum for thermoanalysis*. Bern: Systag; 1998. 36 p.

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European Science and Research Institute (Original Research Paper)

On equivalent fire exposure

Vladimír Mózer[a]

[a]*University of Žilina, Faculty of Special Engineering, Ul. 1. mája 32, 010 26 Žilina, Slovakia*

ABSTRACT

The ability to accurately determine fire resistance requirements is one of the most important aspects of fire safety science. Since fire resistance depends on fire severity, which is a rather ambiguous term, there have been continuous efforts to develop a method for quantifying the term fire severity itself as well as converting it into fire resistance requirements. This paper evaluates two approaches – equivalent temperature and normalised heat load methods, by employing computer fire simulation. It is shown that the latter is a more appropriate quantifier, as it does not rely on an arbitrarily selected reference point and allows for a comparison in an unlike enclosures.

Keywords: Fire severity · Fire resistance · Equivalent fire exposure · Enclosure thermal properties · Heat load

1. Introduction

Fire resistance is the basic property of any fire resisting construction. As straightforward as this requirement may seem, there is ambiguity in the way of translating real-world fire severity into a fire-resistance requirement. Even the term *fire severity* itself is somewhat ambiguous; is it the temperature history of a fire, its overall heat output, or the heat absorbed by the enclosure boundaries?

At the moment, standardised fire-resistance tests, such as those referenced in [1], are used to determine the fire resistance of a construction product or building element. These tests methods simulate a fire by following one of standardized time-temperature curves. Such an approach controls the temperature within the test furnace and allows for comparison under reference conditions.

On the other hand, there are real-world fires in buildings, the construction elements of which must withstand the exposure. These fires vary greatly with the nature and layout of the fuel and geometry and construction materials of the building. Therefore, the open question that needs close attention is: *"Which real-world fire severity descriptor should be selected and how to translate it into the standardised test conditions?"*

2. Fire resistance and standardised fire resistance testing

Fire resistance is the ability of a construction member to withstand fire exposure for a given time period, maintaining the required fire-resistance criteria. These criteria are most often load-bearing ability, integrity and insulation, but there are other criteria available.

In order to be able to undertake such testing, there are reference time-temperature curves available, which describe the development of a fire. Most often the standard time-temperature curve is employed in fire resistance testing, the development of which follows the equation (1) [1-2]:

$$T = \log_{10}(8t + 1) + 20 \qquad (1)$$

where T is average furnace temperature (°C) and t is time (min).

The actual development of the temperature during a 60 fire exposure is shown in Fig. 1.

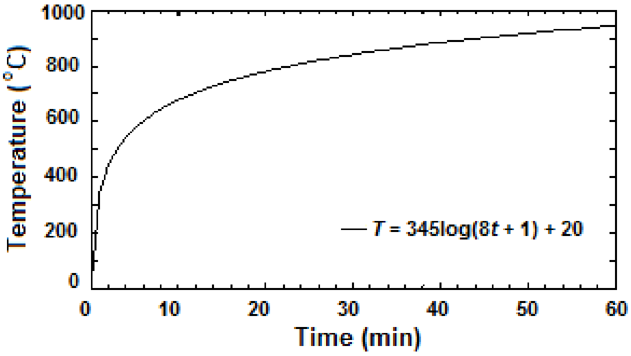

Figure 1 Standard time-temperature curve

From the above, it is obvious that, when testing to this, but also other (C-H, exterior fire, tunnel fire, etc) standard-

Ing. Vladimír Mózer, PhD. (corresponding author)
University of Žilina, Faculty of Special Engineering, Department of Fire Engineering
Ul. 1. mája 32, 010 26 Žilina, Slovakia, e-mail: vladimir.mozer@fsi.uniza.sk

ised time-temperature curves, the furnace temperature conditions change only with time, following the course of the selected curve.

The advantage of this system is that fire resistance is determined on a reference basis and it is therefore possible to compare tested construction members with each other. However, the disadvantage of this approach is that the development and intensity of heat loading in real fires is not identical with the standardised time-temperature curves. Hence, there is a need for a method which would allow conversion of the intensity or development of a fire in a building to the intensity or development of the fire achieved by a standardised time-temperature curve, to specify sufficient resistance levels of the construction elements for a given fire.

3. Fire severity

As mentioned earlier, fire severity is not a standardised term with fixed meaning. It is, however, generally accepted that is should represent the detrimental effect of a fire on the construction element(s) in question. There are a number of methods which, utilizing various input parameters, convert the calculated intensity of a fire, expressed by various means, to the equal duration of exposure in a test furnace under one of the standardised time-temperature conditions. Some of these methods are evaluated in this paper.

3.1. Equality of time-temperature areas

The earliest concept, proposed by Ingberg [3], bases the equivalence of fire severity on the equality of the areas under the time-temperature curves of the real fire and test furnace fire. The proposed equation (2) for determining the equivalent fire duration was:

$$\tau_\mathrm{e} = 0.0205 \cdot L \qquad (2)$$

where τ_e is equivalent fire duration (time-temperature curve) (hours) and L is fire load (kg.m^{-2}). This approach is completely insensitive to the effect of ventilation and the thermal properties of enclosing construction.

Czechoslovakian standard CSN 73 0802 [4] utilized a similar approach adjusted for the nature of fuel and effect of ventilation.

3.2. Equality of temperature in reference points

The second group of methods involves the equality of temperatures achieved in selected parts or depths of a construction element. The basis of this approach is that two fires (real and test) are of an equal severity if the temperature achieved in the selected reference point is identical. A number of methods working on this principle have been developed including those of Law [5], Petterson [6], DIN [7] and Reichel [8-9].

The calculation methods employed for determining the critical temperature vary, but all of them are based on the equation of energy balance. Although of they provide a higher level of accuracy, a great care must be taken when selecting the point of reference, as it cannot be generalised. For reinforced concrete members it is usually the temperature at the reinforcing steel, however, the selection of a reference point for masonry walls may not be as straightforward.

3.3. Normalized heat concept

The basis of the normalized heat concept, developed by Harmathy [10-11], is that the severity of the fire can be expressed as the overall heat penetrating into the enclosure boundaries with the normalization being done through their thermal properties. This way the fire severities of fires in unlike enclosures may be compared. The mathematical equation (3) of normalised heat load is:

$$H = \frac{1}{(\lambda \cdot \rho \cdot c)^{1/2}} \cdot \int_0^\tau q \, \mathrm{d}t \qquad (2)$$

where H is normalised heat load (s$^{1/2}$.K), $(\lambda.\rho.c)^{1/2}$ is thermal absorptivity and τ is time duration of exposure (s).

4. Computer modelling evaluation

In order to evaluate the capabilities and suitability of the methods described in Section 3 of this paper a number of computer simulations were carried out. The results from the simulations are then compared, utilizing both concepts – equal temperature and normalized heat load.

Fire Dynamics Simulator version 6 [12-13] was selected for the simulation. It is a freely available software package by NIST that allows simulation of combustion, heat generation and its transfer in an enclosure. Fire Dynamics Simulator is a suitable tool for both the prescribed time-temperature and free-burning fire (heat generation) scenarios.

4.1. Computer model scenarios

4.1.1. Computer model scenarios

There were three alternative material configuration with different thermal properties simulated. These included – mineral wool as thermal insulator, normal weight concrete as thermal conductor and a layered combination of the above. In the layered alternative the mineral wool was on the exposed side followed by concrete. The thermal properties of the materials are described in Tab. 1.

4.1.1. Simulated scenarios

There were two types of scenarios simulated – test furnace and room fire. In the test furnace simulation, a reduced scale test furnace was created. In both cases the simulation time was 3600s (one hour).

Grid resolution was set to 10 cm in each direction, which provides a good compromise between accuracy and computational times. The thickness of the simulated walls was 20 cm. In the case of the layered configuration the first 10 cm represented mineral wool and second 10 cm concrete.

The furnace was simulated as a cubic enclosure with a side of 1 m in each direction. Five of the walls were specified as heaters, in order to heat the interior of the enclosure as per the standard time-temperature curve. The sixth wall represented the test specimen. The layout of the test furnace model is shown in Fig. 2.

The room fire scenario (Fig. 3) involved a cubic room with a side of 2 m in each direction. Each wall (20 cm thick) was prescribed the same boundary condition, corresponding with the material alternative modelled. The computational domain was extended 1m from the front wall to allow free air circulation into the room. There was a single opening, 1 x 1m (1m^2) in the front wall providing access for fresh air. Given the size of the enclosure, this approximately equals to a ventilation parameter of 0.04 which is the same as that of the standardised time-temperature curve [8]. Therefore the ventilation conditions should not cause deviation from the test furnace results.

The fire was located across the entire floor prescribing it a uniform fixed burning rate of 0.025 kg.m^{-2}, which corresponds to a heat release rate of 471.5 kW.m^{-2} at the heat of combustion of 16.7 MJ.kg^{-1}. The burning rate is an average of values calculated by various methods [8, 15-16] and is a function of the ventilation parameter. It is the maximum burning rate achievable under the ventilation conditions. The fuel used in the model was wood. Research [17] confirms a strong dependence of burning rate on the radiant flux within the compartment which is reflected by the value used in the simulation. Further discussion on thermal degradation of wood under fire conditions may be found in [18].

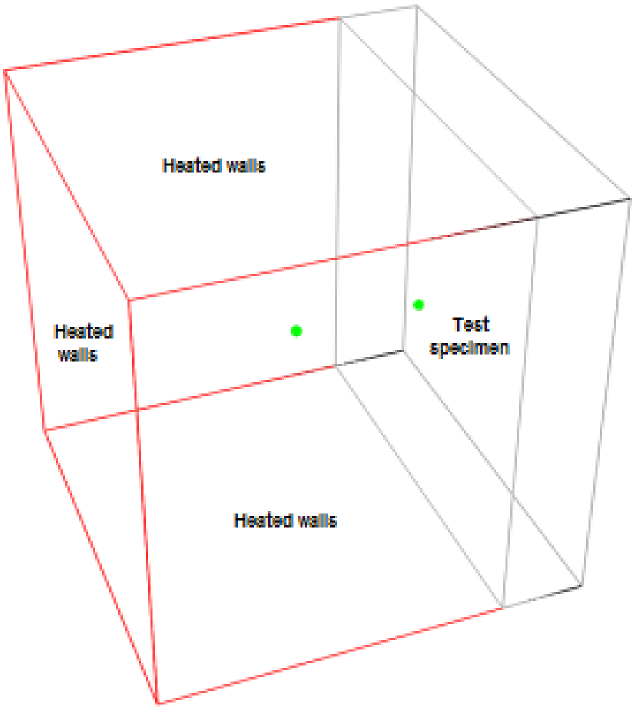

Figure 2 Wireframe visualisation of test furnace model

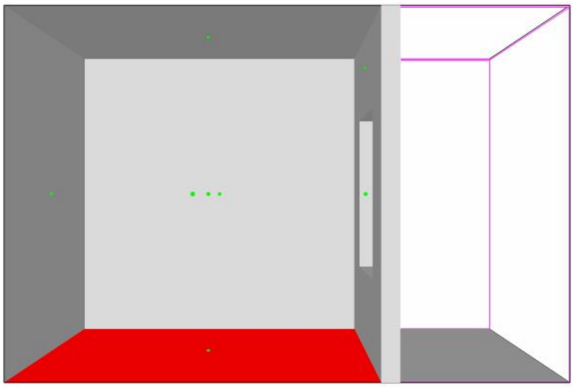

Figure 3 Visualisation of room fire model

Table 1 Thermal properties of construction materials used in model

Material	ρ (kg.m^{-3})	c (J.g^{-1}.K^{-1})	λ (J.m^{-1}.s^{-1}.K^{-1})	$(\lambda\, \rho\, c)^{1/2}$ (J.m^{-2}.s$^{-1/2}$.K^{-1})
Normal weight concrete	2400	837	1.67	1832
Mineral wool	60	880	0.072	62

5. Results and discussion

The results of the simulations were compared in order to evaluate the equivalent exposure using both the equal temperature and normalized heat load methods. The development of temperature in the test furnace was identical to the standard time-temperature curve shown in Fig. 2, regardless of the material simulated. On the contrary, in the room fire simulations, the development of the temperature showed a strong dependence on the thermal properties of the enclosing construction, refer to Fig 4. The scenarios with mineral wool lining reached much higher average temperatures inside the enclosure compared with concrete due to the amount of heat absorbed by the walls.

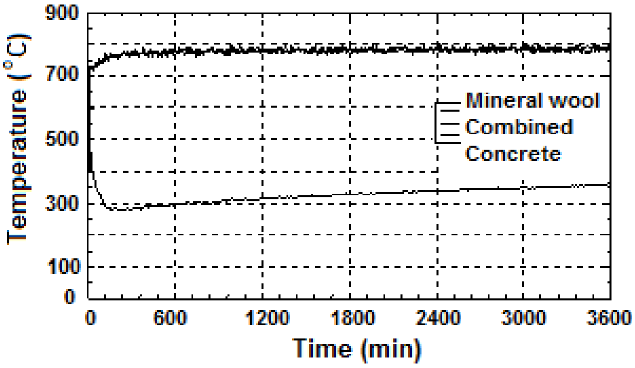

Figure 4 Development of temperatures in room fire simulations

For the evaluation of equal temperatures a number of reference points were monitored in the construction. These were as follows: exposed surface, within construction at 1 cm, 2 cm, 5 cm, 10 cm, 15 cm and rear surface. Fig. 5 - 7 show the temperatures attained in the reference points after 60 minutes of exposure for both scenarios – furnace and room fire.

It is apparent that there are differences in the maximum temperatures reached in the individual reference points, due to the different temperature profiles in the furnace and room scenarios. What is also very obvious, when comparing Fig 5 to Figs. 6 and 7, is the fact that the difference is considerably smaller for scenarios in which the walls were lined (formed) by mineral wool which is an insulator. The differences between the furnace and room fire scenarios also decrease with the increasing depth of the reference points.

The above confirms the importance of the correct selection of the equal-temperature reference point. For materials with higher thermal absobtivity $(\lambda\ \rho\ c)^{1/2}$, represented by normal weight concrete in this paper, the differences are significant. This is confirmed by the values shown in Fig. 8.

The time required to reach an equal temperature in the furnace decreases the closer is the reference point to the exposed side of the wall. For the concrete wall, it takes only 10 minutes to reach the same temperature in the furnace as is reached after 60 minutes in room fire scenario when the reference point is located 1cm beneath the exposed surface. When the reference point is located 5 cm beneath the exposed surfaces the time increases to 23 min and keeps increasing with the depth.

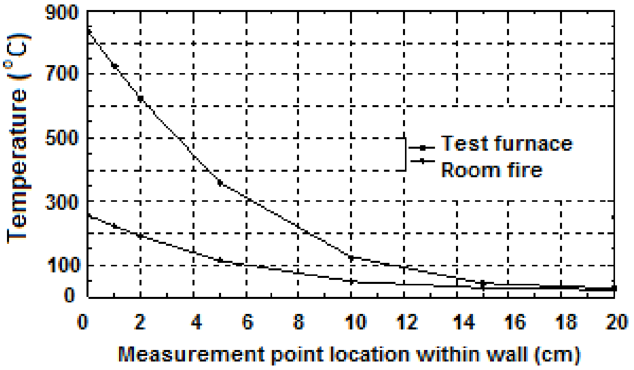

Figure 5 Temperatures achieved in construction after 3600s exposure – concrete

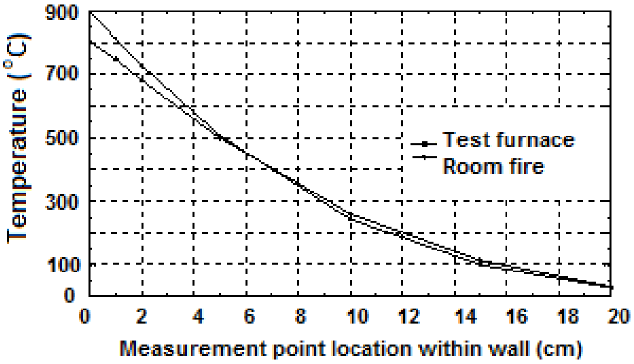

Figure 6 Temperatures achieved in construction after 3600s exposure – mineral wool

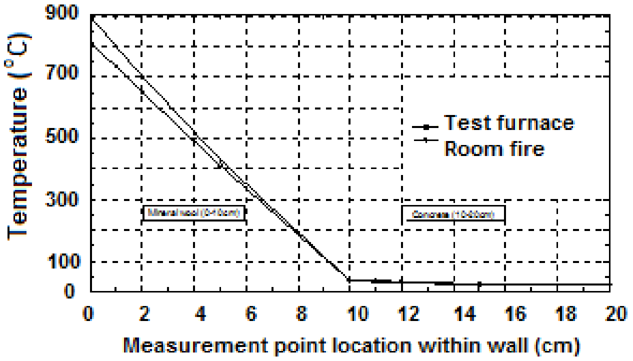

Figure 7 Temperatures achieved in construction after 3600s exposure – combined

A similar trend is observed in the scenario where mineral wool is used as lining material. There is also a difference between the furnace and room-fire scenarios in the individual temperature reference points, but it decreases sharply with increasing depth.

To provide a comparison with the equal-temperature method, the normalised heat load method results are also included. To determine the overall heat flux penetrating into the construction in question, trapezoidal integration of the individual data points in time was employed. The obtained values per square meter of wall surface area are listed in Tab. 2 together with the times required to achieve the same heat load in the furnace.

Again, the times required to reach the same heat load exposure on the concrete specimen is significantly shorter, that those of the insulating linings.

When the two approaches are compared it is obvious that for the non-insulating material with high thermal absorbtivity – normal weight concrete – the equal temperature method seems to yield similar results to the normalized heat load method in reference points closer to the exposed surface. On the other hand, the simulations where walls were lined with an insulator – mineral wool – seem to correspond at greater depths.

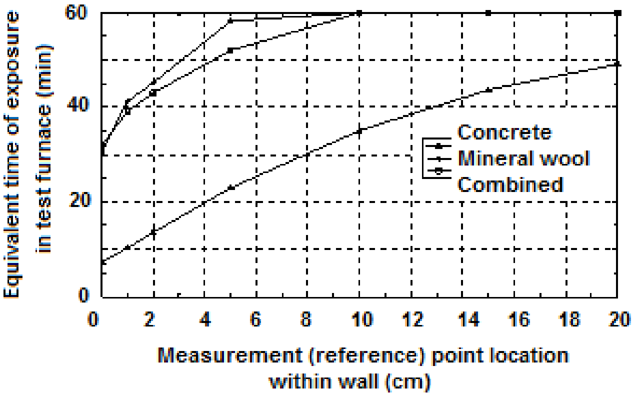

Figure 8 Test furnace equivalent exposure times – 60min room fire

Table 2 Heat loads and normalized heat loads construction evaluation after 60min exposure

Material	Heat load		Normalized heat load		Equivalent time
	Room fire	Furnace	Room fire	Furnace	
	(kW.m^{-2})	(kW.m^{-2})	(s$^{1/2}$.K)	(s$^{1/2}$.K)	(min)
Normal weight concrete	24963	83378	13.6	45.5	18.1
Mineral wool	3282	3315	53.2	53.77	58.4
Combined	3396	3402	55.1	55.18	58.2

6. Conclusion

A series of computer simulations were carried out in order to evaluate two different methods for determining equivalent fire exposure – equal temperature and normalized heat load concepts.

The results have confirmed that it is not possible to base the fire severity solely on the heat output of a fire. Even if ventilation does not change, as it was shown in the room fire simulations, the temperatures within the enclosure vary significantly, depending on the thermal properties of the boundary, which is in agreement a previous study [19].

The other issue highlighted in this paper is the importance of the selection of the equal-temperature reference point. For example, 2 cm beneath the exposed surface may be a crucial depth for prestressed concrete members, however, will not provide any relevant comparison for a brick wall. Furthermore, the differences decrease with the increasing depth of the construction.

In contrast to the equal temperature approach, the normalized heat method does not rely on a specific reference point and allows for comparison of unlike enclosures. This is achieved through the adjustment of the absorbed heat by the thermal properties of the boundary (thermal absorptivity).

Even a comparison of two very simple scenarios – thermal conductor vs thermal insulator – shows that the question of equivalent fire exposure is a complex issue. Given the above results, the amount of heat which a construction member is to withstand in a fire, appears to be a more suitable quantity for conversion into fire resistance requirements.

Acknowledgements

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References

[1] European Committee for Standardization. EN 13501-2:2007. Fire classification of construction products and building elements. Part 2: Classification using data from fire resistance tests, excluding ventilation services. Brussels: CEN; 2007.

[2] European Committee for Standardization. EN 1363-1:2012. Fire resistance tests. Part 1: General requirements. Brussels: CEN; 2012.

[3] Ingberg SH. Tests of the severity of building fires. *NFPA Q* 1928;22(1):43-61.

[4] Federal Office for Standardization and Metrology. CSN 730802:1975. Fire protection of buildings: common regulations. Praha: FUNM; 1975.

[5] Law M. *A relationship between fire grading and building design and contents*. Watford: BRE Trust; 1971. 47 p.

[6] Pettersson O. *The connection between a real fire exposure and the heating conditions to standard fire resistance tests – with special application to steel structures*. Brussels: European Convention for Constructional Steelwork; 1983. 24 p.

[7] German Institute for Standardization. DIN 18320:2012. Structural fire protection in industrial buildings. Part 1: Analytically required fire resistance time. Berlin: DIN; 2012.

[8] Reichel V. *Determination of buildings structures fire resistance*. Praha: Main Fire Protection Office; 1981. 196 p.

[9] Federal Office for Standardization and Metrology. CSN 730804:1991. Fire protection of buildings: industrial buildings. Praha: FUNM; 1991.

[10] Harmathy TZ. The possibility of characterizing the severity of fires by a single parameter. *Fire Mater* 1980;4(2):71-76.

[11] Harmathy TZ. The fire resistance test and its relation to real-world fires. *Fire Mater* 1981;5(3):112-122.

[12] Floyd J, Forney G, Hostikka S, Korhonen T, McDermott R, McGrattan K. *Fire dynamics simulator: user's guide*. Maryland: NIST; 2012. 250 p.

[13] McGrattan K, Hostikka S, Floyd J, Baum H, Rehm R. *Fire Dynamics Simulator: technical reference guide*. Maryland: NIST; 2007. 86 p.

[14] Slovak Office of Standards, Metrology and Testing. STN 730540-3:2012. Thermal protection of buildings: thermal performance of buildings and components. Part 3: Properties of environments and building products. Bratislava: SUTN; 2012.

[15] Drysdale D. *An introduction to fire dynamics*. 2nd ed. Chichester: Wiley; 1999. 451 p.

[16] DiNenno. *SFPE handbook of fire protection engineering*. 3rd ed. Maryland: SFPE 2002. 1606 p.

[17] Martinka J, Hroncová E, Chrebet T, Balog K. A comparison of the behaviour of spruce wood and polyolefins during the test on the cone calorimeter. *Adv Mater Res* 2013;726-731(1):4280-4287.

[18] Martinka J, Hroncová E, Chrebet T, Balog K. Fire risk assessment of thermally modified spruce wood. *Acta Facultatis Xylologiae* 2013;55(2):117-128.

[19] Mózer V. Comparison of selected calculation methods for determination of fire resistance requirements. In: Šenovský M, editor. Fire Safety. 22nd International Scientific Conference on Fire Safety; 2013 4 – 5 Sept; Ostrava, Czech Republic. Ostrava: VŠB TU Ostrava; 2013. p. 169-173.

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Assessment of the impact of heat flux density on the combustion efficiency and fire hazard of spruce pellets

Jozef Martinka[a] · Tomáš Chrebet[a] · Ivan Hrušovský[a] · Karol Balog[a]

[a]*Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava, Paulínska 16, 917 24 Trnava, Slovakia*

ABSTRACT

The presented paper analysed the impact of the heat flux density on the combustion efficiency and fire hazard of spruce pellets. Combustion efficiency and fire hazard were assessed on the base of heat release rate (HRR), carbon monoxide yield (CO), and flash-ignition and spontaneous-ignition temperatures. The experiment was performed using hot-pressed pellets with no added chemicals produced from the wood of Norway spruce (*Picea abies* L.) without bark. The density of pellets at 6 % water content was 665 kg.m^{-3}. HRR and CO yield were measured in the cone calorimeter at three heat flux densities (20, 30 and 40) kW.m^{-2} according to ISO 5660-1:2002. A sample used for one experimental trial weighed 150 ± 0.1 g. The obtained results showed that maximum HRR increased with the increasing density of the external heat flux. Maximum HRR (315 kW.m^{-2}) was determined for heat flux of 40 kW.m^{-2}. CO yield per mass loss (maximum 36.2 g.kg^{-1} obtained for heat flux of 20 kW.m^{-2}) decreased with the increasing heat flux density. CO yield per released heat reached its maximum for the heat flux of 20 kW.m^{-2} (3.42 g.MJ^{-1}) and minimum for the heat flux of 30 kW.m^{-2} (2.18 g.MJ^{-1}). From the measured CO concentrations, the concentrations of polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) in combustion gases were derived using the published statistical relationships. The obtained results proved that the maximum concentration of PAHs and PCBs in combustion gases decreases with the increasing heat flux density. The results also proved that during the combustion of spruce pellets, PAHs and PCBs are produced in the amount that is from the point of the actual toxicity insignificant. Ignition temperature (410 °C) and combustion temperature (460 °C) were determined according to ISO 871:2006.

Keywords: Spruce pellets · Combustion process · Carbon monoxide yield · Polycyclic aromatic hydrocarbons · Polychlorinated biphenyls · Fire hazard

1. Introduction

Recently, energy demands have become increasingly covered by the energy produced from renewable sources. The main reasons for such a shift are attempts to reduce the dependency on fossil fuels and to stabilise the amount of carbon dioxide in the atmosphere. Wood is one of the most important renewable sources. For the economic reasons, energy is mainly obtained from wood residues or wood that is not suitable for construction and carpentry. To use disintegrated wood residues for energy purposes, the manufacturing process called pelletizing is frequently used. Pellets represent a densified form of wood material produced from the disintegrated wood waste (commonly from sawdust and wood shavings). Pellets are in the shape of cylinders with a diameter of 6 mm and a length of several tens of millimetres. They are produced by compression under the simultaneous influence of the increased temperature. Various chemical substances can be added into sawdust and wood shavings to increase the quality of the final product.

In spite of the fact that the production of wood pellets started already in the 20s of the last century, their utilisation in a greater extent has occurred only in the last years. According to Filbakk et al. [1], pellets have several advantages compared to unrefined fuels such as wood chips, including considerably higher density, lower moisture content, more homogeneity, higher transport efficiency and better handling properties. The parameters and attributes that characterise wood pellets are shape and size, volumetric and bulk density, hardness and abrasion resistance, moisture content, calorific value and ash fusibility [2-4].

Ing. Jozef Martinka, PhD. (corresponding author)
Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava
Paulínska 16, 917 24 Trnava, Slovak Republic, e-mail: jozef.martinka@stuba.sk

These properties are affected by the quality of the input material, as well as by the production method and technology [4].

A great number of scientific papers [1-6] analysed how the attributes of the input material and the production technology affect pellet parameters. Jandacka et al. [4] examined the effect of the amount of the concentrate of chemical compounds from defibration on the selected attributes of wood pellets. The presented results prove that the mechanical durability and density of wood pellets can be increased and their ash content can be reduced if chemical compounds from defibration are added. Filbakk et al. [1] analysed how the storage and the drying temperature of Scots pine raw materials affect mechanical properties of pellets. Their results suggest that the pellets from fresh materials dried at low temperature are more durable and have lower density than the pellets from fresh materials dried at higher temperature. Pellets produced from stored materials have higher density.

Similarly, a number of papers [7-17] examined the impact of physical and chemical properties of pellets and combustion conditions (temperature and air access to combustion zone) on the combustion process in wood pellet furnaces. However, only a few papers have dealt with the exact assessment of the effect of heat flux density on the combustion process of pellets. Fire hazard of pellets was also addressed in a very small number of works. Fire hazard of materials can be best examined on the base of the heat release rate (HRR) [18] and the yield of carbon monoxide (CO) [19]. Although a number of studies determined the amount of released heat and CO yield from the pellet combustion in wood pellet furnaces, the published information is not suitable for an exact assessment of maximum combustion efficiency. The reason for this is that combustion conditions in wood pellet furnaces are deliberately set to achieve the maximum combustion efficiency. However, the conditions under the real fire are considerably different. The complexity of the comparison of combustion process under the conditions of industrial combustion and fire conditions is also apparent from the results of Ladomersky et al., and Martinka et al. [20-24]. Moreover, the data obtained from measuring the released heat and selected combustion gases under the conditions of industrial combustion do not allow exact assessment of the impact of heat flux density on the combustion process. The information about the influence of heat flux density on HRR, total heat release (THR) and CO yield are important not only for assessing fire hazard and the impact of wood pellet combustion on environment, but also for the future development of wood pellet furnaces.

The presented paper aims to perform an exact assessment of the impact of external heat flux density in the interval from 20 to 40 kW.m^{-2} on HRR, THR and CO yield (as a toxicologically most important component of combustion of wood and wood based materials) from spruce pellets. The paper also aims to compare the relative fire hazard of spruce wood in the form of solid wood and pellets using the data gathered within this study and the data published elsewhere. Fire hazard will be assessed not only on the base of HRR, THR and CO yield, but also on the base of flash-ignition and spontaneous-ignition temperatures.

2. Material and methods

The experiment was performed using the pellets produced from Norway spruce (*Picea abies* L.) wood without bark by hot compression without the use of the additional chemicals. The properties of the examined pellets determined according to EN 14961-1:2010 [25] are presented in Table 1. Prior to the experiment, the samples were conditioned at 6 % moisture.

HRR, THR and CO yield were determined in the cone calorimeter according to ISO 5660-1:2002 [26]. A sample for one experimental trial weighed 150 ± 0.1 g. The samples were bulk distributed and loosely aligned in a sample holder of the cone calorimeter normally used for testing solid materials in a horizontal position. The area of the sample was 100 x 100 mm. The sample area directly exposed to the external heat flux from the cone heater was 88.4 cm^2, which is in accordance with the cited standard. The samples were analysed at three densities of the external heat flux (20, 30 and 40) kW.m^{-2}. The trial time was 3600 s. The experiment was repeated five times for each density of the external heat flux, from which average values were calculated and are presented below.

Fire hazard was assessed not only on the base of HRR, THR and CO yield, but also on the base of ignition and combustion temperatures determined on the experimental apparatus according to ISO 871:2006 [27].

Table 1 Basic characteristics of the examined pellets according to EN 14961-1:2010

Physical and chemical properties	
Diameter (mm)	6
Length (mm)	10 - 40
Moisture (%)	6
Mechanical durability (%)	99.4
Fines F1.0 (%)	0.3
Bulk density (kg.m^{-3})	665
Net calorific value (MJ.kg^{-1})	18.6
Ash (%)	0.19

3. Results and discussion

Fig. 1 presents HRR and specific mass loss rate (SMLR) of the examined samples at three heat flux densities (20, 30 and 40) kW.m^{-2}. The first peak of HRR and SMLR was recorded in the ignition phase, and the second peak was observed in the phase of flaming combustion of gaseous degradation products and flameless combustion (incandescence) of carbon layer. The comparison of the obtained data with the published studies [28-30] showed that the pattern of HRR and SMLR of spruce pellets is similar to solid wood materials. The temporal performance of HRR and SMLR of pellets differed from solid wood materials in the shape (peakedness) of the first and the second peak. Spruce pellets showed more moderate decrease after the first peak, corresponding to ignition. This was caused by the greater surface area of the bulk pellet samples when compared with the solid wood samples. The second, even more significant difference between the pellets and the solid wood was that the pellets reached the second peak of HRR and SMLR faster. This can also be explained by the greater surface area of bulk pellets, from which the release of degradation products is faster. Secondly, the surface of bulk pellets is not homogeneous due to which a compact charred layer cannot be created. This state slows down the overheating of pellets by the heat flux from the cone heater as well as from the reverse heat flow from the flame. The impact of the charred layer on the combustion process of wood and wood materials is thoroughly described in Majlingova et al. [31]. Another significant difference was revealed by comparing the gathered data with the results published elsewhere [28-30]. The cited papers prove that compact coniferous wood reaches its maximum HRR and SMLR in the first peak, while pellets reached their maximum HRR in the second peak. This can again be explained by the greater surface area of the pellets. The comparison of the obtained results with the published values [28-30, 32-33] revealed that the behaviour of spruce pellets during the experiment in the cone calorimeter is more similar to the behaviour of broadleaved wood than coniferous wood.

Kamikawa et al. [34] determined HRR of wood pellets placed in the sample holder only in one layer. The comparison of their results with our data showed that the temporal performance of HRR strongly depends on the way how pellets are placed on the sample holder. The performance of the pellets distributed in one layer showed only one major peak. However, bulk distribution of pellets in the sample holder resembles the real storage of pellets better. Thus, the measured values of HRR and SMLR of bulk pellets are of higher informative value for assessing the fire hazard and the impact of combustion process on environment.

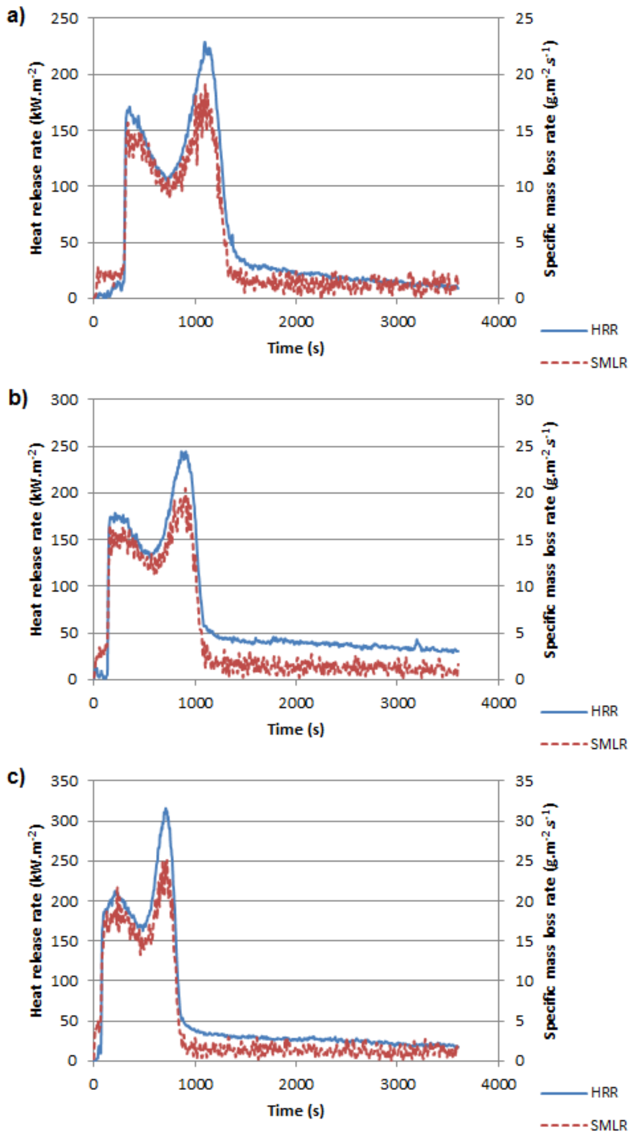

Figure 1 Temporal performance of heat release rate and specific mass loss rate of spruce pellets loaded with the heat flux of density a) 20 kW.m^{-2}; b) 30 kW.m^{-2} and c) 40 kW.m^{-2}

Visual analysis of Fig. 1 indicated high combustion efficiency of the examined samples under the experimental conditions (highly significant dependency of HRR from SMLR). To prove this hypothesis the graphs of the linear statistical relationship between HRR and SMLR (Fig. 2) has been created. The coefficient of determination R^2 described the goodness of fit (tightness) of the relationships. In the case of perfect combustion, the coefficient of determination is equal to 1, i.e. HRR depends only on SMLR. This statement is valid only for chemically pure liquids (e.g. methanol or ethanol). For wood materials, this statement is valid only partially, since the calorific value of the degradation products changes in time. Moreover, it is common that during the combustion of wood materials, both the flaming combustion of gaseous degradation products and the flameless combustion of the charred layer occur simultaneously, while the gaseous degradation products have a lower calorific value than the charred layer.

A relatively low value of the coefficient of determination of the statistical relationship between HRR and SMLR at 30 kW.m^{-2} heat flux density was caused by several extreme values in the ignition phase (SMLR from 3 to 5 g.m^{-2}.s^{-1}, while HRR values were almost zero). Therefore, it is not possible to make a final conclusion about the combustion efficiency under these conditions.

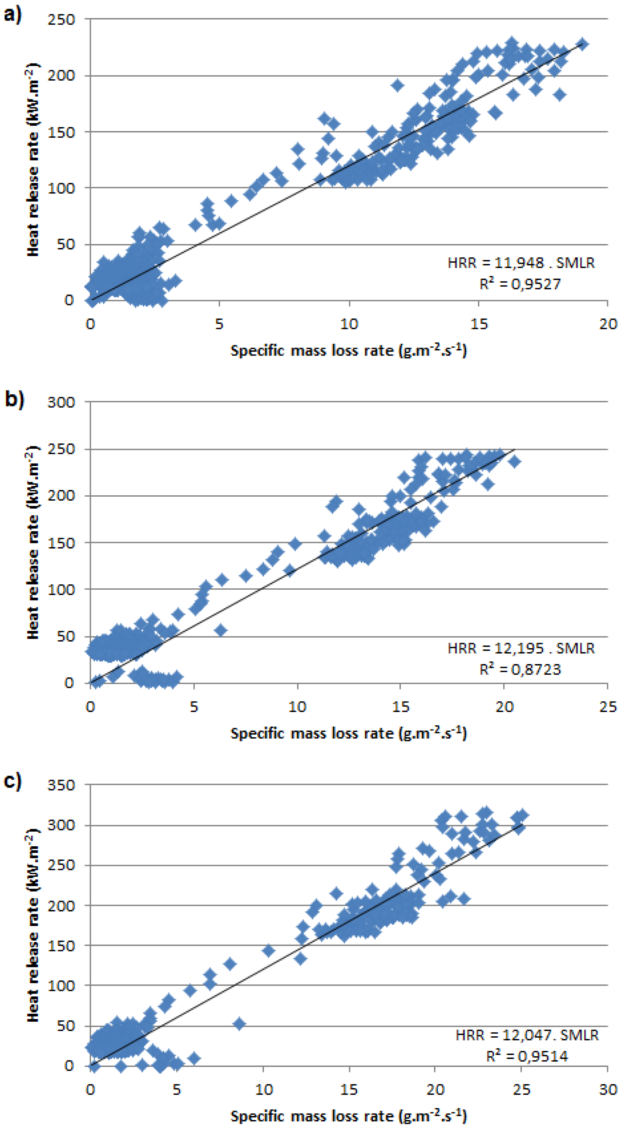

Figure 2 Statistical relationship between heat release rate and specific mass loss rate of the sample loaded with the heat flux of density a) 20 kW.m^{-2}; b) 30 kW.m^{-2} and c) 40 kW.m^{-2}

The influence of heat flux density on the combustion efficiency was exactly assessed on the base of CO yield per sample mass loss (Fig. 3) and released heat (Fig. 4).

Data shown in Fig. 3 and 4 proved that the combustion efficiency of spruce pellets increased with the increasing heat flux density. The only exception is the CO yield per released heat at heat flux densities of 30 and 40 kW.m^{-2}. However, CO yield at 40 kW.m^{-2} heat flux density is only slightly higher than CO yield at density of 30 kW.m^{-2} (by less than 14 %). The given results can be explained by the fact that the heat flux with 40 kW.m^{-2} density caused faster degradation of the analysed samples than the heat flux with 30 kW.m^{-2} density, which resulted in the increase of the fuel – air equivalence ratio (i.e. the coefficient of the air excess was reduced). This caused that the oxidation of the degraded products up to CO_2 decreased, due to which the total heat release was reduced and CO yield (per released heat) increased. For the comparison, Martinka et al. [35] present that CO yield per sample mass loss of the solid spruce wood, which was loaded with the external heat flux from the interval between 20 and 40 kW.m^{-2}, is from 28.3 g.kg^{-1} to 33.2 g.kg^{-1} (it decreases with the increasing density of the external heat flux). Hence, during the experiment in the cone calorimeter CO yield (per mass loss) of spruce pellets was 10 % higher than of solid spruce wood. This is due to the greater surface area of bulk pellets in the sample holder than the area of the solid wood, which allowed faster thermal degradation of samples (faster release of degradation products) resulting in higher fuel (degradation products) - air (air oxygen) equivalence ratio.

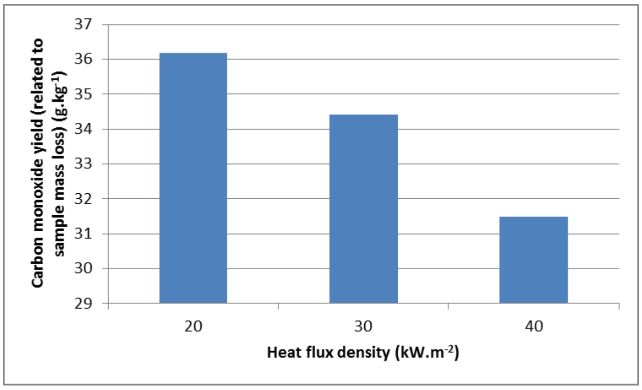

Figure 3 CO yield from spruce pellets (per sample mass loss) determined for the samples loaded with the heat flux of density a) 20 kW.m^{-2}; b) 30 kW.m^{-2} and c) 40 kW.m^{-2}

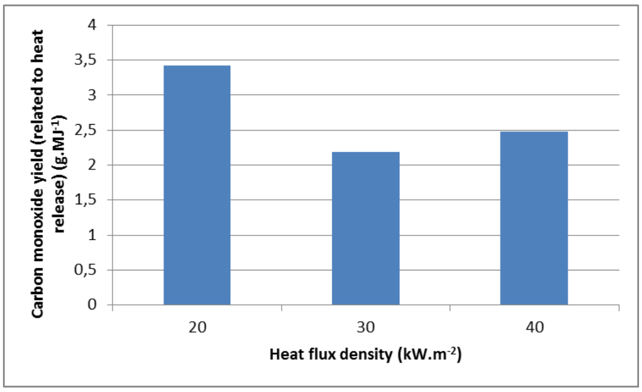

Figure 4 CO yield from spruce pellets (per released heat) determined for the samples loaded with the heat flux of density a) 20 kW.m^{-2}; b) 30 kW.m^{-2} and c) 40 kW.m^{-2}

The results of Atkins et al. [36] prove close statistical relationship of the concentrations of polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) to the concentration of CO in combustion gases from biomass pellets. The authors derived equations (1) and (2) from which PAHs and PCBs concentrations can be calculated, respectively. The respective coefficients of determination R^2 are equal to 0.72 and 0.82. The temporal performance of PAHs and PCBs concentrations calculated from the measured CO concentrations using equations (1 and 2) is presented in Fig. 5. Although the values of PAHs and PCBs concentrations given in Fig. 5 are only of indicative nature, they are very similar to the measured values in [36]. Considering the calculated concentrations of PAHs and PCBs in combustion gases we can state that under fire conditions of spruce pellets, the listed compounds will not be toxicologically significant. This conclusion is in accordance with the results of the scientific study [37]. The results presented in Fig. 5 also indicate that the maximum concentration of PAHs and PCBs in combustion gases of spruce pellets decreases with the increasing density of the external heat flux.

$$\mathrm{PAHs} = 0.096 \cdot c_{\mathrm{CO}} + 33 \tag{1}$$

$$\mathrm{PCBs} = 0.0024 \cdot c_{\mathrm{CO}} + 0.22 \tag{2}$$

Where PAHs is the concentration of polycyclic aromatic hydrocarbons (μg.m^{-3}), PCBs is the concentration of polychlorinated biphenyls (μg.m^{-3}) and c_{CO} is the concentration of CO (mg.m^{-3}) in combustion gases.

Thermal resistance of spruce pellets was assessed on the base of the temporal development of the sample mass at examined heat flux densities (Fig. 6). The obtained data suggested that heat flux density caused significant increase of the mass loss rate of spruce pellets, particularly in the phase of flaming combustion. In the phase of flameless combustion (glowing of carbon residues), the influence of heat flux on the mass loss rate of spruce pellets decreased. This is caused by a relatively low value of the coefficient of thermal conductivity of the charred layer (low coefficient of thermal conductivity prevents rapid overheating of material by the external heat flux).

The flash-ignition and spontaneous-ignition temperatures of spruce pellets were 410 °C and 460 °C, respectively. Zachar et al. [38] present 370 °C and 400 °C as flash-ignition and spontaneous-ignition temperatures for solid spruce wood. The difference between the pellets and the solid spruce wood was caused by higher density of pellets, due to which pellets were more resistant to ignition. The increasing resistance of wood and wood materials to the ignition with the increasing density is in accordance with the results of Kacikova et al. [39].

Table 2 presents the comparison of the basic parameters describing the fire hazard of the material from the point of heat development during fire (maximum HRR, average HRR – during the first thirty minutes of experiment, and THR during the first thirty minutes of experiment) determined for the analysed spruce pellets with the results presented in [35] for the solid spruce wood. Higher values of maximum HRR, average HRR and THR of pellets result from the greater surface area of pellets bulk distributed in the sample holder.

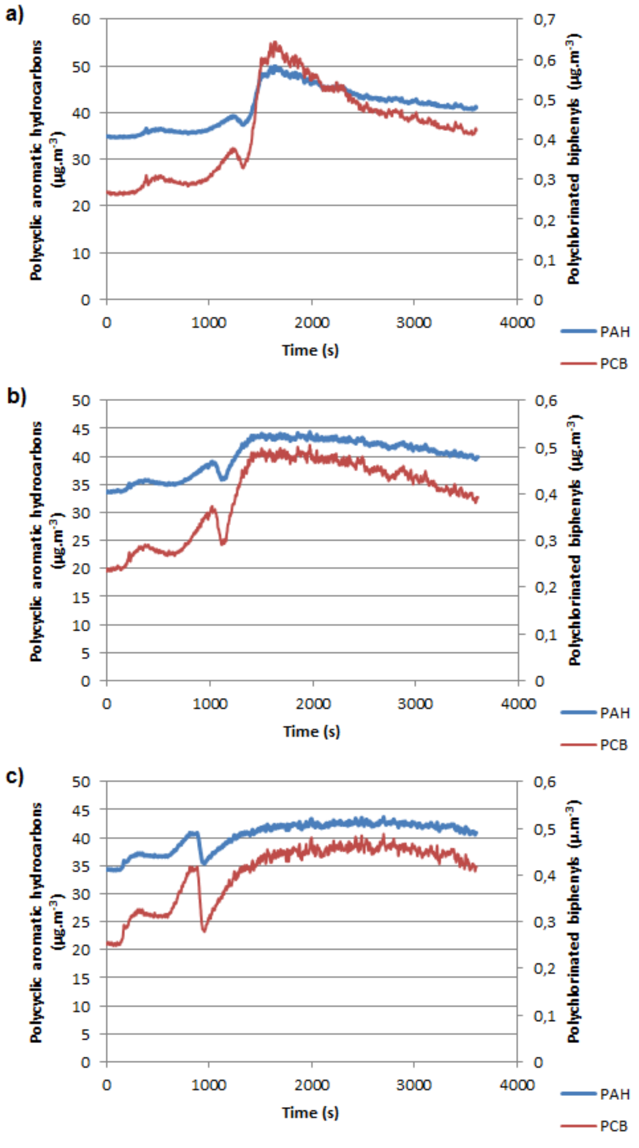

Figure 5 Concentration of polycyclic aromatic hydrocarbons and polychlorinated biphenyls in combustion gases from spruce pellets loaded with the heat flux of density a) 20 kW.m^{-2}; b) 30 kW.m^{-2} and c) 40 kW.m^{-2} (values calculated from CO concentrations)

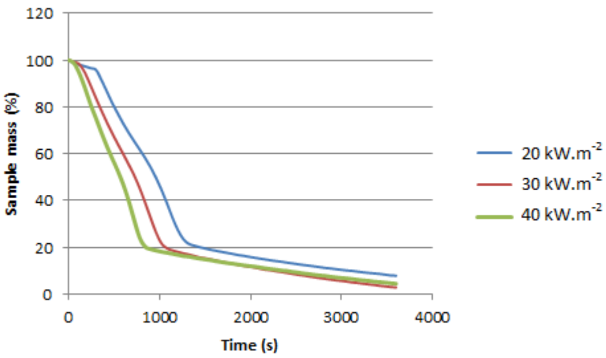

Figure 6 Temporal development of the relative sample mass of spruce pellets loaded with the external heat flux a) 20 kW.m^{-2}; b) 30 kW.m^{-2} and c) 40 kW.m^{-2}

Table 2 Maximum and average HRR and THR during the first 30 minutes of experiment in the cone calorimeter of spruce pellets and solid spruce wood under loadings of heat flux (20, 30 and 40) kW.m^{-2}

Heat flux / Parameters	Pellets	Solid wood
20 kW.m^{-2}		
Maximum HRR (kW.m^{-2})	229	222
Average HRR (kW.m^{-2})	94	51
THR (MJ)	169	93
30 kW.m^{-2}		
Maximum HRR (kW.m^{-2})	244	176
Average HRR (kW.m^{-2})	106	62
THR (MJ)	191	111
40 kW.m^{-2}		
Maximum HRR (kW.m^{-2})	315	254
Average HRR (kW.m^{-2})	106	67
THR (MJ)	191	120

3. Conclusion

The presented paper examined the impact of heat flux density on the combustion efficiency and the fire hazard of spruce pellets. Combustion efficiency was assessed on the base of CO yield (per mass loss and released heat). Fire hazard was assessed by comparing maximum and average HRR and THR of pellets and solid wood. The resistance to ignition was assessed on the base of ignition and combustion temperatures.

The obtained results showed that CO yield (per mass loss) of spruce pellets decreased with the increasing heat flux density. CO yield from spruce pellets was by approximately 10 % higher than from solid spruce wood (in the interval of heat flux densities from 20 to 40 kW.m^{-2}). In the given interval, spruce pellets had also higher values of average and maximum HRR and THR during the first 30 minutes of the experiment in the cone calorimeter. Hence, considering the toxicity of combustion gases and the values of HRR and THR, spruce pellets represent a greater fire hazard. The increase of maximum and average HRR and THR is sufficient to have a significant influence on the dynamics of fire development. From the point of the resistance to ignition, pellets represent a lower fire hazard.

The obtained results show that the material form has a significant influence on its fire hazard (its behaviour during fire), and partially contradict the generally accepted opinion that the behaviour of the wood with higher density during fire is more favourable. The results can be efficiently applied not only for assessing the fire risk of pellets using fire engineering tools (thoroughly described in Osvald and Mozer [40] and Mozer [41]), but also for examining the impact of the industrial combustion of spruce pellets on environment.

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References

[1] Filbakk T, Skjevrak G, Høibø O, Dibdiakova J, Jirjis R. The influence of storage and drying methods for Scots pine raw material on mechanical pellet properties and production parameters. *Fuel Process Technolog* 2011;92(5):871-878.

[2] Dzurenda L, Slovák J. Energy properties of wood pellets. *Acta Mech Slovaca* 2001;5(3):201-206.

[3] Jandačka J, Papučík S, Nosek R, Holubčík M, Kapjor A. *Environmental and energetic aspects of biomass combustion*. Žilina: Georg; 2011. 137 p.

[4] Jandačka J, Holubčík M, Malcho M, Nosek R. Modification of wood pellets parameters by using of concentrate of organic compounds from defibration. *Acta Facultatis Xylologiae* 2013;55(2):105-115.

[5] Uasuf A, Becker G. Wood pellets production costs and energy consumption under different framework conditions in Northeast Argentina. *Biomass Bioenergy* 2011;35(3):1357-1366.

[6] Stahl M, Berghel J. Energy efficient pilot-scale production of wood fuel pellets made from a raw material mix including sawdust and rapeseed cake. *Biomass Bioenergy* 2011;35(12):4849-4854.

[7] Kjallstrand J, Olsson M. Chimney emissions from small-scale burning of pellets and fuelwood–examples referring to different combustion appliances. *Biomass Bioenergy* 2004;27(6):557-561.

[8] Klason T, Bai XS. Computational study of the combustion process and NO formation in a small-scale wood pellet furnace. *Fuel* 2007;86(10-11):1465-1474.

[9] Han J, Kim H, Minami W, Shimizu T, Wang, G. The effect of the particle size of alumina sand on the combustion and emission behavior of cedar pellets in a fluidized bed combustor. *Bioresour Technol* 2008;99(9):3782-3786.

[10] Atkins A, Bignal KL, Zhoua JL, Cazier F. Profiles of polycyclic aromatic hydrocarbons and polychlorinated biphenyls from the combustion of biomass pellets. *Chemosphere* 2010;78(11):1385-1392.

[11] Gil MV, Oulego P, Casal MD, Pevida C, Pis JJ, Rubiera F. Mechanical durability and combustion characteristics of pellets from biomass blends. *Bioresour Technol* 2010;101(22):8859-8867.

[12] Mandl C, Obernberger I, Scharler IR. Characterisation of fuel bound nitrogen in the gasification process and the staged combustion of producer gas from the updraft gasification of softwood pellets. *Biomass Bioenergy* 2011;35(11):4595-4604.

[13] Miranda T, Arranz JI, Montero I, Román S, Rojas CV, Nogales S. Characterization and combustion of olive pomace and forest residue pellets. *Fuel Process Technol* 2012;103(1):91-96.

[14] Roy MM, Dutta A, Corscadden K. An experimental study of combustion and emissions of biomass pellets in a prototype pellet furnace. *Appl Energy* 2013;108(1):298-307.

[15] Rabaçal M, Fernandes U, Costa M. Combustion and emission characteristics of a domestic boiler fired with pellets of pine, industrial wood wastes and peach stones. *Renew Energy* 2013;51(1):220-226.

[16] Persson T, Riedel T, Berghel J, Bexell U, Win MK. Emissions and deposit properties from combustion of wood pellet with magnesium additives. *J Fuel Chem Technol* 2013;41(5):530-539.

[17] Fernandes U, Guerrero M, Millera A, Bilbao R, Alzueta MU, Costa M. Oxidation behavior of particulate matter sampled from the combustion zone of a domestic pellet-fired boiler. *Fuel Process Technol* 2013;116(1):201-208.

[18] Babrauskas V, Peacock RD. Heat release rate: the single most important variable in fire hazard. *Fire Saf J* 1992;18(3):255-272.

[19] Martinka J, Hroncová E, Chrebet T, Balog. K. A comparison of the behaviour of spruce wood and polyolefins during the test on the cone calorimeter. *Adv Mater Res* 2013;726-731:4280-4287.

[20] Ladomerský J. Emission analysis and minimization from the wood waste combustion. *Wood Res* 2000;45(4):33-44.

[21] Ladomerský J, Hroncová E, Samešová D. Investigation of appropriate conditions for wood wastes combustion on basis of emission. *Drew* 2003;170:90-98.

[22] Ladomerský J, Hroncová E. Evaluation of combustion process of wood waste in combustion chamber based on emissions. *Acta Mech Slovaca* 2003;7(3):595-600.

[23] Martinka J, Kačíková D, Hroncová E, Ladomerský J. Experimental determination of the effect of temperature and oxygen concentration on the production of birch wood main fire emissions. *J Therm Anal Calorim* 2012;110(1):193-198.

[24] Martinka J, Balog K, Chrebet T, Hroncová E, Dibdiaková J. Effect of oxygen concentration and temperature on ignition time of polypropylene. *J Therm Anal Calorim* 2012;110(1):485-487.

[25] European Committee for Standardization. EN 14961-1:2010. Solid biofuels: Fuel specifications and classes. Part 1: General requirements. Brussels: CEN; 2010.

[26] International Organization for Standardization. ISO 5660-1:2002. Reaction-to-fire tests: Heat release, smoke production and mass loss rate. Part 1: Heat release rate (cone calorimeter method). Geneva: ISO; 2002.

[27] International Organization for Standardization. ISO 871:2006. Plastics: Determination of ignition temperature using a hot air furnace. Geneva: ISO; 2006.

[28] Parker WJ. Prediction of the heat release rate from basic measurements. In: Babrauskas V, Grayson, SJ, editors. *Heat release in fires*. London: Interscience Communications; 2009. p. 333-356.

[29] Tran HC. Experimental data on wood materials. In: Babrauskas V, Grayson, SJ, editors. *Heat release in fires*. London: Interscience Communications; 2009. p. 357-372.

[30] White RH, Dietenberg MA. Fire safety. In: Dietenberg MA, Green WD, Kretschmann DE, editors. *Wood handbook*. Madison: Forest Products Laboratory USDA Forest Service; 1999. p. 400-416.

[31] Majlingová A, Osvald A, Osvaldová L. Comparison of carbonized layer thickness and weightless of spruce and larix. In: Kačíková D, editor. *Proceedings of Heat-fire-materials*; 2007 Feb 6-9; Tále, Slovakia. Zvolen: Technical University in Zvolen; 2007. p. 49-55.

[32] Martinka J, Chrebet T, Zachar M, Balog K. Assessment of oak wood age on its fire risk. In: Galla Š, editor. Proceedings of Fireco; 2013 May 2-4; Trenčín, Slovakia. Bratislava: Fire Research Laboratory of Ministry of Interior of Slovak Republic; 2013. p. 235-243.

[33] Xu Q, Majlingová A, Zachar M, Jin C, Jiang Y. Correlation analysis of cone calorimetry test data assessment of the procedure with test of different polymers. *J Therm Anal Calorim* 2012;110(1):65-70.

[34] Kamikawa D, Kuroda K, Inoue M, Kubo S, Yoshida T. Evaluation of combustion properties of wood pellets using a cone calorimeter. *J Wood Sci* 2009;55(6):453-457.

[35] Martinka J, Hroncová E, Chrebet T, Balog K. The influence of spruce wood heat treatment on its thermal resistance and burning process. *Eur J Wood Prod*. Forthcoming 2014.

[36] Atkins A, Bignal KL, Zhou JL, Cazier F. Profiles of polycyclic aromatic hydrocarbons and polychlorinated biphenyls from the combustion of biomass pellets. *Chemosphere* 2010;78(11):1385-1392.

[37] Dvořák O, Charvátová V, Ružička M. Hazard of combustion products of materials. Praha: Ministry of Interior of Czech Republic; 2007. 87 p.

[38] Zachar M, Mitterová I, Xu Q, Majlingová A, Cong J, Galla Š. Determination of fire and burning properties of spruce wood. *Drv Ind* 2012;63(3):217-223.

[39] Kačíková D, Osvald A, Netopilová M. Wood and its thermal degradation. Ostrava: Association of Fire and Safety Engineering; 2006. 79 p.

[40] Osvald A, Mózer V. Fire engineering a method for fire safety design. In: Zachar M, Dúbravská K, editors. Advances in Fire and Safety Engineering. 2nd International Scientific Conference on Advances in Fire and Safety Engineering; 2012 Nov 15-16; Zvolen, Slovakia. Zvolen: Technical University in Zvolen; 2012. p. 211-223.

[41] Osvald A, Mózer V. Comparison of selected calculation methods for determination of fire resistance requirements. In: Šenovský M, editor. Fire Safety. 22nd International Scientific Conference on Fire Safety; 2013 Sept 4 – 5; Ostrava, Czech Republic. Ostrava: Technical VŠB TU Ostrava; 2013. p. 169-173.