ENGINEERING STRUCTURES AND TECHNOLOGIES
ISSN 2029-882X print / ISSN 2029-8838 online
2013 5(4): 167–174
doi:10.3846/2029882X.2014.894666

APPLICATION OF DATA FROM THE CONE CALORIMETER FOR ANALYSES OF THE IMPACT OF A FLAME RETARDANT APPLIED ON POLYMER MATERIAL SUBJECTED TO COMBUSTION ON CHANGES IN SMOKE LOGGING WITHIN A CONFINED SPACE

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Received 18 September 2013; accepted 19 December 2013

Abstract. The paper makes an analysis of the impact of flame retardants on parameters of thermal energy emission and smoke from polymer materials specified with the use of a cone calorimeter and on parameters of the smoke logging zones in a model scheme of compartments. Polymer construction materials modified and without modification by flame retardants, such as oak wood, epoxy resins based on Epidian 5 as well as such equipping materials as polyurethane foams were selected as research materials. On the basis of the conducted analyses, a positive correlation was achieved between the parameters obtained with the use of a cone calorimeter and parameters of smoke logging based on bi-zonal modelling for a simple model scheme of two compartments.

Keywords: flame retardants, heat release rate, smoke, visibility range.

Reference to this paper should be made as follows: Konecki, M.; Półka, M.; Tuśnio, N.; Griškevičius, M. 2013. Application of data from the cone calorimeter for analyses of the impact of a flame retardant applied on polymer material subjected to combustion on changes in smoke logging within a confined space, *Engineering Structures and Technologies* 5(4): 167–174. http://dx.doi.org/10.3846/2029882X.2014.894666

Introduction

Limited visibility impedes or renders impossible the evacuation of people from a building. A reduction in the visibility range due to smoke is in many cases the first risk factor for humans during a fire, even before its thermal impact commences. Reducing the feeling of impending hazard helps enhancing the efficiency of the human organism in an environment affected by a fire, which helps avoiding panic and facilitates organised effective rescuing and extinguishing actions. Statistical data (Guidelines ... 1989) show that 60–80% of fire fatalities arise from smoke inhalation.

Up to now, studies were carried out on smoke emission from natural and artificial polymer materials, but in the majority of cases the studies were limited merely to the measurement of optical density of smoke and the derivative values in conditions where there is an impact of a thermal radiation flux with a required value. Tests were carried out on smoke logging properties of materials, depending on the type of combustion (Drysdale 1985) oxygen concentration (Mulholland 1988), chemical composition and structure of material (Mulholland 1988; Östman 1992) fire resistant additives (Gao *et al.* 2008; Konecki, Półka 2006; Tewarson 1988; Toldy *et al.* 2007, 2008). There are still few research publications that analyse relations between data related to the emission of thermal energy and smoke on a small scale to the smoke logging degree of constrained spaces.

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The objective of this paper was to provide an analysis of values pertaining to visibility ranges in smoke in the selected model scheme of compartments with the use of a single- and double-zone fire model on the basis of data obtained from a cone calorimeter. Such polymer materials as wood, epoxy resins based on Epidian 5 (Ep 5) and synthetic polyurethane foams were selected as for combustion. Those particular materials were selected due to their frequent use in general construction. The research comprised materials modified and unmodified by flame retardants. The analysis focused on the impact of the fire resistant combustible material on changes in the fire environment using a model scheme of two compartments.

1. Experimental part

Based on a small-scale method of the cone calorimeter, this paper determines the following combustible parameters: the mass loss rate during combustion, the heat release rate per material surface unit HRR (kW/m^2), the parameter of smoke emission – the specific extinction area – SEA (m^2/kg). SEA defines the effective light absorbing surface of smoke particles in m^2 produced during the thermal decomposition and combustion of 1 kg of material. In addition, emissions of selected gaseous decomposition products CO and CO_2 have been determined.

The research was carried out for all samples of materials modified and unmodified by flame retardants.

1.1. Description of materials to be tested

Oak wood and applied fire retardants. Experimental studies were conducted using samples of oak wood with the density of 0.62–0.69 g/cm^3 obtained from a sawmill. The choice of impregnates for the testing was predominantly connected with the wood fire resistant formulations, which are most commonly bought and used in the Polish construction industry. The following agents were applied as fire resistant impregnates for wood:

- **Fobos M-4** (manufacturer: Zakłady Chemiczne Luboń sp. z o.o.). Mixture of inorganic salts with a slight addition of organic salts. This medium has a fire resistant action based on endothermic heat absorption during the decomposition and decrease in the concentration of oxygen and flammable gases in the fire zone.
- **Uniepal-Drew** (manufacturer: PPH "ADW" sp. z o.o). Water based polyurethane varnish. This medium has a fire resistant action based on endothermic heat absorption during the decomposition, reduces the heat release rate and does not extend the time until ignition.
- **Ogniochron/Impregnator F40 P** (manufacturer: Przedsiębiorstwo Altax sp. z o.o.). Mixture of inorganic salts. This medium has a fire resistant action based on endothermic heat absorption during the decomposition and decrease in the concentration of oxygen and flammable gases in the fire zone.
- **Prevento Sprayer** (manufacturer: Febbex). This medium has a fire resistant action based on endothermic heat absorption during the decomposition and decrease in the concentration of oxygen and flammable gases in the fire zone.

Polyurethane foams and applied fire retardants. Polyurethane foam manufactured by the Zakłady Chemiczne "Zachem" in Bydgoszcz was used for testing. The output material for the production of the foam was polyurethane produced of isocyanate with two isocyano groups in the particle – TDI (toluene diisocyanate), polyether resin, water and admixtures (catalysts: amine catalyst and organotin compounds and surfactants). The following medium was used for fire retardant modification of the polyurethane for foam:

- **Fyrol PNX LE**, an agent manufactured by ICL Industrial Products. Its main component is an oligomer of the phosphorus polyester type. In addition, the medium contains phosphoric acid and phosphorus oxygen (V).
- **Expanding graphite**, modified, of natural origin, produced of graphite ore in the process of enriching and refinement, and then exposed to strong oxidizers. In the course of that process, the intercalation of the graphite crystal lattice by atoms of another element takes place. During heating, the release of intercalate occurs, breaking interplanar bonding and exfoliation of graphite crystals, as an effect of which grapheme nano aggregations are formed. The graphite undergoes intense volumetric expansion and transforms into a flexible and insulating material called graphite expand, which has a specific surface area close to 100 m^2/g.

Epoxy material made of Epidian 5 and applied fire retardants. Unmodified hardened epoxy resin (Epidian 5, Ep 5) and samples of resin with flame retardant modification were analysed. Epidian 5 is a resin with a relatively small molecular mass, produced by Zakłady Chemiczne "Organika – Sarzyna" in Nowa Sarzyna. The following mediums were used for fire retardant modification of an epoxy material:

- **Apyral** is the commercial name of a mineral that contains aluminium hydroxides (III). This antipyrene carbonises the surface of the material subjected to a heat flux. In the presence of an ignition source, the aluminium hydroxide begins the decomposition reaction with the precipitation of water, which as an effect leads to the formation of a glassy coat on the surface of the epoxy material. Water vapour cools the surface of the material and dissolves the gaseous flammable decomposition products. The produced carbonated coat provides protection from thermal radiation and the access of oxygen as well as limits the release of flammable products generated during thermal decomposition and combustion.
- **Fyrol PNX** is a flame retardant containing phosphorus atoms, which does not contain halogens. Thanks to the high molecular mass, it has a low vapourisation level and impedes on the emission of volatile organic compounds in fire conditions. The fire retardant action is mainly of a physical nature and consists of the formation of a protective layer (carbonated coat) on the material surface.
- **Carbon nanotubes** – carbon nanomaterials. In the epoxy material, during combustion, they contribute to the production of carbonised structures on the surface of the polymer material.

1.2. Research method

Parameters of the emission of thermal energy and smoke from polymer materials were determined with the use of a cone calorimeter produced by FTT (Fire Testing Technology) from Great Britain, which is at the disposal of the laboratory of the Institute of Combustion of the Main School of Fire Service. The measurement procedure was carried out in accordance with Part 1 "Fire tests – reaction to fire-rate of heat release from building products. Cone calorimeter method" of the standard ISO 5660:2002. The measurements were conducted at the external thermal flux of 30 kW/m^2.

The values of selected flammability parameters obtained with the use of the cone calorimeter were presented in Tables 1 and 2.

The results of the conducted research indicate the impact of fire retardant additives of particular materials on the emission of thermal energy and smoke during their combustion.

The conducted research allowed determining the tested flame retardants that lead to an intensified generation of thermal energy and smoke. Wood modified with fire retardant intensifies the emission of smoke particles, regardless of applied admixture. The impact on changes in the heat emission varies (Table 1).

Table 1. The maximum heat release rate HRR_{max} and average SEA_{av} values for wood and polyurethane foam, unmodified and modified with flame retardants

Tested material	HRR_{max} [kW/m^2]	SEA_{av} [m^2/kg]
oakwood	193.91	16.76
oakwood + Fobos (p)*	236.13	78.33
oakwood + Fobos (n)**	185.54	51.23
oakwood + Uniepal (p)	186.52	45.23
oakwood + Uniepal (n)	224.82	47.89
oakwood + Prevento (p)	208.12	27.67
oakwood + Prevento (n)	215.27	32.29
oakwood + Ognioochron (p)	181.40	49.44
Polyurethane foam	189.18	318.55
Polyurethane foam + graphite	136.51	193.4
PNXEL foam	113.85	334.02

* applied by brush; **applied by spraying.

Table 2. The maximum heat release rate HRR_{max} and average values of SEA_{av} for epoxy material, unmodified and modified with flame retardants

Material name	HRR_{max} [kW/m^2]	SEA_{av} [m^2/kg]
UEp 5	1242	904
UEp5+5%Apyral	908	834
UEp5+10%Apyral	890	844
UEp5+3%Fyrol PNX	797	774
UEp5+5%Fyrol PNX	669	723
UEp5+5%Apyral+5%Fyrol PNX	663	762
UEp5+0.1%Nanotubes	802	869

As regards the epoxy material, flame retardants lower the emission of heat and smoke. This is particularly evident in the case of an additive called Fyrol PNX and mixture of Fyrol PNX with Apyral. Additives to polyurethane foam cause a significant reduction of HRR_{max}. The addition of graphite considerably reduces the emission of smoke, while PNXEL slightly increases it (Table 2).

2. Estimations of the visibility range based on a single-zone model

By assuming the emission of smoke particles in dynamic fire conditions determined with the use of the SEA parameter, an equation was obtained that allows the estimation of the visibility range in smoke on the assumption of the formation of a homogenous mixture of smoke particles with air and gaseous products of thermal decomposition and combustion (Półka 2007):

$$Z(t) = \frac{C \cdot V}{SEA_{śr} \cdot \left[m_p - m(t) \right]}, \quad (1)$$

where: C is a constant for objects glowing by reflected light, C = 3.0; V – the volume of a smoke-logged space [m^3]; m_p – the initial mass of the sample [g]; $m(t)$ – mass of the sample in given time.

By adopting a single-zone smoke it was assumed that polymer materials would burn in a compartment model with a capacity of 114.40 m^3 (office space with a corridor) (Fig. 2). For comparative needs with respect to smoke generation, a constant material combustion surface equal to 1 m^2 was adopted, the material thickness equal to the thickness of the sample in the cone calorimeter and combustion conditions equal to those in the cone calorimeter.

Below (Table 3) are the results of calculations regarding the critical times of visibility reduction of 5 m.

The results of analytical calculations show that critical times of the visibility range reduction are mainly a function of the following: the capacity of the compartments, the chemical composition of the material exposed to thermal decomposition and combustion, the smoke release rate from the material, the mass combustion rate and lighting conditions of objects situated inside a smoke logged compartment.

Oak wood without flame retardant modification has achieved the longest time of critical visibility reduction in the modelled compartment scheme. Wood sample impregnated by flame retardants have indicated much lower values of visibility ranges in smoke in the analysed compartment scheme.

Prevento Speryer (applied with a brush) proved to be the most appropriate fire retardant for samples tested with respect to the values of visibility ranges, and Uniepal Drew was found to be the least advantageous one. Values of visibility ranges in smoke for polyurethane foams were very small, which proves that during a fire they pose a significant hazard as elements of interior decoration, especially in class ZL fire zone buildings.

The addition of antipyrenes changes the nature of distribution of epoxy resins in an individual way. From the viewpoint of the visibility range values, as compared to other types of materials, the most advantageous antipyrene parameters have been recorded for Apyral and Nanotubes. The action of Fyrol appears to be less advantageous despite the fact that it lowers the average smoke emission and considerably reduces the generation of heat (Table 1), which is related with the smallest mass loss rate.

Table 3. The critical times of visibility reduction for the tested materials in the model system of compartments

Tested material	Critical time of visibility range [s]	Tested material	Critical time of visibility range [s]
oakwood	94	Ep5	64
oakwood + Fobos (brush)	52	Ep5 + Apyral 5%	88
oakwood + Fobos (spray)	70	Ep5 + Apyral 5 % + Fyrol PNX 5%	60
oakwood + Uniepal (brush)	32	Ep5 + Apyral 10%	82
oakwood + Uniepal (spray)	80	Ep5 + Fyrol PNX 3%	70
oakwood + Prevento (brush)	102	Ep5 + Fyrol PNX 5%	74
oakwood + Prevento (spray)	88	Ep5 + Nanotubes 0.1%	86
oakwood + Ognioochron (brush)	68	polyurethane foam + graphite	8
polyurethane foam	6	PNXEL foam	8

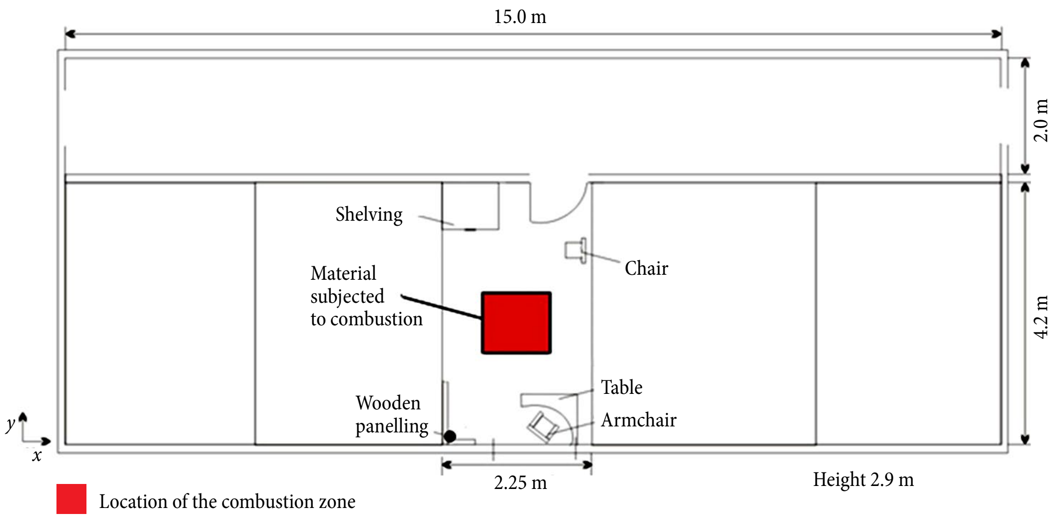

Fig. 1. Diagram of the model: office space – the corridor

3. Smoke logging simulation performed with the use of a bi-zonal model

It was assumed that materials analysed in this paper would be combusted in conditions of a constant combustion (fire) conditions equal to 1 m^2 and that they would be arranged in the geometric centre of the floor of a given compartment (Fig. 1). The thermal decomposition and combustion took place under exposure to a constant heat flux with a capacity of 30 kW/m^2. The data $\dot{Q} = \dot{Q}(t)$ that characterise heat emission from each material and the emission data of various combustion products (smoke particles and gaseous products) have been adopted as data obtained in advance with the use of the method based on a small geometrical scale of the cone calorimeter, with the assumption that 1 m^2 of materials would be combusted. It was assumed that a bi-zonal fire model scheme would be established using the CFAST programme (Jones *et al.* 2006).

The combustion of polymer materials, Epidian and Epidian with additives, leads to a considerable increase in the optical growth of the uppermost smoke layer in the corridor (Fig. 2). The combustion of material consisting of Epidian 5+Z–1+Apyral 5%+Fyrol PNX 5% leads to the quickest reduction in the visibility range. As regards a 5 m evacuation, the critical range was achieved after a time of ca. 80 s. Epidian 5+Z–1 undergoes decomposition with the generation of smoke, which achieves the density conforming to the 5 m visibility range after ca. 120 s. The remaining additives (apart from nanotubes) shorten that time.

As regards the application of nanotubes, the critical smoke logging level was achieved after ca. 260 s. In all cases of thermal decomposition and combustion, the visibility range was reduced to nil.

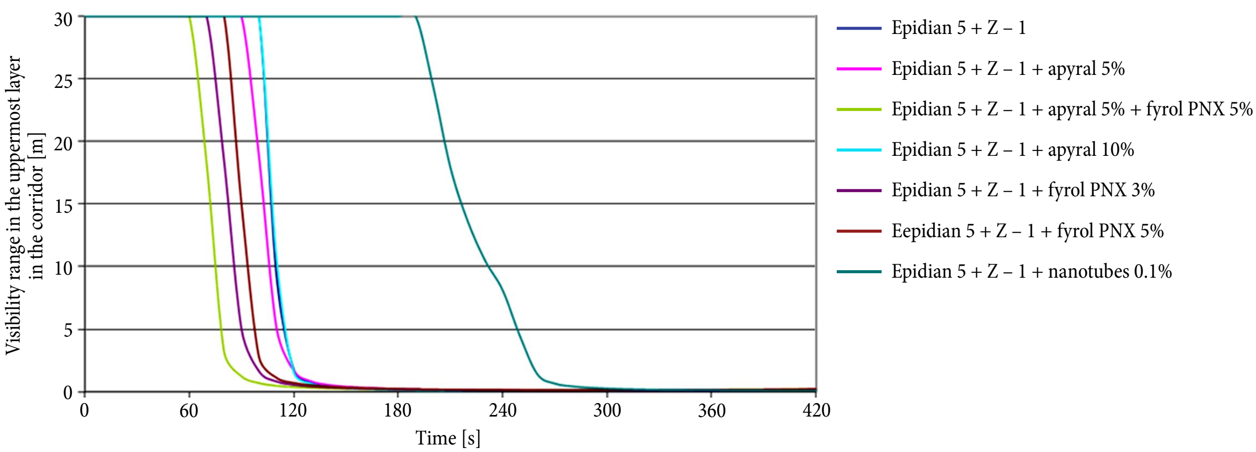

Fig. 2. The range of visibility in the upper layer in the corridor as a function of combustion time of polymeric materials formed from Epidian 5, unmodified and modified with flame retardants

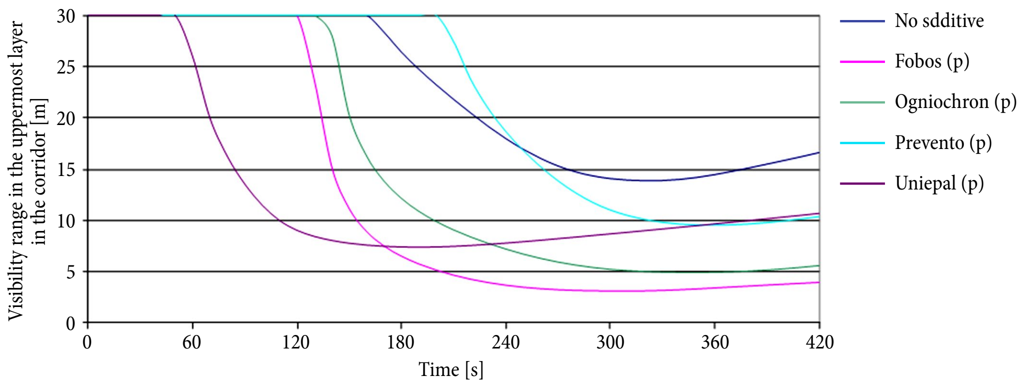

Fig. 3. The range of visibility in the uppermost layer in the corridor as a function of time combustion of oak wood samples modified with flame retardants applied by brush and unmodified

The combustion of pure polyurethane foam and of foam with additives causes a significant increase in the optical density of the uppermost smoke layer in the corridor, which takes place already after a very short time, regardless of the type of additive being tested.

In all cases, the 5 m visibility range was achieved after ca. 20 s, and after the time of ca. 60 s it was reduced to ca. 1 m.

The oak wood modification by fire retardants by selected antipyrenes causes a more intense smoke logging of the corridor than burning unmodified material (Fig. 3).

If Uniepal and Prevento are applied to wood samples, the critical conditions are not exceeded. As regards Fobos, the 5 m level is achieved after 200 s, and for Ogniochron after 360 s. As compared to the brush application method, the spraying method leads to the extension of the time until achieving the minimum visibility range for Fobos and Uniepal. As regards Prevento, the spray application method shortens the time of achieving the minimum visibility range as compared to the brush method.

In conformity to the dynamics of combustion processes, the generation of smoke is a strong function of the combustion rate; and with its increase, emission of smoke particles from materials is hastened. As an effect, the reduction of the visibility range is a process characterised by the highest dynamics among all phenomena comprised by a fire, and in a natural way poses the biggest threats to the process of safe evacuation of humans from a building.

4. Experimental correlations between data from a cone calorimeter and smoke logging parameters

The calculation of smoke emission, which may be characterised by parameters that determine the optical properties of smoke on the basis of the chemical composition and kinetics of the combustion reaction of building materials and products, is presently impossible. For this reason forecasting smoke logging of compartments is based primarily on data obtained in small scale studies, e.g. with the use of a cone calorimeter (Tewarson 1988). The theory of phenomena associated with uncontrolled combustion leads to general dependencies between parameters that determine the emission of thermal energy and smoke from a material in fire conditions and parameters of a fire that control the smoke-logging state in the model scheme of compartments.

From experimental data obtained by applying the cone calorimeter method to describe the dynamics of processes comprising thermal energy dynamics, adopted was the heat release rate parameter $HRR_{Imax}/\Delta t_{Imax}$ determined by the relation between the heat release rate at the maximum point of its first peak to the time of its achieving. The $1/SEA_{av}$ was adopted as a parameter that determines the emission of smoke.

The obtained theoretical correlative dependencies between experimental parameters achieved in the cone calorimeter methods and parameters of the smoke logging rate were used for an analysis of regression, which allows the determination of quantitative dependencies between the specified values.

A strong correlation was achieved between the parameters of the heat release rate and the time needed to achieve the situation of the uppermost layer 1.5 m over the corridor floor level (Fig. 4) and the time until achievement of the 5 m visibility range in the uppermost layer in the corridor (Fig. 5). The theoretically assumed nature of the function dependencies as achieved despite the evident differences in exponents.

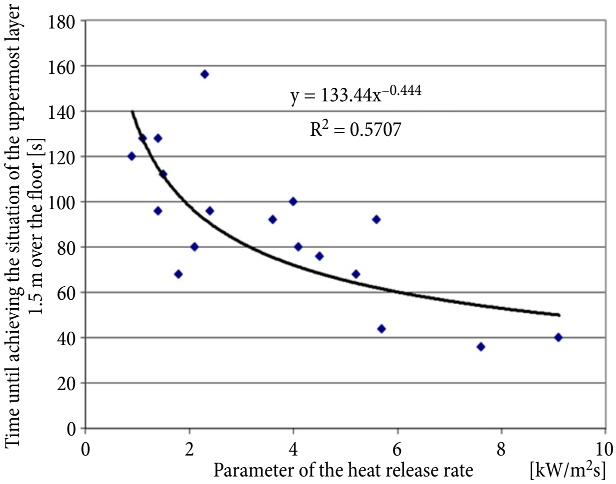

Fig. 4. The correlation between time to achieve the uppermost layer of 1.5 m above the floor corridor and the parameter of heat release rate of materials undergoing combustion

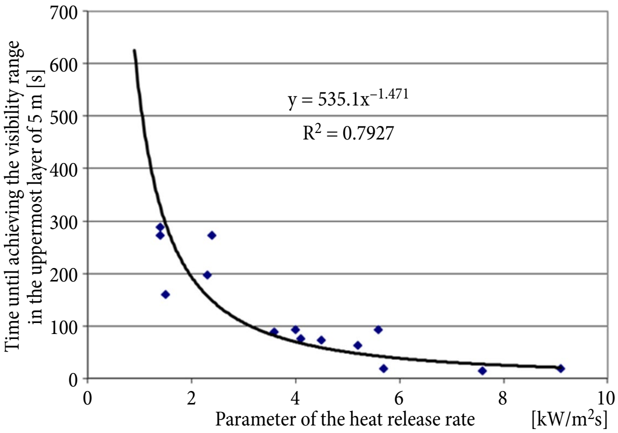

Fig. 5. The correlation between time to achieve the visibility range of 5 m in uppermost layer in the corridor and the parameter of heat release rate of materials undergoing combustion

The correlation between the time of achieving the critical 5 m visibility range in the uppermost layer and the parameter of smoke emission was shown in Fig. 6. A strong correlation was achieved. The following correlating relations where obtained from an analysis of the non-linear regression, as shown in the above-given figures:

$$t(z = 1.5\text{ m}) = \frac{a}{\left(\frac{HRR_{I_{\max}}}{\Delta t_{I_{\max}}}\right)^{b}}, \tag{8}$$

where constants: a = 133.44, b = 0.444 for the model scheme compartment – the corridor.

$$t(Z = 5\text{ m}) = \frac{c}{\left(\frac{HRR_{I_{\max}}}{\Delta t_{I_{\max}}}\right)^{d}}, \tag{9}$$

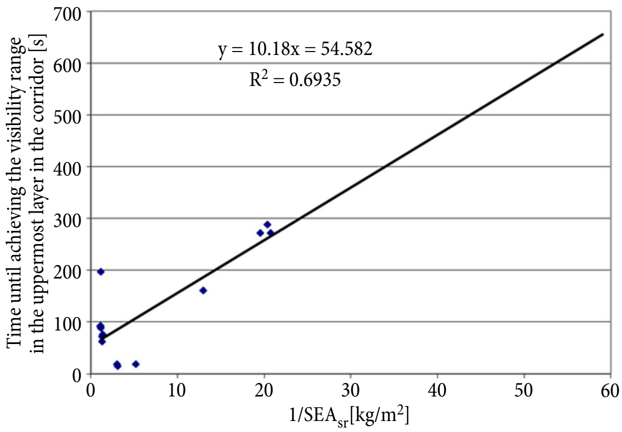

Fig. 6. The correlation between time to achieve the visibility range of 5 m in the uppermost layer in the corridor and smoke parameter of materials undergoing combustion

where constants: c = 535.1, d = 1.471 for the model scheme compartment – the corridor.

$$t(Z = 5\text{ m}) = \frac{e}{SEA_{sr}} + f, \tag{10}$$

where constants: e = 10.18, f = 54.582 for the model scheme compartment – the corridor.

The achieved types of dependencies between the material parameters and smoke logging fire parameters may be used to determine the threshold "safe" values of parameters of heat and smoke emission from materials determined by the cone calorimeter method. Further studies are needed to cover a bigger number of materials, as well as estimations for smoke logging of diverse internal compartments in a building.

Summary

The conducted calculations have proven that zone models averaging fire parameters over fire zones (i.e. control volumes) are sufficiently accurate for needs of carrying out comparative studies related to the impact of diverse additives modifying fire properties of flammable materials and for their ranging with respect to the potential fire hazard related to the increase in the smoke logging degree. Data concerning emission of heat and smoke from the cone calorimeter may be applied to enable a review of the impact of the smoke retardant of the polymer material exposed to combustion on changes of smoke logging of a confined space in the model scheme compartment – the corridor. The extension of obtained interdependencies onto other model schemes of compartments requires further research. For the majority of internal compartments in a building, higher values of critical times may be expected. For this reason, the smoke hazard in the assumed model scheme may be considered to be close to the maximal level. A good correlation was obtained between parameters obtained with the use of a cone calorimeters and parameters of smoke logging modelled on a two-zone basis for various values of the heat release rate. The single zone model may be applied for needs of making an initial assessment of the impact of a fire retardant on the smoke logging degree of a compartment model.

Results of an analysis of regression in correlating dependencies for data from the cone calorimeter and smoke logging parameters in the bi-zonal fire model scheme point to the existence of good correlations between the time until achieving the situation of the uppermost layer 1.5 m over the corridor floor area and the 5 m visibility range in the uppermost layer and the parameter of heat release rate from materials exposed to combustion, defined as a ratio of HRR I maximum to the time it is achieved.

It proved to be possible to achieve the theoretically assumed nature of the functions, despite differences in exponents of the obtained dependencies.

A good correlation was also achieved between the parameter of the smoke particle emission specified as $1/SEA_{av}$ and the time until reaching the visibility range of 5 m in the uppermost layer in the corridor.

The achieved dependencies allow the estimation of times of achieving critical smoke logging conditions for the model scheme compartment – the corridor from data obtained from the cone calorimeter. Consequently the initial assessment is possible of the smoke logging hazard on the basis of the behaviour of materials in conditions that simulate a fire, in a small laboratory scale.

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