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Correlation analysis of cone calorimetry and microscale combustion calorimetry experiments

Rhoda Afriyie Mensah[1] · Qiang Xu[1] · Solomon Asante-Okyere[1] · Cong Jin[2] · Geoffrey Bentum-Micah[3]

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Abstract

Flammability studies are conducted to evaluate the behavior of materials exposed to fire. In this study, microscale combustion calorimetry (MCC) and cone calorimetry methods were applied to acquire the flammability characteristics of red and grey extruded polystyrene (XPS) samples. To understand the effect of changes between parameters, Pearson's correlation coefficient was used to examine their linear relationships. From the research, moderate and weak correlations were recorded between the total heat release rates from both methods for red and grey XPS, respectively. Plotting peak heat release rate against heat release temperature for MCC and ignition temperature for cone test showed that 25, 35 and 50 kW m^{-2} incident heat fluxes of the cone test fall within 0.2 K s^{-1} and 0.5 K s^{-1} heating rates of MCC. Also, all the MCC parameters except char yield and total heat release presented good correlations with the cone calorimetry flammability characteristics. Hence, MCC could be used in conjunction with cone calorimetry to accurately and reliably assess the flammability of materials.

Keywords Flammability · Microscale combustion calorimetry · Cone calorimetry · Pearson's correlation

List of symbols

β	Heating rate in MCC test/K s^{-1}
Bi	Biot's number
c	Specific heat/J g^{-1} K^{-1}
h	Heat transfer coefficient/W m^{-2} K^{-1}
h_g	Heat of gasification/MJ kg^{-1}
k	Thermal conductivity/W m^{-1} K^{-1}
L_c	Characteristic length/m
m_o	Initial mass of sample/g
m_p	Mass of residue/g
η_c	Heat release capacity/J g^{-1} K^{-1}
pHRR	Peak heat release rate/W g^{-1}
pTemp	Temperature at pHRR/°C
q_{in}	Incident heat flux/kW m^{-2}
q_{max}	Maximum value of heat release rate per unit area/ kW m^{-2}
THR	Total heat release/kJ g^{-1}
T_{ig}	Ignition temperature/°C
t_{ig}	Time to ignition/s
ΔT_{ig}	Change in ignition temperature/°C
T_{py}	Pyrolysis temperature/°C
ρ	Density/kg m^{-3}

✉ Qiang Xu
xuqiang@njust.edu.cn

[1] School of Mechanical Engineering, Nanjing University of Science and Technology, Nanjing 210094, China

[2] School of Computer Science and Technology, Nanjing University of Science and Technology, Nanjing 210094, China

[3] School of Management Science and Engineering, Jiangsu University, Zhenjiang 212013, China

Introduction

Microscale combustion and cone calorimetry experiments are thermal analysis methods that play active roles in effective flammability studies. They are carried out to assess the flammability of materials such as polymers. Among other polymers, polystyrene was used as a reference for the calibration of most of the flammability equipment. This material is widely used and has a high production rate around the world. Extruded polystyrene formed by the extrusion of polystyrene beads is normally used as insulation material for energy efficient buildings. Therefore, research on the flammability of extruded polystyrene using flammability test methods is highly beneficial.

Microscale combustion calorimetry (MCC) also known as the pyrolysis combustion flow calorimetry (PCFC) is a small-scale experiment used to test the flammability of milligram-sized samples [1]. The MCC experiment is conducted under an inert gas atmosphere, usually nitrogen or nitrogen mixed with oxygen, in the pyrolysis stage. The gases released from the pyrolysis are combusted in the presence of oxygen under controlled conditions [2]. It is worth noting that MCC has been extensively used in recent years to obtain precise flammability characteristics such as the total heat released, heat release capacity, peak heat release rate and temperature at peak heat release of materials to analyze their flammability [3]. Considering the type of materials that can be tested with this method, MCC has been successfully used to test not only polymeric materials but also wood [4]. Lyon et al. [5] applied MCC as a flammability screening method for flame retardants in plastic materials. On a similar note, Schartel et al. [6] assessed the use of MCC as a tool in evaluating the flammability of blends of polycarbonate (bisphenol A)/acrylonitrile–butadiene–styrene (PC/ABS) with additives and flame retardants. Also, the flammability of cotton and nylon fabrics treated with flame retardants was tested using MCC in the work of Yang et al. [7].

Aside MCC, another equally important and frequently used flammability test method is the cone calorimetry. This is a bench scale method which employs the use of a cone-shaped heater to heat samples of size 100 mm × 100 mm × 10–50 mm under controlled conditions. With this flammability experiment, the sample is positioned under a cone heater and heated within an incident heat flux of 10–75 kW m^{-2}. The sample is ignited by an electric spark which remains lighted until the sample ignites. The gases released are collected in a hood from which the various parameters are measured [8, 9]. Cone calorimetry measures the peak heat release rate and time to ignition of the materials tested. However, unlike MCC, the parameters measured from the cone calorimetry experiment are highly dependent on the ignition source, orientation of sample and its thickness [10]. Cone calorimetry has been widely adopted by several scholars in their research to evaluate fire safety performance of polymeric materials. In the work of Qin et al. [11–13], cone calorimetry experiment was conducted to assess the thermal stability and flammability of polyethylene/clay nanocomposites, polypropylene/montmorillonite composites and polyamide 66/montmorillonite nanocomposites. Similarly, Delichatsios et al. [14] employed cone calorimetry in their research to analyze the flammability of charring materials.

Cone calorimetry could be used to represent specific fire scenarios as well as the prediction of specific parameters in small scale, large scale and real fire behavior [10]. It has been stated in [10] that results from cone calorimetry have the capability of simulating real fire behavior and other fire tests. It has also been proven experimentally in [15] that the result from cone calorimetry can be used to predict full-scale smoke production at 400 kW heat release rate.

In recent studies, both microscale combustion and cone calorimetry methods have been incorporated in research to make a comparison and evaluation of flammability characteristics. Combining the two methodologies gives an in-depth understanding on the flammability of the materials under consideration. Also, the joint assessment could serve as a tool for predicting bench scale flammability measurements from small-scale tests. This is demonstrated in the research of Xu et al. [16] where they compared the flammability of two different extruded polystyrene foams using MCC and cone calorimetry. They made an inference that results from MCC can be used to predict the ignition time of cone calorimeter tests. However, Xu et al. did not consider correlation analysis of the two experiments. It is important to note that correlations between parameters measured from both experiments also offer some advantages in flammability studies by giving details on the relationship between the various flammability characteristics. The methodology described has been used to analyze the flammability of numerous materials by several researchers. Cogen et al. [17] effectively run Pearson's correlations between MCC and other conventional flammability tests on flame-retardant polyolefin compounds free of halogen. A quite similar method was used by Lyon et al. [18] to provide the relationship between heat release capacity from MCC and thermogravity analysis (TG) test for 14 different polymers. Also, Lin et al. [19] conducted flammability analysis of flame-retardant wire and cable compounds using MCC, cone calorimeter, limiting oxygen index and UL94. Subsequently, correlations between the parameters were calculated to show the strength of their relationships.

Despite the fact that cone calorimetry has achieved a high degree of accuracy in flammability studies, it's use is limited due to high cost, equipment complexity and maintenance [17]. MCC has the capability of correlating fire behavior with material properties, but the results do not include ignition parameters. Owing to this, correlations are conducted between cone calorimetry and MCC to ascertain their linear relationships for prediction and joint assessment purposes. Therefore, this research seeks to perform correlation analysis on the flammability characteristics of MCC and cone calorimetry with grey and red extruded polystyrene as the reference materials. In this study, Pearson's correlation coefficients between cone calorimetry and MCC parameters will be derived to evaluate their linear relationships. It attempts to show how MCC could be used in conjunction with cone calorimetry to accurately and reliably assess the flammability of materials.

Experimental

Material

The materials used for the experiments were red and grey extruded polystyrene (XPS) with 3% flame-retardant additives manufactured by Zheng bang Newly Building Material Co. Ltd. in China. The XPS was produced by extrusion of the polystyrene material used as a reference for calibration of most fire equipment. The properties of the XPS used are shown in Table 1.

The thermal properties of the materials were measured with a Hot Disk TPS 2500 s. The samples were taken from large XPS boards and weighed with a Mettler AX205 AX-205 Analytical Semi Micro Balance Delta Range with a readability of 0.01 mg and a weighing range of 81 g.

Microscale combustion calorimetry

The MCC experiment was conducted in accordance with the set guidelines in ASTM D7309-13 [2]. Milligram samples of red and grey extruded polystyrene were tested in an MCC-2 equipment from Govmark Limited at the VTT Technical Research Center of Finland. The masses of the samples of red XPS were in groups of 1 mg, 1.5 mg and 2 mg with standard errors of 0.043, 0.052, and 0.055 mg, respectively. Likewise, the grey XPS samples were prepared in three groups specifically 1.5 mg, 2.5 mg and 3.5 mg with 0.188, 0.101 and 0.159 mg as the standard errors. Twenty-seven samples were tested for each type of XPS.

The samples were heated at nine selected heating rates (β), thus, 0.1, 0.2, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, and 3.5 K s^{-1} under controlled conditions. The method A procedure for MCC experiments as described in [1, 2, 16, 20] was applied. Samples were pyrolyzed in nitrogen atmosphere and combusted in a high-temperature tubular furnace with a mixture of nitrogen and oxygen. The gases used for the experiment were Instrument Nitrogen 5.0, Chemical oxygen 3.5, and Instrument synthetic air 5.0 produced by AGA industrial gases, Finland. The temperature for pyrolysis and combustion was 75–600 °C and 900 °C, respectively. Oxygen consumption calorimetry was applied in calculating the heat release rate from the volumetric flow rate and oxygen concentration of the gases that flowed out of the combustor. During the experiment, three replicate prepared samples were tested for each polystyrene type and an average of the measured results was recorded. Samples were labeled according to their heating rates as xps_red_0.1_1 representing the first sample of red XPS tested under 0.1 K s^{-1}. The time to peak heat release rate, heat release capacity, temperature at peak heat release, char yield and total heat released were measured and recorded.

Table 1 Properties of XPS

Property	Red	Grey
Thermal conductivity/W m^{-1} K^{-1}	0.1316	0.1357
Thermal diffusivity/m^2 s^{-1}	0.4201	0.4401
Specific heat capacity/kJ g^{-1} K^{-1}	1.34	1.39
LOI/%	19.3	20.5
Compressive strength/kN m^{-2}	347	300
Density, ρ/kg m^{-3}	52.6	37.8
Density of molten material, ρ/kg m^{-3}	828	806

Cone calorimetry

The cone calorimeter experiment was performed at the State Key Laboratory of Fire Science in China. Samples of 100 ± 1 mm × 100 ± 1 mm × 20 ± 1 mm were cut from large red and grey extruded polystyrene (XPS) boards and prepared for the experiment. The bottom and sides of the samples were wrapped in aluminum foils, carefully covered to prevent the exposure of excess foils and placed in a specimen holder horizontally. The samples were heated in an open-centered conical heater, while the volatile combustion products flowed into an exhaust tube [8–10, 16]. Three replicate samples of XPS were tested under 25, 35, and 50 kW m^{-2} incident heat flux according to ISO 5660-1 [9]. Samples were labeled according to the tested incident heat flux and color as xps_red_25_1 representing the first red sample tested under 25 kW m^{-2}. To ensure accuracy of the experiment, each test was repeated three times and the average of the three results was recorded. Also, all required preliminary calibrations on the cone calorimeter were performed before conducting the experiments to ensure precision of measurements and readings. The test surface area for this experiment was 0.008848 m^2 as well as a sampling rate of 1 sample s^{-1}.

Pearson's correlation coefficient

Pearson's correlation coefficient is a statistical method used to measure the linearity of two variables [21]. The magnitude of the linear relationship is indicated by an R-value which ranges from − 1 to + 1. Positive linear relationships denote the variables increase or decrease simultaneously, whereas negative R-values signify a decrease in one parameter as the other increases [22]. The Pearson correlation coefficient (r_{xy}) of two variables X and Y having a series of measurements x_i and y_i where $i = 1, 2, \ldots, n$ is calculated by using Eq. (1) [23],

$$r_{xy} = \frac{n(\sum x_i y_i) - (\sum x_i)(\sum y_i)}{\sqrt{\left[n\sum x_i^2 - (\sum x_i)^2\right] - \left[n\sum y_i^2 - (\sum y_i)^2\right]}} \quad (1)$$

where n is the sample size, $(\sum x_i)$ and $(\sum y_i)$ are the sum of the X and Y variables, respectively. Pearson's correlation was used in this research to assess the effect of a change in a parameter over the other flammability characteristics in both MCC and cone calorimetry. The X and Y variables were the flammability characteristics of both methods. In relation to MCC, x_i and y_i were the measurements obtained at different heating rates, i.e., $i = 0.1, 0.2, \ldots, 3.5$. Similarly, with cone calorimetry, variables measured at three heat fluxes were used. Furthermore, this correlation method was applied in matching the parameters of MCC against cone calorimetry. The classification of the range of R-values and their corresponding magnitudes as used in this paper are presented in Table 2. A statistical analysis tool was used in deriving the Pearson correlation coefficients. A two-tailed test of significance was applied with all correlations being significant at the 0.05 level.

Results and discussion

Analysis of MCC data

The parameters used for the MCC analysis in this research were the peak heat release rate (pHRR), heat release capacity (η_c), temperature at pHRR (pTemp), total heat release (THR) and char yield. Results interpretation of MCC data requires some parameters to be calculated from the basic ones that are measured from the experiment. According to method A from MCC standards in [2] the heat release capacity, char yield and total heat released were derived using the equations shown.

η_c describes the thermal stability of a material and it represents the average heat released in the combustion stage per change in temperature [2, 20, 21].

Table 2 The range of R-values and their corresponding magnitudes of correlation

Range of R-value	Magnitude of correlation
0.00–0.19	Very weak
0.20-0.39	Weak
0.40–0.59	Moderate
0.60–0.79	Strong
0.80–1.00	Very strong

$$\eta_c = \frac{Q_{max}}{\beta} \quad (2)$$

where Q_{max} = pHRR/W g^{-1} and β = average heating rate over the measurement range/K s^{-1}.

Char yield represents the mass ratio of the sample which does not undergo combustion in the flame zone [2, 10]. It is defined as the mass of residue (m_p/g) divided by initial mass of sample (m_o/g).

$$\text{Char yield} = \frac{m_p}{m_o} \times 100 \quad (3)$$

THR signifies the heat output at a specific heat release rate and time (t) [10]. It is characterized by the area under the HRR versus time graph.

$$\text{THR} = \int \frac{Q_{max}}{t} \mathrm{d}t \quad (4)$$

Generally, the flammability of materials is characterized by the amount of heat released when the material is exposed fire. Therefore, the most important characteristic that is measured from an MCC experiment is HRR [24]. Specific heat release rates at nine heating rates were plotted against their respective temperatures for red and grey XPS in Figs. 1 and 2, respectively. It is clearly seen in Figs. 1 and 2 that the peaks of the curves representing the peak heat release rate and their respective temperatures increase as heating rate increases. Comparing the two figures revealed that the temperature at peak heat release rate is almost similar for the two materials at the same heating rates. On the other hand, it was observed that red XPS achieved higher specific heat release rate than grey XPS at the same heating rate. This confirms what has been done in literature by Xu et al. [16]. The results obtained for testing 1.5 mg samples of red and grey XPS are presented in Tables 3 and 4.

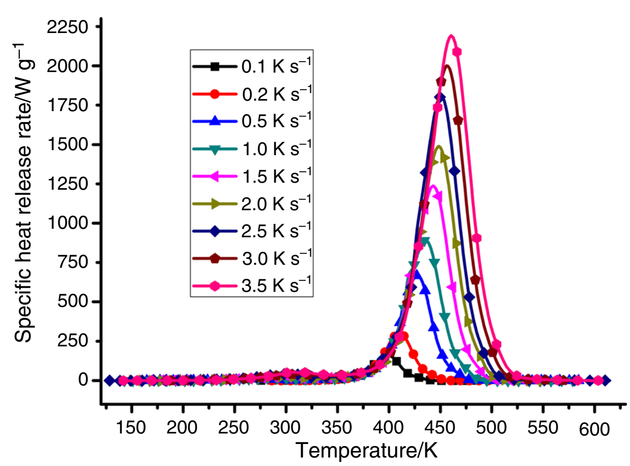

Fig. 1 Graph of specific heat release rate against temperature for different heating rates for red XPS

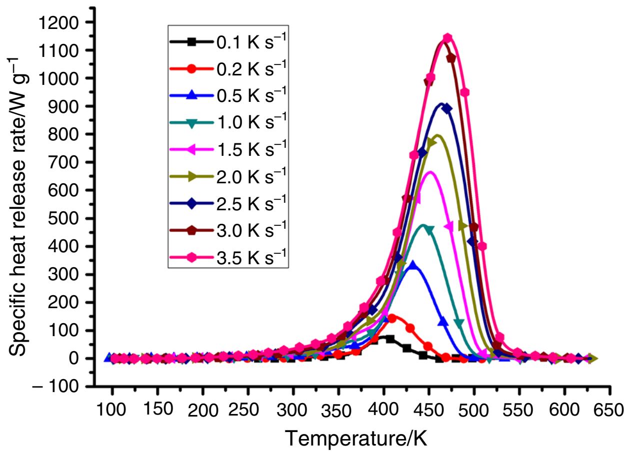

Fig. 2 Graph of specific heat release rate against temperature for different heating rates for grey XPS

Tables 5 and 6 show the *R*-values derived from the Pearson correlation between the MCC parameters obtained for red and grey XPS. The results from the tables clearly show that pHRR had a very strong negative linear relationship (− 0.95 and − 0.98) with heat release capacity. For most polymeric materials, pHRR increases, while η_c decreases with increasing heating rate [19, 20]. This explains the strong negative values presented. pHRR increases with an increase in pTemp and it is evidently shown by a strong positive relationship (0.96 for red and 0.97 for grey) for pHRR/pTemp.

It is worth noting that *R*-values observed for THR and char yield differed for both materials. pHRR had a strong relationship with THR for grey and a weak relationship for red XPS. Char yield for red XPS presented strong linear relationships with all the sets with the exception of THR (− 0.48). On the other hand, the char yield for grey XPS showed very weak linear relationships for pTemp, pHRR and η_c. A moderate relationship (− 0.48) was recorded for char yield/THR in Table 6. From the analysis, similar *R*-values and significance were seen in the pairs of char yield/THR for both materials. The independence of char yield on THR could be due to the forced complete combustion of pyrolysis products from the MCC experiment.

Table 3 Results of testing 1.5 mg red XPS with MCC

β/K s^{-1}	pHRR/W g^{-1}	η_c/J g^{-1} k^{-1}	THR/kJ g^{-1}	pTemp/°C	Char yield/mass%
0.1	163 ± 9	1630 ± 62.4	32.4 ± 0.07	397.9 ± 9.9	0.68 ± 1.1
0.2	300.6 ± 15.58	1503 ± 33.8	31 ± 1.5	410.4 ± 1.3	5.92 ± 3.7
0.5	668 ± 31.56	1336 ± 18.2	34.5 ± 1.8	428.2 ± 6.3	1.45 ± 1.2
1.0	948.8 ± 25.4	948.8 ± 67.3	33.2 ± 0.5	435.8 ± 6.1	2 ± 2.2
1.5	1236.5 ± 54.1	824.3 ± 65.1	33.3 ± 0.5	442.9 ± 5.2	3.45 ± 1.7
2.0	1488.4 ± 63.2	744.2 ± 57.8	32.1 ± 0.7	447.8 ± 3.1	10.14 ± 4.2
2.5	1809 ± 64.8	723.6 ± 21.3	33 ± 0.08	451 ± 2.6	5.88 ± 1.06
3.0	1999.8 ± 93	666.6 ± 5.5	32.8 ± 0.2	455.7 ± 3.2	8.05 ± 0.5
3.5	2192 ± 85	626.3 ± 50.9	32.5 ± 0.5	460.5 ± 3.7	7.80 ± 0.3
Mean	1200.7	1000.3	32.8	436.7	5.04
σ	733.95	385.5	0.96	21.1	3.31

Table 4 Results of testing 1.5 mg grey XPS with MCC

β/K s^{-1}	pHRR/W g^{-1}	η_c/J g^{-1} k^{-1}	THR/kJ g^{-1}	pTemp/°C	Char yield/mass%
0.1	79.2 ± 5.8	792 ± 12.8	25.5 ± 0.02	402 ± 7	11.56 ± 0.08
0.2	149.5 ± 9.2	747.5 ± 3.9	24.1 ± 1.5	412.4 ± 0.5	17.07 ± 5.5
0.5	339 ± 11.8	678 ± 4.1	25.3 ± 0.6	432.1 ± 8.3	9.84 ± 2
1.0	533 ± 23.3	533 ± 30	26.7 ± 0.3	443.2 ± 3.9	13.7 ± 1.4
1.5	728.7 ± 22.1	485.8 ± 10.3	30.1 ± 3.3	451 ± 0.4	1.99 ± 1.7
2.0	806.4 ± 26.3	403.2 ± 7.6	27.4 ± 0.09	456.3 ± 3.7	13.14 ± 0.06
2.5	977.5 ± 31.7	391 ± 21.7	27.4 ± 0.4	462.1 ± 3.1	18.24 ± 4.5
3.0	1062.3 ± 28.9	354.1 ± 3.3	27.8 ± 0.4	467.6 ± 0.6	16.29 ± 2.2
3.5	1206.6 ± 36.8	344.74 ± 10.6	28 ± 0.7	473.4 ± 2	13.64 ± 0.9
Mean	653.6	525.5	26.9	443.8	12.83
σ	403.78	173.3	1.77	23.81	4.86

Table 5 Correlation between MCC parameters for red XPS

	pHRR/W g^{-1}		η_c/J g^{-1} k^{-1}		THR/kJ g^{-1}		pTemp/°C		Char yield/mass%	
	R	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.
pHRR	1.00	–								
η_c	**− 0.95**	**7.01×10^{-5}**	1.00	–						
THR	0.12	0.76	− 0.16	0.69	1.00	–				
pTemp	**0.96**	**2.57×10^{-5}**	**− 0.98**	**5.98×10^{-6}**	0.24	0.53	1.00	–		
Char yield	0.69	0.04	− 0.65	0.058	− 0.48	0.19	0.65	0.06	1.00	–

Bold indicates the strongest correlations and corresponding significance

Table 6 Correlation between MCC parameters for grey XPS

	pHRR/W g^{-1}		η_c/J g^{-1} k^{-1}		THR/kJ g^{-1}		pTemp/°C		Char yield/mass%	
	R	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.
pHRR	1.00	–								
η_c	**− 0.98**	**3.15×10^{-6}**	1.00	–						
THR	0.73	0.02	− 0.75	0.02	1.00	–				
pTemp	**0.97**	**1.22×10^{-5}**	**− 0.98**	**1.36×10^{-6}**	0.74	0.02	1.00	–		
Char yield	0.14	0.71	− 0.13	0.72	− 0.48	0.19	0.087	0.82	1.00	–

Bold indicates the strongest correlations and corresponding significance

Analysis of cone calorimetry data

In analyzing the cone calorimetry test, the parameters used were peak heat release rate (q_{max}), ignition temperature (T_{ig}), time to ignition (t_{ig}), THR, fire growth rate (FIGRA) and fire performance index (FPI). FIGRA and FPI were derived from the basic parameters measured from the experiment. There was no residue left in this test so char yield was not included in the evaluation.

FIGRA is defined by pHRR divided by the time to peak heat release rate. In flammability analysis, a better flame resistance ability is signified by low FIGRA values [9, 16].

$$\mathrm{FIGRA} = \frac{q_{max}}{t_{pHRR}} \quad (5)$$

FPI is the time to peak heat release rate divided by peak heat release rate. Higher FPI values denote better flame resistance [16].

$$\mathrm{FPI} = \frac{t_{pHRR}}{q_{max}} \quad (6)$$

The values of fire performance index of red and grey XPS for the cone calorimetry experiment were plotted against time to ignition in Figs. 3 and 4. The figures show three samples for each incident heat flux tested. From the figures, fire performance index and time to ignition decreases as the incident heat flux increases. Thus, at high incident heat flux, low fire performance and shorter ignition times were recorded. Therefore, it can be deduced from the two plots that rapid ignition produces low fire performance and vice versa. Both materials exhibit good flame resistance and longer time to ignition at low incident heat flux as seen in most polymers.

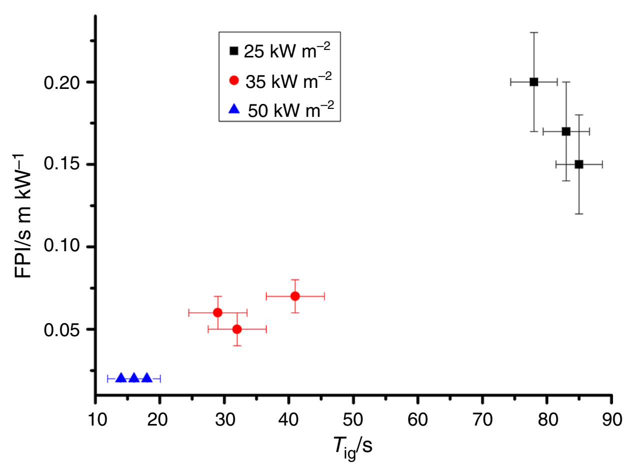

Fig. 3 Plot of FPI versus t_{ig} for different incident heat flux for red XPS

Also, shown in Figs. 5 and 6 are plots of fire growth rate against the time to ignition at 25, 35 and 50 kW m^{-2} incident heat flux of red and grey XPS. From the graph illustrated, FIGRA increases with an increase in incident heat flux, while time to ignition decreases with increasing

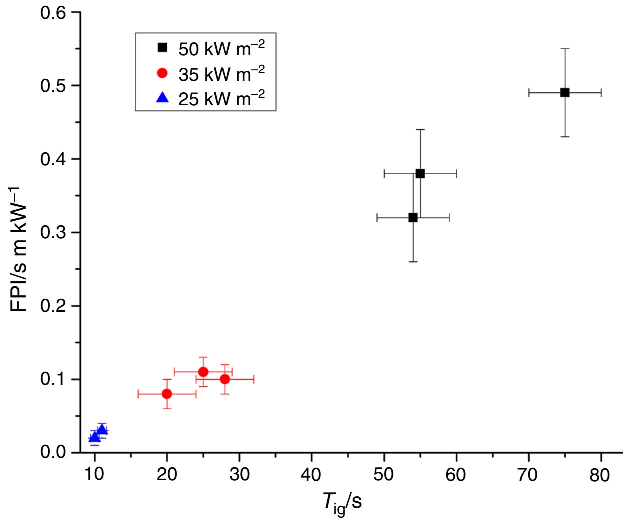

Fig. 4 Plot of FPI versus t_{ig} for different incident heat flux for grey XPS

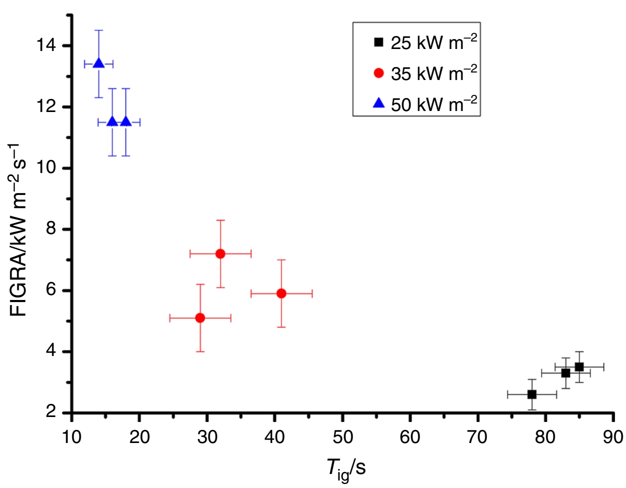

Fig. 5 Plot of FIGRA against t_{ig} different incident heat flux for red XPS

incident heat flux. Therefore, fire growth rate reduces at longer ignition times and vice versa.

To determine whether the foam used for the experiment was thermally thin or thick the Biot's number for the material was calculated using Eq. (7) [25];

$$\mathrm{Bi} = \frac{L_c h}{k} \tag{7}$$

where Bi is the Biot's number with a material having Bi > 0.1 being thermally thick as used in [25], L_c represents the characteristic length defined by the volume of a material per its area, h is the heat transfer coefficient and k, the thermal conductivity of the extruded polystyrene. With a Biot number of 0.6, the XPS material was found to be thermally thick; therefore, temperature varied throughout the material.

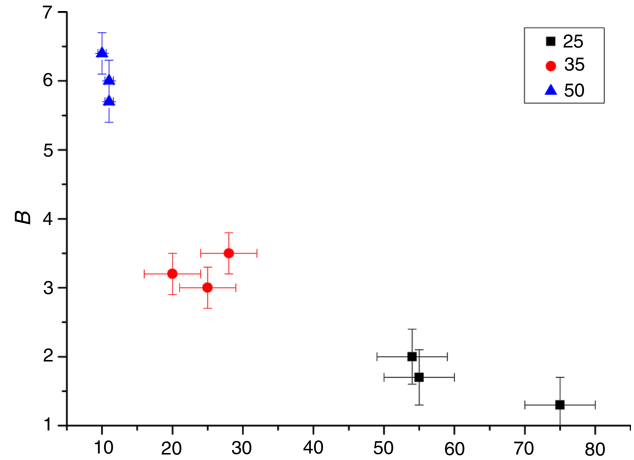

Fig. 6 Plot of FIGRA against t_{ig} different incident heat flux for grey XPS

To compare temperature at peak heat release rate from MCC and cone calorimetry, it was imperative to consider the surface temperature gradients during pyrolysis for the samples tested. Thermally thick materials undergo convection, radiation and conduction processes [26–28]. Thus, all these processes must be accounted for to obtain the surface temperature. Also, the thermal inertia ($k\rho c$) of XPS play a key role in acquiring the surface temperature [29, 30]. The higher the thermal inertia of a material the lower the ignition temperature. Where k is the thermal conductivity of extruded polystyrene, k = 0.1316 W m^{-1} K^{-1} for red XPS and 0.1357 W m^{-1} K^{-1} for grey XPS, ρ is the density of the molten red and grey XPS (828 kg m^{-3} for red and 806 kg m^{-3} for grey) and c represents the specific heat capacity which is 1.34 kJ kg^{-1} K^{-1} for polystyrenes. To predict the ignition temperature for the experiment Eq. (8) from [29] and Eq. (9) are used.

$$(k\rho c) = \frac{4}{\pi}\left[\frac{q_{in}}{\Delta T_{ig}}\right]^2 \times t_{ig} \tag{8}$$

Ignition of materials in flammability experiments occurs after pyrolysis. Hence, the ignition temperature is the pyrolysis temperature in addition to any increment made toward ignition.

$$T_{ig} = \Delta T_{ig} + T_{py} \tag{9}$$

where T_{ig} is the ignition temperature, T_{py} represents the pyrolysis temperature (397 °C for red XPS and 398 °C for grey XPS [16]) and q_{in} is the incident heat flux applied. Tables 7 and 8 show the measured and calculated cone test data for red and grey XPS.

Correlations were conducted on parameters measured and derived from the cone calorimetry test of red and grey XPS in Tables 9 and 10. From the results, q_{max} had strong

Table 7 Results of testing red XPS with cone calorimetry

Sample	q_{max}/W g^{-1}	t_{ig}/s	THR/MJ g^{-1} [16]	T_{ig}/°C	FIGRA/kW m^{-1} s^{-1} [16]	FPI/S m kW^{-1} [16]
25_1	483.45	83	25.8	418.27	3.3	0.17
25_2	386.18	78	26.6	417.62	2.6	0.2
25_3	552.88	85	25.0	418.52	3.5	0.15
Mean ± σ	474.2 ± 83.7	82 ± 3.6	25.8 ± 0.8	418.13 ± 0.5	3.2 ± 0.5	0.17 ± 0.03
35_1	672.57	32	23.6	415.49	7.2	0.05
35_2	527.41	36	24.1	416.61	5.1	0.06
35_3	622.68	41	24.8	417.93	5.9	0.07
Mean ± σ	607.6 ± 73.8	36.3 ± 4.5	24.2 ± 0.6	416.7 ± 1.2	6.1 ± 1.1	0.06 ± 0.01
50_1	818.91	20	25.0	417.88	11.5	0.02
50_2	994.83	17	24.6	416.25	13.4	0.02
50_3	756.04	16	24.6	415.68	11.5	0.02
Mean ± σ	856.6 ± 123.8	17.7 ± 2.1	24.7 ± 0.3	416.6 ± 1.14	12.1 ± 1.1	0.02

Table 8 Results of testing grey XPS with cone calorimetry

Sample	q_{max}/W g^{-1}	t_{ig}/s	THR/MJ g^{-1} [16]	T_{ig}/°C	FIGRA/kW m^{-1} s^{-1} [16]	FPI/S m kW^{-1} [16]
25_1	165.14	54	27.1	415.38	2.0	0.32
25_2	145.64	55	26.4	415.55	1.7	0.38
25_3	149.69	75	24.8	418.49	1.3	0.49
Mean ± σ	153.5 ± 10.3	61.3 ± 5	26.1 ± 1.2	416.5 ± 1.7	1.7 ± 0.4	0.4 ± 0.06
35_1	203.39	25	24.9	414.56	3	0.11
35_2	246.56	28	25.7	415.53	3.5	0.10
35_3	202.52	20	26.0	412.81	3.2	0.08
Mean ± σ	217.5 ± 25.2	24.3 ± 4	25.5 ± 0.6	414.3 ± 1.4	3.2 ± 0.3	0.1 ± 0.02
50_1	359.65	11	23.9	413.69	6.0	0.03
50_2	292.29	10	24.1	412.96	6.4	0.02
50_3	289.10	11	24.8	413.69	5.7	0.03
Mean ± σ	313.7 ± 39.8	10.7 ± 0.6	24.3 ± 0.5	413.5 ± 0.4	6 ± 0.3	0.03 ± 0.01

Table 9 Correlations between cone calorimetry parameters for red XPS

	q_{max}/W g^{-1}		t_{ig}/s		THR/kJ g^{-1}		T_{ig}/°C		FIGRA/kW m^{-1} s^{-1}		FPI/S m kW^{-1}	
	R	Sig.	R	Sig.	R	Sig.	R	Sig.	R	Sig.	R	Sig.
q_{max}	1.00	–										
t_{ig}	− 0.81	0.0085	1.00	–								
THR	− 0.49	0.18	0.65	0.06	1.00	–						
T_{ig}	− 0.70	0.22	0.70	0.03	0.65	0.06	1.00	–				
FIGRA	**0.96**	**4.63 × 10^{-5}**	− 0.89	0.0014	− 0.44	0.24	− 0.54	0.12	1.00	–		
FPI	− 0.84	0.005	**0.97**	**1.6 × 10^{-5}**	0.76	0.02	0.63	0.06	− 0.87	0.002	1.00	–

Bold indicates the strongest correlations and corresponding significance

correlations with all parameters except THR of red XPS. Correlation between FPI and q_{max} was − 0.84 and − 0.83 for red and grey, respectively, since high peak heat release rates demonstrates low fire performance. It is interesting to note that FIGRA and FPI showed very strong negative correlations proving the fact that high FIGRA values also represents a reduced fire performance. Similarly, t_{ig} had very strong negative relationships with FIGRA and rather strong positive correlations with T_{ig} and FPI. Furthermore, an increase in t_{ig} depicts a better fire performance index

Table 10 Correlations between cone calorimetry parameters for grey XPS

	q_{max}/W g^{-1}		t_{ig}/s		THR/kJ g^{-1}		T_{ig}/°C		FIGRA/kW m^{-1} s^{-1}		FPI/S m kW^{-1}	
	R	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.
q_{max}	1.00	–										
t_{ig}	− 0.84	0.0045	1.00	–								
THR	− 0.72	0.02	0.51	0.16	1.00	–						
T_{ig}	− 0.62	0.07	**0.90**	**9.9 × 10^{-4}**	0.22	0.56	1.00	–				
FIGRA	**0.95**	**1.02 × 10^{-4}**	− 0.89	0.001	− 0.70	0.03	− 0.74	0.02	1.00	–		
FPI	− 0.83	0.006	**0.99**	**1.62 × 10^{-7}**	0.48	0.19	0.87	0.002	− 0.86	0.003	1.00	–

Bold indicates the strongest correlations and corresponding significance

shown by a correlation coefficient of 0.97 and 0.99. All the pairs from the cone calorimetry experiment either had strong or very strong linear correlations with the exception of the set of THR correlations. The weakest correlations were recorded between pairs of THR/T_{ig} and THR/FIGRA.

Correlations between MCC and cone calorimetry parameters

To establish relationships between the cone calorimeter and MCC experiments, correlations were conducted on the parameters from both methods for red and grey XPS. From the correlations presented in Tables 11 and 12, pHRR, pTemp and η_c had strong positive and negative linear relationships with all the cone calorimetry parameters analyzed except THR and T_{ig}. THR and char yield from MCC did not show strong correlations with any of the parameters which could likely be the forced complete combustion in MCC. Also, considering correlations between THR from both methods, a weak correlation was recorded for grey XPS, while a moderate *R* was obtained for red XPS. The difference in correlations between the THR pair can be attributed to the different char formation ability in grey and red XPS which resulted in higher THR values for red XPS in the MCC experiment. The incomplete combustion in cone calorimetry being compared with the complete non-flaming combustion in MCC could possibly be the reason for the poor correlations.

From the ongoing discussion it can be deduced that almost all the parameters thus, pHRR, pTemp and η_c from MCC correlates very well with most of the parameters from cone calorimetry. In view of this MCC experiment and cone calorimetry in some degree can be used together to assess flammability of materials.

Additionally, an analysis between pHRR and pTemp as well as q_{max} and T_{ig} were made for both grey and red XPS. Graphs are drawn in Figs. 7 and 8 between these parameters to show how the results from MCC differed from the cone test. From the plots, it was observed that the cone test achieved moderate temperatures and peak heat release rates at the various incident fluxes applied as compared to the MCC results at nine heating rates. The data from the cone test fell within 0.2 K s^{-1} and 0.5 K s^{-1} heating rates of MCC for both red and grey XPS.

With this analogy, a model for predicting q_{max} was derived using linear regression. A very strong correlation (0.84 for grey and 0.86 for red XPS) was observed between q_{max} and pHRR indicating a strong relationship between the two parameters. In view of this, a linear regression analysis was conducted between pHRR values of MCC at

Table 11 Correlations between cone calorimetry and MCC parameters for red XPS

	pHRR/W g^{-1}		η_c/J g k^{-1}		THR/kJ g^{-1}		pTemp/°C		Char yield/mass%	
	R	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.
q_{max}	0.84	0.004	− 0.77	0.02	0.24	0.53	0.78	0.01	0.46	0.21
t_{ig}	**0.95**	**5.3 × 10^{-4}**	**0.95**	**9.7 × 10^{-5}**	− 0.04	0.91	− 0.89	0.001	− 0.56	0.11
THR	− 0.52	0.14	0.69	0.04	− 0.59	0.09	− 0.65	0.06	0.01	0.97
T_{ig}	− 0.51	0.16	0.57	0.11	0.016	0.97	− 0.52	0.14	− 0.19	0.63
FIGRA	**0.90**	**7.9 × 10^{-4}**	− 0.81	0.008	0.09	0.81	0.82	0.007	0.53	0.14
FPI	**0.91**	**4.6 × 10^{-4}**	**0.96**	**5.2 × 10^{-5}**	− 0.26	0.49	**− 0.92**	**4.9 × 10^{-4}**	− 0.46	0.21

Bold indicates the strongest correlations and corresponding significance

Table 12 Correlations between cone calorimetry and MCC parameters for grey XPS

	pHRR/W g^{-1}		η_c/J g k^{-1}		THR/kJ g^{-1}		pTemp/°C		Char yield/mass%	
	R	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.	*R*	Sig.
q_{max}	0.86	0.003	− 0.83	0.006	− 0.57	0.11	0.81	0.009	0.24	0.52
t_{ig}	− 0.87	0.002	**0.9**	**8.4×10^{-4}**	0.29	0.44	− 0.82	0.006	− 0.25	0.51
THR	− 0.71	0.03	0.69	0.04	− 0.36	0.35	− 0.77	0.01	− 0.31	0.4
T_{ig}	− 0.67	0.05	0.72	0.03	0.25	0.51	− 0.59	0.09	− 0.45	0.23
FIGRA	**0.91**	**6.7×10^{-4}**	**− 0.87**	**0.002**	− 0.67	0.05	0.84	0.005	0.34	0.37
FPI	− 0.86	0.03	**0.90**	**0.001**	0.24	0.54	− 0.83	0.006	− 0.14	0.72

Bold indicates the strongest correlations and corresponding significance

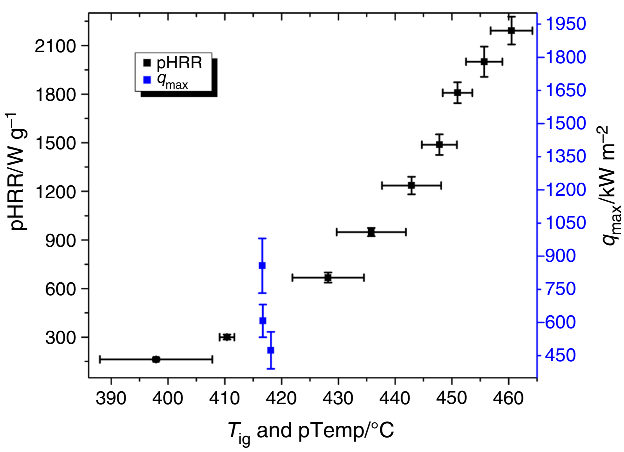

Fig. 7 Comparison of pHRR and pTemp with q_{max} and T_{ig} for red XPS

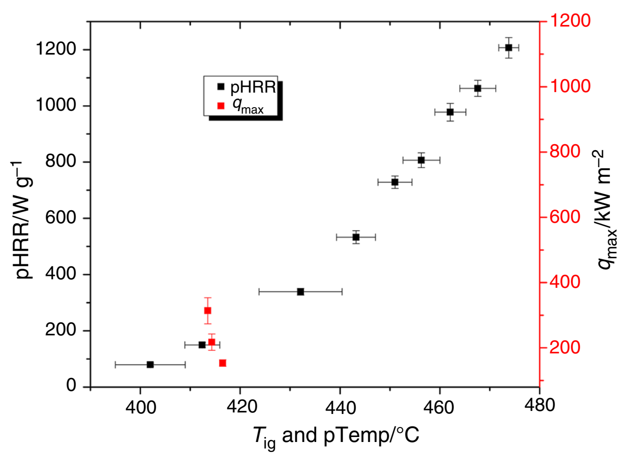

Fig. 8 Comparison of pHRR and pTemp with q_{max} and T_{ig} for grey XPS

the heating rates of 0.2 and 0.5 K s^{-1} and the averages of the cone calorimeter test data for each heat flux. This was done to obtain the regression equation in the form $y = ax + b$, where y is the dependent variable (q_{max}), a is the slope of the line, b is the intercept and x is the

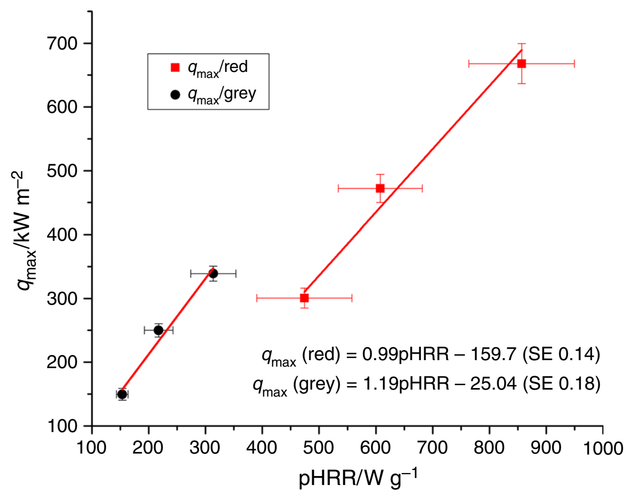

Fig. 9 Regression analysis of pHRR and q_{max} for red and grey XPS

independent variable (pHRR) for both red and grey XPS (Fig. 9). The prediction equation obtained for red XPS was $q_{max} = 0.99\text{pHRR} - 159.7$ and the model for grey XPS was $q_{max} = 1.18\text{pHRR} - 25.04$.

Conclusions

Correlations between flammability experiments are conducted to evaluate the dependence and linear relationship between the parameters. In this research, correlations were made between the results obtained from cone calorimetry and MCC experiments of red and grey samples of extruded polystyrene. Strong correlations were observed by most of the flammability characteristics analyzed. Averagely, cone calorimetry parameters displayed the strongest correlations for both materials. The weakest correlation in MCC was recorded by THR and pHRR pair. Also, correlating MCC characteristics with cone calorimetry results produced moderate and weak correlations between the two THR parameters for red and grey XPS, respectively. Analyzing the q_{max}, T_{ig}, pTemp and pHRR plots for the two methods showed that cone calorimetry data of both red and grey XPS at 25, 35 and 50 kW m^{-2} fell within 0.2 and 0.5 K s^{-1} heating rates of MCC.

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