



Test Method

The rapid mass calorimeter: Understanding reduced-scale fire test results



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ABSTRACT

The effects of reducing specimen size on the fire behavior of polymeric materials were investigated by means of the rapid mass calorimeter, a high-throughput screening instrument. Results from the rapid mass calorimeter were compared with those from the cone calorimeter. Correlation coefficients between the different measures of each method and between the two methods are discussed to elucidate the differences and similarities in the two methods. Materials with characteristic heat release rate (HRR) curves in the cone calorimeter were evaluated in detail. The rapid mass calorimeter produces valuable and interpretable results with HRR curve characteristics similar to cone calorimeter results. Compared to cone calorimeter measurements, material savings of 96% are achieved, while maintaining the advantages of a macroscopic fire test.

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1. Introduction

Modern polymeric materials are multicomponent systems. Many variables must be considered during the development of a novel material. Additives, plasticizers [1–3], adjuvants, synergists [4,5] and so forth can be varied in kind, concentration, particle size distribution [6,7] and other parameters, yielding an extensive matrix of possible systems. To assess the fire performance of these formulations and to find the best performing material, high throughput screening methods are essential. Until now, the performance of such materials in terms of fire behavior under flaming conditions has been evaluated with the cone calorimeter. For fast evaluation of fire performance, the pyrolysis combustion flow calorimeter (PCFC, or microscale combustion calorimeter, MCC) has been proposed [8]. While the cone calorimeter is a fire test using 100 mm × 100 mm plates, the latter performs fast screening of the pyrolysis of specimens on the milligram scale. Macroscopic modes of action like the formation of protective layers, dripping, wicking and so forth, as well as flame inhibition, cannot be observed in the PCFC [9]. There is a pronounced dilemma between proper fire testing based on macroscopic specimen and accelerated testing demanding reduction of the specimen size. The rapid mass calorimeter aims to reduce specimen size while maintaining fire testing

of a macroscopic specimen. The rapid mass calorimeter has been proposed and discussed in a previous publication [10]. Selected according to their characteristic heat release rate (HRR) curve shapes in the cone calorimeter, various sets of materials were evaluated in the rapid mass calorimeter, and the results compared with those from the cone calorimeter to assess the value of rapid mass calorimeter testing.

Scale reduction is crucial if the forced-flaming combustion test is to be accelerated [11]. The rapid mass calorimeter should still be operated on a macroscopic scale, limiting the minimum size to which the specimens can be reduced. Changes in length scale and thickness of specimens alter the obtained fire properties dramatically [12–15]. Modes of action like heat and fuel transport barriers created from inert filler or from an intumescent system also showed different performance [10]. Further flame retarding effects were not as distinct in the rapid mass calorimeter as in the cone calorimeter. Therefore, the task is to reveal and understand the reasons for this divergent behavior. For this, correlation coefficients derived from the heat release rate (HRR) of the rapid mass calorimeter and cone calorimeter results are elucidated; as well as the correlation coefficients of the results within each method. The differences in correlation strength between the methods are discussed, explaining effects that occur when specimen size is reduced to 20 mm × 20 mm. A set of 73 different materials was used, including a large number of flame retarded materials. Several characteristic materials were selected to discuss the reduced size effect in detail, particularly with respect to flame retardancy.

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2. Experimental

2.1. Rapid mass calorimeter

The rapid mass calorimeter consists of a mass loss calorimeter (Fire Testing Technology, UK) equipped with a chimney with thermoelements to record the heat release rate (HRR) according to ISO 13927 [16]. The balance has been replaced with a linear motion unit (Oriental Motor, JP) to facilitate semi-automatic sample exchange. The 20 mm × 20 mm samples were wrapped in an aluminum tray and placed on the center of the sample holder. The distance from sample surface to cone heater was 25 mm and the heat flux was 50 kW/m². The setup and method were described in detail in a previous publication [10].

2.2. Cone calorimeter

Forced-flaming combustion tests were performed with a cone calorimeter (FTT, UK). Specimens 100 mm × 100 mm in size and encased in an aluminum tray were irradiated with a heat flux of 50 kW/m² at a distance of 25 mm. Thicknesses of the samples ranged from 3 mm to 10 mm. No retainer frame was used [17].

2.3. Correlation analysis

The results from both methods, rapid mass calorimeter and cone calorimeter, were checked for their Pearson R correlation coefficients, within each method and also between the two methods. Pearson R values can range from ±1 for perfect linear correlation to 0 for no correlation at all, as seen in Table 1 [18].

2.4. Materials

In total, 73 different polymeric materials (Table 2) were measured in the cone calorimeter and the rapid mass calorimeter. From those materials certain systematic series were selected for further evaluation and comparison. All investigated systems originate from earlier projects and were provided by partners with high compounding and processing competence [9,19–30].

3. Results and discussion

3.1. Comparison of correlation coefficients within each method

The rapid mass calorimeter allows for accelerated screening of flame retarded polymeric materials. The HRR is measured via the voltage difference in the thermopile, and mass is recorded before and after the test with a separate balance. The test yields similar measures as the cone calorimeter test. To compare the character of both methods, correlation coefficients are evaluated either between the measures of each method or between the measures of both methods.

Tables 3 and 4 clarify the similarity of both methods by providing correlation coefficients of the HRR measurement results.

Results that correlate strongly in the cone calorimeter also show strong correlation in the rapid mass calorimeter. In general, all of the tendencies of the correlation coefficients are the same. This similar correlation pattern proves that the rapid mass calorimeter and cone calorimeter are strongly related tests. The time to ignition shows no significant correlation at all, no matter the method with which the results were obtained, because t_{ig} is not directly related to any of the other fire properties. A flame retardant can achieve a reduction of, e.g. PHRR or THE and prolong the time of burning, but must not necessarily prolong the time to ignition of the material.

At first glance, rapid mass calorimeter HRR curves show a slope, a peak and a decay. The most important characteristic of a peak is its peak height. Flame retardants incorporated in a polymeric material serve to lower the peak height. PHRR and THE do not correlate well with each other, which leads to the conclusion that added flame retardants change more than just the peak height of the HRR curve. In fact, HRR curves obtained with the rapid mass calorimeter show the same or similar characteristics as HRR curves from the cone calorimeter and thus can be interpreted similarly.

Values that show about the same correlation in both methods are MARHE ↔ THE, PHRR ↔ HRRavg and MARHE ↔ FIGRA. The relation between MARHE and HRRavg shows strong correlation with a coefficient of $R = 0.90$ in the rapid mass calorimeter (Fig. 1), and is about the same strength in the cone calorimeter ($R = 0.97$). This similarity shows that MARHE and HRRavg proportions are mainly unaffected by specimen size and method variation. Specimen size does not seem to alter the relation between these two measures very much.

Those averaged measures are also among the few that show any moderate to strong correlation in the rapid mass calorimeter. They correlate well mainly with PHRR, FIGRA and with each other. When HRRavg is calculated from rapid mass calorimeter HRR data, it will always result in a value dependent on burning duration and PHRR. This explains the strong correlation coefficient values with PHRR ($R = 0.90$) and FIGRA ($R = 0.83$). These measures are also among the values that change most obviously in the rapid mass calorimeter results when a flame retardant is integrated or its content varied in a polymeric material. Averaging the value of the HRR from the time of ignition to flameout (HRRavg) is a tool to combine the complete burning behavior into a single value (see Fig. 2).

Correlations between FIGRA and PHRR as well as FIGRA and HRRavg are among the strongest when comparing measures derived from the rapid mass calorimeter. Increasing peak height due to heavily burning specimens often results in a steeper slope of the recorded HRR curve. If a sample shows high flame retardant performance, resulting in a low PHRR, the slope of the curve will also be less steep. However, if the burning behavior of the investigated specimen results in a HRR curve that shows not a distinctive peak, but a rather wide plateau, this relation no longer applies. A good correlation of HRR results over all tested materials is statistical evidence for the significance of measurements with the rapid mass calorimeter. Correlation between measurement results of specific, coherent specimens are stronger, and show more clearly that changes in HRR values are consistent among sample series with similar characteristic curve shapes. Such stronger correlations due to structure-property relationships are depicted for four different sample series in Fig. 3. The sample sequence consisting of glass fiber reinforced PA66 with and without red phosphorus as a char-inducing flame retardant shows the strongest correlation between FIGRA and PHRR, exhibiting a correlation coefficient of $R = 0.99$. Formulations with APP in PP also show a strong dependence of both values, while specimens with MDH and TRGO in PP correlate only marginally for the FIGRA and PHRR. The correlation in the PCABS sample series is weak at $R = 0.32$. The single outlier that lowers the correlation between FIGRA and PHRR was PCABS-

Table 1
Correlation strength definition for the Pearson correlation coefficient R.

Correlation Strength	Pearson R
Poor	$ R < 0.40$
Weak	$0.40 \leq R < 0.55$
Marginal	$0.55 \leq R < 0.70$
Moderate	$0.70 \leq R < 0.85$
Strong	$0.85 \leq R $

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