

Some comments on the use of cone calorimeter data

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Abstract

The cone calorimeter has become one of the most important and widely used instruments for the research and development of fire retarded polymeric materials. The paper addresses three important ways in which the principal setup influences the results — factors which sometimes do not receive due consideration when drawing conclusions. The paper discusses in detail the impact on cone calorimeter results of the choice of external heat flux, the influence on the peak of heat release rate of sample thickness and thermal feedback from the back of the sample, and the influence on irradiance of the horizontal and vertical distances from the cone heater.

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1. Introduction

The cone calorimeter is a performance-based bench scale fire testing apparatus [1,2]. Sample plates 100 mm × 100 mm in size are investigated under forced-flaming conditions [3]. The sample size is of the smallest order of magnitude discussed in fire engineering and of the largest used in polymer analysis. Hence, the cone calorimeter constitutes an important link between fire engineering and polymer science, which is crucial in the interdisciplinary area of fire science. Furthermore, it provides comprehensive insight into not only fire risks such as heat release rate, total heat release, and time to ignition, but also fire hazards such as smoke release and CO production. The cone calorimeter setup [4] was developed thoroughly to target the properties of materials [5] rather than to correspond to a special full-scale scenario of a real fire. Cone calorimeter investigations

can be used as a universal approach to ranking and comparing the fire behaviour of materials. Therefore, it is not surprising that the cone calorimeter is finding increasing implementation as a characterization tool in the research and development of fire retarded polymeric materials.

However, each experimental setup defines a specific fire scenario. As is typical for all fire tests, samples' performance in the cone calorimeter depends on the specific characteristics of the test, including ignition source, ventilation, irradiance (external heat flux), temperature, and the geometry of the specimen. Strictly speaking, the cone calorimeter test characterizes the performance resulting from an interaction of material properties, specimen, and the defined fire scenario. The meaning of the results may have little relevance for other fire scenarios or fire tests that differ in their essential setup. Some of the crucial setup characteristics are obvious, such as horizontal sample positioning, melt dripping prevention, and well-ventilated combustion, and the effects of these characteristics on the results are well known. However, some cone calorimeter characteristics are less obvious and are sometimes neglected

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detrimentally in performing cone calorimeter tests or discussing their results. The author was encouraged to submit this paper as a consequence of discussions on cone calorimeter experiments and data at the ACS meeting in Fall 2004. This paper addresses three different points concerning the performance and evaluation of cone calorimeter tests on polymeric materials.

2. Experimental

A cone calorimeter (Fire Testing Technology, East Grinstead, UK) was used in accordance with ISO 5660 [6]. Samples were measured in the horizontal position using the retainer frame. The data were evaluated using the decreased surface area of the sample (88.36 cm^2). Temperatures were measured using NiCr–Ni thermocouples of type K. Irradiances were measured using the Schmidt–Boelter heat flux meter (GTW-10-32-485A, Meditherm Corporation) of the cone calorimeter equipment.

3. Results and discussion

3.1. Heat flux and temperature

In the cone calorimeter experiment a constant external heat flux is applied, resulting in heating of the sample surface, in some cases in phase transitions in the interphase, and in the formation of a pyrolysis zone where endothermic decomposition of material occurs. Fig. 1 shows the surface temperature of a ceramic fibreboard plotted against time for different external heat fluxes between 10 kW m^{-2} and 70 kW m^{-2} . Since the ceramic and polymer specimens have similarly low

thermal conductivities, the plot shows the approximate temperature curve expected for a polymer surface if no decomposition and thus no ignition occur. The surface temperature increased quite rapidly at the early stage of the tests and levelled off to a steady state as soon as the re-radiation of the surface and the heat transport from the sample plate to the surrounding was equal to the input at its surface. It became clear that — if no ignition occurs — the typical external heat fluxes used for investigations on polymers ($25\text{--}50 \text{ kW m}^{-2}$) lead to maximum temperatures far below those reached in fires characterized by similar heat fluxes.

Reaching surface temperatures above the decomposition temperature results in the formation of a pyrolysis zone, so that ignition subsequently occurs, at the critical temperature or at the corresponding critical mass loss rate. The highly exothermic reactions in the flame zone are the source of an additional heat flux affecting the sample surface. During a cone calorimeter test, external heat flux, flame heat flux, and re-radiated heat flux are all of the same order of magnitude [7]. In contrast to the surface temperature without decomposition, which is illustrated in Fig. 1, the pyrolysis temperature is determined by a complex equilibrium during the fire performance of the sample, controlled by external heat flux, heat transfer, endothermic polymer decomposition, gasification, mass loss rate, effective heat of combustion, thermal feedback of the flame, and re-radiation of the surface. Typical surface temperatures during a cone calorimeter test were reported [8,9] and characterized by increase until decomposition temperature is reached, ignition occurs at time to ignition (t_{ig}), and the temperature soars up to the pyrolysis temperature. The discontinuity in temperature indicates the crossover from an ignition scenario to a forced-flaming scenario. It also marks the difference between ignitability and flammability. Ignition occurs when the heat flux resulting from the difference of external heat flux and re-radiated heat flux results in a mass loss rate that exceeds the critical mass loss rate of a distinct ignition scenario. A sustained flame (flammability) is detected if the differences in external heat flux, heat flux from the flame, and re-radiated heat flux result in a mass loss rate that exceeds the critical mass loss rate of extinguishing. Fig. 2 shows a rather rough scheme of the surface temperature and effective heat flux behaviour at the surface according to the results reported in the literature [8,9] and based on a number of simplifying assumptions. Indeed, Fig. 2 is a description reasonable only for a thermally thick sample of a non-charring material that is characterized by a steady-state burning after ignition. Hence, the system can be described in terms of constant heat release rate, mass and heat transfers, and temperatures. Three main zones characterize the system: the flame zone, the pyrolysis zone, and the polymer. In a rough approximation, the pyrolysis zone is characterized by a constant

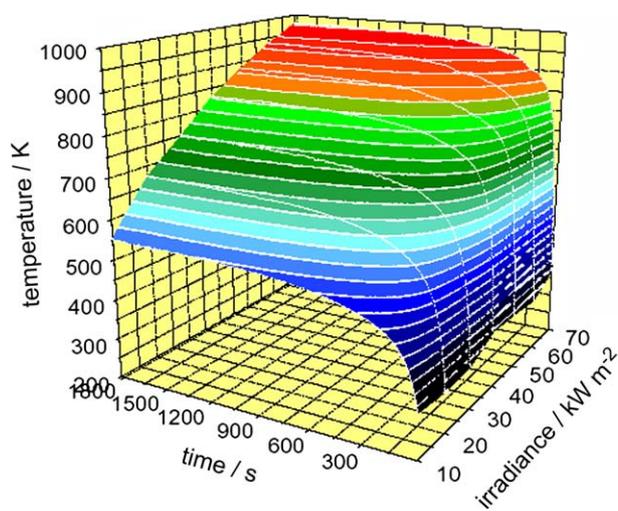


Fig. 1. Surface temperature of a ceramic plate plotted against the external heat flux applied and the time in a cone calorimeter test run.

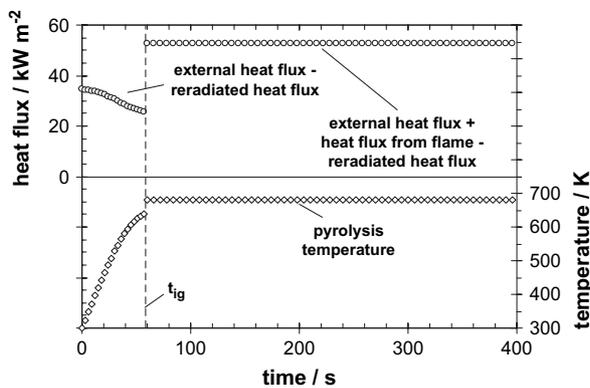


Fig. 2. Scheme for the surface temperature and effective heat flux at the surface for a thermally thick sample of a non-charring material. Surface temperature and heat flux at the surface are sketched according to the experimental results reported in the literature [8,9] and based on simplifying assumptions such as steady-state burning, excluding minor influences and differences.

thickness and an average temperature that corresponds to the characteristic decomposition temperature of the material. As time progresses, the pyrolysis zone penetrates through the polymer by feeding the flame zone with fuel.

Although only the easiest case of performance in the cone calorimeter was discussed here, this example makes clear that the sample works as a kind of “black box” between the controlled input (external heat flux), and the monitored output (heat release rate), since typically a whole set of crucial characteristics of the sample is neither known nor monitored during the test, including heat capacity, thermal conductivity, ignition temperature, pyrolysis temperature, surface temperature, sample temperature, surface emissivity, thermal feedback of the flame zone, and heat transfer to the sample holder. Based on this lack of description, it is concluded that the heat flux and the temperatures relevant in the cone calorimeter test should be discussed separately at some length, as they are two of the most relevant parameters defining the fire scenario.

This conclusion has no truly obvious impact on the standardized performance of cone calorimeter tests and data evaluation. Hence, although generally not communicated, this performance becomes important, for instance, when it is attempted to correlate cone calorimeter tests with other fire tests. In the easiest approximation, the UL 94 V-0/V-1/V-2 test is characterized by temperatures occurring during decomposition and combustion, but uses either a very low external heat flux or none at all, depending on how the applied ignition source is taken into account. Because of the different sample geometry and ignition source, an equivalent combination of temperature and external heat flux is not possible in the cone calorimeter. This example shows that, in principle, a test corresponding directly to UL 94 cannot be performed in the cone

calorimeter used according to ISO 5660 (ASTM E 1354). A scientifically convincing approach to correlate cone calorimeter data with UL 94 data was recently proposed by Lyon [7] and adopted by other groups [10]. The steady heat release of the material was determined for different relatively high external heat fluxes and then extrapolated to an external heat flux of zero in order to factor out the contribution to the heat flux originating from the combustion of decomposed material.

In ISO 5660 [6] an external heat flux of 35 kW m^{-2} is recommended for initial tests, and in absence of further specifications, tests at 25 kW m^{-2} , 35 kW m^{-2} , and 50 kW m^{-2} . Furthermore, it is important to note that test results may not be statistically significant unless the external heat flux is substantially (10 kW m^{-2}) higher than the minimum irradiance required for sustained flaming to occur. In practice, often only one external heat flux of 35 kW m^{-2} , or — increasingly popular today — 50 kW m^{-2} , is used to ensure reproducible and significant data. In general it should be noted that the cone calorimeter setup targets a forced-flaming combustion scenario that is typical of developing fires or developed fires. Therefore, cone calorimeter data on heat release are much more useful in discussing how to rank the flame spread and the heat release contribution of materials' combustion in a fire than in assessing the flammability properties of materials that determine their LOI and UL 94 classification. Using external heat fluxes higher than 50 kW m^{-2} has the advantage that the results can be reproduced more easily, show a smaller relative error, and reveal much more significant differences between different materials in terms of the heat release rate. However, using 50 kW m^{-2} as the external heat flux yields only the fire response to a well-developed fire. For many applications of polymeric materials, especially electronic and electronical ones, the goal is to optimize fire retardancy in terms of ignition and flammability. Some of the successful materials may fail in tests with a flux of 50 kW m^{-2} , because this crucial change in fire scenario amounts to an essential change in the concept of fire retardancy. For instance, char-forming systems may show decreasing fire retardancy with respect to total heat evolved as the external heat flux increases, since the char may decompose at higher temperatures, as was reported for glass-fibre reinforced PA 66 fire retarded with phosphorus [11,12]. Intumescent systems may show a crucial decrease in their corresponding insulation effect as char decomposes faster [13,14] with increasing external heat flux and temperature. Both the absolute and relative reductions in the peak of heat release rate are often higher for polymers than for their fire retarded counterparts as external heat flux increases. This is quite a common effect for all fire retarded systems, because the fire retardancy mechanisms work by increasing char-forming or by flame inhibition, such that the thermal

feedback from the flame zone is decreased, thus also decreasing the effective heat flux on the sample surface. Therefore, fire retardancy mechanisms are increasingly self-reinforcing with increasing external heat flux. Furthermore, for systems that form a physical barrier or an insulating interphase, such as charring and intumescent systems, heat re-radiation or reduced transport rates can even lead to sharply increasing effects with increasing external heat flux. The peak of heat release was reported to be reduced by between 40% and 80% for external heat fluxes of between 35 kW m^{-2} and 70 kW m^{-2} applied to layered silicate PP-g-MA nanocomposites [15] that show only a physical barrier effect in the cone calorimeter. An extrapolation to a heat flux of 0 kW m^{-2} even indicated a vanishing fire retardancy effect, which corresponded to the less convincing performance of this material in flammability tests such as UL 94.

Indeed, using higher external heat fluxes is suitable to promote certain kinds of fire retardants like halogenated substances, since their gas-phase mechanism is less dependent on the external heat flux, barrier mechanisms, or even non-polymeric materials such as ceramics or metals. It should be noted that prEN 45545 demands cone calorimeter tests of 50 kW m^{-2} for interior materials in railway applications, and that cone calorimeter setups using up to 100 kW m^{-2} are used for naval applications, while many aviation regulations stipulate external heat fluxes of only 35 kW m^{-2} . The choice of external heat flux is not a question of trying to achieve the best possible fire safety, but rather of striking a balance between protection against realistic fire scenarios and other technological economical conditions such as specific weight and costs.

3.2. Peak of heat release rate

The results of cone calorimeter investigations are a comprehensive characterization of the performance of the tested samples in a rather well-defined fire test scenario. Since the cone calorimeter was developed in order to approximate an ideal performance-based bench scale fire testing method, some of the results even allow an accurate description of the materials' properties, such as the effective heat of combustion, for instance. However, in terms of other measures and the results for distinct samples, the data are hardly suitable to convey the intrinsic properties of materials.

Obviously, the strong influence of low sample thickness must be taken into account when thermally thin samples are investigated. Even the cone calorimeter standards address this problem by proposing special procedures for samples thinner than 6 mm. However, since the cone calorimeter is used not only for academic tasks in basic research, but mainly in applied research and product development, ISO 5660 covers all realistic

thicknesses with respect to application. When thermally thin samples are investigated, not only are the time to ignition decreased and peak of heat release influenced [16,17], but crucial mechanistic changes may occur as well. For instance, Kashiwagi et al. [18] reported that the fire retardancy mechanism of layered silicate polypropylene nanocomposites disappears completely in thermally thin samples, such that no significant reduction in peak of heat release occurs at all. Furthermore, the influence of thickness differs greatly for different measures and for different kinds of materials. Fig. 3 shows the heat release rate for high-impact polystyrene (HIPS) and HIPS/15 wt.% $\text{Mg}(\text{OH})_2$ as it is reported in the literature [19] for 3 mm and 6 mm thick samples. HIPS showed the typical behaviour of thermally intermediate thick non-charring material, whereas HIPS containing 15 wt.% $\text{Mg}(\text{OH})_2$ displayed the typical behaviour of thermally thick charring material. For such a residue-forming material, even the 3 mm thick sample can be interpreted as thermally thick. Increasing the thickness increases the burning time, but affects neither the shape of the heat release rate curve nor the magnitude of the peak heat release rate or time. For the non-charring HIPS material, not even 6 mm amounted to a thermally thick sample. The peak of heat release increases in magnitude and extends over longer periods of time.

The peak of heat release rate is not only the cone calorimeter result probably most communicated to the polymer science community, but also a characteristic that displays a very strong and quite complex dependence on the fire scenario defined by the cone calorimeter setup. The reasons for this complex dependence are that the origin of the peak of heat release rate is very different for different kinds of materials, and that these different kinds of peak of heat release rates are sensitive to the fire scenario in different ways. Char and

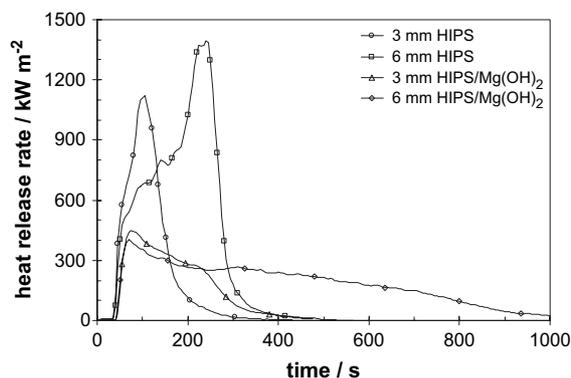


Fig. 3. Heat release rate plotted against time for HIPS and HIPS/15 wt.% $\text{Mg}(\text{OH})_2$ with sample thicknesses of 3 mm and 6 mm. The peak of heat release is strongly dependent on thickness for the thermally intermediate thick non-charring HIPS system, but not for the charring material, which achieves thermally thick behaviour even at the lower thickness.

residue-forming materials immediately show a peak of heat release rate after the initial strong increase in the heat release rate (Fig. 3) due to the formation of a protective surface layer. Some of these materials, such as cellulosic materials [20], show a second peak of heat release rate, presumably when the char structure breaks up. Many samples, including most of the non-charring thermoplastics, are characterized by a peak of heat release rate at the end of the burning phase (Fig. 3), which is caused by the thermal feedback that occurs when the pyrolysis zone reaches the back of the sample. Strictly speaking, this particular behaviour may even be triggered by the sampler holder setup in the cone calorimeter, which is in this respect the most essential part of the fire scenario. In order to illustrate this more clearly, Fig. 4 displays the heat release rate of high-impact polystyrene (HIPS) and that of the same material measured in a modified sample holder setup that rules out the thermal feedback from the back of the sample by using a 10 mm thick Cu block. Since the sample was not insulated in the modified sample holder, but had contact with a thermally conductive material, a lot of heat was actually transferred. There was no thermal feedback from the back of the sample. The shape of the heat release rate curve for the non-charring melting material changed completely. The peak at the end of the burning disappeared; the origin of the peak of heat release rate changed to a steady-state heat release; and the peak of heat release rate decreased from 1121 kW m^{-2} to 502 kW m^{-2} for this HIPS example at an external heat flux of 50 kW m^{-2} . Fig. 5 displays the heat release rate of polypropylene-graft-maleic anhydride (PP-g-MA) and a 5 wt.% layered silicate PP-g-MA nanocomposite as they were reported in the literature [15], and of the same materials measured in the sample holder setup modified with the Cu block to rule out the thermal feedback from the back of the sample. Burning times increased strongly and heat release rates decreased

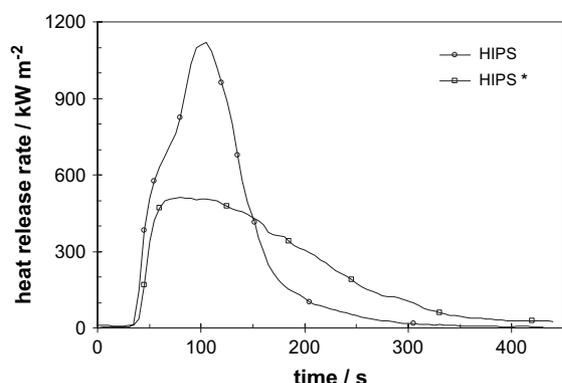


Fig. 4. Heat release rate plotted against time for HIPS, measured using a sample holder setup according to ISO 5660 and a modified sample holder setup (*) to avoid thermal feedback from the back of the sample. External heat flux was 50 kW m^{-2} .

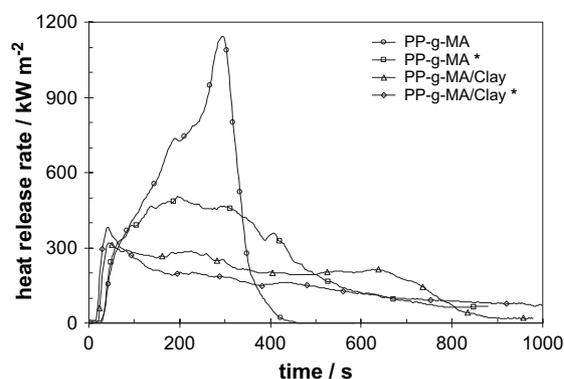


Fig. 5. Heat release rate plotted against time for PP-g-MA and 5 wt.% layered silicate PP-g-MA nanocomposite, measured using a sample holder setup according to ISO 5660 and using a modified sample holder setup (*) to avoid thermal feedback from the back of the sample. External heat flux was 50 kW m^{-2} .

significantly for both materials. However, whereas the shape of the nanocomposites' heat release rate curve showed the same behaviour in principle, the shape of the heat release rate curve of the non-charring melting materials changed completely. The peak of heat release rate decreased by 70% for nanocomposites using the cone calorimeter sample holder setup, whereas the reduction was only 27% when the modified sample holder was used. The varying influence of the setup on different kinds of peak of heat release rates became clear and should be taken into account, especially when comparing such different kinds of setups.

It should be noted that the thermal insulation on the back of the material in the cone calorimeter sample holder is used intentionally, with the purpose of approximating free-standing applications. The enhancement of pyrolysis is an important feature resulting from the thermal feedback of the back of the component samples. However, it is strongly determined by the fire scenario or components' geometry. Therefore, one of the major aims of this paper is to propose a method of quantitative comparison and ways to present peak of heat release rate data that are more sensible than those common in today's investigations of the fire retardancy of polymers.

3.3. Distance between cone heater and sample surface

The appropriate distance between the cone heater and the sample surface is a key factor in the cone calorimeter setup, since it ensures a uniform and defined external heat flux. ISO 5660 stipulates that "the distance between the bottom surface of the cone heater and the top of the specimen shall be adjusted to be $(25 \pm 1) \text{ mm}$, except for dimensionally unstable materials for which the distance shall be $(60 \pm 1) \text{ mm}$ " and "the irradiance shall be uniform within the central $50 \text{ mm} \times 50 \text{ mm}$

area of the exposed specimen surface, to within $\pm 2\%$ ". Originally, the cone calorimeter test standard specified that the sample surface for deforming or intumescent systems be fixed by using a grid. This procedure has been strongly criticized because the prevention of deformation or intumescence changes fire performance crucially, and thus apparently was never well accepted by the scientific community.

Despite the clear definition set out in the standard, many questions have arisen about what kind of setup is appropriate for measuring intumescent systems. One idea proposed is to keep the distance between the sample surface and the cone heater constant by moving the sample holder away from the heater during the test. Besides the technical problems this procedure would entail — not to mention the problem of how to define the sample surface in view of consequent deformation — such a procedure also raises a principal question. Although keeping the distance and thus the heat flux constant with respect to the sample surface seems convincing at first glance, moving the sample away means decreasing the impact of the heat flux on the original surface. Hence, it would become unclear whether resulting fire retardancy effects were due to the insulation of the char or to the repositioning of the sample away from the heater. Clearly, measuring intumescent systems in the cone calorimeter is critical since the same external heat flux can be maintained reliably neither at the surface nor with respect to the original surface in such a small-scale test method. Following the conservative approach, it is clear that the distance between the specimen and the heater should not be changed.

Figs. 6 and 7 illustrate the problem of a changing external heat flux with changing distance to the cone heater. Fig. 6 displays the irradiance in the middle, at the edge, and at the corner of the sample plate as a function

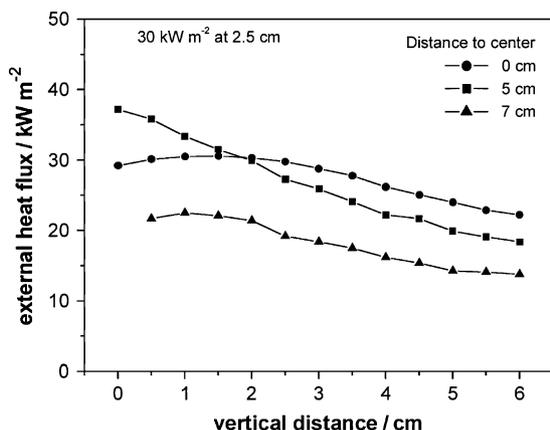


Fig. 6. External heat flux plotted against vertical distance from the cone heater, ranging between 0 cm and 6 cm for the three horizontal positions middle (0 cm), edge (5 cm), and corner (~ 7 cm) of the sample. An external heat flux of 30 kW m^{-2} is adjusted at a distance of 2.5 cm.

of the vertical distance from the cone heater, with irradiance adjusted to 30 kW m^{-2} in the middle of the sample plate for a vertical distance of 2.5 cm. Analogous results are obtained for other external heat fluxes. Fig. 7 shows the irradiance of the sample surface for two different vertical distances to the cone heater. Irradiance is plotted as a percentage of the heat flux, adjusted in the middle of the sample for each distance. The data were determined by averaging the results for different external heat fluxes, all of which show the same reduction in percent. The data shown in Figs. 6 and 7 are certainly a function of the setup, but the qualitative change and the order of magnitude are representative of every cone calorimeter setup according to ISO 5660. The heat flux exhibits linear dependence on neither the vertical nor the horizontal distance. The cone heater creates a homogeneous irradiance on the inner area ($50 \text{ mm} \times 50 \text{ mm}$) in compliance with the ISO 5660 standard when the vertical distance is between 0 cm and around 4 cm. Furthermore, it differs less than 10% for vertical distances between 0 cm and 3.5 cm when adjusted for 2.5 cm.

It can be concluded that samples with a deforming or intumescence effect of less than 2.5 cm can be measured reasonably using a distance of 2.5 cm. Both the heat flux impact on the surface raised by deformation and that on the original surface are roughly constant. If a starting distance of 3 cm or 3.5 cm is used, even samples with deformation or intumescence of up to 3.5 cm can be measured accurately. Performing accurate cone calorimeter tests on a large number of deforming and intumescence systems reveals no practical problems, since the external heat flux is quite insensitive for a distinct distance.

The problem becomes relevant when the extreme horizontal and vertical distances are discussed. At very close vertical distances, the external heat flux on the edge (5 cm) of the sample outstrips the heat flux in the middle of the sample (Fig. 6). At larger vertical distances, the irradiance at the edge and the corner are characterized by significantly lower values. If the external heat flux for a distance of 2.5 cm is averaged for the whole $100 \text{ mm} \times 100 \text{ mm}$ sample surface, it reaches around 97% of the external heat flux adjusted in the middle of the sample. At a distance of 2.5 cm, it should be noted that the inner surface of $50 \text{ mm} \times 50 \text{ mm}$ exhibits a homogeneous external heat flux (Fig. 7). At a vertical distance of 5 cm, the average external heat flux may be less than 92%, and the inner surface showing a homogeneous external heat flux ($\pm 2\%$) is less than $50 \text{ mm} \times 50 \text{ mm}$ (Fig. 7).

4. Conclusions

This paper addresses details in performing and discussing cone calorimeter measurements and results

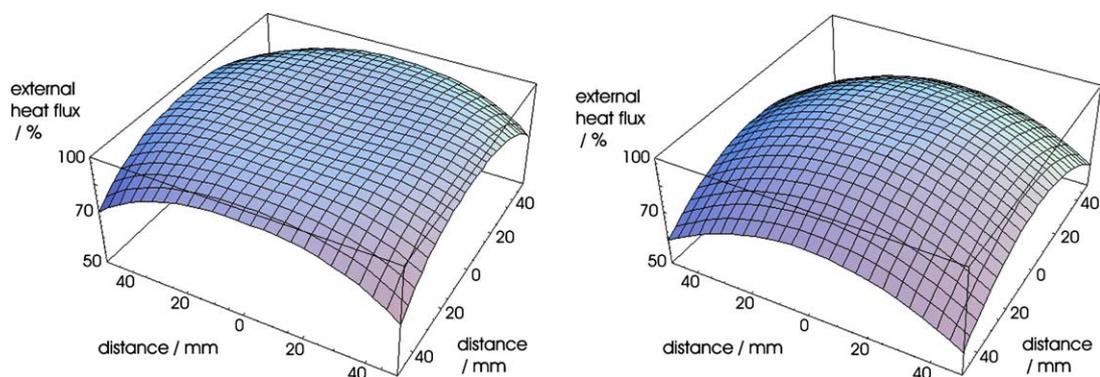


Fig. 7. Irradiance (external heat flux) plotted in percent for the surface of the sample at vertical distances of 2.5 cm and 5 cm from the cone heater.

in polymeric science. The influence and impact of choosing an external heat flux are discussed. The different external heat fluxes correspond to different fire scenarios, so that the challenge changes significantly along with the materials. Lower external heat fluxes such as below 20 kW m^{-2} target ignition and flammability, whereas higher external heat flux such as 50 kW m^{-2} target flame spread and combustion properties. The relevant and active mechanisms can change significantly. Hence, the external heat flux influences the conclusions implied by the cone calorimeter studies. The frequently used irradiances of 35 kW m^{-2} or even 50 kW m^{-2} deliver reproducible results for forced-flaming conditions with respect to flame spread and combustion properties.

The paper points out that the different results of cone calorimeter tests are influenced significantly by the setup. Some cone calorimeter data portray materials' properties, whereas others are strongly dependent on the particular setup. The peak of heat release rate is one of the best-known cone calorimeter test measures, but shows a strong and complex dependence on the setup, which often does not receive sufficient attention. The influence of thickness and thermal feedback is discussed in detail.

The paper illustrates the dependence of the irradiance on the vertical and horizontal distances between the sample surface and the cone heater. It is concluded that specimens with an intumescence of up to 3.5 cm can be measured accurately using a starting specimen distance of 3.5 cm. For larger deformations, the cone calorimeter setup does not deliver irradiance that is uniform in the vertical and horizontal distances. In any case, the conservative approach favours measurements with a fixed sample holder over a moving specimen. The above discussion may assist researchers in performing accurate measurements and data evaluations of deforming and intumescent materials.

Some parts of the above discussions and conclusions may be familiar to cone calorimeter experts, but they are not well known in the scientific community that uses the cone calorimeter extensively to investigate the fire

retardancy of polymeric materials. Therefore, this paper makes a valuable contribution to improving the interpretation of data and to raising the quality of the scientific discussion.

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