

# APPLICATION OF A HEAT RELEASE CRITERION FOR NONCOMBUSTIBLE MATERIALS

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## ABSTRACT

A method for extrapolating the time dependent heat release rates obtained from Cone Calorimeter measurements is presented. This method is implemented by fitting model parameters to experimental heat release rate data obtained at a specified level of irradiance and evaluating the resulting function at the values of interest. The method was validated by comparing the predicted heat release rate curves for wallboard and ceiling tiles, which were calibrated using data obtained at an irradiance of 95 kW/m<sup>2</sup>, to experimental measurements performed at irradiances of 65 kW/m<sup>2</sup>, 75 kW/m<sup>2</sup>, and 85 kW/m<sup>2</sup>. The model was then applied to determine whether these materials met the criteria for combustibility proposed by Alpert and Khan.

## INTRODUCTION

A noncombustible material is defined in the NFPA 101 Code for Safety to Life from Fire in Buildings and Structures as a material that, "in the form in which it is used and under the conditions anticipated, will not aid combustion or add appreciable heat to an ambient fire." This designation is commonly used in building codes. Materials are generally tested for noncombustibility using ASTM E 136, "Standard Test Method for Behavior of Materials in a Vertical Tube Furnace at 750° C," although a similar ISO test (1182) is also used. Small samples of the material are subjected to a stream of air heated to 750° C. These are pass/fail tests. The criteria for classification as a noncombustible material are based on temperature rise and ignition behavior, and low values of each are permitted. Thus, some materials that are currently classified as noncombustible do have nonzero heat release rates and can contribute to a fire. This degree of involvement is not quantified in the standard test methods.

Recently, Alpert and Khan proposed an alternative criterion based directly on heat release rate (HRR) measurements.<sup>1</sup> More specifically, they proposed that a material should have a peak HRR of less than 53 kW/m<sup>2</sup> when exposed to an incident flux of 50 kW/m<sup>2</sup> to be classified as noncombustible. This criterion is based on an analysis that determines the minimum HRR needed to support flaming combustion on the surface of a continuous disk of fuel 1 meter in diameter. These conditions are thought to be representative of the environment that building components made from these materials would experience in an actual fire.

Cone calorimetry<sup>2</sup> has become the de facto standard for measuring the heat release rate (HRR) of products and materials. In this method, the HRR is determined indirectly from the depletion of O<sub>2</sub> while the material is burning under well-ventilated flaming conditions. This works well for materials with high or moderate HRR values where the amount of oxygen consumed by combustion is substantial. However, for materials with low combustibility, the changes in the concentration of O<sub>2</sub> are small and may be difficult to detect reliably. Even more problematic is the fact that small samples (needed for Cone Calorimeter tests) of many of the products of interest, including wallboard and ceiling tiles, fail to ignite when the incident flux is as low as 50 kW/m<sup>2</sup>. In an effort to circumvent this problem, Alpert and Khan suggested making HRR measurements in an atmosphere enriched with oxygen.<sup>1</sup> They demonstrated that the peak HRRs of several materials that are classified as noncombustibles (by ASTM E 136) approximately double when the oxygen concentration is increased from ambient (21 %) to 40 % so that the requirement for noncombustibility becomes a HRR of 106 kW/m<sup>2</sup> or less when irradiated at a thermal flux of 50 kW/m<sup>2</sup> in a atmosphere containing 40 % oxygen.

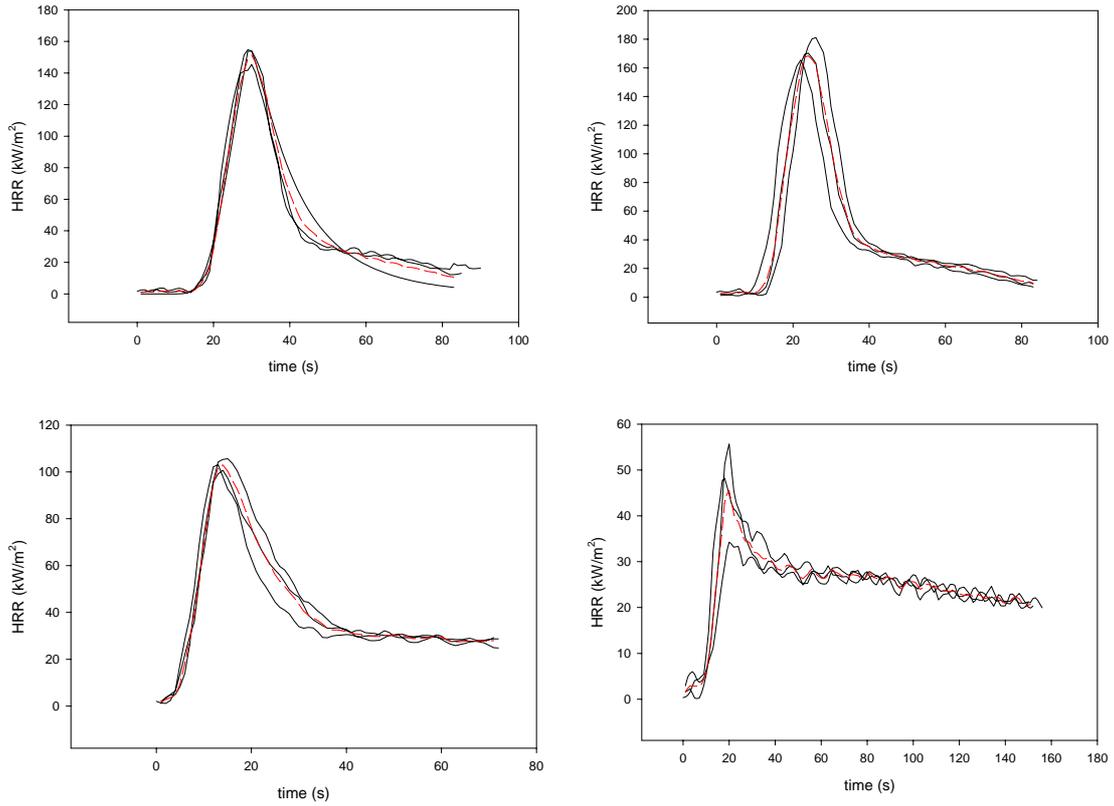
In this paper, we examine a different approach. Rather than increase the oxygen concentration to promote ignition, we consider the possibility of extrapolating the HRR curves obtained from Cone Calorimeter measurements made in ambient air at irradiance levels that are sufficiently high to ensure flaming combustion to the value the material should have at an applied flux of 50 kW/m<sup>2</sup>.

## **PROCEDURES**

### **Measurements**

Cone Calorimeter HRR measurements were made on two types of gypsum wallboard (C and X core) and two types of ceiling tiles (mineral fiber and fiberglass) at 3 levels of irradiance. Samples were cut into squares having a surface area,  $S = 1.0 \times 10^{-2} \text{ m}^2$ . The measurements on the wallboard and fiberglass based ceiling tiles were obtained while exposing the samples to incident fluxes of 95 kW/m<sup>2</sup>, 75 kW/m<sup>2</sup> and 65 kW/m<sup>2</sup>. Since the mineral fiber ceiling tiles did not ignite at 65 kW/m<sup>2</sup>, the measurements were made at 95 kW/m<sup>2</sup>, 85 kW/m<sup>2</sup> and 75 kW/m<sup>2</sup>.

Three measurements were made at each incident flux for each of the 4 materials. A comparison of the curves obtained at 95 kW/m<sup>2</sup> is shown in Figure 1. The comparisons of the data obtained at 75 kW/m<sup>2</sup> and 65 kW/m<sup>2</sup> (or 85 kW/m<sup>2</sup>) are similar. Usually, peak HRRs obtained from Cone Calorimeter measurements do not vary by more than 10 %<sup>3</sup>. However, the discrepancies observed for the mineral fiber ceiling tiles are considerably larger than this value. This lack of reproducibility may be due to their very low combustibility (note the difference in the ordinate). We also observed significant discrepancies in the replicates of the HRR measurements made on the C type wallboard, which may be due to variations in the water content of the samples. Nevertheless, the relative deviations (from the mean) of the peak HRRs are within  $\pm 10 \%$  for all of the samples except the mineral fiber ceiling tiles. The ignition times, as indicated by the peak positions, appear to be reproducible with a comparable level of certainty.



**Figure 1.** Comparison of the 3 HRR measurements obtained at an incident flux of  $95 \text{ kW/m}^2$  for the X (upper left) and C (upper right) type ceiling tiles and fiberglass (lower left) and mineral fiber (lower right) ceiling tiles. The average of the 3 curves is shown as a dashed red line.

### Extrapolation Method

The extrapolations are based on a simple model, which presumes a steady burning solid with a single pyrolysis zone. With the additional assumption that the thermal decomposition of the solid can be described by first order kinetics<sup>4</sup>, the mass-loss rate (MLR) can be represented by Eq.[1].

$$\dot{m}(t) = \frac{m_b}{S} k(T(t)), \quad [1]$$

where  $\dot{m}(t)$  is the mass flux of volatiles leaving the burning object and  $m_b$  is the mass of the solid that is undergoing thermal decomposition at time,  $t$ . The temperature dependence of the rate constant is accounted for by the Arrhenius expression

$$k(T(t)) = A \exp\left(-\frac{E_a}{RT(t)}\right), \quad [2]$$

where  $R$  is the gas constant,  $E_a$  is the global activation energy, and  $A$  is the corresponding pre-exponential factor for the reactions responsible for the mass loss. The temperature of the sample increases with time in accordance with Eq.[3].

$$T(t) = T_{\infty} + (T_0 - T_{\infty}) \exp(-\beta t), \quad [3]$$

This function, which increases exponentially from ambient,  $T_0 = 300$  K, and approaches a maximum,  $T_{\infty}$ , as  $t \rightarrow \infty$ , is thought to capture the qualitative features of the thermal response of the sample to the incident flux.

During ignition, and while the sample is burning steadily, the mass of material that is undergoing decomposition is presumed to be constant as indicated in Eq.[4].

$$m_b = \rho \delta S, \quad [4]$$

where  $\rho$ , and  $\delta$  are the density of the burning material and the depth of the pyrolysis zone, respectively. At some point, however, the residual mass of the sample is insufficient to sustain steady burning and the MLR falls-off. This is accounted for by the substitution of Eq.[5] for Eq.[4] when the residual mass becomes less than  $\rho \delta S$ .

$$m_b = m_r(t) = m_0 \exp(-k_p t) + m_{\infty} \quad [5]$$

Thus, the implementation of the model requires keeping track of the mass lost during the burn, which can be accomplished by numerical integration of Eqs.(1) - (4). In Eq.[5],

$k_p = A \exp(-\frac{E_a}{RT_p})$ , is the rate constant at the temperature,  $T_p$ , of the sample when the

critical value of the mass is reached (which is presumed to be equal to  $T_{\infty}$ , but can be less if the material is consumed before it reaches its maximum temperature) and  $m_0$  is approximately equal to the amount of combustible material in the sample. In all cases, we observed that the HRR did not fall to zero at the end of the Cone Calorimeter measurements as expected. This nonzero baseline effect was accounted for by introducing a time independent contribution to the sample mass,  $m_{\infty}$ .

The HRR,  $\dot{q}_c$ , is given by multiplying the MLR by an effective heat of combustion,  $h_c$ , as indicated in Eq.[6].

$$\dot{q}_c(t) = \begin{cases} h_c \delta \rho A \exp(-\frac{E_a}{RT(t)}) & m_r(t) \geq \rho \delta S \\ h_c \frac{m_r(t)}{S} A \exp(-\frac{E_a}{RT(t)}) & m_r(t) < \rho \delta S. \end{cases} \quad [6]$$

## RESULTS

### Model Calibration and Validation

The extrapolations are performed by fitting the model parameters to experimental measurements obtained at a high incident flux ( $95 \text{ kW/m}^2$ , in this study) and evaluating Eq.[6] at the lower values of interest. The parameters determined from fitting are  $T_{\infty}$ ,  $m_0$ ,  $m_{\infty}$ ,  $\beta$ , and  $A' = h_c \delta \rho A$ . The pre-exponential factor, activation energies, and effective heat of combustion are regarded as variables that can be obtained from experimental measurements. Only two of the parameters,  $A'$ , through its relationship to  $\delta$ , and  $\beta$ , are considered to depend on the incident flux.

The dependence of  $\delta$  on  $\dot{q}$  is expressed in Eq.[7].

$$\delta(\dot{q}) \approx \frac{\kappa R T_p^2}{E_a \dot{q}_{net}}, \quad [7]$$

where  $\kappa$  is the thermal conductivity,  $\sigma$  is the Stefan-Boltzmann constant and  $\dot{q}_{net} \approx \dot{q}_{ext} - \sigma T_p^4$ , is the net heat flux into the sample. Following the derivation by Lyon<sup>5</sup>, the pyrolysis of the sample is assumed to be confined to a region over which the temperature ranges from  $T_p$  at  $x = 0$  to  $T_\delta$  at  $x = \delta$ , where  $T_\delta$  is the temperature when the MLR first becomes negligible. From Eqs.(1) and (2),  $\Delta T = T_p - T_\delta \sim RT_p^2/E_a$ . Substitution of this expression for  $\Delta T$  into the boundary condition (at  $x = 0$ ),  $-\kappa \Delta T / \delta = \dot{q}_{net}$ , results in Eq.[7]. In the extrapolation model, the dependence of  $\delta$  on external flux is approximated as  $\delta(q) \approx \frac{\alpha T_p^2}{\dot{q}_{net}}$ , where  $\alpha$  is determined from the fit to the experimental HRR curve.

The dependence of  $\beta$  on incident flux is derived by assuming that at the onset of the Cone Calorimeter measurements, all of the thermal energy is deposited into the pyrolysis zone. That is,  $\Delta T \approx \frac{\dot{q}_{net} t}{\rho \delta C_p}$  for  $t \approx 0$ , where  $C_p$  is the average heat capacity of the material in the pyrolysis zone at ambient pressure. Taking the limit of Eq.[3] as  $t \rightarrow 0$  and comparing this to the expression for  $\Delta T$  derived above, we obtain

$$\beta(\dot{q}) \approx \frac{\dot{q}_{net}}{\rho \delta C_p \Delta T}. \quad [8]$$

After substituting for  $\delta$  from Eq.[7], we find that  $\beta \propto \dot{q}_{net}^2$ . However, it turned out that a linear relationship,  $\beta = b \dot{q}_{net}$ , where  $b$  was determined by fitting the experimental HRR curve, actually gave better results.

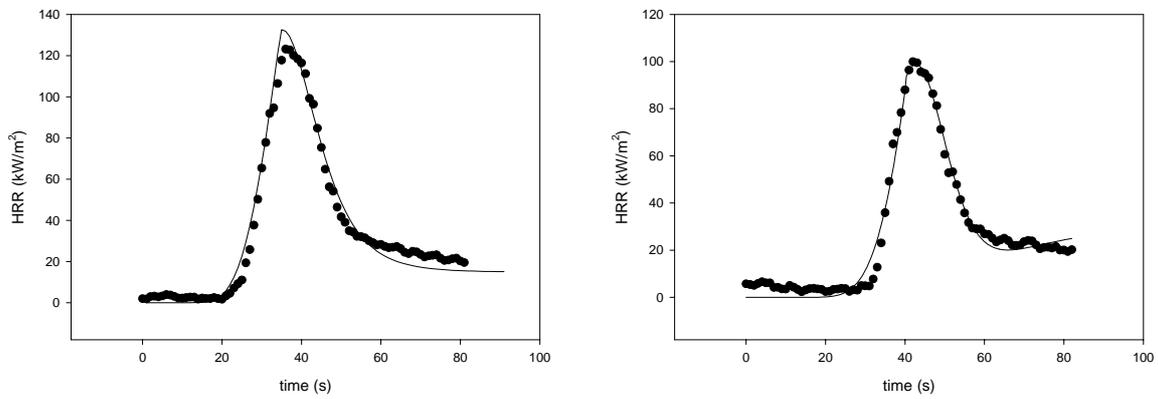
The values of the parameters and experimental variables used to fit the HRR curves measured at 95 kW/m<sup>2</sup> are listed in Table 1. The effective heats of combustion were obtained from the Cone Calorimeter measurements. In all cases, we simply guessed the pre-exponential factors assuming that any inaccuracy would be corrected for in the fitting process. In the absence of data from thermal gravimetric measurements, we did treat the activation energies for the ceiling tiles as parameters, allowing them to vary to obtain the best fits. However, this practice is not recommended in general because of the obvious interdependence between  $E_a$  and  $T$ . Nevertheless, this procedure did seem to provide a unique solution for the ceiling tiles. In the case of the wallboard, we used  $E_a = 209$  kJ/mol, which is comparable to values reported in the literature for the thermal decomposition of cellulose<sup>6</sup>.

A comparison between the HRR curves, obtained by substituting the values listed in Table 1 into Eq. [6], and the experimental results (obtained by averaging the 3 measurements made at each thermal flux) are presented in Figures 2 - 5 for irradiances of 75 kW/m<sup>2</sup> and 65 kW/m<sup>2</sup> for all of the materials except the mineral fiber ceiling tiles. For these; the curves obtained at 75 kW/m<sup>2</sup> and 85 kW/m<sup>2</sup> are compared. Although the actual fits to the experimental data at 95 kW/m<sup>2</sup> are not shown, they are comparable in accuracy to the curves in Figures 2 - 5. Surprisingly, the results do not indicate a systematic increase in error as we move further away from the value of the incident flux used to calibrate the model (95 kW/m<sup>2</sup>). In fact, the

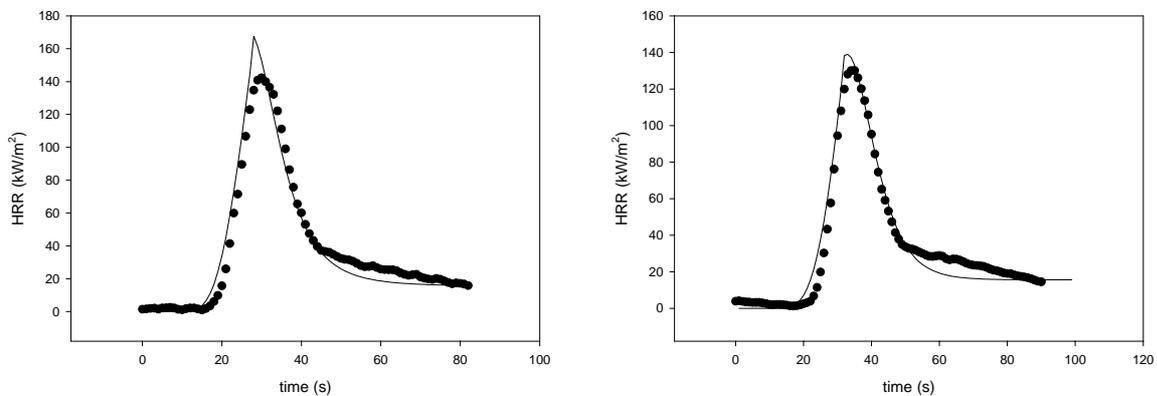
errors evident in Figures 2 - 5 are generally smaller than the differences between the experimental curves measured at the same incident flux.

**Table 1.** Model Parameters and Variables

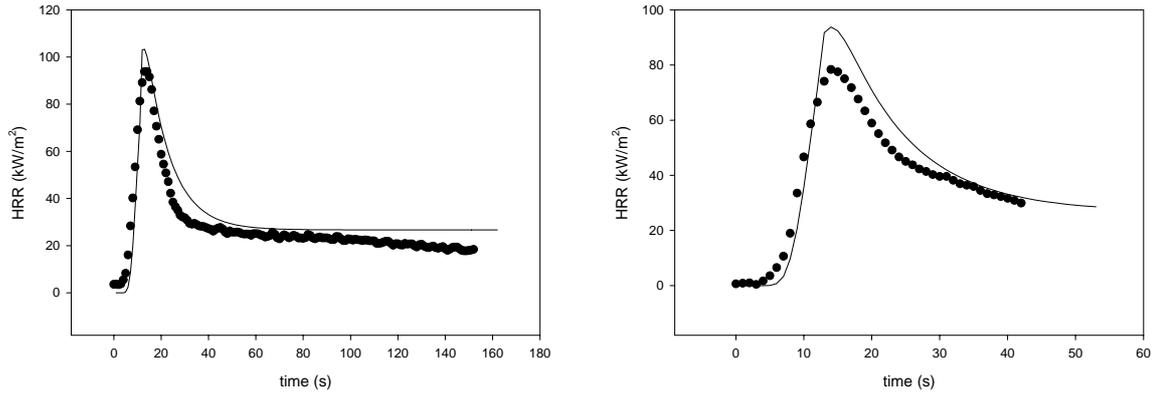
Parameter/Variable	Wallboard (type X)	Wallboard (type C)	Ceiling Tile (fiberglass)	Ceiling Tile (mineral fiber)
$E_a$ (kJ/mol)	209	209	205	213
$A$ ( $s^{-1}$ )	$5 \times 10^{11}$	$5 \times 10^{11}$	$5 \times 10^{16}$	$5 \times 10^{16}$
$h_c$ ( $kJg^{-1}$ )	14	14	8	8
$T_\infty$ (K)	857	861	605	623
$\alpha$ ( $kWm^{-1}K^{-2}$ )	$2.5 \times 10^{-8}$	$1.9 \times 10^{-8}$	$3.4 \times 10^{-8}$	$1.2 \times 10^{-7}$
$b$ ( $m^2kJ^{-1}$ )	$1.8 \times 10^{-3}$	$2.5 \times 10^{-3}$	$5.1 \times 10^{-3}$	$2.5 \times 10^{-3}$
$m_0$ ( $gm^{-2}$ )	8.7	8.5	3.5	1.5
$m_\infty$ ( $gm^{-2}$ )	0.12	0.11	0.35	0.43



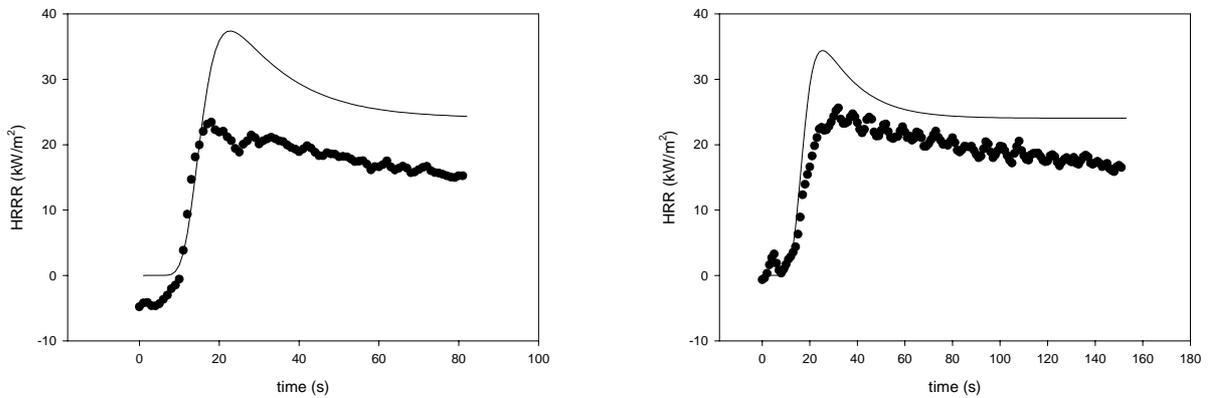
**Figure 2.** Comparison of the extrapolated (lines) and experimental HRR (points) curves for X type wallboard at incident fluxes of  $75 \text{ kW/m}^2$  (left) and  $65 \text{ kW/m}^2$  (right).



**Figure 3.** Comparison of the extrapolated (lines) and experimental HRR (points) curves for C type wallboard at incident fluxes of  $75 \text{ kW/m}^2$  (left) and  $65 \text{ kW/m}^2$  (right).



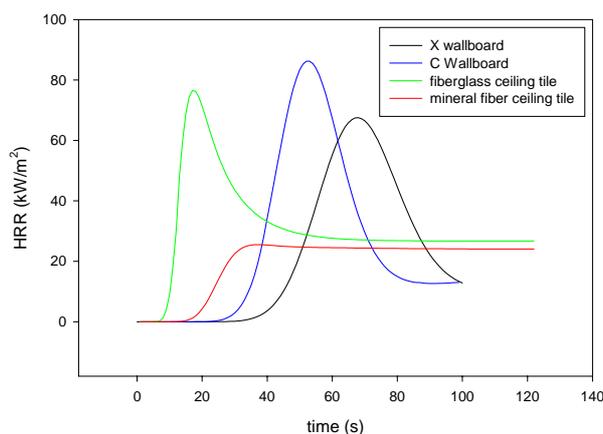
**Figure 4.** Comparison of the extrapolated (lines) and experimental HRR (points) curves for fiberglass ceiling tiles at incident fluxes of 75 kw/m<sup>2</sup> (left) and 65 kw/m<sup>2</sup> (right).



**Figure 5.** Comparison of the extrapolated (lines) and experimental HRR (points) curves for mineral fiber ceiling tiles at incident fluxes of 85 kw/m<sup>2</sup> (left) and 75 kw/m<sup>2</sup> (right).

### Predicted HRR Curves

An extrapolated HRR curve corresponding to an irradiance of 50 kW/m<sup>2</sup> was obtained for each of the 4 materials using the values of the parameters obtained by fitting Eq.[6] to the experimental measurements at 95 kW/m<sup>2</sup> (Table 1). These curves are shown in Figure 6. Only the mineral fiber ceiling tiles strictly meet the criterion proposed by Alpert and Kahn, but the X type wallboard is close. The peak HRRs of the C type wallboard and fiberglass ceiling tiles clearly fall outside of the acceptable range (i.e., within 10 % of 53 kW/m<sup>2</sup>).



**Figure 6. Predicted HRR curves for both types of wallboard and ceiling tiles at 50 kW/m<sup>2</sup>.**

## SUMMARY AND CONCLUSIONS

A method for extrapolating the time dependent heat release rates obtained from Cone Calorimeter measurements was presented. The extrapolation method is based on a simple model of a steady burning solid with a single pyrolysis zone. The model is calibrated by fitting the parameters to experimental HRR data obtained at a specified level of irradiance. The extrapolations are performed by evaluating the calibrated model to obtain the HRR of the material at another value of the incident flux. The method was validated by comparing the predicted HRR curves for wallboard and ceiling tiles, which were calibrated using data obtained at an irradiance of 95 kW/m<sup>2</sup>, to experimental measurements performed at irradiances of 65 kW/m<sup>2</sup>, 75 kW/m<sup>2</sup>, and 85 kW/m<sup>2</sup>. This approach may be more convenient for some laboratories to implement than the enhanced oxygen method demonstrated by Alpert and Kahn.

This method was used to predict the time dependent HRRs for the burning of two types (X and C core) of wallboard and two types (fiberglass and mineral fiber) of ceiling tiles at an irradiance of 50 kW/m<sup>2</sup>, which is below the threshold needed for the ignition of these materials in the Cone Calorimeter. The results indicate that the mineral fiber ceiling tiles meet the criterion for classification as non-combustibles proposed by Alpert and Kahn, whereas the C type wallboard and fiberglass ceiling tiles do not. The X type wallboard is borderline; having a peak HRR very close to the value of 53 kW/m<sup>2</sup>, which according to Alpert and Kahn's analysis is the minimum HRR needed to support flaming combustion of building components made from these materials in a room scale fire scenario.

## REFERENCES

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