Flammability of Building Thermal Insulation Materials Using Self-designed Adiabatic Specimen Holder in Cone Calorimeter

Abstract: Flammability of typical building insulation materials, such as polyurethane (PU), were investigated experimentally and theoretically in a cone calorimeter using a self-designed adiabatic specimen holder to create an ideal insulation testing condition. Effect of fire retardant level on the ignition and flammability of PU was also analyzed. It is observed that comparing to standard specimen holder the self-designed one intends to increase the heat release rate and decrease the extinguish time. It is also known that the total heat release of PU is rather high. Afterglow happened at different fire retardant levels of PU, which is much dependent on material itself but not the fire retardancy.

Keywords: Ignition, Heat release rate, Thermal insulation material, Adiabatic specimen holder

1 Introduction

Polyurethane (PU) as effective thermal insulation materials are widely used in external construction façade on the background of world energy crunch for the critical needs of materials with light weight, low thermal conductivity, compression and perfect physical properties. However, high flammable behaviors of these polymers are great threat to human lives, which have attracted much attention from engineers and related researchers [1–3]. Cone calorimeter supports a well-defined flaming condition, forced by external radiation, typical for a developing fire scenario. Ignition properties and combustion characteristics of various materials, such as wood [4], PMMA [5], are measured and discussed using Cone Calorimeter. Xu et al. [6]
evaluated the fire safety of EPS foam employing multi-scale methods including the cone calorimeter test. A more ideal insulation in cone calorimeter tests would benefit the analysis of thermal properties of material themselves by avoiding heat losses. Insulation conditions of the standard tests are not good as claimed. For example, aluminum foil was usually used acting as an insulation material. Kim et al. [7] wrapped the tested wood samples using very thin aluminum foil, with the shiny side of the foil facing the sample. Shi and Chew [8] covered the edges and rear surface of the specimens with aluminum foil for insulation for the fire behavior tests of wood samples under autoignition conditions. Patel et al. [9] followed the standard cone calorimeter set up and used a 1.5 cm thickness layer of glass wool for the insulation of bottom surface of polymer samples to minimize heat loss effect. An ideal insulation of testing condition in cone calorimeter enables the test to focus on material itself by reducing the outside influences, such as the heat losses at edges or bottom. It will also provide reference data to the relevant numerical modeling.

In the aspect of fire retardant, previous studies have much focused on formulating the most effective combination of the material and flame retardants from the fundamental. For example, small-scale tests such as thermogravimetric (TG) and differential thermogravimetric (DTG) were usually utilized to examine the outcomes of the combinations [10, 11]. Bench-scale experiments, aiming to show a more general idea about their fire characteristics, were attracted much less attention. Bench-scale experiment is critical important to address the fire-resistance capacity [12, 13] in buildings or related areas, which is a part that may be more effective to address their behaviors in practical applications. It is then significant to have the comparison and mechanism investigation of XPS and PU with the development of fire retardant as well.

To address the fire characteristics of PU under ideal insulation conditions, a self-designed insulation holder was employed in this study, which could minimize heat losses as well as prevent entrance of fresh air (oxygen) from any face other than the heated top face of the tested samples. Characteristics parameters such as ignition time, heat release rate, together with the deduced quantities like fire growth rate index (FIGRA), total heat evolved (THE), were observed to assess fire performance in terms of fire prevention and energy conservation purposes. This study provides guidance to the fire behaviors testing of practical applications of building thermal insulation materials.
2 Experimental Apparatus

2.1 Apparatus

The Cone Calorimeter Test (ISO 5660–1) [14] was employed to investigate the ignition and flammability of the tested materials. A calibrated cone heater above the sample provides a uniform heat flux over surface of sample. The sample was held in a self-designed kaowool holder (an adiabatic specimen holder) for insulation to minimize heat losses as well as to prevent the entrance of fresh air (oxygen) from any face other than the heated top face, seen in Fig. 1. It is critical to examine if the ideal insulation conditions (self-designed specimen holder) could benefit the fire characteristics tests when comparing to the standard holder.

Before each test, the sample was placed horizontally on the adiabatic specimen holder, with a fixed distance of 2.5 cm from cone heater to the sample surface. In addition, the edges and rear surface of samples were wrapped with aluminium foil to avoid heat loss and melt dripping. Irradiance levels of 25, 35, 45 and 55 kW/m² were chosen, representing various radiation levels in buildings. As soon as the heater and spark plug switch on, the program in the computer was triggered to record the experiment. Once the visible flame appeared, the sample was then considered to be ignited. Each test was repeated at least two times to minimize experimental error and verify the repeatability.
2.2 Materials

The current study involved 25 mm thick PU supplied by a local manufacturer in Hefei, China. PU-B1, PU-B2 and PU-B3 were classified according to the Chinese standard GB 8624-2012 [15]. The bulk densities of PU were 28, 34 and 35 kg m$^{-3}$ under fire retardant level of B1, B2 and B3, respectively, and Tris (2-chloropropyl) phosphonate (TCPP) was added as flame retardant. Moisture was ignored since the closed-cell structure of these insulation materials. Thermal conductivity of all test samples remains in the range of 0.030 and 0.033 W m$^{-1}$ K$^{-1}$ at the room temperature. The sample size for cone calorimeter tests was 100 mm$\times$100 mm.

3 Results and Analysis

The decomposition of PU in fire is accompanied with char production and release of combustible. Ignition of PU happens always at the beginning of charring. The char layer cracks and plumps up under sustained heat input. Under a higher external heat flux, burning process becomes much stronger with a higher consumption rate. For certain foams that contain a higher percent of flame retardant, the decomposition rate becomes slower, showing a longer ignition time. The decomposition of samples affect both burning behavior and flammability properties, such as ignition time and heat release rate.

3.1 Comparison with Sample Holders (Effect of Boundary Condition)

Heat transfer process of sample can be described by a simplified theoretical model under a specified heat flux. The effective heat flux, $\dot{q}_{\text{net}}$, is a result of external heat flux $\dot{q}_{\text{ex}}$ and the sample’s response, including the thermal feedback of flame heat flux $\dot{q}_{\text{flame}}$ and heat loss $\dot{q}_{\text{loss}}$. And the heat loss contains thermal conduction $\dot{q}_{\text{cond}}$ and re-radiation $\dot{q}_{\text{re-rad}}$:

$$\dot{q}_{\text{net}} = \dot{q}_{\text{ex}} + \dot{q}_{\text{flame}} - \dot{q}_{\text{loss}} \quad (1)$$

$$\dot{q}_{\text{loss}} = \dot{q}_{\text{cond}} + \dot{q}_{\text{re-rad}} \quad (2)$$

An idealized heat release rate (HRR) during a “steading-state” burning [16] (approximately equals to average steady HRR) can be described as:
HRR = \chi (1 - \mu) \frac{\beta}{h_g} \hat{q}_{\text{net}}'' = \chi (1 - \mu) \frac{\beta}{h_g} (\hat{q}_{\text{ex}}'' + \hat{q}_{\text{flame}}'' - \hat{q}_{\text{loss}}'')

= \chi (1 - \mu) \frac{\beta}{h_g} (\hat{q}_{\text{flame}}'' - \hat{q}_{\text{loss}}'') + \chi (1 - \mu) \frac{\beta}{h_g} \hat{q}_{\text{ex}}'' = \text{HRR}_0 + \text{HRP}\hat{q}_{\text{ex}}'' (3)

Hence, there is a linear dependency of the HRR from the expected external heat flux. The slope (referred to as the heat release parameter, HRP) is interpreted as a fire response parameter, whereas the intercept (HRR$_0$) is considered as a flammability parameter.

The self-designed kaowool holder provides an ideal insulation situation through reducing $\hat{q}_{\text{loss}}''$ comparing with the standard steel-made holder. The HRR using kaowool holder is theoretically higher than that from standard steel-made holder, indicating that the self-designed holder shows an advantage in describing the intrinsic behaviours of a material. Samples of PU-B3 average-weighting 8.5 g were tested under an external heat flux of 35 kW/m$^2$ using these two specimen holders, respectively. The ignition time is almost the same of 3.4 s, while the extinguish time is 112 s for the standard holder and 74 s for the case with self-designed holder. The HRR increases after ignition, reaches the first peak similarly and then reduces because of the blockage of char and consumption of the sample, seen in Fig. 2. Divergence appears as the HRR returns directly back to a transient quasi-steady state with standard holder, and continues declining under the heat conduction at the sample edges. The second peak of HRR occurs in the case of self-designed holder other than standard one, which is mainly caused by decomposition under the feedback heat from the bottom and the edge as well. It is shown that the adiabatic boundary condition has limited effect on ignition time and the first peak HRR, but shortens the extinguish time and strengthens the second peak HRR. This phenomenon is just followed the expectation that the heat available for unit mass of sample increase when the heat transfer at the sample edges is largely reduced after utilizing the self-designed specimen holder. It is also indicated that the standard way of using aluminium foil could be improved in the aspect of avoiding heat transfer at the sample edges.
3.2 Heat Release Rate (HRR)

Heat release rate history data are much obvious to represent fire behaviors of materials determined by their specific properties, influences from the specimen deformation and physical and chemical mechanisms, specifically cracking char layer, endothermic reactions, release of pyrolysis products, residue and so on.

The HRR histories of PU under different external heat fluxes are plotted in Figs. 3. As a thermally thick charring material, PU tends to show the first HRR peak at the beginning of burning, which is because of the increasing thickness of char layer tends to block the heat from outside. The second HRR peak appears later on, which is probably caused by the crack in char or increasing pyrolysis reaction rates of both virgin material and char, seen in Fig. 9. HRR curves of PU-B1, PU-B2 and PU-B3 show two peak values under various heat fluxes, with much clearer peaks under high external heat flux. As for PU-B3, the first peak is higher than the second, while the PU-B2' first peak is lower than the second, but the two peaks of PU-B1 are well-matched except the situation under 25 kW/m². It is observed that XPS require limited heat and time for melting process, and a sharp peak is shown in HRR which is mainly the decomposition of the remaining material companied with pyrolysis gases releasing. Fire hazards of PU is mainly reflected during the second peak that showing a sustaining long risk time period.

The peak of heat release rate (PHRR) in the cone calorimeter is strongly dependent on the fire scenario as well as the intrinsic fire properties of the samples, such as thermal conductivity and specific heat capacity. Thermally thin and intermediate thick samples show a larger PHRR than thermally thick samples. The sequencing of
PHRR for different FR levels of PU is obtained as: PU-B3 > PU-B2 > PU-B1. It is indicated that the influence of flame retardant (FR) of PU on its HRR curves cannot be ignored.

**Fig. 3:** HRR curves for PU with levels of fire-retardant: PU-B1, PU-B2 and PU-B3 varying with time.

The fire load (THE) and the flame spread or HRR are found having the critical influence on fire hazard. Since flame spread cannot be measured through cone calorimeter tests, Petrella then proposed plotting THE (as fire load) against PHRR/$t_{ig}$ (as fire growth index) to give a comprehensive assessment of the fire hazard. Even though the PHRR/$t_{ig}$ index probably makes a better attempt in addressing flame spread than FIGRA, its application is still hampered because of its disadvantages of oversimplifying empirical indices. Fig. 4 shows the situation of using Petrella’s approach to assess the fire hazards of PU series under different heat fluxes. Hence, this is provided to be a useful way to reflect the flame retardancy effect on PU over a range of external heat fluxes.
Fire behavior of each material added with flame retardant is illustrated under different fire scenarios. The risk of fire hazard can be reduced by adding flame retardants such as by reducing the THE or the fire growth rate. From PU-B1 and PU-B2 to PU-B3, the slope growth in Fig. 4 means that flame retardant benefit greatly in reducing their fire risk by decreasing the THE (or the fire load). However, its effect tends to be diminished under a higher external heat flux. The THE decreases the most for different FR level of PU such as PU-B1 and PU-B2 at low heat flux. And the fire growth index is almost halved. Since the XPS series is completely burnt out under all the heat fluxes, the THE of XPS is reasonably constant. Flame retardant plays a critical role in reducing the fire risk of materials.

### 3.3 Residue Analysis

The residue of PU after flame out is shown in Fig. 5. The fire characteristics of PU are reflected by its char formation, char cracking, melting and bubbling with small range of bubble explosions. Char residue of PU series remains a lot after flame out. The char layer at the top surface of PU intends to block the external heat and mass transfer, which could result in a drop of HRR during the burning. The residue cracks gradually happened around the second peak in HRR. At the low level of radiant heat flux, the surface keeps nearly integrity except edges tear. At a radiance level of 35 and 45 kW/m², minute cracks are observed at the charred layer. As for PU-B1 and PU-B2, the charred layer appears to be rougher than PU-B3. The collapse occurs when residual stress is beyond the structure stability.
### Conclusion

The flammability investigation of PU with the development of fire retardant in Cone Calorimeter was carried out by using a self-designed adiabatic specimen holder. Both the self-designed holder (kaowool holder) and the standard holder were used to verify the insulation condition of cone calorimeter tests aiming to minimize heat losses as well as prevent entrance of fresh air (oxygen) from any face other than the heated top face. Thermal properties of different kind of thermal insulation materials were obtained through cone calorimeter tests. Thermal conditions, HRR, as well as the peak of heat release rate PHRR and total heat release THR represent the fire behavior controlled by the material specific properties were investigated under a range of heat fluxes and level of fire retardancy. THE (as fire load) against PHRR/$t_{g}$ (as fire growth index) denotes a comprehensive assessment of fire hazard under the effect of fire retardant. And these solutions can benefit the numerical modeling of current or downstream flame spread. The residue were also analyzed to
address the flame behaviors. The addition of flame retardants tends to increase the critical heat flux and ignition temperature.

**Acknowledgement:** This work was supported by National Natural Science Foundation of China (No. 51323010) and Fundamental Research Funds for the Central Universities under Grant (No. WK2320000035).

**References**


