Flame retardant properties of flexible polyurethane foams containing expanded graphite by Cone Calorimetry

Juan-juan Zhao\textsuperscript{1,a}, Shun Chen\textsuperscript{2,b}, Ming Gao\textsuperscript{1,c}

\textsuperscript{1}School of Environmental Engineering, North China University of Science and Technology, Box 206, Yanjiao Beijing 101601, China
\textsuperscript{2}School of Safety Engineering, North China University of Science and Technology, Box 206, Yanjiao Beijing 101601, China
\textsuperscript{a} zhjj@ncist.edu.cn, \textsuperscript{b} 528784219@qq.com, \textsuperscript{c} gaoming@ncist.edu.cn

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Abstract. Expanded graphite (EG) was used as a flame retardant and mixed with the flexible polyurethane foam (FPUF). The effects of EG on flame retardant properties of FPUF were studied by limiting oxygen index (LOI), rate of heat release (RHR), total heat release (THR), total mass loss (TML) and mass loss rate (MLR), yield of CO, yield of CO\textsubscript{2}, smoke production rate (SPR) and total smoke production (TSP).

Introduction

Flexible polyurethane foam (FPUF) is soft polyurethane foam. Due to its low density, well elasticity, sound-absorbing, air permeability, heat preservation performance, etc. It is widely used as furniture mat, seat cushion, various kinds of soft liner of laminated composite materials, as filtering materials, insulation materials, shock-proof materials, decoration materials, packaging materials and heat insulation materials, etc. The use of FPUF in house interiors, building or public transport constitutes a potential hazard for people in case of fire [1-2]. The need for consumer protection, coupled with the new regulations and environmental concerns, increases the interest in flame-retardant treatments. Because EG has certain flame retardant effect, when EG in the process of heating, it will formation of a carbonized layer on the materials blank off the materials and heat source. And in the process of expanding, EG will absorption of a lot of heat to reduce the temperature of the system. In this work, FPUF was treated with the EG, the flame retarding behavior of these samples was evaluated by cone calorimeter and TG [3].

Experimental

Materials

Borax (Na\textsubscript{2}B\textsubscript{4}O\textsubscript{4} \cdot 10H\textsubscript{2}O; analytical reagent) was supplied by Tianjin Yongda Chemical Reagent Co. Polyether polyols mixture (Cst-1076A/B) and isocyanurate (MDI; Cst-1076A/B) was supplied by Shenzhen Kesheng Trading Company ltd.

Measurements and Characterization

The specimen size for the LOI measurement was 90 \times 10 \times 10 \text{mm}^3 by JF-3 LOI apparatus (Nanjing Jiangning Analytical Instrument Factory). Thermogravimetry (TG) was carried out on a HCT-2 thermal analyzer (Beijing Hengjiu Scientific Instrument Factory) under a dynamic nitrogen (dried) atmosphere at a heating rate of 10°C min\textsuperscript{-1}. The specimen size for the cone calorimetry experiments was 10cm\times10cm\times30mm by PX-07-007(Phoenix quality inspection instrument co.,
LTD). At least three samples were tested to obtain average values.

**Preparation of modified flexible polyurethane foam samples**

Isocyanate, polyether polyols, and dimethyl silicone oil of were well mixed in a 1 L beaker. Next EG was added into the beaker with vigorous stirring for 10s. FPUF was treated with 2g, 4g, 6g, 8g, 10g of EG, respectively. The mixture was immediately poured into an open mold (300×250×150 mm$^3$) to produce free-rise foam. Foam blocks so obtained were kept in an oven at 70$^\circ$C for 24 h to complete the polymerization reaction. Samples were cut into the desired shape and size by rubbing with fine emery paper, and these test species were used for the evaluation of different properties.

**Results and discussion**

**Thermal stability of flexible polyurethane foams**

Limiting oxygen index (LOI) were used to evaluate the fire-resistant behavior of FPUF and FPUF/EG [4], as shown in Fig.1. It is clear that LOI value of samples has been increasing when added EG. FPUF was treated with EG significant improvement its flame retardant behavior.

**Degradation stability of flexible polyurethane foams**

The simultaneous DTG and TG curves of FPUF and FPUF/EG were carried out in dynamic nitrogen from ambient temperature to 1000$^\circ$C and are shown in Fig.2-3. Compare Fig.2 with Fig.3, we can know the FPUF/EG is more stable than FPUF.

The increase of char yields agrees with mechanism of flame retardant [5]. From the Fig.4, it can be seen that there is a main and quick decomposition stage of the mass loss behavior of FPUF and FPUF/EG. Compared char yield of FPUF with FPUF/EG have slightly higher char yield. These results indicate that the EG can increase the flame retardancy of FPUF.

![Fig.1 LOI of FPUF containing different content of EG](image1.png)  ![Fig.2 Thermogravimetric curve of FPUF](image2.png)
Heat Release

From Fig. 5, it can be seen that the heat release rate is different between FPUF and FPUF/EG. Heat release rate of FPUF is higher than that of FPUF/EG. In the short time, the FPUF release more heat, because it quickly burns. Due to the EG in the burning formation of a carbonized layer on the FPUF blank off the materials and heat source, so the FPUF is hard to burn, therefore the FPUF/EG in the burning release heat is slowly.

From Fig. 6, we can see that the total heat release of FPUF is greater than the total heat release of FPUF/EG. Indicate after added EG into FPUF can influence total heat release of FPUF in the process of combustion. This may be also attributed to the EG blank off the heat source. EG in the material of the flame retardant behavior showed good flame retardancy.

Gas and Smoke Release

Carbon dioxide yield, carbon monoxide yield, carbon dioxide yield, smoke production rate (SPR) and total smoke production for samples are shown in figures 7-10.

Fig. 7 shows the more carbon dioxide is release in the process burning of FPUF, and it can in a short time quickly burn. FPUF/EG burning is slowly and the CO$_2$ content significantly decreased in the burning produce. Indicate the EG can blank off a lot of heat in FPUF/EG burning.

From Fig. 8, we can see that the FPUF in the process of burning release of CO, at the beginning of the amount of CO at a rapidly rising stage and always increase. In a short time CO content of FPUF burning produce reached maximum peak, and this value less than CO content of FPUF/EG.
burning produce maximum peak. Usually, the flame retardant materials produce more carbon monoxide per mass unit burned than untreated materials. The carbon monoxide formation at the expense of carbon dioxide is however an important fire retardant principle [6].

From Fig.9-10, it can be seen that smoke production rate of FPUF/EG and FPUF are very different. The smoke production rate of FPUF in a short time reached maximum peak, and this value higher than smoke production rate of FPUF/EG. At the beginning the total smoke production rate growth faster of FPUF, approximately after the 300 s the grow trend become slowly. The total smoke production rate always growth faster of FPUF/EG. This indicate EG on suppressing smoke is not good.

![Fig.7 CO₂ yield profile of samples](image)

![Fig.8 CO yield profile of samples](image)

![Fig.9 Smoke production rate of samples](image)

![Fig.10 Total smoke production of samples](image)

**Conclusions**

With the arguments above, we can safely come to the conclusion that EG is not suitable for separate as flame retardant. EG at the increase LOI, increase thermostability, decrease release of CO and CO₂, decrease heat release show a good effect. But EG is not decrease smoke release of FPUF burning. So if EG with other flame retardants jointly improve FPUF would be better effect.

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