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Synthesis and Characterization of Flame-Retardant Polyurethane Elastomers Using Phosphorus-Containing Chain Extender†

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Phenylphosphonic diamine was synthesized from phenylphosphonic dichloride and characterized by Fourier transform infrared spectroscopy and nuclear magnetic resonance. Then phenylphosphonic diamine was used to prepare a series of phosphorus containing polyurethane elastomers (PPUEs) with different phosphorus contents. Combustion behaviour and thermal degradation behaviour of the PPUEs and polyurethane elastomer (PUE) were investigated by macroscale combustion calorimetry and thermogravimetric analysis. The macroscale combustion calorimetry result reveals that the value of peak heat release rate and total heat release of PPUEs are lower than those of PUE made by 3,3'-dichloro-4,4'-diaminodiphenylmethane (MOCA). The TGA result shows that PPUE2 has poorer thermal stability than PUE when they are obtained from the same soft segment, but the char yield of PPUE2 is higher than that of PUE.

Key Words: Phosphorus-containing diamine, Polyurethane, Flame retardancy, Thermal stability.

INTRODUCTION

Phosphorus is an effective element for imparting good fire resistance to polyurethanes (PUs)[1-4]. Compounds containing phosphorus are an important group of environmental friendly flame retardants which have several advantages such as low toxicity, no release of poison gas, *e.g.*, dioxin or halogen acids during combustion, as well as low smoke production during burning. Generally, modifications of flame retardant polyurethane can be roughly grouped into two categories *i.e.*, physical modifications and chemical modifications.

The physical modification approach provides a simple means of flame retardation, but polyurethane materials containing nonchemical bonding phosphorus are limited in several areas and the loss of phosphorus compounds is continuous during storage and processing. Thus, a better approach may be the incorporation of phosphorus into polyurethane materials in which phosphorus-containing structures are an integral part of the polyurethane molecule[5-8].

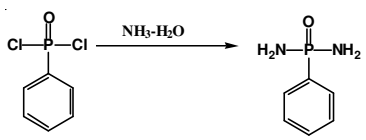
In this paper, we report the synthesis and characterization of a phosphorus-containing chain extender phenylphosphonic diamine and a series of phosphorus containing polyurethane elastomer (PPUEs) synthesized by adjusting the soft segment. The structures and properties of PPUEs were studied by FTIR, TGA and macroscale combustion calorimetry.

EXPERIMENTAL

Polyester diol (M_n = 1213, 1975, 2600) was obtained from Shandong Xinhuarun Chemical Co. Ltd., 3,3'-dichloro-4,4'-diaminodiphenylmethane (MOCA) was purchased from Zhangjiagang Daily Chemical Co. Ltd.

Synthesis of phenylphosphonic diamine: In a four-necked reactor 80 g phenyl phosphonic dichloride was added dropwise to 210g ammonia under constant stirring at 0 ºC. A constant flow of nitrogen gas was maintained to remove the HCl produced. The reaction solution was maintained at 5-10 ºC for 6 h to complete the reaction. The crude product was separated from the reaction mixture by filtration. Finally the product was recrystallized from absolute ethanol.

Synthesis of phosphorus-containing polyurethane elastomers: A certain amount of molten phenylphosphonic diamine or MOCA was added to stir prepolymer and the mixture was put in a Teflon mould and heated at 75 ºC for 2 h and 115 ºC for another 4 h. Table-1 gives a description of PPUEs and PUE.



Scheme-I: Synthesis scheme of phenylphosphonic diamine

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