Iran Polym J (2017) 26:21–30
DOI 10.1007/s13726-016-0495-8

ORIGINAL PAPER

Thermal and combustion behavior of phosphorus–nitrogen and phosphorus–silicon retarded epoxy

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Received: 2 June 2016 / Accepted: 16 November 2016 / Published online: 27 December 2016
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Abstract A novel trizine ring-based phosphorus–nitrogen flame retardant, 1,3,5-tris(3-(diphenylphosphoryl) propyl)-1,3,5-triazinane-2,4,6-trione (PN), was synthesized by the reaction of diphenylphosphine oxide and triallyl isocyanurate with triethylborane as catalyst. Chemical structure of the target compound was confirmed by Flourier transform infrared spectrum, nuclear magnetic resonances, matrix-assisted laser desorption/ionization time-of-flight mass spectrum measurements. The newly developed PN was used in the flame retardancy of *o*-cresol novolac epoxy/phenolic novolac hardener system. For comparison, another analogous phosphorus–silicon flame retardant, [(1,1,3,3-tetramethyl-1,3-disiloxanediyl)-di-2,1-ethanediyl]-bis(diphenylphosphine oxide) (PSi), was also applied in the same system. Experimental results revealed that PN showed superior flame retardant efficiency to that of PSi. In addition, the incorporation of flame retardants was in favor of the char formation during the thermal degradation process of epoxy thermosets. With the same flame retardant content, the char residue of epoxy thermosets with PSi was higher than that of epoxy thermosets with PN at 750 °C. Cone calorimeter results indicated that PN contributed to gas phase flame retardancy while PSi was more likely to take part in flame retardancy in the condense phase. X-ray photoelectron spectroscopy data revealed that the binding energies of phosphorus changed in different ways in PN and PSi after combustion. This implied that phosphorus exhibited different combustion behaviors when combined with nitrogen or silicon.

Keywords Flame retardant · Epoxy · Phosphorus · Nitrogen · Silicon

Introduction

As fire risks have been greatly increased for the increasing application of polymeric materials, flame retardancy has become an urgent issue. For example, the use of epoxy resin for electric and electronic application is extremely limited due to its flammability [1]. In view of environmental concerns, legislations are driving the search of new flame retardants instead of brominated compounds [2]. Phosphorus-based flame retardants have become the main focus as their decomposition products are less toxic and are considered environmental friendly [3]. In the meantime, nitrogen-containing compounds have been reported as great flame retardants [4]. Flame retardants based on triazine ring are synthesized and applied in various polymers [4–6]. Furthermore, the combination of phosphorus and nitrogen has an excellent synergistic effect and high flame retardant efficiency. Therefore, phosphorus–nitrogen flame retardants based on triazine ring are synthesized and studied extensively. Zeng et al. [7] synthesized a novel phosphorus–nitrogen polymeric flame retardant containing triazine ring and used in the flame retardancy of polypropylene. High flame retardancy and intumescent effect were observed. Recent report revealed that mass ratio of phosphorus to nitrogen also had effect on the flame retardant efficiency in the application of triazine ring-based flame retardant [8, 9].

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