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Intumescent flame retardant spirophosphates: pyrolysis-GC-MS studies

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Abstract

Different spirophosphates containing phenol and substituted phenols are synthesized and their structures are characterized using FT-IR and ¹H-NMR. Pyrolysis - Gas Chromatography - Mass Spectrometry (Py-GC-MS) studies are made at two different temperatures (500 and 700°C) for constant time interval (5s) and the isothermal pyrolysis products are identified so as to understand the effect of the substituents present in the phenolic part on the thermal degradation of the spirophosphates. The main volatile products identified are methacrolein, phenol, o-cresol, m-cresol and p-cresol. The amount of formation of phenol and substituted phenols from degrading spirophosphates are low, where as the amount of volatile aromatic hydrocarbons increase considerably when the samples are pyrolysed at higher temperature (700°C). The reactive spiro-[2,2]-pent-1,3-diene (spirodiene) and benzyne formed during the pyrolysis process may account for the formation of different volatile aromatic hydrocarbons. Based on these factors, the mechanism of intumescence of spirophosphates is presented and discussed.

Keywords: Degradation mechanism, flame retardancy, intumescence, isothermal degradation, Py-GC-MS.

Introduction

World moves on polymers due to their excellent properties such as light weight, durability, mechanical performance and resistant towards chemicals etc. However, the main drawback of the polymeric materials is fire risk, since all the synthetic polymers are derived from petroleum products[1]. With increasing consumption of polymeric materials in house hold products, aerospace parts and marine parts, etc., it has to be remarkably flame retardant depending on their applications[2]. In 1980s halogenated flame retardants are highlighted extensively to polymer flame retardancy. Latter many researchers recognized that halogenated flame retardants produced noxious and corrosive halogenated dibenzodioxins and dibenzofurans on fire condition[3]. So research has been focused on to develop environmental friendly flame retardant systems having efficiency similar to halogenated flame retardant materials. The search for alternate of halogen flame retardants results the hydroxides of metal, phosphorus, boron, silicon, nitrogen as well as inorganic nanopaticles containing compounds as flame retardants. The growing patent and literature on halogen free flame retardants points out the significance of phosphorus based compounds as flame retardants. The function of phosphorus compounds for imparting polymer flame retardancy originated with ammonium polyphosphate (APP)[4].

In recent years researchers paid much attention to phosphorus based intumescent system due to the evolution of a lesser amount of smoke, less toxic and corrosive gases and small amounts are enough to impart sufficient flame retardancy. Moreover the heat of combustion of the system by about 70 kcal mol^{-1} were reduced by phosphorus based flame retardants and is depend on the nature of polymers[5]. Intumescent system produces multi-cellular charred layer on degradation which prevents the underlying materials by restricting the oxygen diffusion to the underneath materials. Typically intumescent system comprises three main ingredients which includes acid source (phosphoric acid, polyphosphoric acid, etc.), carbon rich compound (sorbitol, pentaerythritol, etc.) and blowing agent (urea, urea-formaldehyde resin, dicyandiamide, melamine, polyamides, etc.). The multifaceted intumescent flame retardant mechanism is noted, since intumescent system is a combination of compounds having different functional groups[6].

Rudi Ratz et al. synthesised and studied the compound spirodichlorodiphosphate (SDDP)[7]. Halpern et al. prepared intumescent material by treating SDDP with melamine and established its effectiveness towards polypropylene[8]. In the year 2002, Vijayakumar et al. elucidated the chemical mechanism of intumescence of SDDP and spiro acids by investigating pyrolysis and pyrolysis products of the same[9]. Sivasamy et al. synthesised a series of polymer of aromatic spirophosphates by treating SDDP with different dihydric phenols and studied their thermal behaviour[10,11].

The authors have extended this study by synthesizing series of spirophosphates by reacting spirodichloro-diphosphate with phenol and substituted phenols.

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