THERMAL DEGRADATION OF COTTON CELLULOSE


Department of Chemistry, Hebei University, Baoding 071002, People's Republic of China

Abstract

The thermal degradation of cotton cellulose treated with chemical mixtures containing P and N was studied by thermal analysis, infrared spectroscopy, Char yield and limiting-oxygen-index (LOI). Our experiments demonstrated the following facts. The temperatures and activation energies of pyrolysis were lower for cotton cellulose treated with flame retardants than those for untreated samples and the values of Char yield and LOI were greater for treated cotton than those for untreated one.

Keywords: degradation, DTA, flame retardant, IR, pyrolysis, TG

Introduction

With the development of society, the improvement of people's living standard and the increase of city population the consumption of textiles used in industry and civilian life increases swiftly. Because fire disasters caused by textile always cause enormous losses, more and more people begin to think highly of studies on flame retarded textiles. Many papers have been published on the treatment of cotton cellulose with flame retardants [1, 2]. However, these investigations are mainly centred on looking for new flame retardants, systematic studies on flame retardant mechanisms are relatively rare. The main purpose of this paper is to study the thermal degradation of cotton cellulose treated with a series of flame retardants [that is, Tetrakis (hydroxymethyl) phosphonium chloride, ThPC+Dicyanamide, ThPC+urea and phosphoric acid+formaldehyde+Dicyanamide]. In order to solve this problem, thermal analysis, infrared spectroscopy, limiting-oxygen-index (LOI) and Char yield were used.

Experimental

Raw material

Cotton cellulose (Hebei Province Dingzhou Sanitary Plant, P. R. China) was immersed into water and heated for 30 min. After this procedure, the cotton cel-
lulose was dried at 333 K, and then treated with a series of chemicals containing P and N as follows:

**Preparation of flame retardant (I)**

15.0 g of triethanolamine was slowly added into a solution containing 129.0 g of tetrakis (hydroxymethyl) phosphonium chloride (ThPC) (62% concentration) and 200 ml of water was then added and the mixture efficiently stirred. 50.0 g of urea and 47.0 g of hexamethylamine were added, and the mixture was filled up to 1000 ml with water. The temperature of this solution was controlled in the range from 343 to 353 K for 10 min. The reaction product contained the following components: 8% ThPC, 5% of urea and 1.5% triethanolamine.

**Preparation of flame retardant (II)**

129.0 g of ThPC (62%), 167.0 g of dicyanamide and 25.0 g of phosphoric acid (85% concentration) were stirred together in water at room temperature. The total volume was 1000 ml. The temperature of the mixture was controlled in the range from 333–343 K for 30 min to obtain the reaction product containing 8% of ThPC and 16.7% dicyanamide [2].

**Preparation of flame retardant (III)**

A solution of hydroxymethyl dicyanamidine phosphate was prepared with phosphoric acid, dicyanamide and methyl aldehyde. A solution of hydroxymethyl dicyanamide was prepared with phosphoric acid and formaldehyde. Before using the two solutions were mixed in the mole ratio 1:1.5. The method has been described in [3].

**Thermal analysis**

DTA and TG experiments were carried out on a DT-40 thermal analyzer (Shimadzu, Japan), working under static air at a heating rate of 10 K min⁻¹. Calcined alumina was used as a reference material.

**Infrared spectrometry**

For the IR studies (Hitachi 260-50 IR spectrometer, Japan), the residue of cotton cellulose with and without flame retardant treatment was analyzed by the KBr technique. The residual samples were prepared by heating them in a DTA cell. The heating temperatures were 473, 523, 573, 623 and 673 K, respectively.

**Limiting-oxygen-index (LOI)**

The LOI value is the minimum amount of oxygen in an oxygen-nitrogen mixture required to support complete combustion of a vertically held sample that