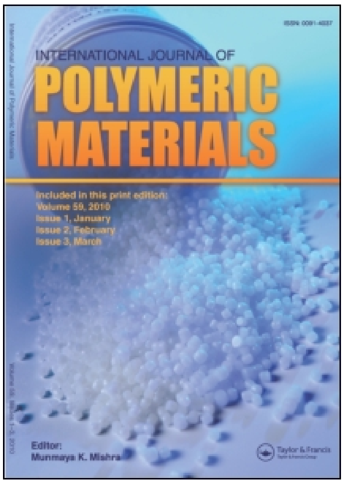

International Journal of Polymeric Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

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To cite this Article Mostashari, S. M. and Mostashari, S. Z.(2008) 'Ammonium Chloride as a Flame Retardant in a Polyester Fabric and its Detection', International Journal of Polymeric Materials, 57: 4, 355 — 361

To link to this Article: DOI: 10.1080/00914030701420178

URL: http://dx.doi.org/10.1080/00914030701420178

International Journal of Polymeric Materials, 57:355–361, 2008
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ISSN: 0091-4037 print/1563-535X online
DOI: 10.1080/00914030701420178

Ammonium Chloride as a Flame Retardant in a Polyester Fabric and its Detection

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The effect of ammonium chloride as a nondurable finish on the flammability of 100% polyester fabric has been investigated. The laundered, bone-dried, weighed fabrics were impregnated with a suitable concentration of aqueous ammonium chloride solution by means of squeeze rolls and drying at 110°C for 30 min. They were then cooled in a desiccator, re-weighed in an analytical balance and kept under ordinary conditions before the fulfillment of the vertical flame spread test. The optimum add-ons to impart flame retardancy were about 20.21–21.87 g anhydrous ammonium chloride per 100 g fabric. These results comply with the Free Radical Theory. Finally, the Mohr analytical test method was used to detect the salt content in finished fabrics.

Keywords: ammonium chloride, chemical action theory, condensed phase retardation, flame retardancy, free radical theory

INTRODUCTION

Polyester undergoes flaming combustion when ignited by an open flame. Once an amount of the material is consumed by the flaming reaction, the charred residue will often continue to react through a solid-state glowing or smoldering oxidation. In general, many organic polymers, with few exceptions, are flammable and a fire hazard is probable.

As far as the household environment is concerned, domestic fires kill more than 3000 people in the United States annually. These statistical data were released on March 31, 2005 [1]. Furthermore it is stated that between 1994 and 1998, an average of 4,400 Americans lost

Received 5 April 2007; in final form 14 April 2007.

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their lives and another 25,100 were injured annually as the result of fire [2]. In fact the elderly are at most risk of fatalities due to ignition of polymers.

While fatalities due to clothing fires are obviously linked to the flammability of polymers, there are other hazards caused by fires, including the effects of smoke and toxic gases. It is stated that over half of the deaths were due to the combined aforementioned side effects [3]. The cost, both in human lives and in financial terms, leads to pressure on the industry to develop flame-retardant fabrics and polymers that meet minimum legislative safety standards [4]. Hence the incorporation of flame retardants into consumer products has gained great significance these days. The aim of this research is to investigate the effect of deposited ammonium chloride as a nondurable finish impregnated onto a polyester fabric.

It is notable that solid ammonium chloride has a crystalline structure in which chloride ions at the eight corners surround the ammonium ion at the center of a cube structure (the adopted cesium chloride structure). At above 184°C it changes to the simple rock-salt structure [5]. Ammonium chloride has different applications such as in textile industries and laboratories as an important reagent for qualitative and quantitative analysis, protecting metal from oxidation during the soldering, cleaning metal surfaces and in the manufacture of dry electrical cells [6]. It has diuretic and diaphoretic properties and is also used as an expectorant, so it has also pharmaceutical applications. However the pharmaceutical applications are small compared with its major uses [7].

EXPERIMENTAL

Materials (Sample Preparation)

All fabrics were a woven plain construction, weighing 160 g/m^2, unfinished 100% polyester, laundered and dried. They were 22 × 8 cm strips cut along the warp direction and pre-washed in hot distilled water. The samples were dried at 110°C for 30 min in an oven, allowed to cool in a desiccator and weighed with an analytical balance.

Bath Treatment

With the exception of the first set, all specimens were impregnated with suitable concentrations of ammonium chloride at 20–22°C. They were then squeeze–rolled and dried horizontally in an oven at 110°C for 30 min. Afterwards they were cooled in a desiccator and re-weighed with analytical precision. The treated fabrics were kept overnight under an relative humidity of between 65 and 67% and an average temperature ranged between 20°C and 22°C before the fulfillment of the flammability test, so that humidity was regained during this period.

Flammability Test

This test method has been described in previous published investigations by the corresponding author [8–17]. It is a vertical tester for the estimation of thermoset fabric's flammability, called Mostashari's Flammability Tester, (Figure 1). The structural details of this tester are specified in references 8 to 17.

The burning time to the nearest 0.1 sec was measured with a stopwatch and the char length was determined to the nearest cm. Repeatability of burning time was ±5% for untreated fabric. This value was somewhat smaller for the salt-treated specimens.

Determination of Ammonium Chloride via the Mohr Analytical Method

At this stage, the ammonium chloride content in selected specimens with the related salt treatment was determined via the Mohr analytical

FIGURE 1 Mostashari's flammability tester with low add-ons applied onto a polyester fabric after the end of the experiment. Uneven burned and carbonaceous areas in the fabric are illustrated in the figure.

method. Pieces of finished polyester fabrics, 22 × 8 cm, with appropriate treatments by 3, 2.75 and 2.65 molar ammonium chloride solutions were chosen. This selection was based on their flammability characteristics (see Table 1). The counterbalanced portions of fabrics were dried in an oven at 110°C for 30 min, cooled in a desiccator and weighed in an analytical balance. They were then put into a 100 ml conical flask containing 90 ml of distilled water. Afterwards they were boiled for 30 min. Each specimen was cooled and shaken in the conical flask. Each remained extracted solution was transferred into a 100 ml volumetric flask and diluted with distilled water to the appropriate volume. The amount of anhydrous ammonium chloride in finished fabrics was calculated by titration of chloride anion via the Mohr method.

The chloride anions were determined by titration with a standard 0.01 normal solution of $AgNO_3$, using 5% K_2CrO_4 solution as an indicator. The results are shown in Table 1. Data illustrate that about 20.21–21.87% anhydrous ammonium chloride is around the optimum value to impart flame retardancy to a polyester fabric.

RESULTS AND DISCUSSION

The experimental results are listed in Table 1. The vertical flame spread test was carefully conducted to ascertain the burning times in sec (column 3). In column 7 the states of fabrics at the completion of flame tests are given, CB for completely burned, FR for flame retarded. The char lengths in cm are presented in column 4. The total percent of the salt and/or their chloride anions determined by the Mohr analytical method are given in columns 5 and 6, respectively.

TABLE 1 The Influence of Ammonium Chloride on the Flammability of a Polyester Fabric (Woven Construction Weighing 160 g/m^2)

Set No*	Treating solution NH_4Cl molarities	Burning time (s)	Char length (cm)	Percent of NH_4Cl (Mohr)**	Percent of chloride (Mohr)**	State*** of the fabric
A	Untreated	12	–	–	–	CB
B	2.65	33	–	19.67	13.05	CB
C	2.75	–	0.5	20.21	13.41	FR
D****	3	–	0.1	21.87	14.51	FR

Note: For flame-retarded (FRs) samples the char length ≤2.0 cm.

*Average of 5 tests for each set of sample.

**Mohr stands for experimental data obtained via the Mohr analytical method.

***CB means completely burnt. FR stands for flame-retarded.

****Confirmatory tests using excessive amounts of ammonium chloride.

It can be deduced from the experimental results, of anhydrous ammonium chloride per 100 g of fabric, that the optimum level of the salt required to impart flame retardancy for a polyester fabric is approximately 20.21–21.87% present in the dried sample.

The experimental observation showed that a black carbonaceous texture remained after the burning process of the treated fabrics when sufficient quantities of ammonium chloride were applied. Moreover, combustion characteristics of the flame-retarded specimens also indicated an effectiveness in suppressing the afterglow. In fact the acidic nature of the above-mentioned salt justifies this action for its catalytic dehydration, i.e. to favor the formation of solid char rather than volatile pyrolysis products, subject to polymer's thermal degradation. Hence the solid phase during the thermal decomposition is favored through the catalytic dehydration.

It is noted that the mode of action of halogen-containing compounds to act as flame retardants is in compliance with the free-radical theory [3,18]. According to this theory, the mechanism taking place in the gas phase during combustion is believed to involve the generation of high-energy OH^*, H^*, $^*O^*$ and the like radicals formed during burning, which can support the process, so that their removal or conversion can help to suppress the flame. To achieve this target it would be helpful if these active radicals could be converted to less active ones. In fact this suppression is believed to occur via chlorine or halogen compounds when applied as flame retardants:

$$RX + H^* \rightarrow HX + R^* \quad (X = Cl \text{ or } Br)$$

It is known that R^* is a less active radical than H^*. It is also worthy to note that the above-mentioned halogenated flame retardants absorb the required quantities of energy and the bonds between carbon-halogen will break homolytically. The free halogen atoms generated by this process react with the hydrogen atoms of the polymer, generated in the combustion's zone, forming hydrogen halide molecules. These molecules play a significant role in quenching the chain reaction that happens during the propagation of fire or burning process. They may, for instance, deactivate or inhibit OH^* radicals [3,19].

$$OH^* + HX \rightarrow H_2O + X^*$$

Then the newly formed free halogen radicals regenerate HX by capturing free hydrogen radical present in the gaseous phase or by reacting with the body of polymers:

$$H^* + X^* \rightarrow HX$$

$$RH + X^{*} \rightarrow HX + R^{*}$$

Other reactions have also been proposed in the literature. The significant overall effect is that the heavy halogen atoms consume energy from the combustion-propagation zone or from the combustion's region of the substrate, thus HX acts as a negative catalyst. Moreover hydrogen halides as nonflammable gases could also generate noncombustible protective layers interfering with or quenching the burning process [3,19–21], so the goal of flame retardancy could be achieved.

CONCLUSION

Ammonium chloride deposited as a flame retardant onto polyester fabric demonstrated a tendency towards attaining this goal. The above-mentioned salt's capability may be rationalized by the formation of free chlorine radicals ascribed in "Free Radical Theory". Moreover, the acidic nature of this salt could justify its action causing the formation of char; this phenomenon has been observed during treated samples' combustion. Although, due to nondurability and probably the effects of the aforesaid deposited salt on the handle, aesthetic and mechanical properties of the fabric, it will never be used for clothing. Nevertheless the obtained results may be put into practice and generalized for some other commercial and industrial purposes in various materials such as insulators and possibly for plastics and polymers.

Selecting polyester fabric, however, as a handy and easy to obtain polymeric substrate may be beneficial for the purpose of a reliable, rapid and useful detection for most flame retardants to be applied in most thermoset fabrics and polymers. Moreover by using polyester fabric an economical assessment of the synergistic effect concerning flame retardants could be made.

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