Asian Journal of Chemistry Vol. 17, No. 3 (2005), 1469–1472

Impartation of Flame Retardancy to Cotton Fabric by the Application of Ammonium Magnesium Phosphate

SEYED MORTEZA MOSTASHARI* and MOHAMMAD REZA BABAEI CHAIJAN
Department of Chemistry, Faculty of Science, Guilan University, Rasht, Iran
E-mail: smmostashari@yahoo.com

The effect of ammonium magnesium phosphate on the flammability of cotton fabric (cotton serge, 220 g/m^2) was investigated. The samples were impregnated with suitable concentrations of aqueous solutions of disodium hydrogen phosphate by means of squeeze rolls and dried in an oven at 110°C for 30 min, then immersed and stirred in a bath of admixed solution of suitable concentrations of magnesium sulphate, ammonium chloride and aqueous solution of ammonia. The treated specimens were again squeezed rolled and dried in an oven at 110°C for 30 min. Afterwards, they were thoroughly washed with a dilute solution of aqueous ammonia for removal of uninvited sodium sulphate formed together with ammonium magnesium phosphate and also to avoid the undesirable hydrolysis of ammonium magnesium phosphate, then the specimens were again dried at 110°C for 30 min, cooled in a desiccator, weighed in an analytical balance and kept under ordinary conditions before the fulfillment of the vertical flame test. The optimum add-on values to impart flame and glow retardancy were about 11.97–13.19 g anhydrous ammonium magnesium phosphate per 100 g fabric. The results compile with chemical theory, coating theory and gas theory.

Key Words: Flame retardancy, Ammonium magnesium Phosphate, Chemical theory, Coating theory, Gas theory.

INTRODUCTION

The incorporation of flame-retardants into consumer products such as fibres, fabrics, plastics etc. has gained a great importance in these days. There has been considerable progress in detailed evaluation of a large number of flame retardants in various materials including textiles.

In general, there are two ways of flame retardancy textiles. One consists, the application of a solution of a soluble flame retardant, for instance, ammonium phosphate and ammonium sulphate, etc. While this method will not greatly alter the feel and colour of the textile but it will lead to a soluble non-permanent finish. Furthermore, crystallization and some decomposition of the ammonium salt on ageing may damage the feel and the tensile strength of the material. The second consists the application of a mixture of an insoluble inorganic pigment, especially antimony oxide, with a resinous binder, especially chlorinated paraffins to the cloth. Though this way will lead to a permanent treatment and appreciably alter the feel and the colour of the cloth.

The value of chemicals sold for use as flame-retardants in Europe in 2003 (the split of income between three main categories) is as follows:

The halogen-based organics 26% inorganic compounds including $Mg(OH)_2$, $ZnSnO_3$, Sb_2O_3 and borates 36%; phosphorus-based chemicals 38%[1-3].

In present study, the multiple-bath method for precipitation of ammonium magnesium phosphate on cotton fabric was employed. A desirable sufficiency of this additive to impart flame-retardancy on cotton fabric has been observed.

EXPERIMENTAL

All fabrics were of a "serge" construction weighing 220 g/m^2, unfinished 100% cotton, laundered and dried. The fabrics were 22 × 8 cm strips cut along the waft direction and pre-washed in hot distilled water[3-5].

Bath treatment

Each group of samples were dried in an oven at 110°C for 30 min, cooled in a desiecator, weighed in an analytical balance, then they were impregnated independently at 20°C with aqueous solutions of 0.75, 0.85 and 1 M disodium hydrogen phosphate (Na_2HPO_4) by means of squeeze rolls and dried horizontally in the oven at 110°C for 30 min. The dry specimens were dipped and stirred in an admixed bath of suitable concentration of magnesium sulphate ammonium chloride and aqueous ammonia (Table-1). The total volume of each bath was 500 mL. The treated specimens were again squeezed, rolled and dried horizontally in an oven at 110°C for 30 min. Afterwards they were immersed in separate dishes of tap water and distilled water containing 0.1 M ammonia to remove sodium sulphate from the fabric [eqns. (1) and (2)] and also to avoid undesirable hydrolysis equilibrium[6] yielding $MgHPO_4$ [eqn. (3)].

$$Na_2HPO_4 + MgSO_4 \longrightarrow MgHPO_4 + Na_2SO_4 \quad (1)$$

$$MgHPO_4 + NH_4OH \longrightarrow MgNH_4PO_4 + H_2O \quad (2)$$

$$MgNH_4PO_4 + H_2O \rightleftharpoons MgHPO_4 + NH_4OH \quad (3)$$

Flammability Test

A vertical test method similar to the procedure in DOC, FF, 3–77 was applied. The conditions of the specimens and environment were in average temperature ranged between 20–22°C and the average of relative humidity ranged between 65 and 67%. The aforementioned tester is an aluminium frame: two strips of 3 mm aluminium double-sheet, 22.5 × 1.5 cm, were cut, perforated and welded at right angles to a shorter 9 cm strip. The samples were pinned tightly to the frame and held vertically in a retort stand by clamps with the lower edge 1.9 cm above the top of a 3 cm yellow flame of a Bunsen burner so as the harsh ignition circumstances are avoided.

Repeatability of burning time was ±5% for untreated fabric. This repeatability for salt treated fabrics was much lower. In fact, the pad squeeze process is known to give a certain amount of variability. After an ignition time of 3 s at the bottom edge, the total burning time was measured with a stop-watch at the nearest 0.1 s and also the char length was measured.

TABLE-1
EFFECT OF DEPOSITED AMMONIUM MAGNESIUM PHOSPHATE ON THE FLAME-RETARDANCY IMPARTED TO COTTON FABRIC (SERGE 220 g/m^2)

Test No.*	Treating solution first bath Na_2HPO_4 molarity	Treating solution second (admixed) bath ($MgSO_4 \cdot 7H_2O$, NH_4Cl, NH_3 respective molarities	Per cent add-on drying at 110°C and weighing (ranging)	Burning time (s) (ranging)	State of the fabric	Char length (cm)	Burning rate (cm/s). (interval)
1	Untreated			35.2 (34–36)	CB		0.625 (0.611–0.647)
2	0.75	0.75, 0.75, 1.5	9.5 (8.37–11)	22.2 (21–23)	CB		0.992 (0.956–1.05)
3	0.85	0.85, 0.85, 1.7	12.65 (11.97–13.19)		FR	0.2	
4†	1	1, 1, 2	16.09 (14.81–16.96)		FR	0.1	

*Average of 5 tests. CB = Completely burned; FR = Flame retarded.
†Confirmatory tests using excessive amounts of additives.

RESULTS AND DISCUSSION

The experimental results are listed synoptically in Table-1. Vertical flame test was carefully conducted to determine the burning times (column 5). In columns 6 and 7 the state of the samples and the char length (after the tests) are given respectively. In column 8, the burning rates are calculated by means of dividing the length of the specimens (22 cm) by the burning times (in seconds).

It can be attributed from the results of the fourth row that a range of 11.97–13.19% ammonium magnesium phosphate is quite sufficient to impart flame-retardancy for cotton fabric. Adverse effects on the thermal stability and physical properties of the fabric are minimized, this being due to the low add-on value required and to a high degree of dispersion achievable in the structure of textile fibres during precipitation.

The results of the third row show that inadequate quantities of the flame-retardant, *i.e.*, a range of 8.37–11% of magnesium ammonium phosphate decreased the burning time and increased the burning rate. The results are in favour of literature[8].

This can be generalized for the action of numerous flame-retardants including the inefficient quantities of ammonium magnesium phosphate which has been deposited on the cotton fabric.

On the other hand, the burning behaviour of the treated specimens also indicates a suitable effectiveness in suppressing the after glow. The plausible mechanism of such flame and glow-retardancy is in favour of the chemical theory stated by Little[9, 10]. According to this theory, the action of certain flame and glow retardants is to promote the formation of solid char rather than volatile pyrolysis products when the polymer is subjected to thermal degradation. Ideally, the carbon content of a fibre, *e.g.*, carbon presented in cellulose could be confined

to the solid phase, then decomposition could occur *via* the catalytic dehydration shown below[9–13]:

$$(C_6H_{10}O_5)_x \longrightarrow 6xC + 5xH_2O \quad (4)$$

Alexeyev[14] indicated that when $MgNH_4PO_4$ is ignited, the precipitate loses ammonia and water and the salt is converted into magnesium pyrophosphate $Mg_2P_2O_7$:

$$2MgNH_4PO_4 \longrightarrow Mg_2P_2O_7 + 2NH_3 + H_2O \quad (5)$$

Therefore, the atmosphere in the vicinity of the inflamed fabric will be modified either to dilute the flammable gases produced during the combustion or to play the role of a blanket which prevents or makes very difficult the access of oxygen. Hence, it can be deduced that a part of flame-retardancy fulfilled by ammonium magnesium phosphate is to lose ineffective diluent gases and this is also in favour of the gas theory[9, 10]. Alexeyev[14] has also stated that ammonium magnesium phosphate ($MgNH_4PO_4$) is a fairly fusible salt and when it is ignited, it melts and covers the unburnt carbon particles and thus protects them from the air, so that complete combustion of the carbon becomes impossible.

The above mentioned description is in favour of the Coating Theory explained as early as 1820 by Gay-Lussac. He called attention to the fact that among the well known flame-retarding salts there exists a group which have a low melting point and which upon contact with a flame fuse to cover the fabric with a glassy layer[9–13, 15, 16]. He proposed that the flame retardancy produced by such compounds was due to their capability to form a coating not easily penetrated by oxygen.

REFERENCES

1. C. Martin, *Chem. Br.*, **34**, 20 (1998).
2. C.E. Housecraft and A.G. Sharpe, Inorganic Chemistry, London Pearson Education, p. 382 (2001).
3. S.M. Mostashari, *Intern. J. Chem.*, **13**, 115 (2003).
4. F.M. Farhan, S.M. Mostashari and G. Ghazi-Moghaddam., *Intern. J. Chem.*, **1**, 117 (1990).
5. ———, *Intern. J. Chem.*, **2**, 163 (1991).
6. V. Alexeyev, Qualitative Analysis, Mir Publishers, Moscow, p. 100 (1987).
7. U.S. Department of Commerce, Standard for the Flammability of Children's Sleepwear (DOC FF 3–71), Federal Register 36, No. 146 (July 19, 1971).
8. W.A. Reeves and M.A. Hammons, *Text. Res. J.*, **50**, 245 (1980).
9. R.W. Little, Flame Proofing Textile Fabrics, American Chemical Society Monograph Series, No. 104, Reinhold Publishing, Nes York (1947).
10. R.W. Little, *Text. Res. J.*, **21**, 901 (1981).
11. S.M. Mostashari, The Production of Flame-Retarded Acetate Rayon, M. Phil. Thesis, University of Leeds, pp. 1, 11, 12 (1978).
12. Z.E. Jolles and G.I. Jolles, *Plastics and Polymers*, **40**, 319 (1972).
13. A.R. Horrocks, *J.Soc Dyers Colorists*, **99**, 191 (1983).
14. V. Alexeyev, Quantitative Analysis, Mir Publishers, Moscow, pp. 176–178 (1969).
15. J.E. Ramsbottom, Fire-proofing of Fabrics, His Majesty's Stationary Office, London (1947).
16. M. Kesner and W. De Vos, *J. Chem. Educ.*, **78**, 41 (2001).

(*Received*: 22 June 2004; *Accepted*: 1 March 2005) AJC-4124