Asian Journal of Chemistry Vol. 17, No. 4 (2005), 2331–2334

Synergistic Effect of Boric Acid-Urea on the Flame-Retardancy Imparted to Cotton Fabric

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

The synergistic effect between boric acid and urea on the flame-retardancy of cotton fabric (plain construction weighing 168 g/m^2) has been of interest in this investigation. The laundered, oven-dried, weighed specimens were impregnated with suitable concentrations of individual aqueous boric acid or urea solutions and some bunches were impregnated with suitable admixed solutions of both chemicals. An acceptable synergistic effect was experienced on applying an admixed bath containing 1.5 M equi-molar solutions of boric acid and urea for impartation of flame-retardancy to cotton fabric. The optimum add-on value of the mixture expressed in gram anhydrous additive per 100 g fabric was about 15%. The results obtained are in favour of gas theory and also in favour of chemical theory as well as condensed phase retardation.

Key Words: Boric acid, Urea, Synergism, Flame-retardancy, Gas theory, Condensed phase retardation, Chemical theory.

INTRODUCTION

Different inorganic compounds including $Mg(OH)_2$, $ZnSnO_3$, Sb_2O_3 and borates had been categorized as an important group of flame-retardants applied in Europe in 2003 at 36% split of income of total chemicals sold for this purpose. Among them, halogen-based organics and phosphorus-based chemicals had been forecasted to be used as 26 and 38%, respectively[1, 2].

The synergistic effect of elements for their applicability to flame-retardancy of polymers has also been exemplified in literature, *e.g.*, nitrogen-phosphorus, antimony-halogen, phosphorus-halogen, etc.[3–5] Although very early use was made of antimony oxide in conjunction with chlorine-containing compounds, the methods being somewhat empirical, but attempts were then made in various publications to explain the favourable results, which were the outcome[6].

The synergism of boric acid and borax was described by Little[7, 8]. He proposed that these combinations are capable of forming layers which are impervious to air. However, he concluded that a surface foam structure is more effective than a glassy layer since not only is air excluded from the fibre but also the foam can trap volatile tars during combustion.

The purpose of this study is to prove the influential synergistic combination of boric acid and urea on the flame-retardancy imparted to cotton fabric.

EXPERIMENTAL

All fabrics were of plain construction weighing 168 g/m^2, unfinished 100% cotton, laundered and dried. The fabrics were 22 by 8 cm strips cut along the wrap direction and pre-washed in hot distilled water. Afterwards, the fabrics were dried horizontally at 110°C for 30 min in an oven, cooled in a desiccator and weighed.

Bath Treatment

With the exception of the first bunch, all other bunches of samples were impregnated with suitable concentrations of boric acid or urea or their combinations at 20–22°C. The treated specimens were squeezed, rolled and dried in an oven at 110°C for 30 min, cooled in a desiccator and again weighed.

The treated fabrics were kept overnight under ordinary conditions before the accomplishment of the flammability test, so that the humidity regain was obtained. The laboratory's environment in average temperature ranged between 20 and 22°C and the relative humidity ranged between 65 and 67%.

Flammability test

The vertical test method similar to the procedure described[9] was employed. According to this test, an aluminium frame with the following specifications was used: two strips of 3 mm aluminium double-sheet 22.5 by 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 Bunsen burner with a three centimetre yellow flame and ignition time of 3 s. This performance was conducted in order to avoid harsh ignition circumstances. Afterwards, the total burning time was measured with a stop-watch to the nearest 0.1 s and the ignition time was subtracted from it. Repeatability of burning time was ±5% for untreated fabrics. This figure was much lower for treated samples. In fact, the pad squeeze process gave a certain amount of variability.

RESULTS AND DISCUSSION

The experimental results are listed in Table-1. Vertical flame test was carefully conducted to ascertain the add-on values on the subject of thc burning time in seconds (column 6). In column 7, the state of the specimens after the accomplishment of tests is given. It can be deduced from the experimental results that the efficient quantity of boric acid expressed in g per 100 g dried fabric is about 25%. This figure for urea treated fabric has also been obtained *ca.* 25%. However, the combination of 1.5 M of each individual bath donated about 15% gaining on the weight into the samples. This add-on value is a sufficient quantity for impartation of flame-retardancy to cotton fabric.

TABLE-1
SYNERGISTIC EFFECT OF DEPOSITED BORIC ACID-UREA ON THE FLAME-RETARDANCY IMPARTED TO COTTON FABRIC (PLAIN 168 g/m^2)

Bunch No.*	Treating solution boric acid† molarity	Treating solution urea molarity	Treating solution (admixed bath) boric acid†, urea respective molarities	Per cent add-on drying at 110°C and weighing	Burning time (s)	State of the fabric	Burning rate (cm/s)
1	Untreated	—	—	—	31.0	CB‡	0.709
2	2.0	—	—	13.43	47.0	CB	0.468
3	2.5 at 80°C	—	—	24.90	—	FR‡	—
4	—	2.5	—	19.48	32.3	CB	0.681
5	—	2.75	—	24.42	—	FR	—
6	—	—	1.25, 1.25	10.39	33	CB	0.666
7	—	—	1.5, 1.5	14.84	—	FR	—

*Average of 5 tests for each bunch.

†Consumable fabrics burnt with green flame, white smoke and without after-glow.

‡CB stands for completely burnt and FR stands for flame-retarded.

According to the chemical theory[7, 8, 10], if the carbon content of a fibre, *e.g.*, the carbon present in cellulose, could be confined to the solid phase during the thermal degradation, then decomposition could occur *via* the catalytic dehydration shown below:

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

This is agreeable to the proposition of molecular rearrangements catalyzed by acids[10]. Therefore, the action of boric acid may be explicable as a catalyst *via.* a carbonium ion mechanism; it removes water from the material. Hence, the production of char is promoted.

But as a nitrogen-containing compound, urea has a little effect on the flame-retardancy[11], due to its high nitrogen content (46.7%).

However, it is also mentionable that the mode of action of nitrogen-containing flame-retardants is still not well understood in scientific literature[12]. Nevertheless, there are some explanations to clarify their performance.

Nitrogen-based flame-retardants such as melamine and melamine derivatives act by intumescence. They are most often applied in conjunction with other flame-retardants. Gases generated from the compounds make the material swell and form an insulating char on the surface[13].

The gases subject to intumescence are mainly CO_2 and NH_3 which are liberated in the vicinity of thermal degradation zone of cellulose and comply with the gas theory[7, 8, 10, 14].

The reaction seems to be accomplished *via* the following equation:

$$H_2N—CO—NH_2 + H_2O \longrightarrow NH_3 + CO_2$$

The water required to sustain the above mentioned reaction, when urea is used by itself may plausibly be supplied by the humidity regain subject to the fabric's conditioning process. However, if urea is used in conjunction with boric acid, the aforementioned reaction is catalyzed by acid. Therefore, the synergistic effect of boric acid-urea is justifiable. The experimental observation fortifies the above-mentioned hypothesis. This is referred to a black remaining texture which has been left after the fulfilment of the flammability tests. Furthermore, no after-glow was observed during the accomplishment of tests. Hence, their synergy has been observable.

Conclusion

The synergism between urea and boric acid deposited into the cotton fabric to impart flame-retardancy, demonstrated acceptable flame- and glow-proofing properties. This is due to their cooperation to promote the formation of solid char rather than volatile pyrolysis products, when the polymer is subjected to thermal decomposition. Therefore, the chemical theory concerning the condensed phase retardation is justified. Furthermore, the gas theory because of the intumescence and subject to the generation of inert or not easily oxidizable gases, seems to play some crucial role in the above-mentioned retardation. These gases modify the atmosphere in the fabric's vicinity and dilute the flammable gases produced during the combustion, or play the role of a blanket which prevents or makes very difficult the access of oxygen. Hence, the target of flame-retardancy is achieved.

REFERENCES

1. C. Martin, *Chem. Britain*, **34**, 20 (1998).
2. C.E. Housecroft and A.G. Sharpe, Inorganic Chemistry, Pearson Education Ltd., London, p. 382 (2001).
3. G.C. Tesoro, S.B. Sello and J.J. Willard, *Textile Res. J.*, **38**, 245 (1968).
4. G.C. Tesero, *Textile Res. J.*, **46**, 152 (1976).
5. M. Lewin, S.M. Atlas and E.M. Pearce, Flame-Retardant Polymeric Materials, Plenum Press, New York, pp. 43–61 (1978).
6. P. Thiery, Fire Proofing, Elsevier Publishing Company Limited, p. 81 (1970).
7. R.W. Little, Flame Proofing Textile Fabrics, American Chemical Society Monograph Series, No. 104, Reinhold Publishing, New York (1947).
8. ———, *Textile Res. J.*, **21**, 901 (1951).
9. U.S. Department of Commerce Standard for the Flammability of Children's Sleepwear (DOC. FF 3-11), Federal Register, Vol. 36, p. 146, July 19 (1971).
10. S.M. Mostashari, M. Phil. Thesis, Leeds University, p. 11 (1978).
11. J.W. Lyons, The Chemistry and Uses of Fire Retardants, Wiley-Interscience, London (1970).
12. J.H. Troitzsch, *Chimica Oggi/Chemistry Today*, **16**, 14 (1998).
13. A. Tohka and R. Zevenhoven, Processing Wastes and Waste-Derived Fuels Contaning Brominated Flame-Retardants, Helsinki University of Technology, Department of Mechanical Engineering, Espoo, p. 11 (2001).
14. M. Kesner and W. de Vos, *J. Chem. Edu.*, **78**, 41 (2001).

(*Received*: 4 October 2004; *Accepted*: 15 June 2005) AJC-4254