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NOTE

Synergistic Effect of Sodium Oxalate-Urea on the Flame-Retardancy Imparted to Cotton Fabric

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The synergistic effect between sodium oxalate and urea on the flame-retardancy of cotton fabric (plain construction weighing 168 g/m^2) has been of interest in this study. The laundered bone-dried weighed samples were impregnated with appropriate concentrations of individual aqueous sodium oxalate or urea solutions and some bunches were impregnated with suitable admixed solutions of both chemicals. An acceptable synergistic effect was experienced using an admixed bath containing 1.0 M equi-molar solutions of sodium oxalate 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 17%. The results obtained are in favour of gas theory and also in favour of chemical theory and condensed phase retardation.

Key Words: Sodium Oxalate, Urea, Synergism, Flame-retardancy, Cotton fabric.

The different inorganic compounds including $Mg(OH)_2$, $ZnSnO_3$ and Sb_2O_3 had been categorized as an important group of flame-retardants used in Europe in 2003 at 36% split of income of total chemicals sold for this purpose. The halogen-based organics and phosphorus-based chemicals had also been forecast to be used as 26 and 38% respectively[1, 2]. One of the most interesting subjects of flame-retardancy is the synergistic study between elements and their related components; among them, the synergism of nitrogen-phosphorus, antimony-halogen, phosphorus-halogen, etc. has also been exemplified in scientific literatures[3–5]. While the additive effect is the sum of the individual actions, the effects of synergism are higher than this sum[6]. It is worthy to mention that in this regard 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[7]. However, antagonism in the flame-retardation of polymers is less known and documented[8]. The intention of this investigation is to prove the influential synergistic combination of sodium oxalate and urea on the flame-retardancy donated to cotton fabric.

All samples were plain constructions weighing 168 g/m^2, unfinished 100% cotton, laundered and dried. The specimens were 22 × 8 cm strips cut along the warp direction and pre-washed in hot distilled water. Afterwards, the samples 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 specimens were impregnated with suitable concentrations of sodium oxalate or urea or their combinations at 20–22°C. The treated samples were squeezed, rolled and dried in an oven at 110°C for 30 min, cooled in a desiccator and reweighed. The treated fabrics were kept overnight under ordinary conditions before the fulfilment of the flammability test, so that the humidity was regained during this period.

Flammability test

The vertical flammability test method similar to the procedure described in DOC FF 3–71[9] was applied.

The results are summarized in Table-1. It can be deduced from the experimental results that the efficient amount of sodium oxalate expressed in g per 100 g dried fabric is about 55%. This figure for urea treated fabric has been obtained *ca.* 25%. However, the combination of 1.0 M of each individual bath donated about 17% gain of weight to the specimens. This add-on value is an efficient amount for impartation of flame-retardancy to cotton fabric.

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

Bunch No.*	Treating solution sodium oxalate (M)	Treating solution urea (M)	Treating solution (admixed bath) urea-sodium oxalate (M)	Per cent add-on drying at 110°C and weighing	Burning time (s)	State of the fabric†	Burning rate (cm/s)
1	Untreated	—	—	—	31.00	CB	0.70
2	2.75	—	—	32.80	26.00	CB	0.67
3	3.00	—	—	54.28	—	FR	—
4	—	2.50	—	19.48	32.30	CB	0.68
5	—	2.75	—	24.42	—	FR	—
6	—	—	0.5, 1	11.34	26.66	CB	1.94
7	—	—	0.75, 1	13.39	30.00	CB	1.64
8	—	—	1, 1	17.08	—	FR	—

* Average of 5 tests for each bunch.
† CB = completely burnt; FR = flame-retarded.

According to the chemical theory[10, 11], the glow and flame-proofing action of some chemicals was explained. It stated that 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:

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

Although urea has a high nitrogen content of 46.7%[12], but shows little effect on flame retardancy[13]. It is noticeable that the mode of action of nitrogen containing flame-retardants is still not well understood in scientific literature[14]. They are most often used in combination with other flame-retardants. Nevertheless there are some explanations to clarify their performance, *i.e.*, nitrogen-based flame-retardants such as melamine and melamine derivatives act by intumescence[6]. The gases subject of intumescence are mainly CO_2 and NH_3 which are liberated in the vicinity of thermal degradation zone of cellulose, make the material swell and form an isolating char on the surface. This complies with the gas theory[10, 11].

The desired reaction of urea seems to be accomplished *via* the following equation[15]:

$$H_2N—CO—NH_2 + H_2O \longrightarrow 2NH_3 + CO_2$$

The required water to sustain the above mentioned reaction, when urea is applied by itself, may probably be supplied by the humidity regained subject to the fabric's conditioning process. However, if urea is used in conjunction with sodium oxalate, the synergistic effect on flame-retardancy between sodium oxalate-urea is achievable. Regarding sodium oxalate, it is worthy to mention that the metal oxalates lose carbon monoxide on heating; thereby the afore-mentioned salt forms sodium carbonate[16].

$$Na_2C_2O_4 \longrightarrow Na_2CO_3 + CO$$

The above mentioned endothermic reaction absorbs heat from the combustion zone of the substrate and assists in quenching the flame. Furthermore, the remaining sodium carbonate decomposes into oxide and carbon dioxide[17]. Therefore in the combustion and thermal degradation zone of cotton substrate[18] which is about 350°C, the remained sodium oxide could act as a dust or wall, in order to dissipate the heat from the combustion zone. According to the dust or wall effect theory[19], "if a high enough concentration of dust is present in the adjacent atmosphere, no flame may propagate". This is due to the absorption and dissipation of heat by the dust, causing a lowering of temperature.

Though it seems that no spectacular effect appeared by using this salt in itself, however, its synergism using an admixed bath of equimolar solutions of (1 : 1) concerning these compounds donated 17.08% dry add-on to the fabric. This was efficient for the impartation of flame-retardancy.

Conclusion

The synergism between urea and sodium oxalate deposited into the cotton fabric to impart flame-retardancy confirmed an acceptable flame and glow proofing property. This is due to their collaboration to promote the formation of solid char rather than volatile pyrolysis products, when the polymer is subjected to thermal decomposition. Hence the chemical theory and the condensed phase retardation may be involved to justify this action. Moreover the gas theory because of the intumescence and subject to the production of inert or not easily oxidizable gases, seems to play some decisive role in the above-mentioned synergism. These gases modify the atmosphere in the fabric's vicinity and reduce the flammable gases produced in the duration of the ignition or play the role of a cover which prevents or makes very difficult the access of the air oxygen. Therefore, the target of flame-retardancy is achieved.

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