Application of flame retardant products to knitted fabric

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The effect of durable flame retardant products on knitted fabric flammability has been studied. Firstly, the non-durable, semi-durable and durable flame retardants were applied to the bleached knitted cotton fabrics to observe their effects on fabric flammability and then phosphorus-based durable flame retardant products were applied to bleached fabrics at different concentrations, curing temperatures and curing times alternatively to evaluate the effect of their treatment parameters on fabric burning behaviour. The burning behaviour and weight loss values of all the fabrics were evaluated using the standard vertical flammability test method. The results evaluated using an SPSS statistical program show that the flame retardant concentration, curing temperature and curing time are very important parameters to obtain FR fabrics having good flame resistance and acceptable handle and appearance.

Keywords: Cotton, Flame retardant fabric, Knitted fabric
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1 Introduction
Flame retardancy may be conferred on textile materials by the use of inherently flame resistant fibres, changing of fibre molecular structures by co-polymerization and chemical modification methods (as in modacrlylic fibres or FR-polyester), incorporation of flame retardant additives during the production of man-made fibres (FR-viscose) or application of flame retardant products to textile substrates by a chemical after-treatment processes.

Chemical after-treatments include surface or topical treatments, coatings and functional finishes, which become a part of final fibre structure. Durability of flame retardant finishes generally depends on their adherence or bond strength to the fibre surfaces or molecular structures. Generally, the flame retardant (FR) products and their flame retardancy effects are classified as non-durable, semi-durable and durable. Durability is determined by launderability, aftercare and defined cleansing requirements, weatherability, and exposure to light, heat and atmospheric agents.

Non-durable FR products are deposited on and between the fibres and no crosslinking takes place. These products adhere on fibre surfaces. Some non-durable FR finishes can resist dry-cleaning process depending on their constitution. Diammmonium phosphate, borax and boric acid mixtures, ammonium sulphate, ammonium bromide and ammonium salts of amidosulphonic acid are often used as non-durable flame retardant products. Semi-durable flame retardant products may crosslink to fibre reactive groups. Flame retardancy can be maintained for 5-15 times to dry or aqueous cleaning processes, depending on their constitution. Halogenated products, such as chlorinated paraffin, polyvinyl chloride, polyvinylidene chloride, metal oxides (especially antimony trioxide in combination with halogen compounds such as hexabromocyclododecane or chlorinated paraffin) and simple phosphorus/nitrogen compounds, are often used as semi-durable FR products unless applied together with a resin. Durable FR products bond to the reactive groups of fibre molecules or penetrate inside the fibre. Durable FR products often consist of organophosphorus condensates especially in the presence of cellulosic fibres.

The efficiency of the FR finish on a textile fabric depends on a number of factors, e.g. fibre composition, fabric construction, FR compatibility with other finishes, end use and durability. Successful FR finishes should combine acceptable levels of flame retardancy at an affordable cost and be applicable to textile fabrics using conventional textile finishing and coating equipment.

Flammability is of major concern with respect to apparel fabrics, furnishing textiles and clothing for defence personnel. By the appropriate application of flame retardant the probability of a textile material catching fire and danger of fire spread is considerably

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reduced, so that it is possible to find enough time to escape or extinguish the fire. More recently, considerable number of studies on the effects of flame retardant on pyrolytic degradation of cellulosic materials and on burning behaviour of textiles has been reported.\textsuperscript{4–16} The present study is aimed at observing how and at which level of application parameters (such as flame retardant concentration, curing temperature and curing time), the flame retardant finishes affect the flame resistancy of the cotton knitted fabric.

2 Materials and Methods

To investigate the effects of flame retardant type on flame resistance of knitted cotton fabric, non-durable, semi-durable and durable flame retardant products were chosen and applied to the bleached knitted fabrics at recommended levels by their suppliers. Phosphorus-based durable flame retardant species were applied to the bleached cotton fabric each at five different concentrations, five different curing temperatures and five different curing times to evaluate the effect of their treatment parameters on fabric burning behaviour. The burning behaviour and weight loss values of all the fabrics were evaluated using the BS 5438 (1976) vertical flammability test method. Finally, the experimental results were evaluated using an SPSS statistical program.

2.1 Materials

SU 40—diammonium phosphate-based (non-durable), PF—phosphorus/nitrogen compound-based (semi-durable) and PR 20—organophosphorus-based (durable) flame retardants were obtained from SETAS Chemical Company and Lefasol P448—organophosphorus-based (durable) flame retardant was obtained from Lefateks Chemical Company. Phosphoric acid was used for catalysing the finishing process and Na\textsubscript{2}CO\textsubscript{3} was used for neutralizing stages of durable flame retardant applications for PR 20. Wet pick-up of the fabrics was about 80% and drying temperature was 120°C for all FR finishes. The curing temperature was kept constant at 150°C for the durable flame retardant (PR20).

In this study, FR type, FR concentration, curing time, curing temperature and washing process of FR fabrics were considered as variables for analysis. The fabrics used were Ne 30/1 cotton single jersey knitted and bleached. All finishing processes were applied to the fabrics according to their anticipated commercial application and were performed in laboratory type padding and dyeing machine. Finishing formulations and after-treatments are given in Table 1. Flammability test was replicated four times for each sample and a total of 84 samples were tested to evaluate the effects of finishes.

2.2 Methods

Vertical flammability test method [BS 5438 (1976) Part 2] was used to evaluate the fabric flammability. The test specimen strips (17 cm x 22 cm) were conditioned at 20±2°C and 65±2% RH for 24 h before testing. The conditioned specimens were mounted on a suitable clamp and placed in a standard cabinet, which allows 2 mm/s airflow. A standard flame was applied at the bottom edge of the fabric for 30 s under controlled conditions. All flammability tests were conducted under ambient temperature and

After the treatment, the fabrics were washed with 10g/L sodium carbonate at 85°C for 2 times, again washed with 5g/L sodium carbonate at 40°C and then rinsed in cold water 4 times.

<table>
<thead>
<tr>
<th>Table 1—Finishing formulations and after treatments</th>
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<tbody>
<tr>
<td><strong>Bleaching</strong></td>
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<tr>
<td>H\textsubscript{2}O\textsubscript{2} (ml/400 ml), 1.6</td>
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<tr>
<td>Na\textsubscript{2}CO\textsubscript{3}(g/400 ml), 0.4</td>
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<tr>
<td>Wetting agent (g/400 ml), 0.4</td>
</tr>
<tr>
<td>Sodium silicate (ml/400 ml), 0.4</td>
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<tr>
<td></td>
</tr>
<tr>
<td>Liquor ratio, 1:20</td>
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<tr>
<td>Temperature (°C), 85</td>
</tr>
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</table>
relative humidity in the laboratory. This flammability test method normally measures char and damaged length. However, these measures do not always represent the real destroyed area of the fabric for many cases. Therefore, it was decided to use another parameter (weight loss), which will give more realistic approach to evaluate fabric destruction. It is clear that the parameters selected are interrelated or dependent on each other; high weight loss would definitely show a high area destroyed, though the reverse would not necessarily be true. For these reasons, char length and weight loss were measured for all specimens. Before and after testing the test specimens were weighed and the weight loss value was determined using the following relationship:

\[
\text{Weight loss} = \frac{\text{Initial weight of specimen} - \text{Last weight of specimen} \times 100}{\text{Initial weight of specimen}}
\]

The results were evaluated statistically with the help of analysis of variance (ANOVA) tables to indicate the significance of the effect of finishing treatments and then regression analysis was used to show the relations between applied finishing process and the fabric flammability. Each variable used in the study was considered significant if the probability level was 0.05 or less and highly significant if the probability level was 0.01 or less. Polynomial regression analysis results are shown in Table 2.

### 3 Results and Discussion

#### 3.1 Effect of Flame Retardant Type

With respect to the effect of non-durable, semi-durable and durable FR finishing agents on vertical strip test behaviour, results from the analysis of variance and polynomial regression analysis \((R^2 = 98.9\% \text{ and significance } < 0.001)\) show that the weight loss values of the fabrics are highly affected from FR types (Table 2). When the FR type changes to a durable FR product, there is a decrease in the weight loss of the fabrics. Figure 1 shows the relation between FR type and weight loss behaviour of the fabrics.

This decrease in the weight loss values can probably be attributed to the enhanced active agent content of the flame retardant products generated by the durable FR product. The increase in active agent content results in an increase in degree of flame retardance consuming more combustion energy and producing less flammable volatiles and hence reduced weight loss.

#### 3.2 Effect of FR Concentration

To determine the effect of FR finish concentration on flame resistance of the knitted fabric, the durable FR finish (organo phosphorus based, PR20) was selected and applied to the fabrics with five different add-ons (200, 250, 300, 350 and 400 g/L). Each sample was dried at 120°C and cured at 160°C for 3.5 min. Linear regression analysis \((R^2 = 73.3\% , F = 49.41, \text{ and significance } < 0.001)\) indicates that the concentration of finishing agents considerably

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**Table 2—Polynomial regression analysis results**

<table>
<thead>
<tr>
<th>Independent variable</th>
<th>Rsq</th>
<th>df</th>
<th>F</th>
<th>Sigf</th>
<th>b₀</th>
<th>b₁</th>
<th>b₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>FR type</td>
<td>0.988</td>
<td>9</td>
<td>382.15</td>
<td>0.000</td>
<td>54.22</td>
<td>-42.17</td>
<td>8.39</td>
</tr>
<tr>
<td>FR concentration</td>
<td>0.960</td>
<td>17</td>
<td>205.53</td>
<td>0.000</td>
<td>389.15</td>
<td>-2.21</td>
<td>0.003</td>
</tr>
<tr>
<td>Curing temperature</td>
<td>0.235</td>
<td>17</td>
<td>2.61</td>
<td>0.103</td>
<td>225.96</td>
<td>-2.61</td>
<td>0.008</td>
</tr>
<tr>
<td>Curing time</td>
<td>0.919</td>
<td>17</td>
<td>96.53</td>
<td>0.000</td>
<td>31.96</td>
<td>-11.79</td>
<td>1.21</td>
</tr>
<tr>
<td>Washing cycle</td>
<td>0.977</td>
<td>9</td>
<td>191.14</td>
<td>0.000</td>
<td>53.55</td>
<td>3.53</td>
<td>-0.062</td>
</tr>
</tbody>
</table>

Rsq—Coefficient of determination; df—Degree of freedom; F—Ratio between the treatment mean square and the error mean square; Sigf—Significance; and \(b₀, b₁, \text{ and } b₂\) are the regression coefficients.

Mathematical equation—Quadratic model whose equation is \(Y = b₀ + (b₁ t) + (b₂ t^2)\), where \(Y\)—Dependent variable, and \(t\)—Independent variable.
influences flame resistance of the fabrics (Table 2). Figure 2 shows that the weight loss decreases significantly as the FR concentration is increased.

Flame retardant application from bath liquor concentration above 350 g/L generates excessive harshness of handle although high flame resistance is obtained.

3.3 Effect of Curing Temperature

Keeping the FR concentration fixed at 300 g/L, the temperature of curing was varied from 140°C to 180°C for 3.5 min for durable FR Lefasol P448. Polynomial regression analysis ($R^2=13.2\%$, $F=2.75$, significance = 0.115) shows that the curing temperature has no significant effect on flame resistance of the fabrics (Table 2). It is observed that the highest curing temperature of 180°C does not add to flame resistance when compared with the similar sample cured at 160°C. All the samples that were cured above 160°C are found to show undesirable yellowing. The samples that were cured under 150°C were ignited and continued to burn. This can be attributed to incompletely FR curing process and removal of retardant after washing off. The effect of change in curing temperature on flame resistance in term of weight loss of the knitted fabrics is shown in Fig. 3.

3.4 Effect of Curing Time

Keeping the FR concentration fixed at 300 g/L and the curing temperature at 160°C, the time of curing process was varied from 2 min to 6 min. Polynomial regression analysis ($R^2=91.9\%$, $F=96.5$, significance<< 0.001) indicates that the flame resistance of the fabrics is influenced by the curing time (Table 2). Figure 4 shows that the flame resistance of the fabrics increases as the curing time increases from 2 min to 5 min. The lowest weight loss values are obtained at 4 min and 5 min. It is assumed that the observed high weight losses at 2 min and 3 min are probably caused by the incomplete condensation of the flame retardant during these curing times. On the other hand the samples cured at 6 min show a slight reduction in FR properties and undesirable yellowing. This clearly indicates that the time and temperature of curing stage of the FR finish are very important parameters to obtain FR fabrics having good flame resistance, and acceptable handle and appearance.

3.5 Effect of Washing Process

Many FR cotton products are washed under severe conditions. In this study, after the durable FR finishing agent was applied to the knitted fabrics according to the supplier recommendations for commercial use (300 g/L), the FR fabrics obtained were washed 5 and 50 times according to the ISO 105 C06 washing standard at 40°C for 30 min. Polynomial regression analysis shows that as the washing cycles increase, the weight loss also increases which is inversely proportional to the flame resistance. This clearly indicates that with every additional wash there is a definite amount of loss in flame retardant finish. Figure 5 shows the relation between the washing process and the fabric flame resistance.

It is interesting to note that after 5th washing cycle
the weight loss after ignition of the fabrics increases by approximately 5 times compared to their initial value. The highest weight loss value is obtained after 50th wash cycle.

4 Conclusions

The studies show that the applied FR types and application conditions considerably influence knitted fabric flame resistance. With the durable flame retardant, an increase in FR concentration causes flame resistance to increase although fabrics become harsh to handle above 350 g/L of FR application. Curing temperature has no significant effect on flame resistance of the fabric, but all samples that were cured at above 160°C are found to show unacceptable yellowing. This effect most likely has resulted from damage to cotton cellulose above this temperature. Flame resistance of the fabrics increases as the time of curing increases and samples cured for 6 min show the considerable yellowing. In normal commercial flame retardant finishing at 160°C, the curing times of the order of 3-5 min are acceptable and little or no yellowing occurs. Yellowing observed in this work is probably a consequence of poor temperature control and higher heating rates typically observed in small-scale experimental work. These results clearly indicate that the FR concentration, curing temperature and curing time are very important parameters to obtain FR fabrics having good flame resistance and acceptable handle and appearance.

The increase in weight loss with every additional wash of the FR fabrics also shows that every additional wash of the FR-finish fabrics causes considerable loss of flame retardant finish although they keep some level of flame retardance.

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