N-Methylol Melamine Resins in Protective Finishes for Cotton Fabrics

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Methylol melamine resins along with various inorganic salt combinations impart flame and microbial resistance to celluloses. To impart water repellency, silicone resins were used in the same as well as subsequent baths. The finish withstood drycleaning action and there was an improvement in protective properties owing to drycleaning.

Keywords: Cotton fabrics, Finishes, Flame retardancy, Methylol melamine resins, Microbial resistance, Water repellency

1 Introduction

Trimethylol melamine is the principal resin used to give clear weather-resistant finishes and it may be used with zinc acetate as catalyst and with titanium dioxide as sunlight screen1. The N-methylol melamine condensate with zinc or zirconium compounds may be polymerized in the fabric by the wet-cure technique with a high efficiency of polymer deposition and no loss in fabric strength2. According to Berard et al.3, a freshly prepared acid colloid of methylol melamine formaldehyde may be used to produce excellent rot and weather resistance. These colloids provide a surface barrier to the contact between the destructive organism and the cellulose substrate4. Cotton fabrics so treated could resist decomposition, in soil burial test, to microbial attack over the years5. According to the presently understood role of nitrogen in flame retardancy, nitrogen acts as a flame-retardant only in the presence of phosphorus, e.g. in compounds such as N-methylol dimethylphosphanopropionamide (NMDMPPA) and diammonium phosphate6.

Most of the resins, especially the melamine formaldehyde, impart stiffness to the finished fabric, so the finish necessitates the use of a softener. A silicone emulsion used along with the resins imparts softness as well as water repellency.

The combined water-repellent and flame-retardant finish is usually regarded very difficult as wherever such a combined finish was tried it was of poor quality in both the respects. The reason is that the active substances responsible for the two opposing effects counteract each other.

Attempts have been made in the past to prepare a finish which could impart flame, water and rot resistance to cotton textiles. Aflamman fast finishes7 imparted outstanding flame resistance with adequate water repellency. An aftertreatment with aluminium formate6 improved water repellency. Leblance8 developed a combined flame-, water-, weather-, and mildew-resistant finish using chlorinated paraffin, antimony oxide, calcium carbonate, aluminium stearate and pentachlorophenol. The finish, expected to be durable, adversely affected hand, drape, flexibility and colour of the fabric.

The present work explores the use of N-methylol melamine resin in combination with inorganic salts and silicone resins for imparting simultaneous9 flame, water and rot resistance to cotton textiles.

2 Materials and Methods

Closely woven cotton fabric having the following specifications was used:

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fabric weave</td>
<td>2/2 matt</td>
</tr>
<tr>
<td>Weight/m²</td>
<td>237 g</td>
</tr>
<tr>
<td>Count of yarn</td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>9.8 tex/2</td>
</tr>
<tr>
<td>Weft</td>
<td>13.4 tex/2</td>
</tr>
<tr>
<td>Threads/cm</td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>63</td>
</tr>
<tr>
<td>Weft</td>
<td>33</td>
</tr>
<tr>
<td>Breaking strength (100cm × 18cm)</td>
<td></td>
</tr>
<tr>
<td>Warp</td>
<td>227.5 kg</td>
</tr>
<tr>
<td>Weft</td>
<td>154.5 kg</td>
</tr>
<tr>
<td>Tolerance</td>
<td>5%</td>
</tr>
</tbody>
</table>

The experiments were divided into three groups. The first group consisted of different inorganic salts,
melamine formaldehyde and silicone resins. The amounts of different constituents are given in Table 1. The catalyst for silicone was 25% o.w.f. resin and that for formaldehyde resin, 20% o.w.f. resin.

The second group consisted of ammonium dihydrogen phosphate with melamine formaldehyde and silicones in different proportions (Table 2). The quantities of catalyst for silicone and melamine formaldehyde resin were the same as for group I.

The third group consisted of a two-bath method, where silicone was applied in the second bath with intermediate drying and curing (Table 3). The melamine formaldehyde resin, silicones and their catalysts used were Hico products, i.e. Hicofor MF resin and Katrang SI 40 respectively.

Drying and curing were done in all the cases at 100°C and 150°C for 3 min.

2.1 Determination of Air Permeability
The air permeability of the treated samples was determined as per BS 3424: 1973(71) by using WIRA air permeability tester.

2.2 Determination of Breaking Strength
The breaking strength of the fabric was determined by using the constant rate loading method as per IS : 7016 (pt II) 1973 and BS : 3424(IS) 1973.

2.3 Determination of Flame Retardancy
The flame retardancy of the samples was determined by using the vertical test method as per BS : 3119-1969.

2.4 Determination of Microbial Resistance
The culture growth on the treated and untreated samples was noted after exposure to microorganisms as per IS : 1389-1959 for a fortnight. The microbial resistance rating was done as per the culture growth on the fabric.

2.5 Determination of Water Repellency
The water repellency of the fabric was determined by using the spray rating method as per IS : 390-1975.

3 Results and Discussion
3.1 Air Permeability
The presence of melamine formaldehyde resins on the fabric has little effect on air permeability. The decrease in air permeability depends on the resin add-on. Different inorganic salts, when incorporated with melamine formaldehyde resins, did not interfere with the air permeability (Table 1).

The presence of phosphates in varying concentrations (Table 2) also confirms our earlier finding that the inorganic salts do not affect air permeability. The same result has been found true in the case of two-bath treatments (Table 3). The reason is that while the inorganic salts loosely adhere to the surface of the fabric, the resins form a polymer on the surface which is responsible for the decrease in air permeability of the fabric.

3.2 Flame Retardancy
The flame retardancy of cellulosics is improved by the presence of nitrogen. The nitrogen of melamine formaldehyde resin used in all samples imparts flame retardancy to the cotton fabrics. This property has been improved by the incorporation of sulphamic acid salts. These salts are stabilized with phosphate or dicyanamide (samples c and d). However, the results obtained with dicyanamide-stabilized sulphamic acid salts are not as good as those obtained with the phosphate-stabilized sulphamate (Table 1). The increase in the amount of either sulphamate or phosphate in the bath improved flame retardancy of cellulosics. These samples did not show any after-flame or after-glow.

Of the number of combinations of inorganic salts used for treatment, phosphate was selected for further investigation under group II owing to better flame retardance exhibited. The varying proportions of resin and phosphate show that the nitrogen-phosphorus synergism is responsible for good flame retardancy in almost all the samples (Table 2). The samples in which resin was absent (samples m and n) only phosphate could not impart the flame retardancy of a reasonable degree. The relationship between the flame retardancy and the amount of phosphate concentration (Fig. 1) shows that as the phosphate concentration in the treatment bath increases, the char length decreases. The effect of melamine formaldehyde concentration on flame retardancy (Fig. 2) shows that an increase in melamine formaldehyde concentration decreases char length and so improves the flame retardancy of the fabric.

The results of flame retardancy are not affected by the presence of silicones in the treatment bath. The silicones, whether used in the same bath with resin and inorganic salts or in a subsequent bath, do not affect flame retardancy.

3.3 Water Repellency
The water repellency improved by the use of zirconium and zinc salts but when phosphates were used, water repellency suffered a setback (Table 1, sample c). This was an expected result because flame retardancy and water repellency are incompatible characteristics.
However, when different concentrations of phosphorus compounds were used with methylol melamine formaldehyde and silicones, good water repellency was observed. In the case where no resin was used (Table 2, sample m), water repellency could not be improved without increasing the silicone resin concentration (Table 2, sample n).

In the case of two bath-treated samples (Table 3, samples I and II) it was observed that after the first bath if silicone treatment was not given to samples,
the treatment with zirconium and zinc salts did not show good results with regard to water repellency but as soon as the same samples were treated with silicone in the second bath an excellent water repellency was observed.

3.4 Microbial Resistance
All the samples treated with melamine resin showed good microbial resistance. However, the samples not treated with the resin but treated with ammonium dihydrogen phosphate also showed good microbial resistance. This may be attributed to the presence of phosphorus compounds, which are the strong inhibitors of both mildew and decay organisms.

3.5 Simultaneous Flame, Water and Rot Resistance
The results of various treatments with different inorganic salts show that in most of the cases it was possible to get two of the three retardancy requirements. Generally, the samples showed good water and microbial resistance and lacked in flame retardancy, and a few showed good flame and microbial resistance but poor water repellency.

A combination of zirconium nitrate, diammonium phosphate, melamine and silicone resin imparted good flame retardancy, with no after-flame and after-glow, along with very good water repellency and good microbial resistance (Table 1, sample f). In other cases, out of the various combinations of inorganic salts, only a reasonably high concentration of phosphoric acid salts imparted flame and rot resistance together with water repellency. In view of these results, further investigations were made with phosphate (Table 2) and it was observed that except in cases where resin was not used (samples m and n) all the samples showed good simultaneous flame, water and rot resistance.

When all the three constituents of the treatment bath, i.e. phosphates, silicone and melamine formaldehyde resins, were equal in amount, good flame, water and rot resistance was observed (Table 1, sample k). The increase in the phosphate concentration in the treatment bath increased flame retardancy at the cost of water repellency (Table 1, sample l). To compensate it, increased silicone concentration (Table 4, sample o) served the purpose. Thus, any increase in phosphate concentration should be balanced by an increase in the amount of silicones.

The double-bath results (Table 3) only support our earlier observations with the single bath that the presence of silicones in the formulation is a must for obtaining excellent water repellency. This also corroborates the conclusion that increase in phosphate concentration improves flame retardancy in single as well as two-bath treatment method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Active treatment constituents</th>
<th>Before dry-cleaning</th>
<th>After dry-cleaning</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Phosphate g/l</td>
<td>MF resin g/l</td>
<td>Silicone g/l</td>
</tr>
<tr>
<td>o</td>
<td>80</td>
<td>60</td>
<td>80</td>
</tr>
<tr>
<td>p</td>
<td>80</td>
<td>80</td>
<td>80</td>
</tr>
<tr>
<td>q</td>
<td>80</td>
<td>120</td>
<td>150</td>
</tr>
<tr>
<td>r</td>
<td>100</td>
<td>120</td>
<td>150</td>
</tr>
<tr>
<td>s</td>
<td>120</td>
<td>120</td>
<td>150</td>
</tr>
</tbody>
</table>
In all the above cases, nitrogen/phosphorus synergism from melamine resin and phosphates provide flame and rot resistance and the addition of silicones provides water repellency. Thus, a simultaneous finish is achieved in a number of cases.

3.6 Fastness of Treatment

The combination and treatments have been found fast to dry-cleaning agents. Also, after the dry-cleaning the flame retardancy and water repellency are improved (Table 4). Improvement in flame retardancy is owing to the nitrogen/phosphorus/chlorine synergism. Water repellency is improved both by dry-cleaning and leaching. The dry- and wet-cleaning wash off the crystalline material from the surface of the fabric and the smoothened surface is responsible for better water repellency.

4 Conclusion

N-methylol melamine resins could be used in various combinations with inorganic salts and silicones to produce concurrent protective finishes on cotton textiles. Nitrogen/phosphorus and nitrogen/phosphorus/chlorine synergism owing to these resins are responsible for protection against flame and microorganisms. Silicones play a significant role in these concurrent protective finishes for imparting water repellency.

References