RECENT DEVELOPMENT IN COMBINING FLAME-RETARDANT AND EASY-CARE FINISHING FOR COTTON

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Received August 2, 2012

Flame retardancy and easy-care properties are desired in several articles produced from cotton fiber. Therefore, chemical finishing of cotton fiber, using reduced consumption of water and energy, is obviously important. Apparently, developments in combining the flame-retardant finishing and easy-care finishing would be useful for the textile finishing sector. Any similarity in the type of reagents used in the finish bath and processing conditions, including padding, drying and curing, presents interest as to combining the two separate finishing processes. Flame-retardant and easy-care finishing of cotton fibers requires an appropriate cross-linking agent and catalyst. The finish composition may be applied through the pad-dry-cure processing sequence. Studies have shown the possibility of achieving flame retardancy and crease recovery properties in cotton fiber through combined flame-retardant, and easy-care finishing. Low-formaldehyde based cross-linking agent, or formaldehyde-free cross-linking agent including dimethyloldihydroxyethyleneurea and selected polycarboxylic acids are used. This paper describes some of the important recent development achieved in producing the flame-retardant and crease recovery properties in cotton fabric using combined finish liquor application.

Keywords: easy-care finishing, flame-retardant finishing, cellulose, phosphorous, cross-linking agents, catalysts, polycarboxylic acids

INTRODUCTION

Water and energy conservation in textile processing are deemed important. It is known that an energy saving of up to 10 percent has been achieved in the textile industry through basic house-keeping enhancement, including energy management and auditing. Textile dyeing and finishing is the major energy-consuming sector. Starting from fiber production to clothing manufacturing, 25 percent of the total energy is consumed in dyeing and finishing. Typical examples for the consumption of energy in the textile industry can be observed in the figures produced for each specialized section of the Japanese textile industry, shown in Table 1.

The energy consumption is different in dry and wet processing, increased energy being consumed in wet processing. The energy consumed in dry processes, such as knitting and weaving, has been found to be of 1.2 kWh/kg, and 6.2 kWh/kg, respectively. However, for finishing, it rises to 17.9 kWh/kg. Steaming, drying, curing and other heating operations in finishing consume a significant amount of energy.

Mattioli et al. investigated the use of water in textile processing in ten selected textile processing industries and identified preparation, dyeing, printing and fabric washing as the major water consuming processes.

The Asian continent houses a significant number of traditional textile processing industries working as commissioned processing agents for brand producers. Significant opportunities exist in processing at reduced energy and water levels. Research and innovation are used for minimizing water and energy consumption in the design and manufacturing of machines and in the development of finishing and dyeing processes.

A number of approaches are being investigated in textile finishing for developing processes that would consume reduced levels of water and energy. These approaches are based on combining the dyeing or printing with finishing, and
combining the two or more preparatory or special finishing processes. For example, combining dyeing and easy-care finishing has been investigated for cellulose fibers using acid, direct, vat and reactive dye types with dimethylhydantoinurea, melamineformaldehyde, ureaformaldehyde, cyclic urea derivatives and nitrogen methylolated finishing agents. In preparatory processes, oxidative desizing provided combined desizing and scouring using hydrogen peroxide or persulphate.

Nitrogen methylolated finishes are increasingly replaced by polycarboxylic acids in dyeing of cotton, using direct dyes.

The continuous textile finishing processes are accompanied by drying and curing stages at increased temperature, resulting in emissions of gases and vapors harmful to the environment. Such gas and vapor emissions would be possibly reduced in combined finishing operation. For example, with increasing industrial development, China has become one of the major countries producing higher carbon dioxide emissions than the United States. Realizing the significance of energy and emission reduction, the eleventh five-year plan of China was targeting to reduce energy consumption per unit of gross domestic product by 20 percent, and to cut total emissions by 10 percent for major pollutants by 2010.

Obviously, the idea of performing combined finishing processes attracted interest in textile processing industries for reducing expenses of water and energy and introducing an enhanced environment-friendly technology, provided the desired effects of finishes in fibers were achievable. The possible advantages are summarized in Table 2.

### Table 1
Energy consumption (in million Yen*) in the Japanese textile industry

<table>
<thead>
<tr>
<th>S. no.</th>
<th>Specialized section</th>
<th>Fuel</th>
<th>Electricity</th>
<th>Total</th>
<th>Percentage share in total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Fiber production</td>
<td>32551</td>
<td>21498</td>
<td>54049</td>
<td>21.0</td>
</tr>
<tr>
<td>2.</td>
<td>Spinning</td>
<td>3224</td>
<td>44262</td>
<td>47480</td>
<td>18.4</td>
</tr>
<tr>
<td>3.</td>
<td>Twisting</td>
<td>219</td>
<td>1660</td>
<td>1879</td>
<td>0.7</td>
</tr>
<tr>
<td>4.</td>
<td>Textured yarn production</td>
<td>120</td>
<td>1543</td>
<td>1663</td>
<td>0.6</td>
</tr>
<tr>
<td>5.</td>
<td>Weaving</td>
<td>4467</td>
<td>24848</td>
<td>29315</td>
<td>11.4</td>
</tr>
<tr>
<td>6.</td>
<td>Knitting</td>
<td>4059</td>
<td>11709</td>
<td>15858</td>
<td>6.1</td>
</tr>
<tr>
<td>7.</td>
<td>Dyeing and finishing (knitted and woven)</td>
<td>37661</td>
<td>28412</td>
<td>66073</td>
<td>25.0</td>
</tr>
<tr>
<td>8.</td>
<td>Clothing manufacturing</td>
<td>8240</td>
<td>15420</td>
<td>23660</td>
<td>9.2</td>
</tr>
<tr>
<td>9.</td>
<td>Others</td>
<td>5959</td>
<td>12000</td>
<td>17959</td>
<td>7.0</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>96500</td>
<td>161442</td>
<td>257942</td>
<td>100.0</td>
</tr>
</tbody>
</table>

*1 JPY = 0.0111203780929 USD, exchange rate as on February 12, 2010
(Source: http://www.bestcalculator.org)

### Table 2
Saving in combined finish operation

<table>
<thead>
<tr>
<th>S. no.</th>
<th>Possible saving</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Time</td>
<td>Finish bath preparation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Overall processing time</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Drying</td>
</tr>
<tr>
<td>2.</td>
<td>Heat energy</td>
<td>Curing/Fixation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Washing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Drying</td>
</tr>
<tr>
<td>3.</td>
<td>Water</td>
<td>Water in liquor bath preparation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Water consumed in washing</td>
</tr>
<tr>
<td>4.</td>
<td>Vapor and gaseous emissions</td>
<td>Drying</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Curing</td>
</tr>
<tr>
<td>5.</td>
<td>Labor</td>
<td>Overall process performance</td>
</tr>
</tbody>
</table>
A number of cotton articles produced are required to exhibit easy-care and flame-retardant properties. This paper identifies and describes the possibility of combining easy-care and flame-retardant finishing of cotton fabric using combined finish bath containing the finishes and any desired auxiliary. The fabric can be finished through the pad-dry-cure processing sequence. Important finishing agents used in easy-care and flame-retardant finishing of cotton and their effects on the desired properties of cotton are described. Possible objectives in this area may be: (i) to improve the desired effects on the fabric; (ii) to reduce energy/water consumption; (iii) to reduce the process cost.

Any similarity in the finish bath composition, application and processing conditions used would be interesting in developing combined finish processing. Such similarity could be possibly achieved in easy-care finishing and flame-retardant finishing of cotton fabric (Table 3). The cotton fabric might be padded through finish liquor containing the reagents used in easy-care and flame-retardant finishing followed by drying and fixation steps.

The important finishing reagents used in easy-care and flame-retardant finishing of cotton are described in the following sections. Subsequently, studies addressing the possibility of combining the flame-retardant and easy-care finishing are discussed.

**FORMALDEHYDE-FREE EASY-CARE FINISHING**

The requirements for reducing or eliminating creasing, and time-consuming ironing of cotton and other cellulose products determined the application of easy-care finishing, using cross-linking agents. This aspect was approached in the early 20th century, when cellulose esterification emerged as a remedy to creasing in cellulose fibers. The permanent cross-linking of cellulose molecules may be described as one of the greatest breakthroughs in the textile chemistry of all times. Cross-linking of cellulose macromolecules (i.e. introducing bonds between two or more adjacent cellulose macromolecules by the application of chemical finish) is now an established finishing, enabling fibers to recover from creasing.

An interesting review by Peterson discussed the chemistry of cross-linking agents and the performance in cross-linking the cellulose fibers. It was indicated that the cross-linking reaction occurred exclusively in the non-crystalline region of cellulose. Therefore, the cross-linking agent with high reactivity and low molecular weights would produce an improved cross-linked structure of cellulose. Important finishing agents having the desired reactivity included N-hydroxymethyl and N-alkoxymethyl compounds of urea, cyclic urreas, carbamates and aminotriazines.

Recently, formaldehyde- and non-formaldehyde-based cross-linking agents, as well as new approaches in using cross-linking agents, including the nanocomposite technology and biotechnology, have been discussed. Several reviews on cotton and cellulose finishing have been reported. The earliest research attempts to produce easy-care cellulose fibers by utilizing formaldehyde-based products were focused on formaldehyde, dimethylolurea, dimethylolethyleneurea (DMEU), dimethyloldihydroxyethyleneurea (DMDHEU) etc.

However, the seriousness of health hazards associated with formaldehyde, which is suspected to be a human carcinogen causing nasal cancer and lung cancer, required the search for alternative finishes. The mutagenic activity of various aldehydes made it imperative to find finishes with very low levels of formaldehyde, or alternative formaldehyde-free finishes. Dimethyloldihydroxyethyleneurea (DMDHEU), and its methylated, and glycolated derivatives still form the basis for a large number of cross-linking agents used in processing industries. 1,3-dimethyl 4,5-dihydroxy 2-imidazolidinone (DHDMI) reacts through its hydroxyl groups with cellulose to form stable ether linkages, utilizing the same catalysts and application procedure required for DMDHEU. However, the absence of -CH\_2OH groups in DHDMI results in zero-level formaldehyde release.

Polycarboxylic acids, particularly 1,2,3,4-butaneetetracarboxylic acid (BTCA), and citric acid have attracted significant research interest as formaldehyde-free easy-care finishing agent. These acids can form an ester cross-link structure with cellulose. By forming two or more ester bonds with cellulose macromolecules, these can produce a cross-linked fiber structure possessing a high level of crease recovery. In the case of dyed or printed fabric, the yellowness, and shade changes produced were desired to be minimum in the finished fabric, when using BTCA or citric acid.
Table 3
Similarity between easy-care and flame retardant finishing of cotton fabric

<table>
<thead>
<tr>
<th>S. no.</th>
<th>Method/ Material</th>
<th>Easy-care finishing</th>
<th>Flame retardant finishing</th>
<th>Possible similarity</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Padding</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>Done at defined wet pick-up</td>
</tr>
<tr>
<td>2.</td>
<td>Drying</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>Conventionally at 60 °C for 5 minutes</td>
</tr>
<tr>
<td>3.</td>
<td>Fixation/ Curing</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>Conventionally at 160 °C for 3 minutes, however other fixation conditions may be employed depending upon flame-retardant requirements</td>
</tr>
<tr>
<td>4.</td>
<td>Washing</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>-</td>
</tr>
<tr>
<td>5.</td>
<td>Cross-linking</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>For improving crease recovery properties, and wash durability of flame-retardant finish</td>
</tr>
<tr>
<td>6.</td>
<td>Catalyst</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>Catalyst required for cross-linking reaction</td>
</tr>
<tr>
<td>7.</td>
<td>Auxiliaries (wetting agent, anti-foaming etc.)</td>
<td>√</td>
<td>√</td>
<td>√</td>
<td>-</td>
</tr>
</tbody>
</table>

Conventionally, weakly basic catalysts were used to enhance the cellulose cross-linking produced by polycarboxylic acids at a selected elevated temperature. These catalysts might be the alkali metal salts of phosphorous-containing mineral acids, including the salts of phosphoric, phosphorous, hypophosphorous, and polyphosphorous acids.

Phosphorous-containing compounds were used in imparting flame retardancy to cellulose fibers. Phosphorous-based flame-retardant (FR) finishes functioned when the fiber structure was combustible through the char formation.

Importantly, at present the research work in easy-care finishing of cellulose fibers using polycarboxylic acids is concentrated on the study of crease recovery and strength properties. Therefore, it might be interesting to assess the effects of polycarboxylic acids, phosphorous-containing reagents, and any additives if necessary, to explore the level of dry crease recovery angles, and flame retardancy achieved in finished cotton fabric.

Studies focusing on polycarboxylic acids, particularly 1,2,3,4-butane-tetracarboxylic acid (BTCA) indicated an appreciable level of crease recovery angles in the finished fabric. It showed comparable finishing effects of crease recovery on cellulose fibers relative to DMDHEU, however, it required a relatively higher production cost. Its isomer, 1,1,4,4-butane-tetracarboxylic acid, was also used, but it was found less effective. Some of the means attempted to enhance the BTCA finishing effects included the two-step finishing (first finishing with citric acid and then with BTCA), and combining BTCA with boric acid in finishing treatment, using various catalysts. The two-step method produced some improvement in crease recovery relative to the combined application of BTCA and citric acid.

The combined application of BTCA and citric acid produced significant crease recovery, and an enhanced retention of tensile strength relative to the separate applications. Unsaturated acrylic and maleic acids were also used as non-formaldehyde crease recovery finishing agents. These two acids were copolymerized and used to finish cotton. This copolymer imparted crease recovery angles comparable to those achieved with DMDHEU, while reducing fabric strength and whiteness.

Selected carboxylic acids, including succinic acid (SA), malic acid (MA) and tartaric acid (TA), imparted flame-retardant finishing effects to cotton fleece fabrics. Any of the said carboxylic acids can esterify cotton fibers in the presence of sodium hypophosphite used as catalyst. The flame retardancy produced in cotton fleece using any of these carboxylic acids was assessed in accordance with the Federal Flammability Standard for general apparel (16 CFR 1610). It was observed that the mentioned...
three carboxylic acids were effective in reducing the flammability of cotton fleece fabric from ‘class 3’ to ‘class 1’.

Succinic acid was found to be the most reactive and the most hydrolysis-resistant, and showed the highest bursting strength retention and the least fabric yellowing. However, no data have been reported to indicate any effect on the easy-care or durable press properties of finished fabrics. Flame retardancy in cotton fleece imparted by succinic acid was produced through the reduction in peak heat release rate and heat release capacity.

Catalyst systems in polycarboxylic acids based finishing

The influence of catalyst in cellulose cross-linking was not limited to crease recovery, also showing significant effects on various fabric properties, including strength, wettability and whiteness etc. The cross-linking of cellulose fibers using polycarboxylic acids required the use of an appropriate catalyst system. The most convenient catalysts were the alkali metal salts of phosphoric, phosphorous, hypophosphorous, and polyphosphoric acids. Sodium hypophosphite (SHP) was observed as a more appropriate catalyst. In the order of decreasing effectiveness, these catalysts are described as follows:

\[
\text{Na}_2\text{HPO}_4 > \text{Na}_3\text{HPO}_3 = \text{NaH}_2\text{PO}_3 > \text{NaH}_2\text{PO}_4 > \\
\text{Na}_4\text{P}_2\text{O}_7 > \text{Na}_2\text{P}_2\text{O}_7 > \text{Na}_3\text{P}_2\text{O}_{10} = (\text{NaPO}_3)_2 > \\
\text{Na}_2\text{HPO}_4 = \text{Na}_2\text{PO}_4 > \text{Na}_2\text{CO}_3
\]

This order is based on the durable press rating, rate of curing, durability of finish to laundering with an alkaline reagent.

In the case of finishing using citric acid, the required quantities of SHP catalyst can be replaced up to 30% by diethylenetriaminepentamethylenephosphoric acid (DETPMA), and up to 50% by 1-hydroxyethane 1,1-diphosphonic acid (HEDPA). Such a replacement produced similar wrinkle recovery angles obtainable with the catalyst SHP alone in an equivalent quantity.

Flame-retardant finishing

Phosphorous-containing organic and inorganic compounds have been long known for improving the flame retardancy of cellulose fibers. In 1821, Gay-Lussac obtained a finish for linen and jute based on a mixture of ammonium phosphate, ammonium chloride, and borax. It was inferred that the most effective flame-retarding salts either exhibited low melting points, producing a glassy coating over the substrate, or decomposed into non-flammable vapors, resulting in the dilution of flammable gases released from cellulose combustion. Any of these effects retarded the flame.

The variety of phosphorous salts available provided a range of flame-retardant finishes for cellulose fibers in terms of imparting flame-retarding performance, and durability to washing or laundering.

The non-durable finishes are appropriate for disposable items, including fabrics, insulation wallboards, theatrical scenery, packaging material etc. These finishes consist in inorganic salts or acids, such as phosphoric acid, boric acid, borates, zinc chloride, and bases like sodium hydroxide, and potassium carbonate.

The corrosive nature of acids and bases limits their use and the salts preferred are mainly ammonium and diammonium phosphates, urea phosphates, and ammonium sulfamates. Generally, they produce a viscous melt on heating on the fiber surface, which creates a physical barrier and promotes char formation.

**Phosphorous-nitrogen-based flame-retardant finishing**

Insoluble phosphate salts of tin, zinc, and aluminum, and salts containing high molecular polyphosphate ions, e.g. ammonium polyphosphate, provide semi-durable finishing effects (finish durable to 20 or more laundering cycles). The durable finishing effects are provided by the organophosphorous compounds, which chemically bond to cellulose or polymerize in the fiber matrix. The known phosphorous-nitrogen synergism, observed in flame-retardant finishing, led to the creation of finishes and formulations based on phosphorous and nitrogen. The two widely used types of such flame-retardants are dimethyl (3-[(hydroxymethyl) amino]-3-oxopropyl) phosphonate (Pyrovatex CP), and tetrakis (hydroxymethyl) phosphonium chloride (THPC) (Proban). The reaction conditions necessary to achieve higher levels of phosphorylation in cellulose, and its char-forming behavior using Proban Rhodia were discussed.

One of the mechanisms proposed in the functioning of P-N containing flame-retardants was the Lewis acidity of electrophilic phosphorous, which promotes cellulose
dehydration and char formation. The overall performance of such flame-retardants are summarized below:

1) nitrogen-synergized phosphorylation of cellulose to block levoglucosan formation;
2) Lewis acid-catalyzed dehydration, and some chain degradation, which is not conducive to levoglucosan formation; and
3) Cellulose cross-linking through phosphorylation, which enhances char formation.

Phosphorous is known as the most versatile constituent in flame-retardant formulations, organo-phosphorous finishes being today the subject of significant research studies for applications on cotton. The presence of melamine formaldehyde as cross-linking agent was found to increase the flame retardancy of finished cotton, and DMDHEU enhanced the finish durability to multiple laundry cycles when used with organo-phosphorous oligomers.

COMBINED EASY-CARE AND FLAME-RETARDANT FINISHING

Similarity in one or more constituents of two separate processes may be an opportunity to combine them in a single process (Table 3). For example, cross-linking agents are used in crease recovery finishing, and in pigment printing, and it was shown that these two processes can be combined. There have been made attempts to combine the easy-care finishing and flame-retardant finishing through a single bath application using the pad-dry-cure process.

Each of the flame-retardant and easy-care finishing processes induced cellulose cross-linking in cotton fibers, in the presence of a selected catalyst, using the dry heat fixation conditions at a selected elevated temperature. Typically, in a conventional finishing operation through the pad-dry-cure sequence, the conditions used might be 60 °C for around five minutes in drying, and 160 °C for 3 minutes in curing.

The earliest attempts in evaluating combined flame-retardant and easy-care finishing used the formaldehyde-based cross-linking agent, or a cross-linking agent that might release formaldehyde at any subsequent stage of processing or during the service life of the finished fabric. However, recent research investigated the formaldehyde-free cross-linking agents.

Possibly, an example of the earliest attempt of combining the effects of flame-retardant and easy-care finishing for cotton fabrics was made in 1968. A combination of cross-linking agent and tetracis(hydroxymethyl) phosphonium hydroxide (THPOH), urea and trimethylolmelamine (TMM) was applied through padding cotton fabric at wet pick-up of 75%, followed by drying at moderate temperature and curing at an increased temperature. The combination of THPOH/urea/TMM in aqueous solution had a total solids content of 24-34% at a molar ratio of 2:4:1. The finished cotton showed flame retardancy, minimal reduction (5-9%) in breaking strength and a relatively higher loss (20-27%) in tearing strength. Wrinkle recovery angles (warp + weft) of 280°-306° were obtained in the finished fabric, which showed no yellowing when bleached using sodium hypochlorite solution, and scorched between hot plates. Importantly, the flame retardancy was retained following the boiling of finished fabric in an aqueous solution containing soap and sodium carbonate for 3 hours or after 15 laundering cycles.

In 1970, another study showed the achievement of crease recovery (220°-270°) and durable flame retardancy (for over 50 home laundering) on 100% cotton fabric. The fabric was finished using the pad-dry-cure process through a single bath system comprising the reaction product of guanidines, dimethyl phosphate, and formaldehyde (GDMPF). The cotton fabric, finished with partially methylolated acrylamide, THPC, and zinc acetate, was evaluated. The fabric was cured and washed, and in the second step the fabric was padded with ceric ammonium nitrate catalyst, cured and washed. The treated fabric showed the desired conditioned crease recovery angles (warp + weft) and tensile strength retention. At crease recovery angles greater than 260°, the tensile strength loss was significant. However, in producing the desired properties of crease recovery angles and tensile strength, the phosphorous content in fabric was not sufficient, resulting in reduced flame retardancy.

There have been reported studies on flame retardancy in terms of ignition resistance to small flame exposure, for 12 seconds, and durable press properties using reagents, including oligomeric vinyl phosphonate, and either trimethylacetylenediurea or dimethyl 4,5-dihydroxyethyleneurea. Cotton broadcloth and polyester/cotton broadcloth were finished through
the pad-dry-cure process, and ignition resistance and durable press properties were discussed.\textsuperscript{44}

Diammonium hydrogen phosphate (DAP) and N-methylol resins were applied in combination, and separately through the pad-dry-cure processes to cotton, polyester/cellulose blends.\textsuperscript{45} The phosphorous-nitrogen synergism on phosphorylated cotton, in terms of limiting oxygen index, was reported for urea formaldehyde treatment. Trimethylol melamine treatment on phosphorylated cotton accelerated the $45^\circ$ angle burning rate. However, in the study of 48/52 polyester/viscose, a slight retardation in burning rate was indicated.

Several researchers reported the use of cross-linking agent in flame-retardant finishing of cotton or cellulose fiber blends. BTCA was preferred for its non-formaldehyde character. Cotton fleece fabric finished using a flame-retardant hydroxyl-functional organophosphorous oligomer (HFPO) and BTCA as bonding agent. Micro-scale calorimetry indicated the effectiveness of HFPO and BTCA in reducing cotton flammability. BTCA was efficient in reducing the peak heat release rate of finished cotton.\textsuperscript{46}

HFPO in combination with BTCA further reduced peak heat release rate (PHRR) and the temperature at PHRR, and significantly decreased the decomposition temperature of finished cotton. On adding triethanolamine (TEA) to the HFPO/BTCA, the flame retardancy of treated cotton passed Federal Regulation 16 CFT Part 1610, and achieved ‘class 1’ rating. The use of TEA enhanced the HFPO/BTCA performance without significant influence on the physical properties of the fabric. Moreover, the finish effect was durable to multiple home laundering cycles.

Flame-retardant finishing of Nomex/cotton blend using HFPO in the presence of binding agent BTCA, and TEA was investigated. The HFPO/BTCA/TEA treated Nomex/cotton fabric showed enhanced flame retardancy at reduced add-on levels. TEA was proved to be less hydrolysis-resistant, and to contribute to increased flame retardancy through the phosphorous-nitrogen synergism.\textsuperscript{47}

BTCA may cross-link with HFPO to cotton fibers in the presence of a catalyst, such as sodium hypophosphite (Na$_3$H$_2$PO$_4$). It was indicated that BTCA bound to cotton would produce an insoluble calcium salt during home laundering, resulting in the reduction of flame retardancy of finished cotton fabric. However, the use of triethanolamine (TEA) in the finish formulation produced a reduction in the formation of the said salt.

The free carboxylic acid groups on BTCA, present in treated cotton, were esterified by TEA, causing the suppression in calcium salt formation. Therefore, the flame retardancy was significantly improved.\textsuperscript{48}

The cotton fabric finished with HFPO, BTCA and a new catalyst system consisting of hypophosphorous acid (H$_3$PO$_3$) and TEA showed enhanced flame retardancy in cotton. The flame retardancy was assessed using the vertical flammability test (ASTM D 6413-99) and limiting oxygen index (ASTM D 2863-97).

Another research work studied the flame-retardant finishing of a nylon/cotton blend, with cross-linking agents dimethyloldihydroxyethyleneurea (DMDHEU) and trimethylolmelamine (TMM), used as binders for improving finish performance. The flame-retardant finish used was HFPO. The finish bath containing HFPO/DMDHEU/TMM was applied and evaluated for 50/50 nylon/cotton blend fabric and for nylon 6 6 and nylon- 6 fabrics. It was indicated that about 40% of HFPO applied to nylon fabric was bound through HFPO/TMM cross-linking, resulting in an improved durability to laundering cycles.\textsuperscript{49} The results indicated improved durability and flame retardancy using the cross-linking agents for both cotton and nylon fibers.

Higher flame retardancy levels and an excellent durability to laundering were obtained in the finished nylon/cotton blend fabric. The finished fabric passed the vertical flammability test following 50 home laundering cycles. The study aimed to evaluate flame-retardant finishing system for nylon/cotton blend fabric for use in protective clothing. Durable press finish DMDHEU and TMM were added to enhance the binding of the flame retardant, the research indicating the possibility to combine the application of durable finish – DMDHEU, with flame-retardant finish – HFPO.

In a similar investigation, melamine formaldehyde (MF) was applied to enhance HFPO binding on cotton fabric.\textsuperscript{50} It was shown that an increase in the ratio of DMDHEU/(DMDHEU + MF) with HFPO in finishing the cotton fabric resulted in enhancing the amount of HFPO bound to cotton, improved
durability to laundering and reduced tensile strength retention of finished fabric.

Phosphorous-nitrogen synergism was experienced and DMDHEU and MF were found to increase the nitrogen bound to cotton. However, MF was a more efficient nitrogen provider than DMDHEU.

The achievement of desired properties in finished cotton was related with adjusting the concentration of HFPO, and the ratio of the two binders (DMDHEU and MF) in the finish bath.

The achievements in the combined application of easy-care and flame-retardant finishes may provide the possible advantages of reduced processing time (by a 2-in-1 process), and the consumption of less labor, materials, and energy required, compared to performing the two processes separately. Moreover, the higher production cost of BTCA relative to DMDHEU were noted, therefore any alternative processing using less BTCA in producing higher crease recovery properties would be desired to reduce the process cost and fabric yellowing.

Generally, the studies in using cross-linking agents in flame-retardant finishing have not investigated the comparative merits of various cross-linking agents in achieving flame retardancy levels and crease recovery properties. Moreover, an optimum combined finish bath composition, and associated process conditions for achieving desired flame retardancy and easy-care properties require increased research efforts.

CONCLUSION

Combining the two finishing processes may offer the advantages of reduction in energy consumption, process cost, time and labor provided the desired effects achieved in the finish and fabric performance are similar or enhanced relative to the two processes performed separately.

The approach of combining two or more finishing processes was primarily supported by some level of similarity in the type of reagents, and the conditions used in the finishing process. Both the flame-retardant and easy-care finishes of cotton fibers generally require a cross-linking agent, and selected chemicals and auxiliaries. The finish application may be performed through padding followed by drying and curing. Cross-linking agents are used to enhance the binding and laundering durability of the flame-retardant finish.

Recently, polycarboxylic acids were studied as formaldehyde-free easy-care finishing agents, were evaluated for combined finishing with selected flame-retardants. The combined application of polycarboxylic acids, as cross-linking agent, and selected phosphorous-containing reagent as flame-retardant finish using an appropriate catalyst system would be viable, providing the desired performance levels of crease recovery and flame retardancy properties in fabric are achieved.

The identification of a possible combined finish bath composition, resulting in optimum flame retardancy and easy-care properties coupled with desired fabric strength and abrasion characteristics, would be important for further advancing the subject.

REFERENCES

Cotton