# Flame-Retardant Properties of Soybean Fabric Modified with *N*-Methylol Diakyl Phosphonopropionamide

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**ABSTRACT:** Soybean is a competitive production material for fibers as it is abundant and cost effective. However, an inherent deficiency of soybean fiber is its poor flame-retardant performance. In this study, the effect of *N*-methylol diakyl phosphonopropionamide (Pyrovatex CP) on the flame retardancy of soybean was investigated by the Limiting Oxygen Index (L.O.I.) and the vertical flammability test. Little benefit with regard to flame retardancy was found when soybean was treated with Pyrovatex CP in the absence of additives. However, the incorporation of Lyofix MLF in the finishing treatment (3% w/v) increased the L.O.I. values of soybean fiber and enhanced char formation as indicated by Thermogravimetric Analysis (TGA). Improved fastness to washing was observed at higher application levels of Lyofix MLF (6% w/v). X-ray Photoelectron Spectroscopy (XPS) indicated that surface phosphorus (% atomic) was reduced following washing for all fabrics examined. In addition, the substantivity of Lyofix MLF to soybean surface was exhibited. The flame-retardant treatment presented in this article is cost effective and results in wash-durable flame-retardant fabrics. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 117: 875–881, 2010

**Key words:** soybean; flame retardancy; L.O.I; X.P.S

## INTRODUCTION

The price of petroleum in 2008 reached an unprecedented high[1] with the economies around the world struggling with increasing inflation and industrial recession. In the textile industry, a new approach to this challenge could be the use of competitive, cost effective materials for fiber production. Although natural protein fibers such as wool and silk have good physical properties and have been utilized widely in the textile industry, they are relatively expensive to use and process. On the contrary, unlike most natural protein fibers, soybean is abundant and low-priced and therefore can provide an alternative production material for textiles.

The environmentally friendly production claimed by the manufacturers[2] in conjunction with the high protein content of soybean (40%) compared to peanuts (25%) and corn (10%) can lead to the manufacture of fibers with no theoretical limit in finess. In addition, the potential of plant protein modification by molecular genetic techniques could result in improved fiber properties for specific end-uses. The first attempts to manufacture textile fibers from soybean protein were carried out in Japan[3,4] and U.S.A.[5] in the 1940's. However, lately, the production of soybean fibers has gained ground again due to environmental concerns.[6,7]

One of the most fundamental technical deficiencies of the fiber is its inherent flammability, affecting the safety of the consumer. In our previous work,[8] we demonstrated that the modification of soybean with sulphamic acid provides flame-retardant properties to the fiber. However, low durability to laundering was observed upon washing. A cationic agent (Matexil FC-ER) that was applied subsequent to the finishing treatment was found to be beneficial in terms of durability to laundering. Even though, in our work, sulphamic acid treatment of soybean fabric imparted improved flame-retardant properties, the requirement in textile industry for a cost effective finishing treatment without aftertreatment is still evident. Phosphorus-containing reagents may provide an alternative process to this prospect.

It is well known that phosphorylation with phosphoric acids and derivatives results in hydrolytically unstable ester bonds in flame-retardant finishing.[9] Therefore, to overcome this negative aspect, Jones and Noone[10] prepared more stable flame retardants based on phosphoric acid esters. Although Tesoro et al.[11,12] demonstrated that phosphonoacetamide derivatives of cellulose impart acceptable flame retardancy, it has been shown that the most commercially successful phosphonates are the *N*-methylol diakyl phosphonopropionamides.[13]

Pyrovatex CP (Ciba) has been successfully used as a flame-retardant reagent for textile fibers, such as

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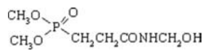

**Figure 1** Structure of Pyrovatex CP.

cotton,[14,15] nylon,[16] and lyocell.[17] It is a phosphorus-containingflame-retardant (*N*-methylol diakyl phosphonopropionamide)[18] with the structure shown in Figure 1. Kapura[19] found using NMR and HPLC analysis that Pyrovatex CP does not consist of an individual compound but of several species based on dimethoxyphosphorylpropionamide.

In this study, the effect of Pyrovatex CP and the role of a melamine formaldehyde additive (Lyofix MLF) on the flame retardancy of soybean fabric are examined. The surface interface is the primary region reached by the flame. As a result, surface protection, and modification are required for effectual flame retardancy and the safety of the consumer. Therefore, XPS was used in this study to determine the effect of Pyrovatex CP modification on soybean fabric, to establish the deposition of phosphorus on the fiber surface and to examine the effect of washing on surface Pyrovatex CP finishing.

## EXPERIMENTAL

### Materials

The soybean yarn, supplied by Huakang, China, was woven into a 1/2 twill, 100% soybean fabric (210g/$m^2$) and the fabric was scoured before use. Pyrovatex CP and Lyofix MLF were supplied by Ciba, USA. The standard ECE detergent was obtained from the Society of Dyers & Colourists, Bradford, UK.

### Chemical treatments

Soybean fabric was padded at 90% wet pick up (w.p.u.) with solutions containing 10–30% w/v Pyrovatex CP, 0, 3 and 6% w/v Lyofix MLF, 2% w/v phosphoric acid, dried at 90°C for 5 min, cured at 150°C for 30 sec, rinsed in running water, and finally tumble dried. The treated fabrics were washed in a Wascator FOM 71P, using a standard cotton 5A cycle, at 40°C with an overall 1.5 kg load of polyester fabric. Each washing cycle corresponded to 10 domestic washings. The standard ECE detergent was used with 1 g/L sodium perborate.

### Characterization of fibers and fabrics

The morphology of untreated and modified soybean fibers was examined by SEM and was carried out on a Hitachi S-300N instrument.

XPS measurements were carried out with a Kratos Axis Ultra XPS instrument. The samples were analyzed with a monochromatic Al*Ka* radiation source (1486.6eV) operating at base pressure of $3 \times 10^{-9}$torr. Wide survey spectra were recorded at a pass energy of 100 eV to determine the surface chemical compositions. High-resolution spectra were obtained at a pass energy of 20 eV to determine the chemical state of phosphorus. All surface analyses were obtained in triplicate in order to ensure reproducibility and confirm uniformity of treatment.

TGA measurements were performed on a TA Q100 instrument with a refrigerated cooling system (RCS). The thermal analyser was operated at a heating rate of 10°C/min and an environment of 100% $N_2$ gas flow with the gas rate being 50 mL $min^{-1}$. The start temperature of measurements was 35°C and 7 ± 0.1 mg of sample was utilized.

The residual amount of char remaining after heating was determined after heating to 700°C. The maximum degradation rate point (MDRP) for each sample was determined as the temperature at, which each differential DTG showed a peak.

The L.O.I. of soybean fabrics was measured according to BS 4599-2:1999 on a Stanton Redcroft FTA flammability test unit. L.O.I. values were calculated based on the following equation.

$$\text{L.O.I.}(\%) = \frac{[O_2]}{[O_2] + [N_2]} \quad (1)$$

The value presented here is the sum of the warp and weft direction values.

The burning behavior of treated soybean fabrics was also assessed with the BS 5438 vertical flammability test method. Fabrics of 180× 650 mm length and width were mounted on a suitable clamp and placed in a standard cabinet with a 2 mm/sec airflow. The bottom edge of the fabric was exposed to a standard flame for 1 sec and after the removal of the flame, the flame spread speed (mm/sec) was determined by the average of 10 measurements for both warp and weft directions. The Yellowness Index, YID, of the soybean fabrics was determined with a Datacolor International Spectrophotometer under illuminant D65/10° observer conditions.

The soybean fabrics were conditioned for 24 h at 20°C and 65% R.H. before testing on an Instron 5564 testing system. Tensile strength of the untreated and Pyrovatex CP modified fabrics were determined as the average of 10 measurements in the warp direction.

The Kawabata Evaluation System for fabrics (KES-F) measures the fabric's mechanical and surface properties at load levels typical of normal handling and end-use applications.[20] A full set of results uniquely defines the characteristics of a fabric and gives a 'fingerprint' of the fabric. Standard sample size was 20 cm x 20 cm with the laboratory conditions being 65% R.H. and 20°C.

**TABLE I**
**Effect of Pyrovatex CP and Lyofix MLF on the L.O.I, Flame Spread Speed, Tensile Strength, and Yellowness Indices of Treated Soybean Fabrics**

| Pyrovatex CP (w/v) | Lyofix MLF (w/v) | L.O.I. (%) ± SD | Flame spread speed (mm/sec) | Tensile strength (kN/m) ± SD | Yellowness index |
|---|---|---|---|---|---|
| Untreated | – | 18.8 ± 0.1 | 17 | 17.6 ± 0.1 | 42.1 |
| 0 | 3 | 19.3 ± 0.5 | 16 | 13.6 ± 0.8 | 44.3 |
| 0 | 6 | 19.4 ± 0.3 | 16 | 15.0 ± 0.6 | 45.2 |
| 10 | 0 | 20.5 ± 0.4 | 10 | 14.5 ± 1.1 | 43.2 |
| 10 | 3 | 23.4 ± 0.5 | 6 | 16.2 ± 0.7 | 46.2 |
| 10 | 6 | 23.7 ± 0.3 | 5 | 16.8 ± 1.1 | 47.4 |
| 20 | 0 | 21.1 ± 0.3 | 9 | 14.4 ± 0.5 | 43.5 |
| 20 | 3 | 24.3 ± 0.4 | 5 | 14.9 ± 0.5 | 48.9 |
| 20 | 6 | 24.6 ± 0.2 | 3 | 16.1 ± 0.6 | 50.2 |
| 30 | 0 | 21.0 ± 0.1 | 9 | 13.9 ± 0.7 | 43.7 |
| 30 | 3 | 23.1 ± 0.3 | 9 | 15.1 ± 0.8 | 50.3 |
| 30 | 6 | 23.5 ± 0.1 | 8 | 16.3 ± 0.5 | 52.1 |

## RESULTS AND DISCUSSION

### Effect of Pyrovatex CP concentration

The aim of this study was to examine the effect of Pyrovatex CP on the flame retardancy of soybean. The results indicated, Table I, a slight increase in the L.O.I. values with increasing concentrations of Pyrovatex CP.

Similar to soybean, little benefit was observed in terms of flame retardancy for cotton fabrics treated with Pyrovatex CP alone,[21] suggesting that additives were required. Tesoro et al.[11] have demonstrated the synergistic effect of nitrogen on phosphorus compounds, resulting in enhanced flame retardancy, with the application of a melamine formaldehyde compound in a phosphonate flame-retardant finishing.

Therefore, in this investigation, an etherified methylolated melamine reagent (Lyofix MLF), Figure 2, was incorporated into the pad formulation with the expectation to enhance flame retardancy.

The inclusion of Lyofix MLF at 3% w/v application level enhanced flame retardancy compared to the Pyrovatex CP treatment alone, Table I. This finding is not unexpected as melamine resins have been reported to be the most effective reagents for P-N synergism for cotton.[22,23] However, at higher concentration of Lyofix MLF (6% w/v), only a marginal improvement in the L.O.I. value was observed with a negligible beneficial effect on the flame-retardant properties. The vertical flammability test data support these findings where, although, the fiber modification with Pyrovatex CP initially reduced the flame spread speed, with increasing concentrations of Lyofix MLF an enhanced flame spread performance was achieved, presented in Table I. However, at higher concentrations of Lyofix MLF an increase in yellowness was observed for all 10–30 % w/v concentrations of Pyrovatex CP examined, suggesting discolouration of the fabric.

### Thermogravimetric analysis

In our study, the DTG curve was utilized to study the thermal behavior of treated fabrics. The DTG curve can be obtained by differentiating the dynamic TG curve in time and plotting the rate of weight loss as a function of temperature. The area under the DTG curve relates to the weight loss denoted by the TG curve.

DTG curve of untreated soybean showed a deep single peak, with MDRP occurring at 320°C. However, the DTG curve of the Pyrovatex CP treated sample demonstrated a shift of the MDRP to a lower temperature (300°C). In addition, with the incorporation of the Lyofix MLF (3% w/v) in the pad formulation, a significant shift of the MDRP to a lower temperature was observed (280°C), Table II.

The shift of MDRP to a lower temperature has been similarly observed for cotton fabrics treated with Pyrovatex CP and hexamethylol melamine.[21] Flame retardants possessing phosphorus-nitrogen bonds increase char production and reduce the formation of volatile combustibles.[24] In our study, it is likely that the nitrogen-containing reagent promoted the formation of P-N bonds in soybean fiber and obstructed the volatilisation of phosphorus atoms[25] resulting in the lowering of the MDRP. TGA-derived residual char levels corroborate the flame-retardant effect of Lyofix MLF. The 20% w/v Pyrovatex CP-6% w/v Lyofix MLF treated sample provides the highest char residues at 700°C (15.2%) compared to that of Pyrovatex CP alone (11.0%) as shown in Table II.

The char residues of the 20% w/v Pyrovatex CP-6% w/v Lyofix MLF treated samples increased about 49% relative to untreated soybean fabric. The

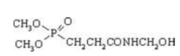


**Figure 2** Structure of etherified methylolated melamine.

**TABLE II**
**Effect of Pyrovatex CP and Lyofix MLF on the Maximum Degradation Rate Point and Char Residue of Treated Soybean Fabrics**

| Pyrovatex CP (w/v) | Lyofix MLF (w/v) | Maximum degradation rate point (°C) | Char residue at 700°C (%) |
|---|---|---|---|
| Untreated | – | 320 | 7.7 |
| 20 | 0 | 300 | 11.0 |
| 20 | 3 | 280 | 14.6 |
| 20 | 6 | 260 | 15.2 |

residual char in the modified fabrics plays the role of a diffusion barrier to volatiles emitted from the fabric, acting as a thermal insulation barrier and therefore forbidding heat transfer in the material.

The surface of the fiber plays an important role in heat transfer within the material. Therefore, it was decided to investigate the effect of the chemical treatment on soybean surface utilizing SEM. SEM analysis of fiber indicated longitudinal striations on the surface parallel to the axis, varying in length, and depth, Figure 3(a). In our trials, prolonged curing time (5 min) altered the structural characteristics of the fiber, Figure 3(b). Soybean became flatter with cracks appearing at the edges of the fiber, an indication of damage.

The inclusion of Pyrovatex CP into the pad formulation alone, did not result in a uniform finishing treatment. The phosphorus-based reagent was found to be mainly deposited along the center of the fiber surface, possibly due to preferential adsorption of the reagent with the fiber, Figure 3(c). However, when Lyofix MLF was used in the finishing treatment, Pyrovatex CP was successfully dissolved into the fiber, resulting in a more uniform treatment, Figure 3(d).

Losses in tensile strength following the flame-retardant finishing of cotton with Pyrovatex CP have been reported in the literature.[14] In our study, the effect of Pyrovatex CP modified soybean on the tensile properties of fabrics is presented in Table I. The results indicated that for all concentrations examined there was an increase in strength loss caused by the Pyrovatex CP treatment, which is possibly due to the acidity caused by phosphoric acid in the pad formulation. The use of an acid catalyst during the finishing treatment has been reported to affect the tensile properties resulting in the molecular degradation of the substrate with its excessive concentration being responsible for the rigidity of the fabric.[14]

In this study, increasing concentrations of Lyofix MLF in the finishing bath improved the tensile properties of the treated soybean due to the rigidity caused by the melamine resin. Nevertheless, in all

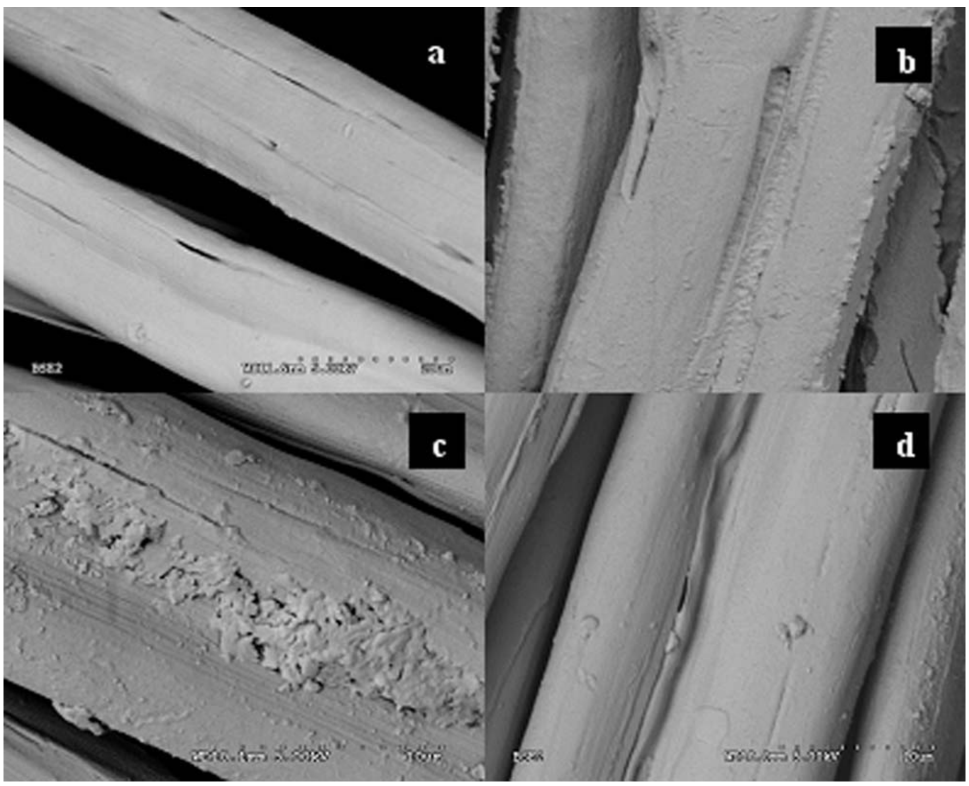


**Figure 3** SEM micrographs of (a) untreated soybean (b) cured at 150°C for 5min, (c) 20% w/v Pyrovatex CP, (d) 20% w/v Pyrovatex CP and 6% w/v Lyofix MLF.

**TABLE III**
**Effect of Pyrovatex CP and Lyofix MLF on the C/N and N/P Surface Atomic Ratios of Treated Soybean Fabrics**

| Pyrovatex CP (% w/v) | Lyofix MLF (% w/v) | C | O | N | S | P | C/N | N/P |
|---|---|---|---|---|---|---|---|---|
| Untreated | – | 74.7 | 19.8 | 3.2 | 0.3 | – | 23.3 | – |
| 20 | 0 | 71.6 | 22.9 | 5.1 | 0.2 | 0.5 | 14.0 | 10.2 |
| x1 | 0 | 71.9 | 23.5 | 4.0 | 0.2 | 0.4 | 18.0 | 10.0 |
| x3 | 0 | 72.4 | 23.7 | 3.3 | 0.2 | 0.4 | 21.9 | 8.3 |
| 20 | 3 | 68.8 | 23.7 | 7.2 | 0.2 | 0.5 | 9.6 | 14.4 |
| x1 | 3 | 68.6 | 24.7 | 6.3 | 0.2 | 0.4 | 10.9 | 15.8 |
| x3 | 3 | 69.6 | 24.9 | 4.9 | 0.2 | 0.3 | 14.2 | 16.3 |
| 20 | 6 | 68.7 | 22.9 | 7.9 | 0.3 | 0.5 | 8.7 | 15.8 |
| x1 | 6 | 68.3 | 23.5 | 7.6 | 0.2 | 0.4 | 9.0 | 19.0 |
| x3 | 6 | 68.6 | 23.9 | 6.9 | 0.2 | 0.3 | 9.9 | 23.0 |

where x is the number of launderings.

trials the tensile strength of the modified fabrics was still lower compared to that of untreated soybean.

## KES-F analysis

The KES-F is thought to be an objective measurement of textile fabric handle that measures the surface, bending, compression, tensile, and shear properties of fabrics.[20] The handle of modified soybean fabrics was assessed with the KES-F method focusing on mechanical properties such as bending hysteresis (2HB) and shear hysteresis at a shear angle of 5° (2HG5).

Following the treatment with Pyrovatex CP, at all application levels examined, the 2HG5 values for treated soybean were reduced indicating softer fabric. At 20% w/v levels of Pyrovatex CP, the 2HG5 value was reduced from 9.68 for untreated soybean to 6.43 gf/cm. Bending hysteresis, 2HB (gf.cm/cm), is a parameter that plays a part to the handle expression "stiffness". With the incorporation of Pyrovatex CP in the pad formulation the 2HB value increased from 0.242 for untreated soybean to 0.304 gf.cm/cm. The addition of Lyofix MLF in the flame-retardant system altered the bending rigidity of the samples. At 3% w/v application levels of Lyofix MLF, the 2HB and 2HG5 values increased significantly (0.949 and 11.05, respectively) indicating stiffer and less soft fabrics.

The observed increase in both 2HG5 and 2HB values for Pyrovatex CP/Lyofix MLF soybean denotes an increase in the interyarn and interfibre friction caused by the application of the melamine resin during treatment.

## XPS analysis

XPS was used to monitor the deposition of Pyrovatex CP at the fiber surface and to identify changes on the atomic composition following the inclusion of the melamine resin. Untreated soybean, Table III, does not contain phosphorus. However, examination of the P(2p) spectrum of Pyrovatex CP modified soybean indicated the presence of phosphorus species located at 135eV, which may be attributed to the $P^{+5}$ form of the phosphonopropionamide derivative,[26] Figure 4.

Increasing the concentration of Pyrovatex CP resulted in an increase in the surface phosphorus, Table III.

Nevertheless, the incorporation of Lyofix MLF into the flame-retardant system, altered the surface atomic composition. An apparent increase in the N/P ratio was observed at all concentration levels of Pyrovatex CP examined. Raising the concentration of Lyofix MLF led to an increase in the surface nitrogen with the intensity of the N(1s) spectrum becoming higher with increasing concentration of Lyofix MLF, Figure 5.

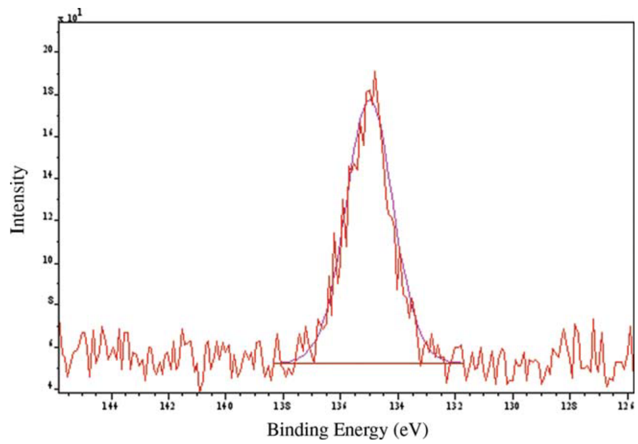


**Figure 4** P(2p) XP spectrum of 20% w/v Pyrovatex CP treated soybean, cured at 150°C for 30 sec. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

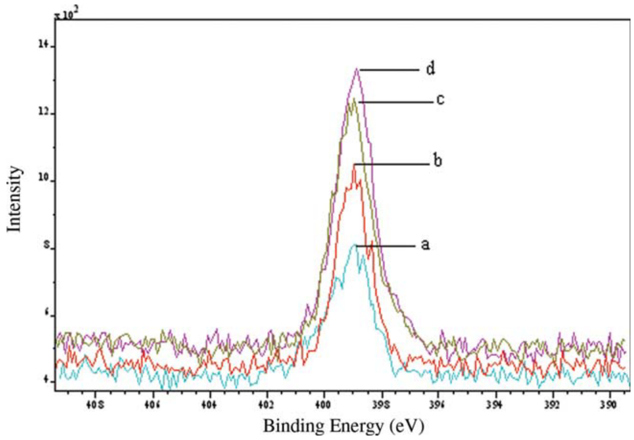


**Figure 5** N(1s) XP spectrum of (a) untreated soybean, (b) 20% w/v Pyrovatex CP modified soybean, (c) 20% w/v Pyrovatex CP, 3% w/v Lyofix MLF, (d) 20% w/v Pyrovatex CP, 6% w/v Lyofix MLF. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

## Durability to laundering

Flame-retardant finishing with Pyrovatex CP/Lyofix MLF system was found to be more beneficial with regard to flame retardancy compared to Pyrovatex CP alone. However, in textiles, it is of great importance that the finishing treatment is durable to laundering. Therefore, the effect of washing on the flame retardancy performance was investigated.

Successive washing cycles reduced the L.O.I. values for all treatments studied. However, after three washing cycles, the L.O.I. values for Pyrovatex CP were significantly reduced to the same value to that of untreated fabric, Table IV. The flame spread speed increased, from 9 mm/sec to 16 mm/sec, resulting in a fabric that had low protection against the flame.

In contrast to Pyrovatex CP treatment, the Pyrovatex CP/Lyofix MLF treated sample provided higher L.O.I. values after three washing cycles, demonstrating wash-durable flame-retardant properties. It is likely that the bonding of Lyofix MLF with Pyrovatex CP and soybean fiber is more resistant to hydrolysis during laundering than that between Pyrovatex CP and soybean. The highest flame retardancy performance was obtained with 6% Lyofix MLF with the loss in L.O.I. values reaching 3.3% after three washing cycles, Table IV. In addition, the flame spread was approximately 70% lower than that of untreated soybean.

The effect of laundering on the phosphorus and nitrogen content of treated cotton is well documented.[27,28] In addition, it is known that Pyrovatex CP is bound to cotton by its methylol group with the bonding being highly resistant to hydrolysis.[23] However, to our knowledge, the effect of laundering on the fiber surface of flame-retardant fabrics has not been investigated. Therefore, the enhanced durability to laundering imparted in the Pyrovatex CP/Lyofix MLF soybean fabric was investigated by the XPS technique to probe changes at the fiber surface following hydrolysis due to laundering.

In the light of XPS analysis it was revealed that for the Pyrovatex CP modified soybean fabric, washing after one and three cycles reduced the N/P ratio at the fiber surface, Table III. In contrast, after three washing cycles, the N/P ratio for Pyrovatex CP/Lyofix MLF treatment increased with increasing concentrations of Lyofix MLF. Although surface phosphorus content (% atomic) was reduced in all treatments, it is evident that nitrogen was retained on the surface. Therefore, the benefits imparted by Pyrovatex CP/Lyofix MLF system compared to the Pyrovatex CP system alone can be attributed to nitrogen "binding" on the surface, complexing with phosphorus and delivering associated enhanced flame retardancy.

## CONCLUSIONS

A phosphorus-based flame-retardant treatment, Pyrovatex CP, was investigated and found to only

**TABLE IV**
**Effect of Laundering on the L.O.I., Flame Spread Speed and Yellowness Indices of Treated Soybean Fabrics**

| Pyrovatex CP (w/v) | Lyofix MLF (w/v) | LOI (%) ± SD | Flame spread speed (mm/sec) | Yellowness index |
|---|---|---|---|---|
| Untreated | – | 18.8 ± 0.1 | 17 | 41.9 |
| 20 | 0 | 21.1 ± 0.3 | 9 | 43.5 |
| x1 | 0 | 19.1 ± 0.2 | 11 | 43.3 |
| x3 | 0 | 18.8 ± 0.3 | 16 | 43.1 |
| 20 | 3 | 24.3 ± 0.4 | 5 | 48.9 |
| x1 | 3 | 23.7 ± 0.1 | 7 | 48.7 |
| x3 | 3 | 23.0 ± 0.3 | 10 | 48.5 |
| 20 | 6 | 24.6 ± 0.2 | 3 | 50.2 |
| x1 | 6 | 24.1 ± 0.3 | 5 | 50.0 |
| x3 | 6 | 23.8 ± 0.1 | 5 | 49.8 |

where x as indicated previously

slightly increase the L.O.I. value of the soybean fabric. However, when a melamine-based resin, Lyofix MLF was incorporated into the pad formulation, the L.O.I. values and the char formation increased, demonstrating enhanced flame retardancy possibly occurring through a P/N synergistic effect.

Although, the flame-resist performance was marginally lower than that of the sulphamic acid/urea modified soybean,[8] this treatment is cost effective as it does not require any aftertreatment and provides wash-durable flame-retardant fabrics.

XPS analysis of the Pyrovatex CP treated soybean showed a P(2p) peak at 135.0 eV attributed to the $P^{5+}$ form of the phosphorus-based reagent. In addition, an increase in the N(1s) spectrum intensity was also found with increasing concentrations of Lyofix MLF incorporated into the pad formulation. The enhanced durability to laundering of the Pyrovatex CP/Lyofix MLF treated soybean was attributed to the formaldehyde-based system resulting in insolubilisation of the P/N flame-retardant at the fiber surface and within the bulk.

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