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The Flame Retardant Nomex/cotton and Nylon/Cotton Blend Fabrics for Protective Clothing

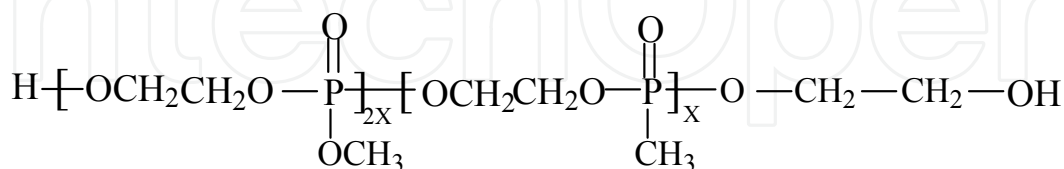
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1. Introduction

Due to its excellent fire-resistant property, Nomex has commonly been used to produce protective clothing [1, 2]. However, the high cost and low comfortability of Nomex have limited its wider uses. Blending Nomex with cotton not only reduces the cost but also improves comfortability of the fabrics. Because cotton is a highly flammable fiber, the Nomex/cotton blend fabric containing more than 20% cotton is not self-extinguishable [3-4]. Therefore, a durable flame-retardant finishing treatment becomes necessary to make the Nomex/cotton blend flame-resistant if it contains more than 20% cotton fiber.

Previously we developed a flame retardant finishing system for cotton based on a hydroxy-functional organophosphorus oligomer (HFPO) shown in Scheme 1. Because HFPO does not have a reactive functional group for cotton, it is necessary to use a bonding agent, such as dimethyloldihydroxyethyleneurea (DMDHEU), trimethylolmelamine (TMM), or 1,2,3,4-butanetetracarboxylic acid (BTCA), to make the flame retardant resistant to hydrolysis [5-12]. In this research, we developed a nonformaldehyde flame retardant finishing system for the Nomex/cotton using BTCA to bond HFPO to cotton by esterifying both HFPO and cotton.

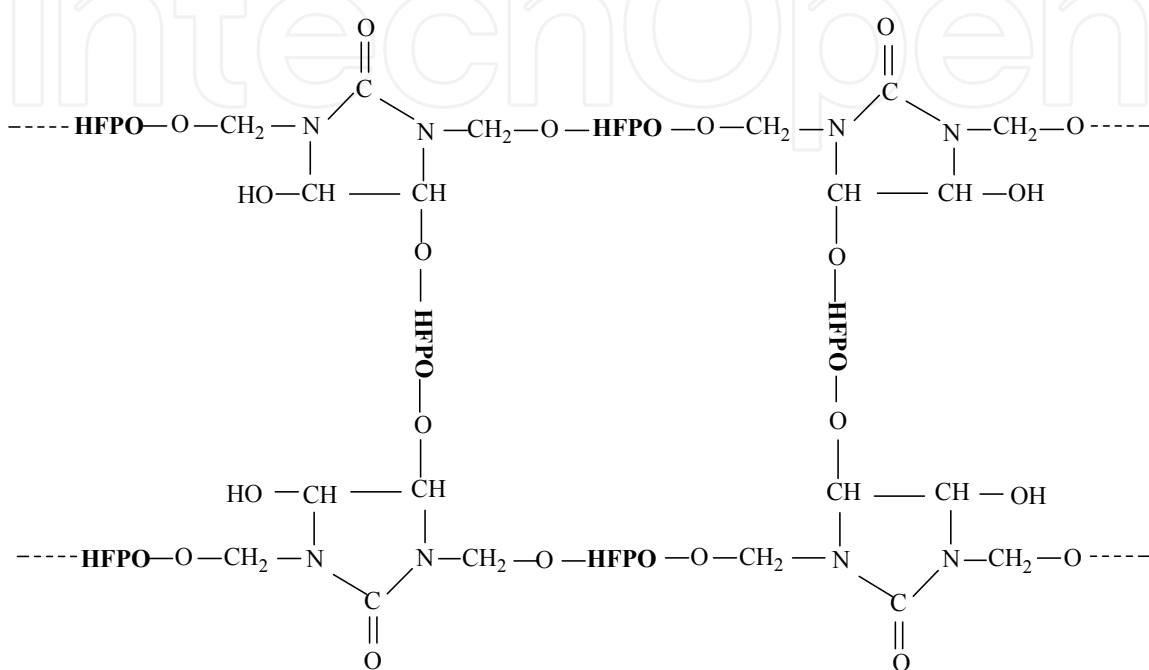


Scheme 1. A hydroxy-functional organophosphorus oligomer (HFPO)

Considering the high cost of Nomex, nylon/cotton blend is a more attractive alternative for use in protective clothing if the nylon/cotton fabric can be successfully flame retardant finished. The industry is still not able to produce flame-resistant nylon fabrics in spite of significant efforts made in the past 40 years [13-16]. It is even more difficult to impart flame retardancy to a blend of cotton and a synthetic fiber, such as cotton/nylon blend, than to each individual component fiber due to "scaffolding effect" [17]. The industry has yet to

develop effective, practical and commercially feasible flame retardant finishing system for nylon/cotton blend fabrics.

In this study, we investigated the bonding of HFPO onto nylon by DMDHEU, and found that HFPO can be bound to the nylon fabric in the presence of DMDHEU by forming a polymeric HFPO/DMDHEU system shown in Scheme 2. We also evaluated the performance of two 50/50 nylon/cotton batter dress uniform (BDU) military fabrics treated with HFPO/DMDHEU flame retardant finishing system.



Scheme 2. Formation of a Crosslinked Polymeric Network on Nylon

2. Experimental

2.1 Materials

The Nomex/cotton (35%/65%) blend fabric with woodland camouflage was a twill weave fabric weighing 219 g/m² produced in China. The nylon fabric was a 100% nylon 6.6 woven fabric (Testfabrics Style 306A) weighing 59 g/m². Two nylon/cotton blend BDU fabrics were used in this study: (1) a 50%/50% nylon/cotton BDU pure finish ripstop fabric printed with three-color "day desert" camouflage weighing 216 g/m² (military specification: MIL-C-44031 CL1); (2) a 50%/50% nylon/cotton BDU pure finish twill fabric with three-color "woodland" camouflage weighing 220 g/m² (military specification: MIL-C-44436 CL3), both supplied by Bradford Dyeing Association, Bradford, Rhode Island. HFPO under the commercial name of "Fyroltex HP" (also known previously as "Fyrol 51", CA Registry No. 70715-06-9) was supplied by Akzo Nobel Phosphorus Chemical Division, Dobbs Ferry, New York. N-methylol dimethylphosphonopropionamide (MDPA) under the trade name of "Pyrovatex CP New" (CA Registry No. 20120-33-6) was supplied by Ciba Specialty Chemicals, High Point, North Carolina. DMDHEU was a commercial product (44% aqueous solution) under the trade name of "Freerez 900" supplied by Noveon, Cleveland, Ohio. BTCA, triethanolamine (TEA) and hypophosphorous acid (H₃PO₂) were reagent-grade chemicals supplied by Aldrich, Wisconsin.

2.2 Fabric treatment and laundering procedures

The fabric was first immersed in a finishing solution, then passed through a laboratory padder with two dips and two nips, dried at 90°C for 5 min and finally cured in a Mathis curing oven. All concentrations presented here were based on weight of bath (w/w %). The wet pick-up of the nylon/cotton blend fabrics was 77±2% whereas that of the Nomex/cotton blend fabric was approximately 60±2%. After curing, the treated fabric was subjected to a specified number of home laundering cycles using a standard reference detergent (AATCC Detergent 1993) according to AATCC Test Method 124. The water temperature for laundering was approximately 46°C.

2.3 Evaluation of the flame retarding performance and stiffness of the fabric

The vertical flammability of the fabrics was measured according to ASTM Standard Method D6413. The limiting oxygen index (LOI) of the fabrics was measured according to ASTM Standard Method D2863. The fabric stiffness was measured according to ASTM Standard Method D6828 using a "Handle-O-Meter" tester (Model 211-300) manufactured by Thwing-Albert, Philadelphia. The slot width was 5 mm, and the beam size was 1000 grams. The fabric stiffness presented in this paper was the mean of measurements of 5 specimens.

2.4 Determination of phosphorus concentration on the treated fabric

Approximately 2 g of a treated fabric sample taken from three different parts of a "10 inches × 12 inches" fabric specimen were ground in a Wiley mill into a powder to improve sample uniformity. 2 ml of concentrated H₂SO₄ were added to 0.1 g of the powder in a beaker. 10 ml of 30% H₂O₂ were added dropwise to the mixture, allowing the reaction to subside between drops. The reaction mixture was heated at approximately 250°C to digest the powder and to evaporate the water until dense SO₃ vapor was produced. The completely digested sample as a clear solution was transferred to a 50 ml volumetric flask, then diluted with distilled/deionized water. The sample thus prepared was analyzed with a Thermo-Farrell-Ash Model 965 inductively coupled plasma atomic emission spectrometer (ICP/AES) to determine the phosphorus concentration. The percent phosphorus retention is calculated by: (the phosphorus concentration of the fabric after laundering) ÷ (that of the fabric before laundering) × 100%.

3. Results and discussion

3.1 Flame retardant finishing of the 65/35 nomex/cotton blend military fabric

The phosphorus concentration of Nomex/cotton blend fabric treated with 24% HFPO, 8% BTCA and 2.5% H₃PO₂ in combination with TEA at different concentrations and subjected to different home laundering cycles is presented in Figure 1. The data presented here show that the phosphorus concentration on the treated Nomex/cotton blend fabric first increased, then decrease as the TEA concentration increases in the range from 1.0% to 10.0% and the maximum phosphorus concentrations on the treated fabric are achieved at 4% TEA (Figure 1). The data indicate that the use of TEA also increases the percent phosphorus retention on the fabric after multiple laundering cycles. TEA has three hydroxyl groups in its molecule and is able to react with carboxylic acid groups of BTCA by esterification. BTCA also reacts with HFPO and cotton to form a BTCA/HFPO/TEA/cotton crosslinked network as shown

in Scheme 3, thus improving the laundering resistance of the HFPO on cotton. The data presented in Figure 1 also show that further increasing TEA concentration from 4% to 10% reduces the retention of HFPO after multiple launderings on the treated fabric. Because TEA, HFPO and cotton all have hydroxyl groups and they compete to react with BTCA, the presence of excessive amount of TEA reduces the reaction of BTCA with HFPO and cotton, thus reducing the bonding of HFPO on cotton as shown in Figure 1.

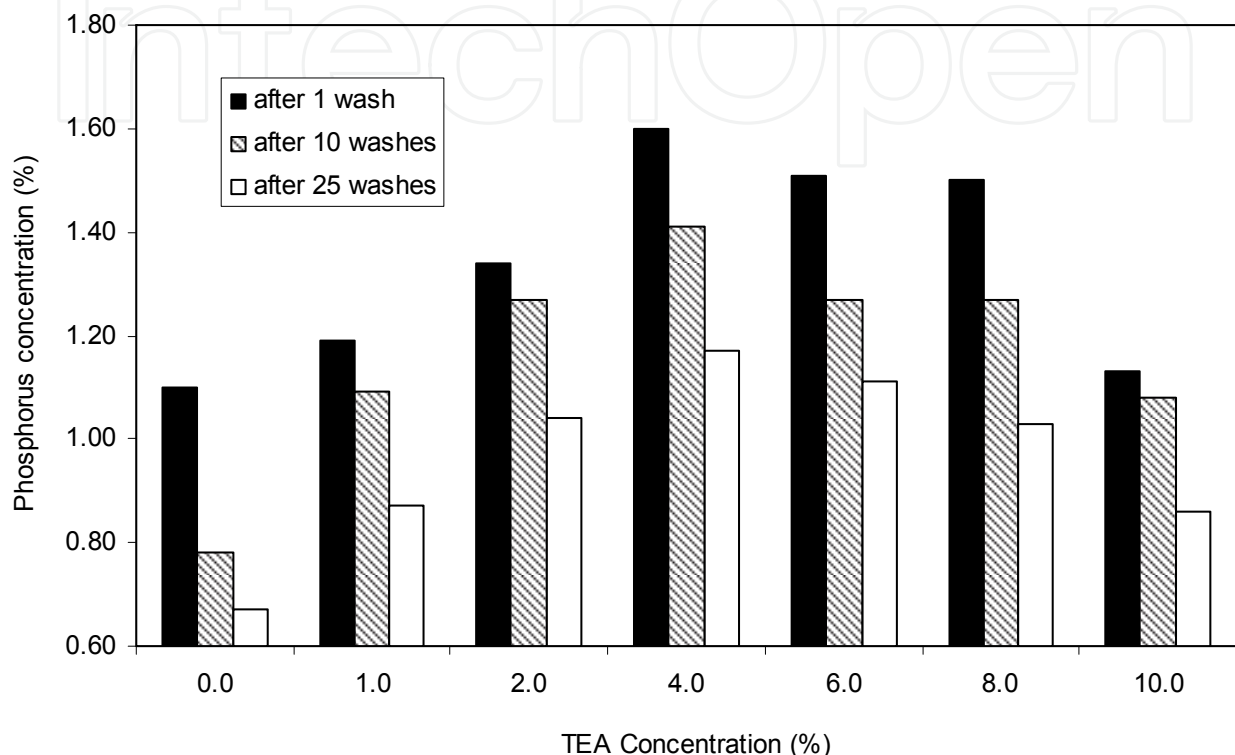
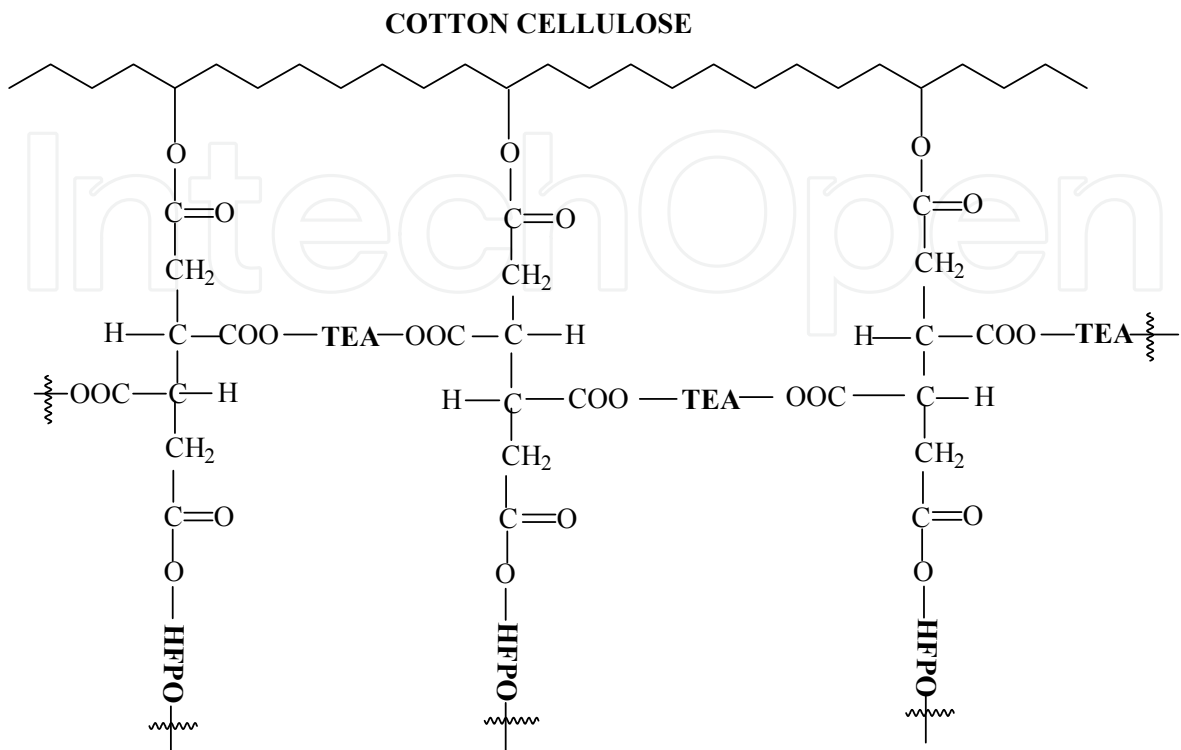


Fig. 1. The phosphorus concentrations of the Nomex/cotton blend fabric treated by 24% HFPO, 8% BTCA and 2.5% H_3PO_2 as a function of TEA concentration.

The Nomex/cotton fabric is treated with 24% HFPO, 8% BTCA, 2.5% H_3PO_2 and TEA at different concentrations. The fabric thus treated is cured at 180°C for 3 min. The LOI of the fabric thus treated (before washing) is plotted against the TEA concentration in Figure 2. Without being subjected to laundering, all Nomex/cotton fabric samples have the same HFPO and H_3PO_2 concentrations but different TEA concentrations. The LOI (%) of the fabric increases from 37.2 to 40.6 as the TEA concentration (%) increases from 0.0 to 8.0% (Figure 2). Thus, the data demonstrate the phosphorus-nitrogen synergistic effect of TEA in the HFPO/BTCA/ H_3PO_2 /TEA system on the Nomex/cotton blend fabric.

Previously, we found that calcium deposit formed on the cotton treated with HFPO/BTCA during laundering diminishes the flame retardant performance of the treated cotton fabric [11, 12]. We also studied the effects of TEA on the calcium deposit on the cotton fabric treated with HFPO/BTCA [12]. The calcium concentration on the treated Nomex/cotton blend fabric increases after multiple launderings is due to the formation of insoluble calcium salt of those free carboxylic acid groups of BTCA bound to cotton. We also found that the calcium concentration on the fabric after multiple launderings decreases as the TEA concentration is increased. The reduction of the calcium concentration on the treated

Nomex/cotton blend fabric as a result of the presence of TEA is attributed to esterification of the free carboxylic acid groups of BTCA on cotton by TEA as shown in Scheme 3.



Scheme 3. Formation of BTCA/HFPO/TEA crosslinked network on cotton

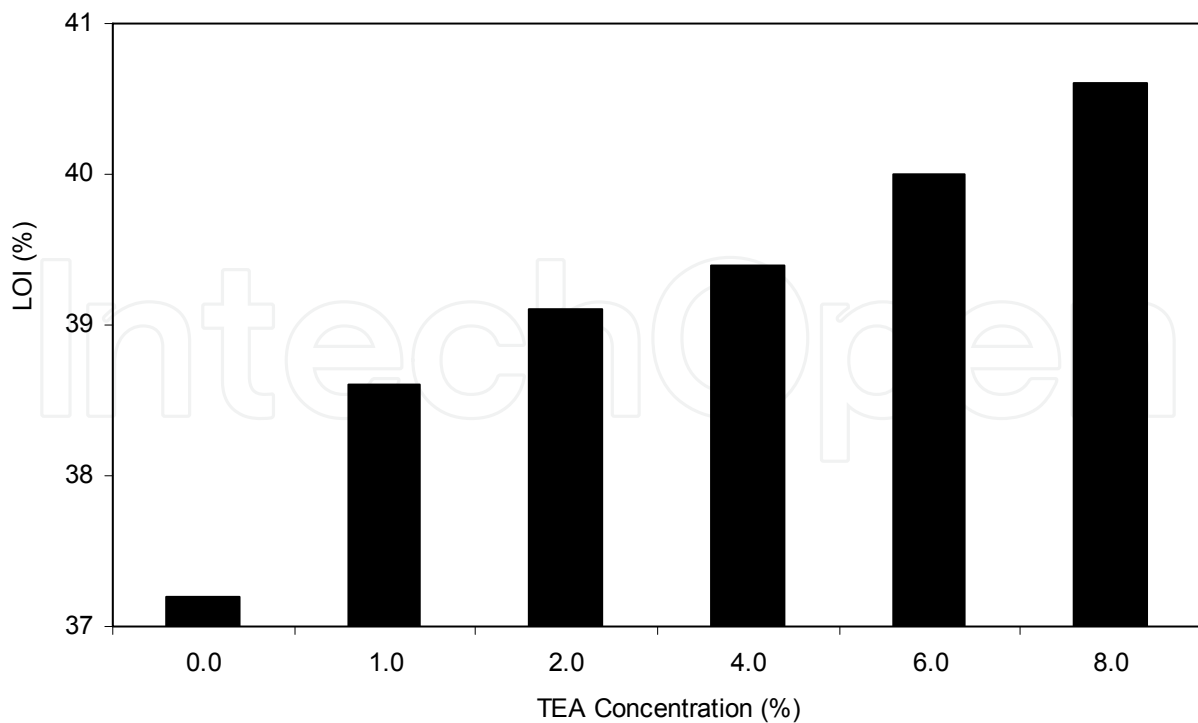


Fig. 2. LOI of the Nomex/cotton blend fabric treated by 24% HFPO, 8% BTCA and 2.5% H₃PO₂ in combination with TEA as a function of TEA concentration.

The Nomex/cotton blend fabrics were treated with 24% HFPO, 8% BTCA, 2.5% H_3PO_2 and TEA at different concentrations. The Nomex/cotton blend fabric thus treated was cured at 180°C for 3 min and finally subjected to 1, 10 and 25 laundering cycles. The LOI (%) of the fabric thus treated is shown against the TEA concentration (Figure 3). After 1 laundering cycle, the LOI of the treated Nomex/cotton blend fabric first increases from 32.1% without TEA to its maximum (36.3%) when 6.0% TEA is used. Further increasing TEA concentration reduces the LOI of treated Nomex/cotton blend fabric. Similar trends are observed on the treated fabric subjected to 10 and 25 laundering cycles. The optimum TEA concentration for the finish solution is in the 4.0-6.0% range. After 25 laundering cycles, the LOI of the fabric treated using 6.0% TEA is 30.5%.

The Nomex/cotton blend fabrics was treated with HFPO/BTCA/TEA (weight ratio: 3.0/1.0/0.75) at different concentrations and curried at 180°C for 3 min. The HFPO concentration increases from 12% to 24%, and the BTCA and TEA concentration are increased accordingly. The LOI and vertical flammability (char length) of the treated fabric after different laundering cycles are presented in Tables 1 and 2, respectively. The LOI of the Nomex/cotton blend fabric without treatment is 22.9% and it fails the vertical flammability test. All the Nomex/cotton fabric samples treated with the four HFPO/BTCA/TEA formulas pass the vertical flammability test after 30 laundering cycles (Table 2). The fabric treated with 12% (w/w) HFPO finishing solution (approximately 8% [w/w] HFPO on the fabric) has LOI of 26.5% and char length of 48 mm after 30 laundering cycles, demonstrating excellent flame retardant performance and superior laundering durability at a small flame retardant concentration on the fabric.

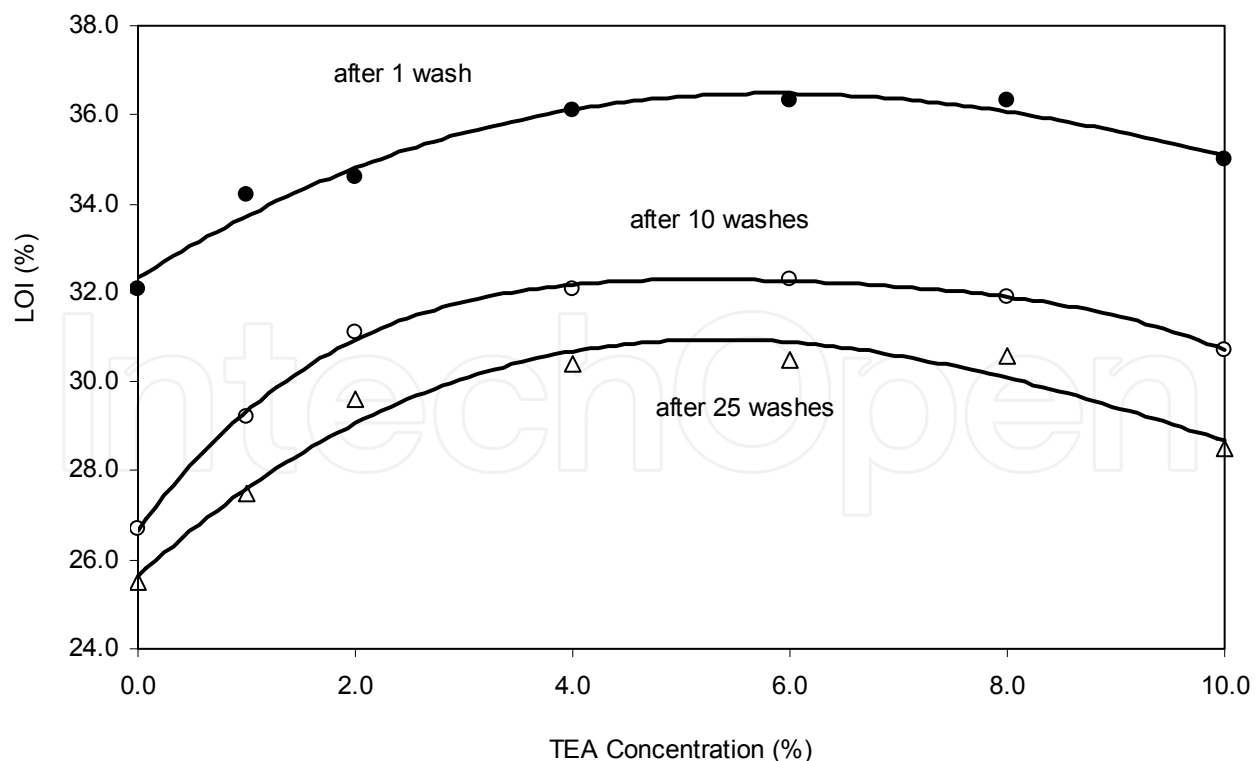


Fig. 3. The LOI of the Nomex/cotton blend fabric treated by 24%HFPO, 8%BTCA and 2.5% H_3PO_2 in combination with TEA as a function of TEA concentration.

HFPO (%)	BTCA (%)	H ₃ PO ₂ (%)	TEA (%)	Number of home laundering cycles				
				before wash	1 wash	10 washes	20 washes	30 washes
12	4	1.25	3.0	35.7	32.7	27.9	27.1	26.5
18	6	1.88	4.5	38.8	35.7	31.1	30.1	28.5
24	8	2.50	6.0	40.7	37.2	32.5	30.5	29.4
30	10	3.13	7.5	40.6	37.8	33.0	32.0	29.5
Control				22.9				

Table 1. The LOI of the Nomex/cotton fabric treated with HFPO/BTCA/H₃PO₂/TEA at the weight ratio of 24/8/2.5/6 and cured at 180°C for 3 min.

HFPO (%)	BTCA (%)	H ₃ PO ₂ (%)	TEA (%)	Number of home laundering cycles				
				No wash	1 wash	10 washes	20 washes	30 washes
12	4	1.25	3.0	37	34	41	44	48
18	6	1.88	4.5	28	29	43	27	35
24	8	2.50	6.0	27	31	35	34	31
30	10	3.13	7.5	27	31	30	38	32
Control				>300				

Table 2. The char length of the Nomex/cotton blend fabric treated with HFPO/BTCA/H₃PO₂/TEA at a weight ratio of 24/8/2.5/6 and cured at 180°C for 3 min.

The tensile strength of the treated Nomex/cotton blend fabric is summarized in Table 3. The tensile strength retention is 73-77% at the warp direction and 77-82% at the filling direction (Table 3). The fabric strength loss is due to acid-catalyzed cellulose depolymerization and crosslinking of cellulose [18]. The fabric strength loss after the flame retardant finishing process is modest. The effect of the treatment on the fabric hand property appears to be negligible. More details about the flame retardant Nomex/cotton blend fabric can be found elsewhere [19].

HFPO (%)	BTCA (%)	H ₃ PO ₂ (%)	TEA (%)	Tensile Strength (N)		Strength Retention (%)	
				Warp	Filling	Warp	Filling
12	4	1.25	3.0	405	262	73	80
18	6	1.88	4.5	414	271	74	82
24	8	2.50	6.0	409	254	74	77
30	10	3.13	7.5	427	270	77	82
Control				556	329	-	-

Table 3. The tensile strength of the Nomex/cotton blend fabric treated with HFPO/BTCA/H₃PO₂/ TEA at the weight ratio of 24/8/2.5/6 and cured at 180°C for 3 min.

3.2 The flame retardant finishing of nylon/cotton blend BDU fabrics

We first studied the bonding of HFPO to nylon fiber using DMDHEU as the bonding agent. The nylon 6.6 fabric was first treated with the combination of 32% HFPO and DMDHEU at

different concentrations, cured at 165 °C for 2 min, and finally subjected to 1 and 10 laundering cycles. The phosphorus concentration and the percent phosphorus retention of the nylon fabric thus treated are shown in Figure 4 and Table 4, respectively. When the DMDHEU concentration is increased from 1% to 8%, the phosphorus concentration of the treated nylon fabric after 1 laundering cycle increases from 0.21% to 1.75%, representing an increase in phosphorus retention from 9% to 75%, respectively. After 10 laundering cycles, 1.04% phosphorus (45% retention) remains on the nylon fabric treated with 8% DMDHEU.

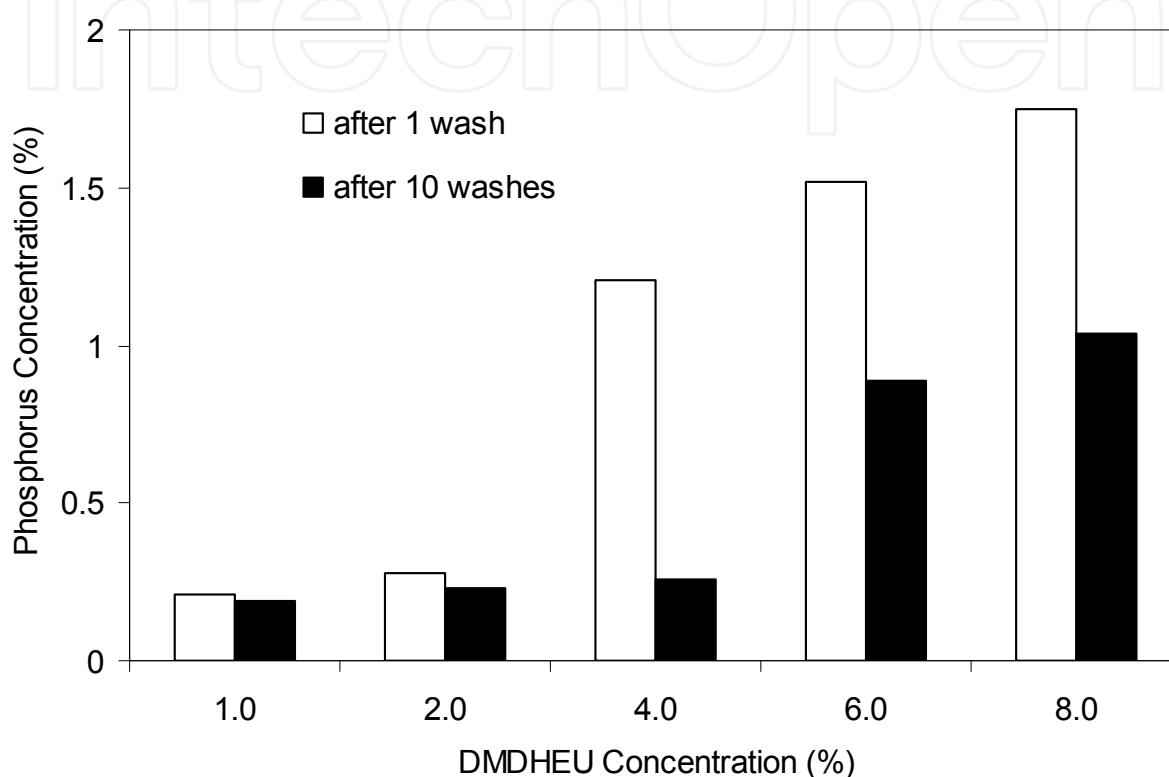


Fig. 4. The phosphorus concentration of the nylon-6.6 fabric treated with 32% HFPO and DMDHEU cured at 165°C for 2 min and finally subjected to 1 and 10 laundering cycles versus TMM concentration.

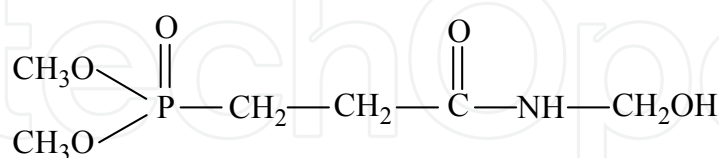
HFPO (%)	DMDHEU (%)	Molar Ratio (hydroxyl/hemi-acetal*)	Phosphorus Retention (%)	
			1 wash	10 washes
32	1	6.19	9	8
32	2	3.10	12	10
32	4	1.55	52	11
32	6	1.03	65	38
32	8	0.77	75	45

* The molar ratio of the hydroxy group of HFPO to the methylol group of DMDHEU.

Table 4. The percent phosphorus retention of the nylon 6.6 fabric treated with 32% HFPO and DMDHEU at different concentrations, cured at 165°C for 2 min and finally subjected to 1 and 10 laundering cycles.

The concentration of the terminal amine groups of nylon 6.6 in the fiber is small. Due to the poor penetration of the finishing solution into the fiber interior and the low reactivity of the terminal amine groups as a result of a high degree of crystallinity and H-bonding in nylon 6.6, the concentration of phosphorus bound to the fabric by nylon's terminal groups should be even smaller. The phosphorus concentration of nylon fabric treated with 32% HFPO without DMDHEU is 0.20% and 0.17% after 1 and 10 laundering cycles, respectively. The phosphorus concentration on the nylon fabric after 1 laundering reaches 1.21, 1.52 and 1.75 when 4, 6 and 8% DMDHEU, respectively, is used for the treatment. The data indicate that HFPO bound to the fabric is durable to multiple laundings. Therefore, the majority of HFPO on the fabric must be bound to nylon by its reactions with DMDHEU other than its bonding to nylon's terminal groups by a DMDHEU "bridge". DMDHEU has four chemically active methylol groups, and HFPO has two hydroxyl groups. The increase in the phosphorus retention as the DMDHEU concentration is increased shown here suggests the formation of a crosslinked polymeric network. A simplified version of the crosslinked polymeric network is shown in Scheme 2. The bonding of HFPO to the nylon fabric and the retention of HFPO on the fabric after multiple laundings can probably be attributed to the formation of the polymeric network on the fabric.

For the purpose of elucidating the bonding mechanism of HFPO to nylon by DMDHEU, we applied N-methylol dimethylphosphonopropionamide (MDPA, shown in Scheme 4) to the nylon fabric. A MDPA molecule has only one methylol group which may react with the terminal amine groups on nylon but it is not able to form crosslinked network with HFPO. The nylon 6.6 fabric was treated with 32% MDPA and DMDHEU at different concentrations, cured at 165 °C for 2min and finally subjected to 1 laundering cycle. The phosphorus concentration of the nylon-6.6 fabric thus treated is presented in Table 5. The phosphorus concentration on the nylon fabric treated using MDPA without DMDHEU is 2.53% before laundering, and it becomes 0.23% after 1 laundering (Table 5). Considering the fact that the same fabric treated with 32% HFPO without a bonding agent is 0.20% after 1 laundering, the phosphorus concentration on the fabric thus treated (0.23-0.26%) is negligible. It is also independent of the DMDHEU concentration used (Table 5). The small amount of MDPA bound onto the nylon fabric is possibly due to (1) the reaction between MDPA and the terminal amine group on the nylon fiber and (2) physical adsorption.



Scheme 4. MDPA

HFPO is a bifunctional compound and it is able to form a crosslinked polymeric network by its reaction with DMDHEU as shown in Scheme 2. Unlike HFPO, MDPA is mono-functional and is not able to form a crosslinked polymeric network in the presence of DMDHEU. The data presented here shows that the amount MDPA bound to nylon is negligible and is independent of the amount of DMDHEU used. Those facts are consistent with the hypothesis that HFPO reacts with DMDHEU on the nylon fabric to form a crosslinked polymeric network shown in Scheme 2, which makes HFPO on nylon resistant to laundering.

MDPA (%)	DMDHEU (%)	Phosphorus (%)*
32	0	0.23
32	2	0.26
32	4	0.25
32	6	0.24
32	8	0.23

Table 5. The phosphorus concentration of the nylon 6.6 fabric treated with 32% MDPA and DMDHEU at different concentrations, then cured at 165°C for 2 min, and finally subjected to 1 laundering cycle. (The phosphorus concentration on the fabric was 2.53% before laundering.)

We applied two different formulas (HFPO/DMDHEU and MDPA/TMM) to the 50/50 nylon/cotton fabric (Table 6). The nylon/cotton fabric samples treated with the two formulas have approximately the same phosphorus concentration (~3.80%) before laundering. The fabric was cured at 165°C for 2 min and finally subjected to 10 laundering cycles. The phosphorus concentration, LOI and char length of the fabric thus treated is shown in Table 6. The nylon/cotton fabric treated with MDPA has 1.20% phosphorus retained, and it has LOI of 23.9% and failed the vertical flammability test after 10 launderings. The fabric samples treated with HFPO/DMDHEU has LOI of 27.3% and passes the vertical flammability test. Evidently, the MDPA/TMM system is not suitable for the flame retardant finishing of the nylon/cotton blend fabric.

Flame Retardant	Bonding Agent	Catalyst	Phosphorus (%)	LOI (%)	Char length (mm)
HFPO 32%	DMDHEU 6%	NH ₄ Cl 0.12%	2.20	27.3	75
MDPA 45%	TMM 6%	H ₃ PO ₄ 2.0%	1.20	23.9	>300

Table 6. The LOI and char length of the 50/50 nylon/cotton blend fabric treated with different flame retardants and different binders and subjected to 10 laundering.

The nylon/cotton fabric (woodland) is treated with 32% HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min. The phosphorus concentration of the fabric thus treated and subjected to different numbers of laundering cycles is shown in Table 7. The phosphorus retention of the treated fabric is presented against the DMDHEU concentration in Figure 5. When DMDHEU increases from 1% to 10%, the phosphorus concentration of the treated fabric after one laundering increases from 0.56% (16% phosphorus retention) to 2.69% (78% phosphorus retention). The percent phosphorus retention after one laundering is also called "percent phosphorus fixation" by the industry. A higher percent phosphorus fixation is an indicator of higher relative quantity of the flame retarding organic phosphorus agent bound to the fabric after curing. Similar trend is observed on the treated fabric after 20 and 40 launderings. The phosphorus concentration and phosphorus retention decrease as the number of laundering cycle increases due to the hydrolysis of the HFPO bound to the fabric. It is noticed that the fabric treated with 32% HFPO and 10% DMDHEU after 40 laundering cycles still retains 1.84% phosphorus (54% phosphorus retention).

HFPO (%)	DMDHEU (%)	Phosphorus concentration (%)		
		1 laundering	20 laundering	40 laundering
32	1	0.56	0.30	0.25
32	2	1.34	0.52	0.42
32	4	1.81	0.72	0.64
32	6	2.47	1.79	1.01
32	8	2.59	2.00	1.13
32	10	2.69	2.18	1.84

Table 7. The phosphorus concentration of the nylon/cotton fabric (woodland) treated with HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min. (The phosphorus concentration of the treated fabric before wash is 3.43%.)

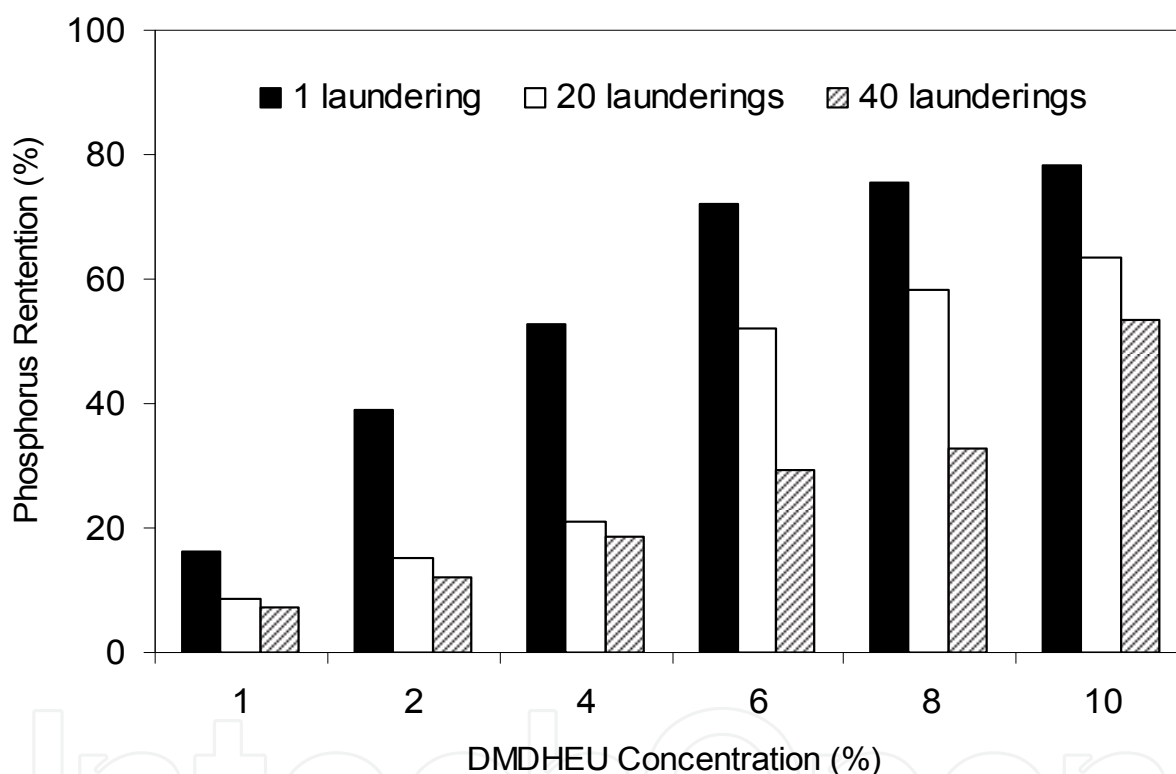


Fig. 5. The percent phosphorus retention of the nylon/cotton fabric (woodland) treated with 32%HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min.

The LOI and vertical flammability of the fabric thus treated is shown in Table 8 and Table 9, respectively. As the DMDHEU concentration is increased from 1 to 10%, the LOI of the treated fabric after 1 laundering cycle increases from 22.9 to 28.0%. The LOI decreases as the number of laundering cycles for the fabric increases. The fabric treated using 6% or higher DMDHEU concentrations passes the test after 40 laundering cycles (Table 9). The LOI of the fabric treated with 32% HFPO and 10% DMDHEU (after 40 laundering cycles) reaches 27.0% with a char length of 81 mm. The data presented here clearly show that DMDHEU concentration plays a critical role in determining the flame retardant performance of the nylon/cotton blend fabric treated with HFPO/DMDHEU. A higher DMDHEU concentration increases the amount of HFPO bound to the treated fabric and it also improves the hydrolysis resistance

HFPO (%)	DMDHEU (%)	LOI (%)			
		1 laundering	10 launderings	20 launderings	40 launderings
32	1	22.9	22.2	21.6	21.2
32	2	25.0	23.2	22.5	22.2
32	4	26.5	25.1	23.0	22.7
32	6	27.7	27.1	26.8	24.8
32	8	27.9	27.5	27.2	25.8
32	10	28.0	28.0	27.4	27.0

Table 8. The LOI (%) of the nylon/cotton fabric (woodland) treated with HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min. (The LOI (%) of the untreated fabric is 20.1.)

of the HFPO bound onto the treated fabric by forming crosslinked HFPO/DMDHEU polymeric network. In our previous research, we have discovered that the nitrogen of DMDHEU does have a synergistic effect for the HFPO-based flame retardant system on cotton [9], but this effect becomes less predominant on the nylon/cotton blends.

We applied the same flame retardant finishing system to a second nylon/cotton BDU fabric ("desert") with the same chemical composition but different structure. The fabric treated with 32% HFPO and DMDHEU at different concentrations is subjected to different numbers of laundering cycles. The LOI (%) and vertical flammability of the nylon/cotton blend fabric (desert) thus treated are presented in Table 10 and 11, respectively. After 1 laundering cycle, the LOI of the treated fabric increases from 28.0 to 28.5% as the DMDHEU concentration is increased from 6 to 10%, respectively, and all three fabric samples pass the vertical flammability test. As the number of laundering cycle increases, the difference among the fabric samples treated using different DMDHEU concentrations becomes more evident. After 25 launderings, the fabric treated with 6% DMDHEU has LOI of 23.8% and fails the vertical flammability test, whereas that treated with 8% DMDHEU has LOI of 26.1% and passes the flammability test. The fabric treated with 10% DMDHEU has LOI of 24.8% and passes the vertical flammability test after 50 launderings. The data presented here again demonstrate that DMDHEU plays a decisive role in determining the flame retardant performance of the nylon/cotton blend fabric treated with HFPO and DMDHEU.

HFPO (%)	DMDHEU (%)	Char length (mm)			
		1 laundering	10 launderings	20 launderings	40 launderings
32	1	>300	>300	>300	>300
32	2	77	>300	>300	>300
32	4	80	94	>300	>300
32	6	77	99	88	114
32	8	79	66	83	105
32	10	49	62	68	81

Table 9. The vertical flammability of the nylon/cotton fabric (woodland) treated with HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min. (The char length of the untreated fabric is >300 mm.)

DMDHEU (%)	LOI (%)			
	1 laundering	25 launderings	40 launderings	50 launderings
6	28.0	23.8	23.1	22.5
8	28.4	26.1	24.4	23.2
10	28.5	27.1	25.5	24.8

Table 10. The LOI of the nylon/cotton fabric (desert) treated with 32%HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min. (The LOI (%) of the untreated fabric is 20.1.)

DMDHEU (%)	Char Length (mm)			
	1 laundering	25 launderings	40 launderings	50 launderings
6	68	>300	>300	>300
8	74	94	103	>300
10	53	81	92	92

Table 11. The vertical flammability of the nylon/cotton fabric (desert) treated with 32%HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min. (The char length of the untreated fabric is >300 mm.)

The nylon/cotton fabric (woodland) is treated with 32% HFPO and DMDHEU at different concentrations and subjected to 1 laundering cycle. The tensile strength of the fabric thus treated after 1 laundering cycle is shown in Table 12. When DMDHEU concentration increases from 2 to 8%, the tensile strength at the warp direction is in the range from 703 N (98% retention) to 685 N (95% retention), respectively. The tensile strength in the filling direction is in the range from 445 N (97 retention) to 454 N (99% retention). Thus, the data presented in Table 12 demonstrate that the fabric treated with HFPO and DMDHEU has negligible strength loss. More details for the flame retardant finished nylon/cotton blend fabrics can be found in our two recent publications [20, 21].

DMDHEU (%)	Tensile Strength (N)		Tensile Strength Retention (%)	
	Warp	Filling	Warp	Filling
2	703	454	98	99
4	694	449	96	98
6	685	445	95	97
8	701	451	97	98
Control	721	458	--	--

Table 12. The tensile strength of the nylon/cotton fabric (woodland) treated with 32%HFPO and DMDHEU at different concentrations and cured at 165°C for 2 min (after 1 laundering cycle).

4. Conclusions

(1) The HFPO/BTCA/TEA flame retardant finishing system applied to the Nomex/cotton blend fabric significantly enhances the performance of the Nomex/cotton blend fabric. The

Nomex/cotton blend fabric treated with HFPO/BTCA/TEA is able to achieve high levels of the flame retardant performance and laundering durability at relatively low add-on levels. The treated fabric also shows modest strength loss and little change in hand properties. This flame retardant finishing system is a formaldehyde-free and odor-free system.

(2) DMDHEU is able to covalently bond HFPO to nylon 6.6 fabrics probably by the formation of a crosslinked HFPO/DMDHEU polymeric network. The combination of HFPO and DMDHEU is an effective durable flame retardant finishing system for the 50/50 nylon/cotton blend BDU fabrics with negligible fabric strength loss. The MDPA/TMM system is not suitable for the flame retardant finishing of the nylon/cotton blend fabric.

5. Acknowledgement

This paper is based on the data included in the dissertation of Dr. Hui Yang, the University of Georgia. Dr. Hui Yang was a graduate student under my supervision and he received his Ph.D. degree in the summer of 2007.

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Advances in Modern Woven Fabrics Technology

Edited by Dr. Savvas Vassiliadis

ISBN 978-953-307-337-8

Hard cover, 240 pages

Publisher InTech

Published online 27, July, 2011

Published in print edition July, 2011

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How to reference

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Charles Q. Yang and Hui Yang (2011). The Flame Retardant Nomex/Cotton, Nylon/Cotton and Polyester/Cotton Blend Fabrics for Protective Clothing, *Advances in Modern Woven Fabrics Technology*, Dr. Savvas Vassiliadis (Ed.), ISBN: 978-953-307-337-8, InTech, Available from:
<http://www.intechopen.com/books/advances-in-modern-woven-fabrics-technology/the-flame-retardant-nomex-cotton-nylon-cotton-and-polyester-cotton-blend-fabrics-for-protective-clot>

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