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Flame-Resistant Polyester/Cotton Fabrics*

GIULIANA C. TESORO, *Fibers and Polymers Laboratory, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139*

Synopsis

New experimental results are reported for the modification of 50/50 polyester/cotton blend fabrics made from bromine-free and bromine-containing polyester with a reactive flame retardant compound of high phosphorus content. Reaction of the cotton in the blend with methyl-phosphonic diamide yields modified fabrics in which flame resistance is attained without impairment of fabric hand. The level of flame resistance depends on the amount of insolubilized phosphorus in the treated fabric, but the hand is essentially unchanged even for fabrics of high phosphorus content which pass the vertical test of DOC-FF-3-71. The results of this work provide a basis for improved definitions of future approaches to the development of flame resistant polyester/cotton blend fabrics.

INTRODUCTION

When the problem of attaining flame resistance in polyester/cotton blend fabrics was reviewed under contract to the National Bureau of Standards in 1972–1973,[1,2] speculation concerning the minimum amount of phosphorus needed to attain self-extinguishing behavior in vertical flammability tests focused primarily on finishes derived from tetrakishydroxymethylphosphonium compounds such as the chloride (THPC). These finishes can be insolubilized on fabrics by polycondensation reactions which proceed with good efficiency, essentially independently of the fiber composition or cotton content of the substrate, under appropriate conditions (coreactants, time, and temperature, etc.).

Good yields of insolubilized phosphorus obtained with such polymer-forming herent problems of interfiber bonding, finish distribution, and adhesion. Undesirable effects such as fabric stiffness and limited durability of the finish are generally associated with this approach in the case of polyester/cotton blends.

By contrast, finishes based on molecules designed for reaction with cellulose hydroxyl groups (e.g., N-methylol-dimethylphosphonopropionamide, Pyrovatex CP) react in low yields on blends of cotton with nonreactive fibers such as polyester, and the amount of insolubilized phosphorus obtained is accordingly low. Yields decrease as the concentration of reagent applied increases and as the cellulose (cotton) content in the blend decreases.[3] It is postulated that, as increasing amounts of the reagent penetrate the cotton component from the aqueous solution and are "crowded" into the limited accessible volume of the

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water-swollen cotton fiber, the mole ratio of reagent to available hydroxyl groups in the cellulose becomes significantly greater than that giving optimum reaction yield.

Perhaps the major problem in the technology of finishing polyester/cotton blends with durable flame retardants is the difficulty in attaining a sufficiently high insolubilized phosphorus content, without impairing fabric properties by introducing excessive amounts of polymer on fiber and yarn surfaces.

This problem can be approached, in principle, by identifying reactive phosphorus compounds or systems, either polymer formers or capable of reaction with cellulose hydroxyl groups, which are more efficient flame retardants than those known to date. With such systems, lower amounts of finish would be used to attain a specified level of flame resistance, and the extent to which surface resin (in the case of polymer formers) or low reaction yields (in the case of cellulose-reactive compounds) constitute insurmountable obstacles would be reduced.

In addition, bromine-containing compounds, either present in the polyester fiber or added as part of the finish, could decrease the amount of phosphorus-containing flame retardant needed and thus further reduce the difficulty in solving the problem of flame resistance in blends.

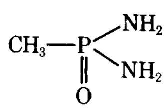
These approaches have been discussed in 1973, based on limited experimental evidence then available, and partly in a speculative vein.[3] Since that time, they have been investigated by several workers. Sello and co-workers[4] have reported results of experiments with a model phosphine oxide (tris-carbamoylethyl-phosphine oxide, CARPO) on 50/50 polyester/cotton blends. These authors investigated the effect of phosphorus content (about 1% to 3% P) from CARPO on oxygen index for a bromine-free blend, and for fabrics in which 3% bromine was introduced either by finishing with tris-2,3-dibromopropyl phosphate (TBPP) or by blending bromine-containing polyester (Dacron 900F) with cotton. At any given phosphorus content, the presence of bromine increased the oxygen index significantly, and an oxygen index of about 28 (% O_2) was reached on fabrics containing about 3% Br + 3% P. In another publication,[5] the same authors compared the relative effectiveness of the model phosphine oxide (CARPO) with that of an insolubilized finish based on THPC, in the presence of aromatic bromine compounds. They concluded that phosphorus from CARPO was more efficient than phosphorus from THPC over a range of bromine contents of 0% to 6% in the 50/50 polyester/cotton blend fabrics studied.

More recently, Barker and his co-workers have investigated[6] the decrease in heat released as a function of phosphorus content for 50/50 blends treated with phosphoric acid (isoperibol calorimeter). In blends made from bromine-containing polyester (Dacron 900F), heat release continued to decrease as phosphorus content increased, up to 2%. In a blend made from bromine-free polyester, heat release initially decreased as phosphorus content increased but leveled off at about ⅔ of the untreated (zero phosphorus) value when the phosphorus content reached about 1%.

The investigations cited[4–6] have utilized model phosphorus compounds to explore the relationships of phosphorus and bromine content in 50/50 polyester/cotton blends to specified parameters of flammability behavior and did not consider insolubilization of the phosphorus-containing flame-retardant compounds. In the present paper, we report the results of a limited study of the durable finishing of 50/50 polyester/cotton blend fabrics made from bromine-free and bromine-containing polyester fibers, by reaction of the cotton component in the blends with an organophosphorus compound which is potentially suitable for application in the mill. At this time, the results of initial work with the system allow better definitions of approaches to the problem than have been possible to date. With further work, viable solutions of the problem may be developed with this system.

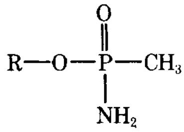
BACKGROUND OF THE INVESTIGATION

The investigation reported here reflects an approach to flame-retardant finishing of polyester/cotton blends which is based on results recently obtained on 100% cotton in a research program supported by Cotton Incorporated. In the study of 100% cotton fabrics, it was postulated that an efficient phosphorus-containing flame retardant should be (a) a small molecule, capable of penetrating the cotton fiber from aqueous solutions; (b) a compound capable of reacting with cellulose hydroxyl groups, so as to attain insolubilization of phosphorus without formation of polymers on fiber surfaces; (c) a compound of high phosphorus content *and* low carbon content, so as to minimize added weight in the treated fabric and minimize the formation of combustible fragments from the added finish; (d) a compound containing, or capable of forming, P–N bonds so as to enhance the rate of cellulose phosphorylation on heating. This definition of the problem, and working hypothesis, led to results reported earlier for a group of phosphonamides[7] which are highly efficient flame retardants, insolubilized by simple pad/dry/cure procedures. Among these reagents, methylphosphonic diamide (MPDA), mol. wt. 94, 33% phosphorus, is particularly promising:



The requirements for an efficient flame retardant for cotton fabrics would also apply to compounds suitable for finishing cellulosic blends, and MPDA is therefore of interest as a candidate flame retardant for blends of cotton with polyester.

The reaction of MPDA with cellulose is postulated, on the basis of analytical data, to yield the following reaction products:



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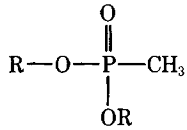

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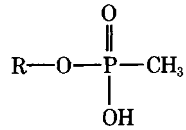

III

where R-OH = cellulose. As a first approximation, the effectiveness of insolubilized phosphorus from MPDA is expected to be comparable to that of phosphorus from diammonium phosphate. An indication of the effectiveness of MPDA could thus be derived from results of model experiments with diammonium phosphate (DAP). The results of such experiments are summarized in Figure 1, which shows the relationship of oxygen index to phosphorus content

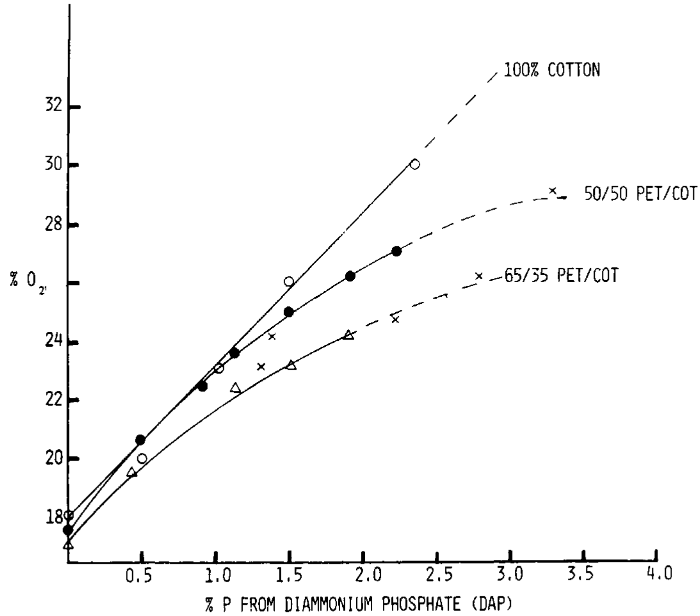

Fig. 1. Effect of DAP phosphorus on oxygen index: (O) 100% cotton, data from ref. 8; (●) 50/50 PET/COT, test fabrics #9503; (▵) 65/35 PET/COT, test fabrics #5404; (×) data from ref. 6.

for several fabrics treated with DAP. The differences between 100% cotton, 50/50 polyester/cotton, and the 65/35 blend are striking. The excellent agreement between our results and those reported by Barker[6] for similar experiments is noteworthy, particularly considering that fabrics of different construction and weight were used in the two sets of experiments.

The results shown in Figure 1 indicate that an oxygen index in the range of interest (about 28% O_2) for DAP treated, 50/50 polyester/cotton blends is reached at a phosphorus content of approximately 3% in the absence of bromine. Assuming analogous effectiveness of DAP phosphorus and MPDA phosphorus, this would correspond to a weight gain of about 10% from MPDA. The objectives of the work reported in this paper were to establish whether MPDA could be reacted with the cotton component in a 50/50 blend to attain this insolubilized weight gain (~10%) and phosphorus content (~3%); whether this level of modification would be sufficient to impart self-extinguishing properties durable to laundering; and how fabric properties would be affected by modification with MPDA. An additional objective of the investigation was to establish to what extent the phosphorus (or reacted MPDA) content required to reach a specified level of flame resistance could be decreased by replacing the polyester in the 50/50 blend with (experimental) polyester copolymer fibers containing aromatic bromine in amount of 6% and 9% on the weight of polyester.

EXPERIMENTAL

Fabrics. The fabrics used for the investigation are listed in Table I. All fabrics were plain weave, scoured, undyed, and free of finish.

TABLE I
Fabrics

Code	Description	Wt:oz/yd²	Source
S/9503	50/50 polyester/cotton	5.4	test fabrics
S/5404	65/35 polyester/cotton	2.7	test fabrics
12581	100% cotton	3–3.2	Celanese Fibers Marketing Company
12579	50/50 Fortrel T-310/cotton	3–3.2	Celanese Fibers Marketing Company
12606	50/50 polyester/cotton (3.12% Br)	3–3.2	Celanese Fibers Marketing Company
12603	50/50 polyester/cotton (4.77% Br)	3–3.2	Celanese Fibers Marketing Company
119 C-1	50/50 Dacron 900F/cotton (3.26% Br)	3.8	E.I. duPont deNemours

Reagents. Methylphosphonic diamide (MPDA) was prepared in the laboratories of the Ethyl Corporation and supplied as a crude reaction product, containing two moles of ammonium chloride per mole of MPDA. The reagent, which is a white solid readily soluble in water, was used for the preparation of the pad bath without purification. Aqueous solutions were prepared at room temperature and used within 1 hr, either without further modification or adjusting the pH with aqueous NaOH, or including additives as specified. Chemicals other than MPDA were reagent-grade materials obtained from a laboratory supply house.

Fabric Treatment. Fabric samples (about 18 × 18 in.) were conditioned at 65% R.H. and 21°C, weighed on an analytical balance, then treated on a three-roll laboratory padder, framed to the original dimensions, and dried in a forced draft oven at 90°C. The samples were then cured as indicated, washed thoroughly with nonionic detergent at 40°C to remove soluble residues, dried on frames, and conditioned for determination of weight gain.

Evaluation. The results reported were obtained by the following procedures: Elemental analyses, carried out by Galbraith Laboratories (Knoxville, Tennessee); Percentages reported are based on conditioned weight. Oxygen index, General Electric apparatus and procedure as specified. Char length, procedure of DOC-FF-3-71 test method, as specified. Rate of heat transfer, procedure of MAFT test method (modified as indicated);[9] see also information supplied by the National Bureau of Standards, as draft 7, dated February 9, 1976.

RESULTS AND DISCUSSION

Reaction Conditions and Insolubilization Yields

The apparent yield of insolubilization of MPDA in the reaction with cellulose can be calculated from the concentration of MPDA in the treating solution, the wet pickup, and the measured weight gain. This procedure provides an approximate value of reaction efficiency, since the relative amounts of MPDA–cellulose reaction products are not known, and each of the derivatives may also affect the moisture regain of the treated fabric to a different extent. With these limitations in mind, it is interesting to examine apparent yields of reaction ob-

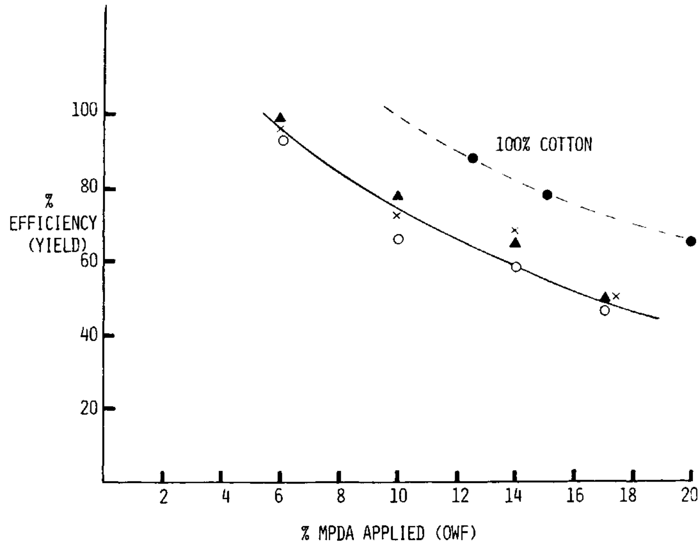

Fig. 2. Apparent insolubilization yield (curing 3 min at 175°C): (○) 50/50 PET/COT, 12579 (no Br); (▲) 12606 (3.12% Br); (×) 12603 (4.77% Br); (●) 100% cotton, 12581.

tained on different fabrics treated with varying concentration of MPDA (cured 3 min at 175°C). The results are shown in Figure 2. For all fabrics, the insolubilization yield decreases with increasing reagent concentration; and at each concentration, the yield is lower on the blends than on 100% cotton.

As expected, it is more difficult to reach high weight gains on the blends, where the efficiency is generally lower, and only modest increases are realized by increasing the reagent concentration applied. The amount of insolubilized

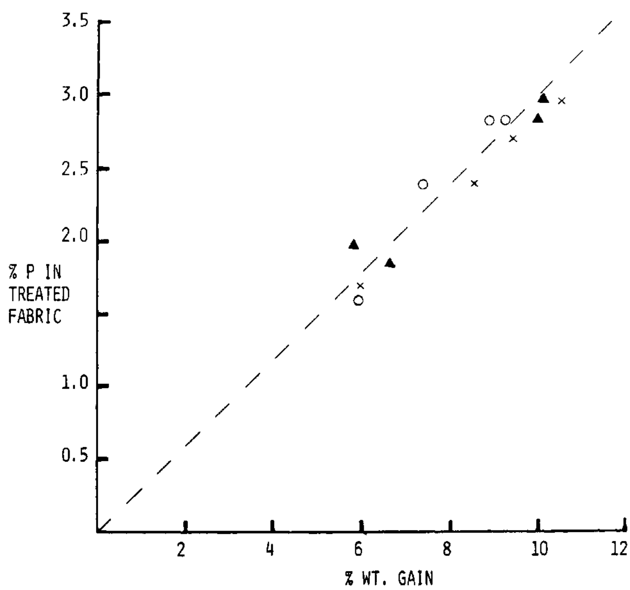

Fig. 3. Weight gain vs. % P (from MPDA): (○) 50/50 PET/COT, 12579 (no Br); (▲) 12606 (3.12% Br); (×) 12603 (4.77% Br).

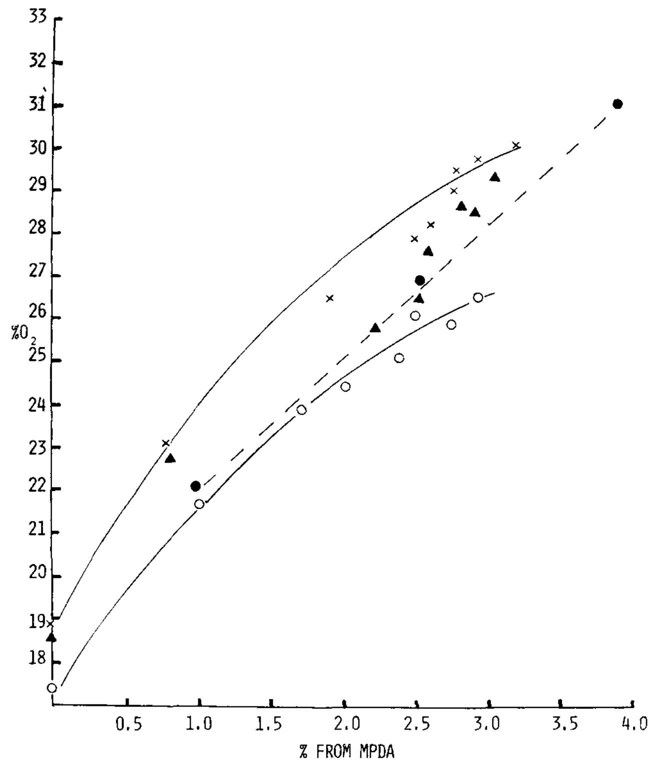

Fig. 4. Effect of MPDA phosphorus on oxygen index: (○) 50/50 PET/COT, 12579 (no Br); (▲) 12606 (3.12% Br); (×) 12603 (4.77% Br); (●) 100% cotton, 12581.

phosphorus from MPDA, determined analytically, is 30% of the weight gain (see Fig. 3): a 3% phosphorus content in the treated fabric requires a 10% weight gain which is not easily reached on blends containing 50% (or more) polyester.

Flammability and Performance Properties of Fabrics Treated with MPDA

The effect of phosphorus from reacted MPDA on oxygen index is shown graphically in Figure 4. As in the case of DAP (Fig. 1), the oxygen index increases linearly with increasing phosphorus content for treated 100% cotton (dotted line). The relationship is far more complex for the treated blends. For each phosphorus content, the oxygen index is higher in the presence of bromine and at higher bromine content (compare data for zero, 3.12% and 4.77% Br in the fabric). Data summarized in Table II indicate that at 2.8% phosphorus treated 50/50 polyester/blend fabrics pass the vertical test of DOC-FF-3-71 if bromine is also present (*OI* = 28.7 at 3.12% Br, 29.2 at 4.77% Br). Insolubilized phosphorus higher than 2.8% was not obtained on the blend fabrics investigated by using conventional procedures.

Variations in *curing conditions* (Table III) have only modest effect on apparent efficiency and oxygen index. The *effect of pH* of the treating solution on efficiency of the reaction is surprisingly small. The results shown in Tables II and

TABLE II
Application of 12% (25% Crude) MPDA Solution Cured 3 Minutes at 175°C

Fabric	% MPDA OWF	% Wt. gain	% Apparent efficiency	% P in treated fabric	*OI*, % O_2	Char length, in.[a]
12581 (100% cotton)	13.2	12.0	93	—	32.5	2.0
12579	12.0	7.3	62	2.6	26.5	BEL
12606	13.0	8.8	68	2.8	28.7	2.5
12603	12.6	9.2	74	2.8	29.2	2.0

[a] DOC-FF-3-71 Vertical Test.

III were obtained with crude MPDA solutions in which the ammonium chloride was not neutralized. Neutralization with sodium hydroxide (to pH 10) gave similar results for efficiency of insolubilization and oxygen index. These are shown in Table IV for two different bromine-containing blend fabrics and two curing conditions.

Some *discoloration* of the fabrics in curing was noted. The extent of discoloration depends on the concentration of MPDA applied, on the curing conditions, and on the bromine content of the fabric treated. In cotton, the addition of small amounts of formaldehyde or other additive is, unexplainably, highly effective in preventing discoloration.[7] This is also true in the case of the 50/50 blends, but the effect is less pronounced in the presence of bromine than in the bromine-free fabric.

Fabric hand is essentially unchanged by the MPDA finishing treatment, even at the highest weight gain and insolubilized phosphorus attainable.

Due to limitations in availability of materials, few data have been obtained to date on *durability* of the finish to laundering and on *strength loss* in treated fabrics. Analytical data on selected samples of treated blends, coupled with results obtained previously on 100% cotton fabrics,[7] indicate that 80% of the

TABLE III
Application of 12% (25% Crude) MPDA Solution: Effect of Curing Conditions

Fabric	Curing		% Apparent efficiency	*OI*, % O_2
	Time	Temp, °C		
12579	80 sec	175	50	24.8
(110% WPU)	2 min	175	62	25.6
	4 min	165	61	25.4
	8 min	150	61	25.6
	80 sec	190	62	26.4
12606	80 sec	175	66	27.4
(108% WPU)	2 min	175	66	27.6
	4 min	165	64	28.0
	8 min	150	68	26.8
	80 sec	190	69	27.6
12603	80 sec	175	58	27.8
(105% WPU)	2 min	175	66	28.2
	4 min	165	64	28.4
	8 min	150	64	27.8
	80 sec	190	70	28.6

TABLE IV
Application of 25% Crude MPDA Neutralized with NaOH to 12606 (3.12% Br) and 119C-1 (3.26% Br)

	12606 (112% WPU) cured at 175°C		119 C-1 (93% WPU) cured at 175°C	
	2 min	3 min	2 min	3 min
% Wt. gain	8.4	8.9	7.6	8.5
% Apparent efficiency	63	68	70	79
OI, % O_2	27.3	27.0	26.8	27.0

phosphorus present on the fabric as finished (process washed) is retained after 50 home launderings. In the treated blends, losses in tensile strength due to the MPDA modification range from zero to 20%, depending on the specific fabric (bromine content), on the MPDA concentration applied, and on the processing conditions. Corresponding losses in tear strength are somewhat higher (20% to 40%). In the work carried out to date, no attempt has been made to minimize these losses through the use of softeners or additives. Additional work on the modification of polyester/cotton blend fabrics with MPDA is essential in order to establish the relationship of formulations and reaction conditions to changes in fabric properties and to finish durability.

Heat Transfer from MPDA-Treated Fabrics

Reports on recent developments in testing of apparel fabrics for flame resistance[9] have stated that the "Mushroom Apparel Flammability Tester" will be used to evaluate several categories of apparel where polyester/cotton blends are important. This test apparatus (referred to as MAFT) was developed at the National Bureau of Standards and has been proposed to the Consumer Products

TABLE V
MPDA on Bromine-Free Fabrics: Crude (5–40% Solution) + 0.25% Formaldehyde; Cured 80 sec at 190°C

Fabric	% MPDA OWF	% Wt. gain	% Apparent efficiency	% P Calcd.	% P Found	*OI* % O_2	Approx. MAFT, joules/ $cm^2 \cdot sec$
	control	—	—	—	—	17.3	1.37
12579	2.5	2.05	87	0.68	1.03	21.8	1.18
50/50	7.5	5.90	77	1.94	2.05	24.4	0.59
PET/COT	13.0	7.68	59	2.53	2.75	25.8	N.I.
	18.0	7.35	40	2.40	2.86	26.4	N.I.
	23.0	8.29	37	2.74	2.89	26.6	N.I.
	control	—	—	—	—	18.1	1.59
100% Cotton	2.8	2.45	92	0.81	1.00	21.6	1.55
	8.0	7.83	95	2.58	2.48	26.0	N.I.
	15.0	11.56	79	3.31	3.89	31.2	N.I.
	20.0	15.29	74	5.04	4.61	32.5	N.I.
	25.0	16.10	63	5.31	5.0	33.2	N.I.

TABLE VI
MPDA on Bromine-Containing Fabrics: Crude (5–40% Solution) + 0.25% Formaldehyde; Cured 80 sec at 190°C

Fabric	% MPDA OWF	% Wt. gain	% Apparent efficiency	% P Calcd.	% P Found	*OI*, % O_2	Approx. MAFT, joules/$cm^2 \cdot sec$
	control	—	—	—	—	18.8	1.50
12606	2.3	2.22	97	0.73	0.82	22.8	0.80
3.12%	7.0	6.00	84	1.98	2.20	25.8	N.I.
bromine	12.5	7.67	62	2.53	2.65	27.4	N.I.
50/50	17.5	8.43	47	2.78	2.91	28.4	N.I.
PET/COT	21.0	8.65	42	2.86	3.01	29.4	N.I.
	control	—	—	—	—	18.8	1.25
12603	2.2	2.15	97	0.71	0.79	23.0	0.17
4.77%	7.0	5.75	81	1.90	1.93	26.4	N.I.
bromine	12.5	7.91	64	2.61	2.75	28.8	N.I.
50/50	17.5	8.23	47	2.71	2.84	28.4	N.I.
PET/COT	21.0	8.68	42	2.86	3.21	29.8	N.I.

Safety Commission as the tool for the measurement of maximum rate of heat transfer in burning of fabric specimens. It has been suggested[9] that fabrics in which the maximum rate of heat transfer in the MAFT test exceeds 0.1 calorie/$cm^2 \cdot sec$ (or 0.4 joule/$cm^2 \cdot sec$) should be restricted as to end-use by future regulations.

While the details of the MAFT test, and of possible apparel flammability standards based on it, are uncertain at this time, it was nevertheless of interest to examine the behavior of polyester/cotton fabrics treated with MPDA at various phosphorus contents in the MAFT apparatus. Some results have been reported[10] for cotton fabrics treated with flame retardants (including MPDA) at various concentrations, but none are published for treated polyester/cotton blends. Results obtained in our work are summarized in Tables V (for bromine-free fabrics) and VI (for bromine-containing fabrics). The MAFT results included are "approximate" because treated fabric specimens were not large enough and were joined by a patch of self-extinguishing cotton in order to reach the dimensions needed for testing. These preliminary results indicate that in the case of fabrics weighing 3–3.2 oz/yd^2, the phosphorus content (from MPDA) required to meet the proposed MAFT criterion of 0.4 joule/$cm^2 \cdot sec$ is between 1.0% and 2.5% for the 100% cotton between 2.0% and 2.7% for the bromine-free blend, between 0.8 and 2.2% for the blend containing 3.12% bromine, and 0.8% or less for the blend containing 4.77% bromine. MPDA finishing of 50/50 polyester/cotton blends to meet the proposed MAFT criteria is indicated to be a viable approach, since phosphorus contents in this range can be attained with reasonable efficiency and without undesirable effects on fabric properties.

SUMMARY AND CONCLUSIONS

One of the major problems in finishing polyester/cotton blends with flame retardants to meet the requirements of DOC-FF-3-71 has been the difficulty in reaching a sufficiently high insolubilized phosphorus content in the treated fabric.

Polymer-forming systems such as those based on THPC chemistry are insolubilized on fiber surfaces, and tend to impart stiffness which is unacceptable at high add-ons for most fabric constructions and end uses. Reactive systems which are insolubilized by covalent bonding with cellulose must be present and react in *very* high concentration in the cotton component of the blend, and the yields of the insolubilization reaction are consequently low. In this context, MPDA is a promising reactive flame retardant because it is a small molecule of high phosphorus content; it penetrates the cotton easily from aqueous solutions; it carries minimal fuel-forming organic groups; and it can be insolubilized without affecting fabric hand.

The work reported in this paper has shown that, for 50/50 polyester/cotton blends in the weight range of 3 to 4 ounces/yd^2 modified with MPDA in a simple pad/dry/cure wash process, the phosphorus content required to meet the criteria of DOC-FF-3-71 is ~3.0% when 3% bromine is present and ~2.5% when 4.7% bromine is present. In the absence of bromine, the amount of phosphorus required is estimated to be approximately 3.5%. Insolubilization of 3.5% phosphorus in 50/50 blends was not attained by reaction of the cotton component with MPDA under the conventional processing conditions investigated.

For blend fabrics in the 3 to 4 ounces/yd^2 weight range, the phosphorus content required to meet the proposed criteria of the MAFT test is less than 2.7% for the bromine-free blend, less than 2.2% for the blend containing 3% bromine, and less than 0.8% for the blend containing 4.7% bromine. Such levels of insolubilized phosphorus can be obtained with MPDA on the blends in reasonable yields under practical processing conditions. The hand of the treated fabrics is excellent, strength losses are small, and discoloration can be controlled by the use of appropriate additives. Much work remains to be done to establish the limits of durability of the finish of its effect on fabric properties and, above all, of its potential usefulness on a broad range of blend compositions and fabric constructions.

Abbreviations Used in the Text

CARPO	tris-(carbamoylethyl)phosphine oxide
COT	cotton
DAP	diammonium phosphate
MAFT	Mushroom Apparel Flammability Tester
MPDA	methylphosphonic diamide
OI	oxygen index
OWF	on weight of fabric
PET	polyester
TBPP	tris(2,3-dibromopropyl) phosphate
THPC	tetrakishydroxymethylphosphonium chloride
WPU	wet pickup

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