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Observations on some Flame-Retardant Treatments of Cotton/Polyester Blended Fabrics

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OBSERVATIONS ON SOME FLAME-RETARDANT TREATMENTS OF COTTON/POLYESTER BLENDED FABRICS

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ABSTRACT

All cotton, polyester and cotton/polyester blended fabrics containing 80, 60 and 40 per cent cotton were treated with tris (2,3-dibromopropyl) phosphate or titanium tetrachloride/antimony oxide. Some fabrics were given a two-step treatment employing both flame-retardants. The effect of the treatments on the limiting oxygen index (LOI) values of the fabrics was determined. Fabrics which had received a two-step treatment with tris (2,3-dibromopropyl) phosphate and titanium tetrachloride/antimony oxide, had LOI values greater than 0,270 after having been washed for 900 minutes (30 washing cycles) at 60°C.

KEY WORDS

Flame-retardant – cotton – polyester – blended fabrics – limiting oxygen index – tris (2,3-dibromopropyl) phosphate – titanium tetrachloride – antimony oxide.

INTRODUCTION

The flammability of cotton fabrics can be reduced by treatment with a wide variety of reagents [1] . The flame-retardant finishing of cotton fabrics is not only technologically feasible, but it has become a commercial and practical finishing routine for many cotton end-commodities. Several flame-retardant treatments are at present employed in the textile industry in the U.S.A. to produce flame-retardant cotton fabrics, mainly for the childrens' sleepwear, automotive, curtain and upholstery markets [2,3] .

It is more difficult to reduce the flammability of synthetic fibres than it is to reduce that of cotton [4] . In the case of synthetic fibres two alternatives are available : inherently flame-retardant fibres can be synthesized or otherwise the conventional fibres can be rendered flame-retardant through the use of certain chemicals. Several fibre manufacturers are at present producing flame-retardant synthetic fibres. In a recent publication the names of 15 flame-retardant fibres, produced by ten different manufacturers, were listed [5] . These fibres are, however, fairly expensive and difficult to dye while some are not yet available in large quantities. Consequently extensive research is still being carried out on the treatment of the conventional

synthetic fibres by various flame-retardant chemicals.

Most of the flame-retardant treatments which have been found satisfactory for cotton fabrics have limited value in the case of synthetic fibres or cotton/synthetic fibre blends. It has, in fact, been demonstrated that the flammability behaviour of fabrics made from blended fibres cannot be predicted from a knowledge of the flammability of the individual fibres [6]. Tesoro and Meiser [7] showed, for example, that cotton/polyester blends had lower LOI values than either pure cotton or pure polyester. Cotton/polyester blends account for a large percentage of the apparel end-use market (approximately 33 *per cent* in the U.S.A.), and there is, therefore, a great need for the development of flame-retardant treatments for these fabrics.

At present only one manufacturer in the U.S.A. produces a flame-retardant polyester fibre [5]. This fibre is, unfortunately, not yet available in South Africa or Europe and conventional polyester fibres must, therefore, be treated with flame-retardant chemicals to reduce their flammability. It is relatively easy to produce a flame-retardant cotton fabric but very few chemicals will produce flame-retardant cotton/polyester blends of any desired composition [8–11]. Normally the efficiency of the treatment decreases with an increase in polyester content. THPC, for example, will produce flame-retardant cotton/polyester fabrics containing up to 50 *per cent* polyester only [9]. By using an oligomeric phosphonium salt and a methylolmelamine resin, blends containing up to 70 *per cent* polyester can be rendered flame-retardant [11].

When the polyester content of the blend exceeds 70 *per cent,* a two-step flame-retardant treatment would probably be required, with one treatment affecting the cotton and the other the polyester. Tris (2,3-dibromopropyl) phosphate (TBPP) has been found to be an effective flame-retardant for polyester [10]. Furthermore, it is known [12–13] that cotton can be rendered flame-retardant by a treatment with titanium tetrachloride and antimony oxide (Ti/Sb). It was therefore decided to investigate the effect of TBPP and Ti/Sb on the flammability of different cotton/polyester blended fabrics.

EXPERIMENTAL

Plain weave all cotton, polyester and cotton/polyester blended fabrics containing 80, 60 and 40 *per cent* cotton were used. Three types of Trevira polyester, namely type 120 (normal), type 340 (low-pilling) and type 140 (high bulk) were used. The fabric mass per unit area was approximately 140 g/m^2. Details of the fabrics can be found in a recent report [14].

One series of the thirteen fabrics was scoured, bleached, dried, heat-set and cropped. In addition, some pure polyester fabrics were finished as

described above, but with the bleaching step omitted. Furthermore, an additional pure cotton fabric was finished as before but no heat-setting was applied. Experimental details of the finishing procedures appear in a recent report [14].

All percentages given are expressed as mass/mass.

The fabrics were treated with various concentrations of titanium tetrachloride plus antimony oxide as described before [12]. Some fabrics were treated with various solutions of tris (2,3-dibromopropyl) phosphate in perchloroethylene, as described by Tesoro and co-workers [10]. These fabrics were dried for 3 minutes at 200° C in an oven [10], or baked on a Hoffman press for 3 minutes at 120°C or 145°C. The baking step was followed by an evacuation step of 10 seconds. Finally some fabrics were treated with titanium tetrachloride plus antimony oxide as well as by tris (2,3-dibromopropyl) phosphate, in a two-step process. After treatment all the fabrics were rinsed and conditioned at 20°C and 65% RH. The mass/per unit area of the treated as well as untreated fabrics was determined after rinsing by weighing.

The limiting oxygen index (LOI) of the rinsed fabrics was determined as described before [13]. The durability of the treatments to washing was studied by washing the fabrics in an automatic washing machine at 60°C. Each washing cycle lasted 30 minutes, and was followed by three rinses in cold water (total rinsing time being 30 minutes) and spin drying for 5 minutes. A domestic washing powder recommended for automatic washing machines was used. In a few cases the degree of whiteness of the fabrics was determined with a Zeiss Elrepho apparatus, using the formula suggested by Berger [15].

RESULTS AND DISCUSSION

In the case of a two-step flame-retardant treatment it was of importance to establish whether the order in which the treatments were carried out had any effect on the flame-retardancy of the fabrics. Some preliminary trials were therefore carried out, using the 40 *per cent* cotton blend. The fabric was first treated with Ti/Sb, followed by a treatment with TBPP in the one case, and treating in the reverse order in the other. The effect of different curing conditions on the LOI values and degree of whiteness of the fabrics was also investigated. The results obtained appear in Table I. A curing time of 3 minutes at 200° C has been suggested for TBPP treatments [10], but Table I shows that a severe yellowing of the cotton/polyester sample occurred under those conditions. Baking of the samples on a Hoffman press at relatively low temperatures produced much whiter fabrics than did curing, without adversely affecting the LOI values, and consequently all other fabrics were baked on a Hoffman press.

TABLE I

THE EFFECT OF DIFFERENT CURING CONDITIONS AND THE ORDER OF TREATMENT ON THE FLAME-RETARDANCY AND DEGREE OF WHITENESS OF COTTON/POLYESTER FABRICS

Treatment *	LOI after		Degree of Whiteness
	0	10 washing cycles	
TBPP, oven-cured (3 min, 200°C)	0,253	0,240	18,9
TBPP, baked on Hoffman press (3 min, 145° C)	0,256	0,235	21,5
TBPP, baked on Hoffman press (3 min, 120° C)	0,245	0,236	22,5
TBPP, baked on Hoffman press (3 min, 145° C) followed by Ti/Sb	0,289	0,245	22,6
TBPP, baked on Hoffman press (3 min, 120° C) followed by Ti/Sb	0,292	0,244	23,1
TBPP, baked on Hoffman press (1 min, 120° C) followed by Ti/Sb	0,292	0,244	22,6
Ti/Sb, followed by TBPP, baked on Hoffman press (3 min, 145° C)	0,296	0,246	18,8
Ti/Sb, followed by TBPP, baked on Hoffman press (3 min, 120° C)	0,299	0,250	18,3
Ti/Sb, followed by TBPP, baked on Hoffman press (1 min, 120° C)	0,295	0,240	19,0
Untreated			27,9

* The fabrics were treated with 4% TBPP or 29% Ti/Sb

Table I also shows that the order in which the treatments were carried out did not have a significant effect on the flame-retardancy of the fabrics. The degree of whiteness of the samples first treated with TBPP, followed by the Ti/Sb treatment was, however, significantly higher than that of the other samples and consequently the former order of treatment was used for all further treatments. Finally it can be seen from Table I that the different baking conditions on the Hoffman press did not have a significant effect on the LOI values and the degree of whiteness of the fabrics. It was, therefore, decided to bake all TBPP-treated samples for 3 minutes at 120 °C.

No significant differences were found between the LOI values of the pure cotton fabrics which had been heat-set, and the cotton fabrics which had not been heat-set and only the former results are presented. The same applies to the pure polyester fabrics which had been bleached, and those

which had not been bleached. There were no significant differences (with one exception, which will be referred to later) between the LOI values of the normal, low pilling and high bulk polyester at various blend levels for the different treatments. Consequently all LOI values given are those obtained for the normal (Trevira type 120) polyester.

The effect of the TBPP and Ti/Sb treatments on the LOI values of the various fabrics is respectively shown in Figure 1 and Figure 2. Several interesting phenomena emerge from the figures. In the case of the TBPP treatments, the LOI values of the fabrics increased with increasing levels of chemical add-on. The blended fabrics generally had lower LOI values than either pure cotton or pure polyester. This is in agreement with the findings of several research workers [9, 10, 11]. It is also of interest to note that the flammability of the treated blends normally increased when the the polyester content increased. Pure polyester was, however, less flammable than the various blended fabrics. An application level of 40 *per cent* TBPP, or higher, increased the LOI of all the different fabrics to values higher than 0,270, which is normally considered to be the minimum level required for a fabric to pass the vertical flame-test. Finally, it would appear that pure polyester had slightly higher LOI values than all-cotton after treatment with TBPP.

In the case of the Ti/Sb treatment, the LOI values generally increased with an increase of the level of chemical add-on. Once again the LOI values of the blends decreased with increasing polyester content. For all practical purposes only the 100 and the 80 *per cent* cotton fabrics would pass the vertical flame-test after treatment with Ti/Sb.

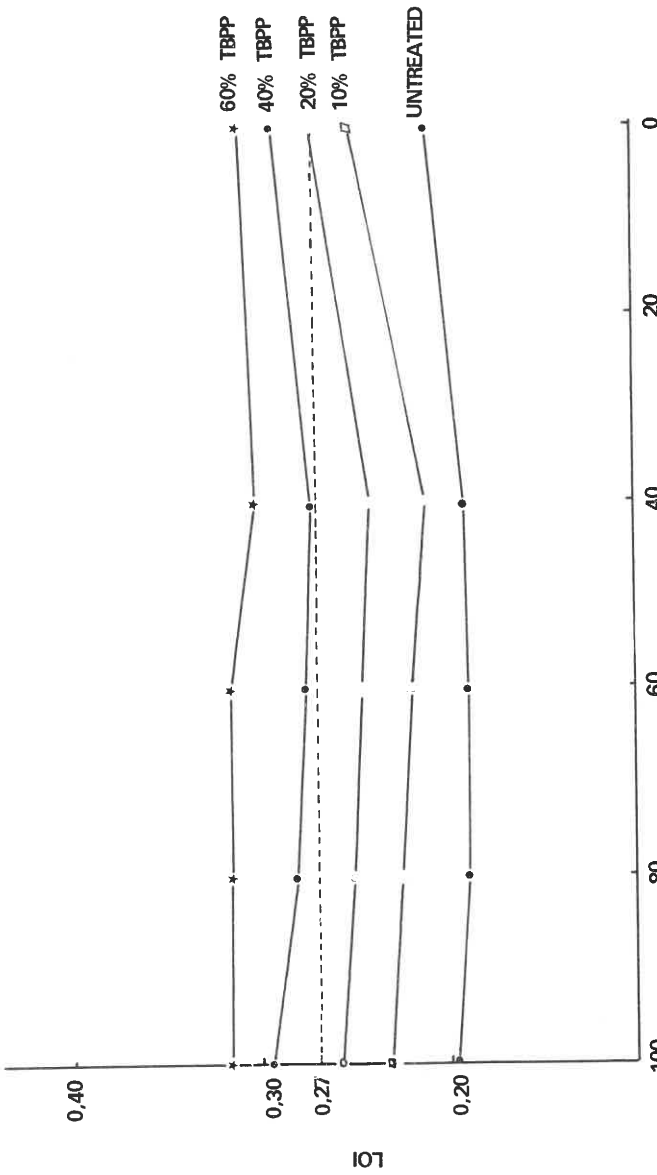

FIGURE 1

The effect of TBPP on the LOI values of cotton/polyester blended fabrics

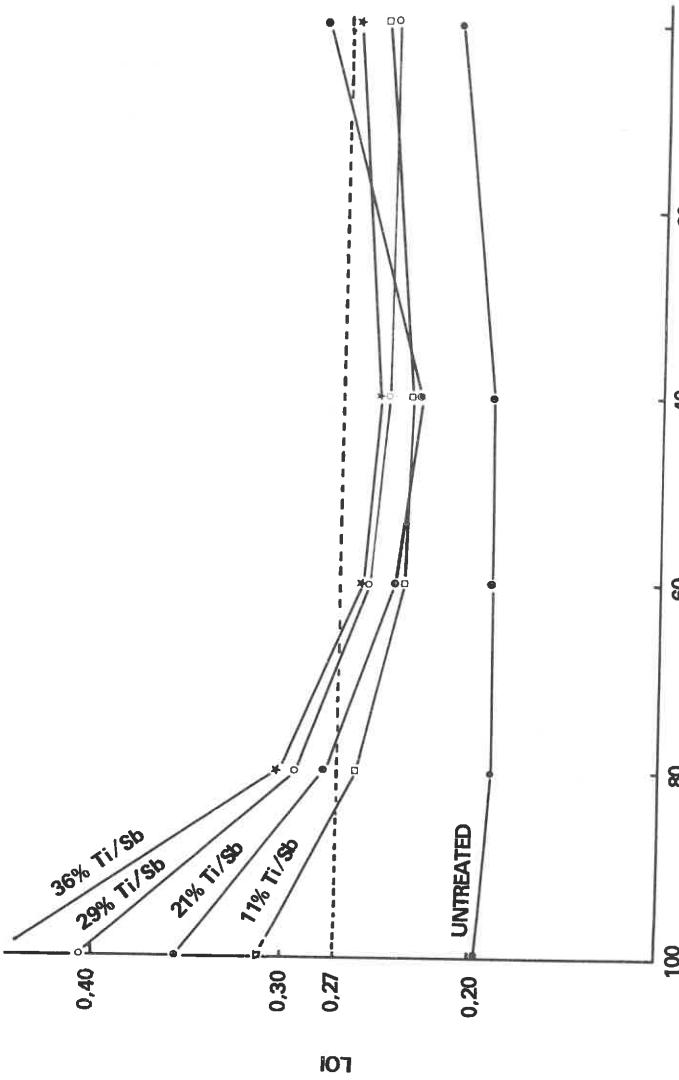

FIGURE 2

The effect of Ti/Sb on the LOI values of cotton/polyester blended fabrics

TABLE II

THE LOI VALUES OF VARIOUS TYPES OF POLYESTER TREATED WITH DIFFERENT CONCENTRATIONS OF Ti/Sb

Add-on level	LOI VALUES					
	Rinsed only			After 10 washing cycles		
	Normal	Low pilling	High bulk	Normal	Low pilling	High bulk
11% Ti/Sb	0,249	0,218	0,210	0,212	0,211	0,210
21% Ti/Sb	0,285	0,241	0,234	0,227	0,215	0,212
29% Ti/Sb	0,247	0,239	0,229	0,223	0,209	0,207
36% Ti/Sb	0,265	0,256	0,239	0,221	0,218	0,211
Untreated	0,210	0,210	0,210			

When pure polyester was treated with Ti/Sb, differences were found between the LOI values of the three different types of polyester. The results obtained are given in Table II. It can be seen that the normal polyester had the highest LOI values after treatment, followed by the low pilling polyester and finally the high bulk polyester. No difference was found between the LOI values of the three types of untreated polyester samples. No explanation can be offered for this phenomenon, which was not observed in the case of the various blended fabrics.

Figures 3 and 4 show the effect of a two-step treatment, using TBPP followed by Ti/Sb, on the LOI values of various fabrics. Once again the efficiency of the treatment was reduced when the polyester content of the sample increased. Here, in contrast to the single step treatments, the pure polyester fabrics had LOI values which were lower than those of all the other samples. An increase in the level of TBPP add-on resulted in an increase in the LOI values of the fabrics. When the level of Ti/Sb add-on was increased, the LOI values of all cotton-containing samples increased.

For all two-step treatments studied the fabrics containing from 100 to 40 *per cent* cotton all had LOI values higher than 0,270, indicating that these fabrics would probably pass the vertical flame-test. In the case of the pure polyester fabric, however, only three treatments produced fabrics with LOI values higher than 0,270. Furthermore, when Figures 3 and 4 are compared with Figure 1, it can be seen that the additional Ti/Sb treatment actually reduced the efficiency of the TBPP treatment of pure polyester. In the case of all other blends, however, the two-step process produced fabrics with higher LOI values than did any of the single treatments.

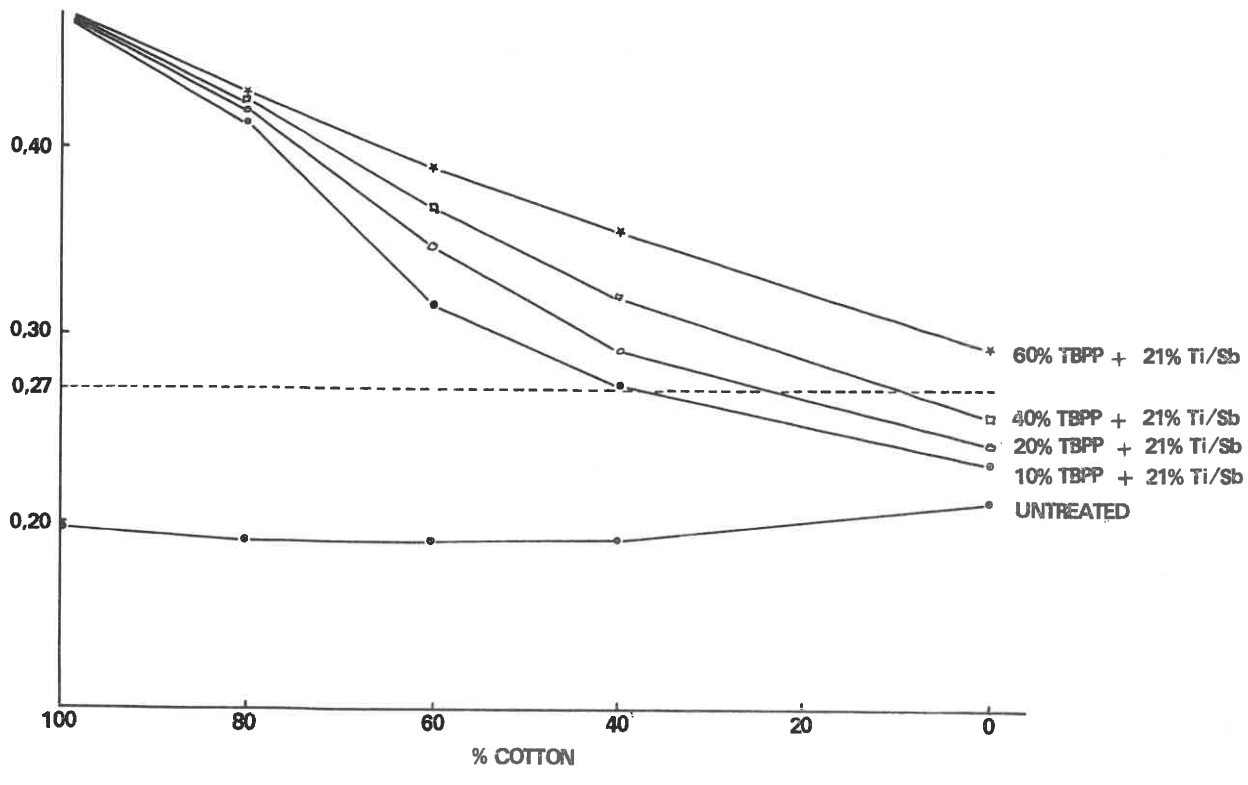

FIGURE 3

The effect of 21% Ti/Sb and different quantities of TBPP on the LOI values of cotton/polyester blended fabrics

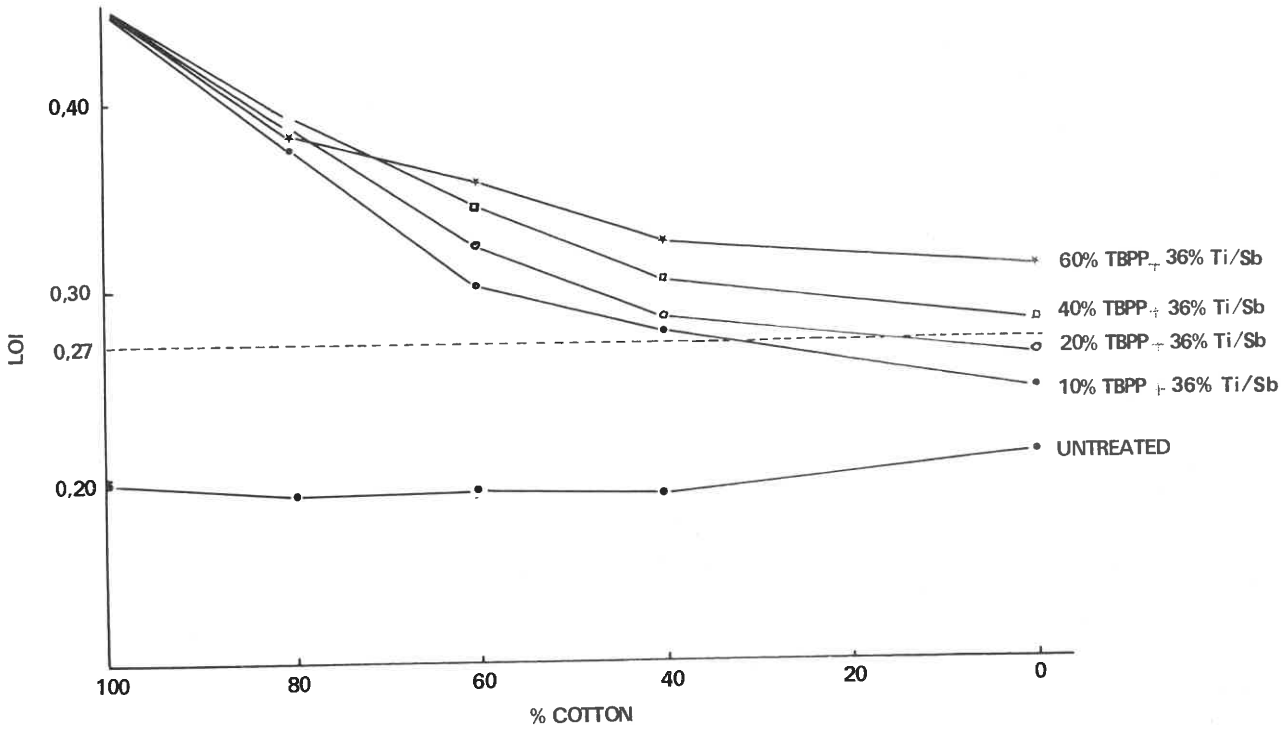

FIGURE 4

The effect of 36% Ti/Sb and different quantities of TBPP on the LOI values of cotton/polyester blended fabrics

TABLE III

THE PERCENTAGE OF FLAME-RETARDANT ACTUALLY PRESENT ON THE FABRICS AFTER RINSING, CALCULATED FROM FABRIC MASS PER UNIT AREA

Fabric	Treatment (% chemical applied)									
	TBPP			Ti/Sb			TBPP + Ti/Sb			
	10	30	60	11	21	36	30+21	60 + 21	30 + 36	60 + 36
100% Cotton	4,6	16,8	19,2	3,9	11,3	22,8	18,3	32,7	28,5	32,9
80% Cotton/20% Polyester	5,2	19,3	18;0	8,4	13,5	26,2	23,6	–	30,1	36,1
60% Cotton/40% Polyester	6,7	15,0	17,1	7,1	12,8	20,8	21,2	32,4	21,4	35,3
40% Cotton/60% Polyester	6,5	14,0	20,4	6,8	6,9	13,9	17,3	30,0	22,7	28,4
100% Polyester	3,4	12,6	27,7	–	0,4	–	14,1	29,2	8,6	21,1

The effect of repeated washing on the LOI values of the treated fabrics can be seen in Figures 5 to 8. The LOI values of all the samples decreased with increasing washing times. Only the all cotton fabrics treated with Ti/Sb had LOI values higher than 0,270 after 10 and 30 washing cycles, respectively. On the other hand, only the pure polyester fabrics treated with TBPP had LOI values higher than 0,270 after 10 and 30 washing cycles. Two-step treatments yielded several samples with LOI values higher than 0,270 after 10 and 30 washing cycles. Blends containing more polyester normally required higher levels of TBPP add-on, while more Ti/Sb was required when the cotton content of the blend was increased. By using 60 *per cent* TBPP, followed by either 21 or 36 *per cent* Ti/Sb, it was possible, however, to render all the different fabrics flame-retardant, even after 30 washing cycles.

Although fairly high levels of chemical add-on were applied in some cases, the handle of the fabrics was still reasonable after treatment. This indicates that probably not all the chemical applied to the fabrics was retained by the fibres, but that some was removed again during the subsequent rinsing process. The *actual* percentage of flame-retardant present on the fibres was, therefore, probably lower than the *applied* add-on levels reported in this publication. To obtain some information about the actual concentration of reagent present on the fibres after rinsing, the mass per unit area of treated as well as untreated fabrics was determined and the percentage of flame-retardant present on the fabrics was calculated. The results obtained are given in Table III. It can be seen that, in the case of

TBPP or Ti/Sb treatments, only approximately one half of the quantity of chemical added to the fabrics remained on the fabrics after rinsing. In the case of the two-step treatments (TBPP followed by Ti/Sb) only one third of the applied chemical remained on the fabrics after rinsing. The quantity of flame-retardant actually present on the fabrics which had been treated with TBPP followed by Ti/Sb, varied from approximately 15 to 35 *per cent* after rinsing. These percentages, although relatively high, are still comparing favourably with the level of 40 to 50 *per cent*, required for a commercially available phosphonium salt flame-retardant [11]

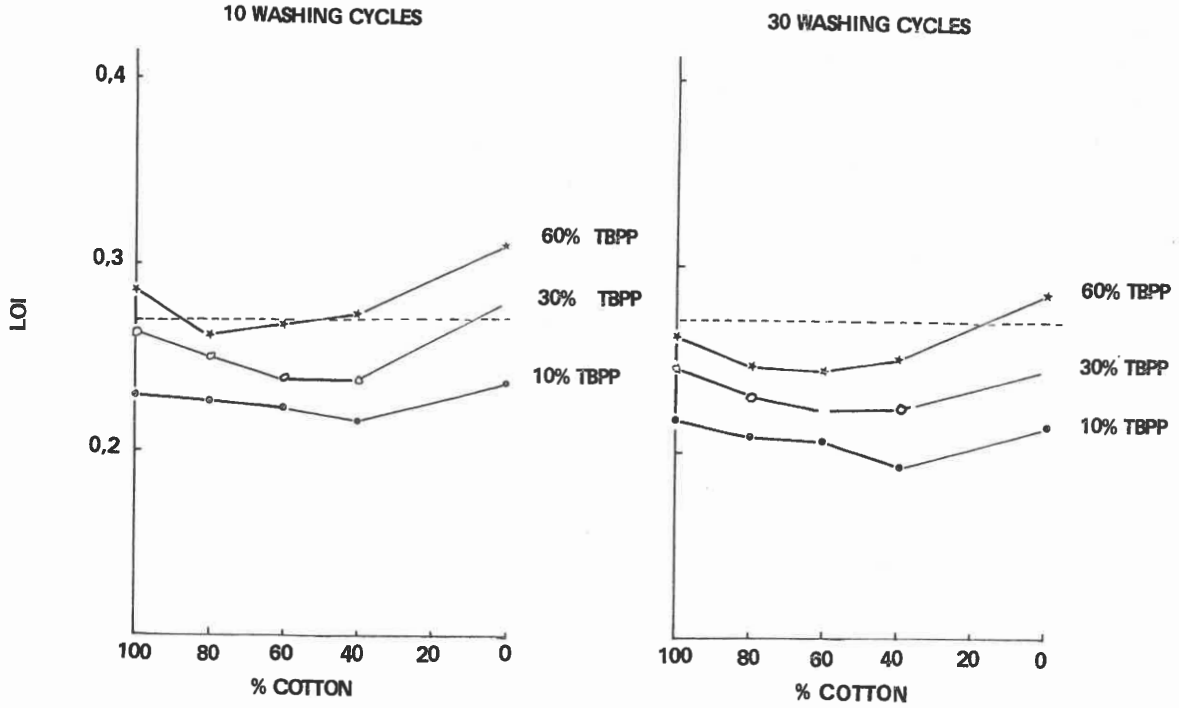

FIGURE 5

The effect of repeated washing on the LOI values of cotton/polyester blended fabrics treated with TBPP

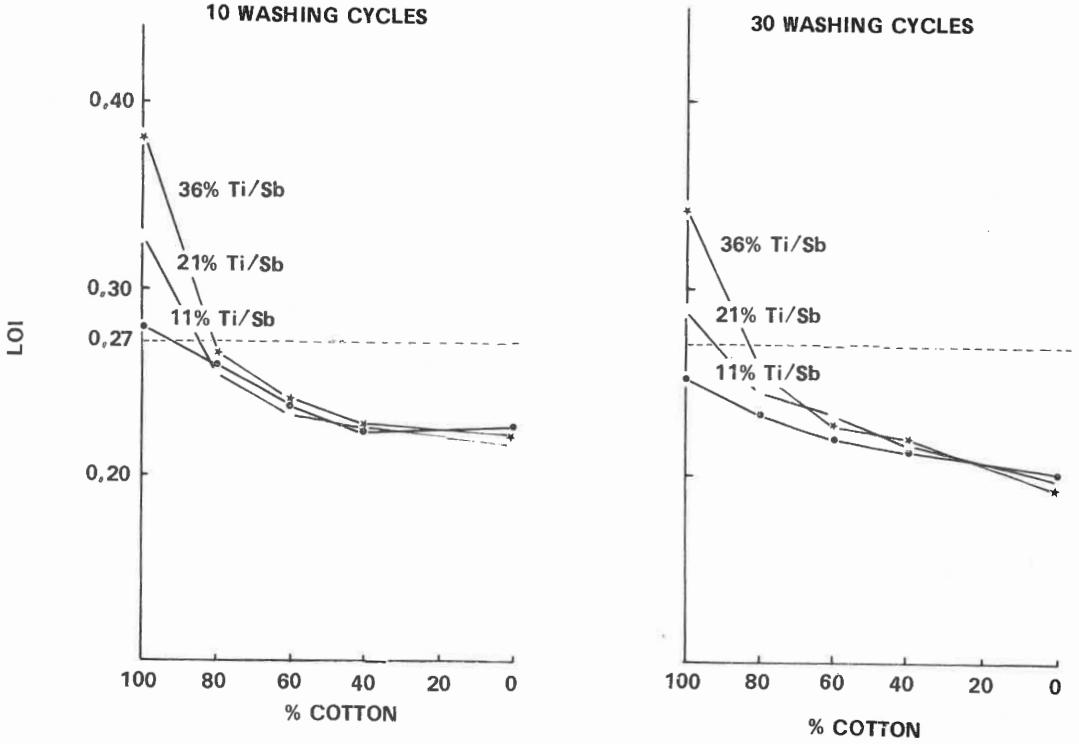

FIGURE 6

The effect of repeated washing on the LOI values of cotton/polyester blended fabrics treated with Ti/Sb

SUMMARY AND CONCLUSIONS

All cotton, polyester and cotton/polyester blended fabrics containing 80, 60 and 40 *per cent* cotton were treated with various concentrations of TBPP, or Ti/Sb, or combinations of the two reagents, and the LOI values of the samples were determined after 0, 10 and 30 washing cycles at 60° C.

Baking of the TBPP-treated samples on a Hoffman press at relatively low temperatures did not adversely affect the flame-retardancy and produced fabrics with a higher degree of whiteness than did curing in an oven. To reduce the yellowing of the samples during a two-step treatment the fabrics should first be treated with the TBPP, followed by the Ti/Sb treatment.

The flammability of the treated blends normally increased with an increase in polyester content. In the case of the TBPP treatments, the pure polyester fabrics generally had LOI values which were higher than those of the various blended fabrics. An application level of 40 *per cent* TBPP, or higher, increased the LOI value of all the different blends to values higher than 0,270. In the case of the Ti/Sb treatments, only the 100 and the 80 *per cent* cotton fabrics would probably pass the vertical flame-test. When the pure polyester fabrics were treated with Ti/Sb, differences were found between the LOI values of the three types of polyester. The normal type of polyester fabric had the highest LOI value, followed by the low pilling polyester and finally the high bulk polyester fabrics.

For all two-step treatments (TBPP followed by Ti/Sb) studied, all the fabrics containing from 100 to 40 *per cent* cotton had LOI values higher than 0,270. In the case of the pure polyester fabric only three treatments produced samples with LOI values higher than 0,270. Furthermore, it was found that the additional Ti/Sb treatment actually reduced the efficiency of the TBPP treatment on pure polyester.

Repeated washing reduced the LOI values of all the treated fabrics. In the case of the Ti/Sb treatments, only the pure cotton fabrics had LOI values higher than 0,270 after 30 washing cycles. In the case of the TBPP treatments, only the pure polyester fabrics had LOI values higher than 0,270 after 30 washing cycles. By using two-step treatments (60 *per cent* TBPP followed by either 21 or 36 *per cent* Ti/Sb) it was, however, possible to render all the different blends flame-retardant (with LOI values higher than 0,270) after 30 washing cycles. The popular blended fabric containing 67 *per cent*. polyester can thus be rendered flame-retardant, even after having been washed for 900 minutes (30 washing cycles) at 60° C.

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PROPRIETARY NAMES

The fact that chemicals with proprietary names have been mentioned in this report does not in any way imply that SAWTRI recommends them or that there are not substitutes which may be of equal value or even better.

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