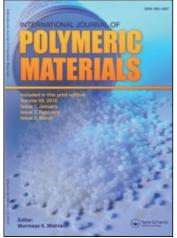
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Plasticized Polyvinyl Chloride Finishes for Durable Flame Retardancy for Cotton Fabrics

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Cotton flannels were padded with flame retardant compositions comprising a vinyl chloride co-polymer resin, a halogenated phosphate ester plasticizer and a small amount of an unsaturated alkyl dimethacrylate monomer to give varying add-on's—up to 60% by weight of the cottons. The padded cotton flannels were then exposed to high energy electron beam irradiation dosages in the presence of air at ambient temperatures. Limiting Oxygen Index (LOI) measurements are reported for the treated cottons for increasing numbers of laundering cycles and correlated with fabric weight changes. Analogous experiments were carried out with the same flame retardant composition padded onto the cotton flannels to similar add-on's but with the resultant samples not subjected to electron beam irradiation. The laundering durability of the irradiated treated cotton flannels was significantly superior and weight losses were lower, especially after extended launderings. Irradiation treatments offer a potentially attractive approach for enhancing the laundering durability of relatively inexpensive flame retardant compositions.

INTRODUCTION

The complex problems associated with the protection of consumers from the hazards of flammable fabrics and other textile materials have become the subject of greatly increased concern to the industries involved particularly so in view of proposed government legislation.¹ Cotton flannels by virtue of their unique construction, are at present especially difficult to render flame retardant without deleteriously affecting esthetics, strength and abrasion resistance almost regardless of the economics of the proposed flame retardant treatment. A very substantial research and development effort has been carried on for some time now in order to develop flame retardant cotton flannels which will be able to meet proposed government standards initially

for children's sleepwear. It is the purpose of this paper to suggest the potential utility of high energy irradiation treatments for upgrading the durability of flame retardant cotton flannels with a relatively simple and economic plasticized vinyl co-polymer resin system and using conventional textile finishing technology.

Compounds containing halogens and phosphorus have been investigated in considerable detail for their flame retardant effects on cellulose and especially on cotton textiles.² The many difficulties involved in the development of high performance durable flame retardant cottons have been identified in considerable detail with laundering fastness up to 50 cycles considered to be a major obstacle. Tris (2,3-dibromopropyl) phosphate is an attractive candidate for flame retardant formulations on account of its high bromine (68.73%) and substantial phosphorus (4.44%) content, good color, improved heat and ultra-violet light stability, low volatility, high degree of compatibility with many polymeric materials and last but by no means least, moderate cost.3 The treatment of cotton cloth with flame retardant formulations comprising tris (2,3-dibromopropyl) phosphate and trimethylolmelamine as the essential ingredients has been reported.⁴ The degree of flame retardancy was found to depend on the ratio of flame retardant to resin employed. After six launderings, 60% of an original 24% add-on based on the weight of the cotton fabrics remained and flame retardancy was lessened. In spite of considerable work with flame retardant formulations with tris (2,3-dibromopropyl) phosphate and quite a number of different resin binders, the treated cottons generally exhibit what might be called semi-durable flame retardancy to prolonged laundering cycles. Emphasis has therefore been shifted to the preparation and evaluation of alternative and frequently much more complex systems for achieving durable flame retardancy.

This paper describes a limited experimental program to ascertain the suitability of high energy ionizing radiation for enhancing the performance of flame retardant cotton fabrics particularly as regards laundering durability. The flame retardant formulation comprised a vinyl chloride co-polymer resin binder and tris (2,3-dibromopropyl) phosphate as the major ingredients. A small amount of an unsaturated dimethacrylate monomer, ethyleneglycol dimethacrylate, was employed as a co-agent to promote irradiation effectiveness.

EXPERIMENTAL

One hundred percent cotton flannels $(3-1/2 \text{ oz/yd}^2)^{\dagger}$ were padded with a flame retardant formulation consisting of emulsions of tris (2,3-dibromo-

† Style 746 Cone Mills.

propyl) phosphate, Geon 222 vinyl chloride—vinyledene chloride co-polymer resin, ethyleneglycol dimethacrylate monomer to various add-on's. One set of the treated samples were exposed to high energy electron beam irradiation dosages of 2 MR and 4 MR at ambient temperatures in the presence of air using a 1-1/2 MeV Electron Beam Dynamitron Accelerator (Radiation Dynamics Inc., Westbury, L.I., N.Y.). The irradiated samples were then subjected to a series of successive laundering and tumble drying cycles using applicable AATCC test procedures.[†] Limiting Oxygen Index (LOI) and weight measurements were performed on the treated fabrics and after various laundering cycles.[†] Phosphorus analyses were carried out on selected samples to ascertain the composition of the treated cotton flannels as a function of laundering cycles. The other set of treated samples was not irradiated but subjected to an analogous series laundering and tumble drying cycles. Similar measurements were carried out to characterize the durability of this flame retardant treatment. Data are presented for tris (2,3-dibromopropyl) phosphate/Geon 222/ethylenedimethacrylate formulations of 2/1/0.05 wt. ratios padded onto cotton fabrics to give add-on's ranging from 30% to 60%based on the weight of the samples to illustrate the durability of the flame retardancy achieved.

RESULTS AND DISCUSSION

Tables I to III and Figures 1 to 7 illustrate the durability of irradiated (2 MR) and unirradiated flame retarded cotton flannels with original add-on's ranging from 37.8% to 60% in terms of LOI values and weight losses vs. laundering cycles. Typically the LOI of one set of the irradiated flame retarded cotton flannels decreased from 0.290 to 0.273 after 30 successive laundering cycles with a concurrent weight loss of 8%. The LOI of unirradiated samples dropped to 0.258 with a 23% weight loss after being subjected to the same number of laundering cycles. After 60 laundering cycles the LOI's of the irradiated and unirradiated samples were 0.262 and 0.223 respectively with corresponding weight losses of 16% and 30%. Similar data are presented for flame retarded cotton flannels with higher add-on's.

In Figure 7 there is shown the minimum original add-on to produce flame retardancy defined arbitrarily in terms of an LOI of 0.290, for various specified numbers of laundering cycles. The irradiated flame retarded cotton flannels are seen to require substantially lower original add-on's than the unirradiated treated samples to obtain this LOI value for equal number of launderings.

[†] AATCC Method 124-1969.

[‡] ASTM D-2863.

Residual % Add-on	Number of Launderings‡	% Weight Loss	LOI§	% P	% C l
		2 MR Irra	adiation		
37.0	0		0.290	1.10	7.80
32.7	15	6.8	0.278	0.95	7.33
31.2	30	8.2	0.273	0.86	8.00
26.8	45	12.8	0.267	0.85	4.93
23.3	60	16.4	0.262	0.70	4.86
		No Irrac	liation		
37.4	0		0.290	1.11	5.33
25.7	15	15.7	0.272	0.94	2.94
23.2	30	17.8	0.258	0.75	4.03
17.9	45	22.5	0.241	0.54	3.65
9.5	60	29.8	0.223	0.28	2.05

TABLE I

† Formulation: tris (2,3-dibromopropyl) phosphate/Geon 222/ethylene glycol dimethacrylate = 2/1/0.05.

‡ AATCC Method 124-1969.

§ ASTM D-2863.

Residual % Add-on	Number of Launderings‡	% Weight Loss	LOI§
	2 MR I	rradiation	
50.2	0		0.320
47.8	15	4.7	
47.7	30	4.8	
43.5	45	10.8	0.299
37.5	60	19.5	0.288
	No Irr	adiation	
53.3	0	_	0.324
44.1	15	17.0	
38.8	30	24.1	_
30.5	45	27.5	0.277
22.5	60	35.0	0.258

TABLE II Laundering durability of flame retarded cotton flannels†

[†] Formulation: tris (2,3-dibromopropyl) phosphate/Geon 222/ethylene glycol dimethacrylate = 2/1/0.05.

‡ AATCC Method 124—1969.

§ ASTM D-2863.

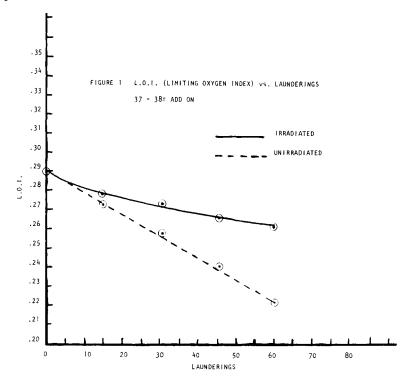
Laundering durability	of flame retarded cotton	flannels†
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Residual % Add-on	Number of Launderings‡	% Weight Loss	LOI§
	2 MR II	radiation	· • ••
61.5	0	-	0.347
59.0	15	6.2	
59.0	30	6.2	—
55.5	45	11.7	0.331
49.7	60	22.1	0.312
	No Irr	adiation	
61.1	0	_	0.342
46.7	15	26.9	
45.1	30	29.0	
38.0	45	37.2	0.288
30.0	60	44.5	0.273

[†] Formulation: tris (2,3-dibromopropyl) phosphate/Geon 222/ethylene glycol dimethacrylate = 2/1/0.05.

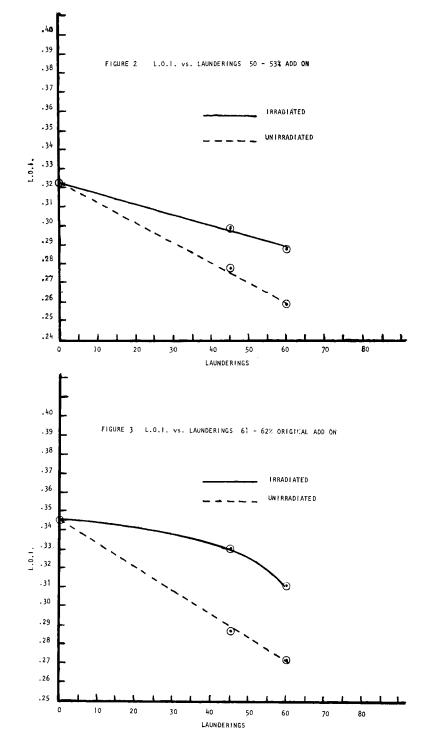
‡ AATCC Method 124—1969.

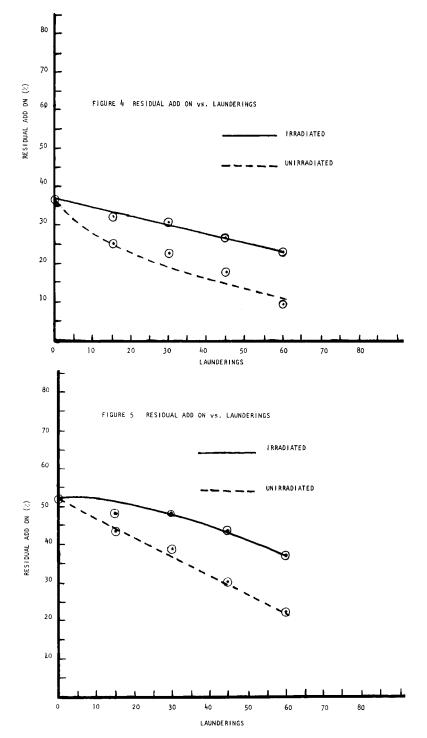
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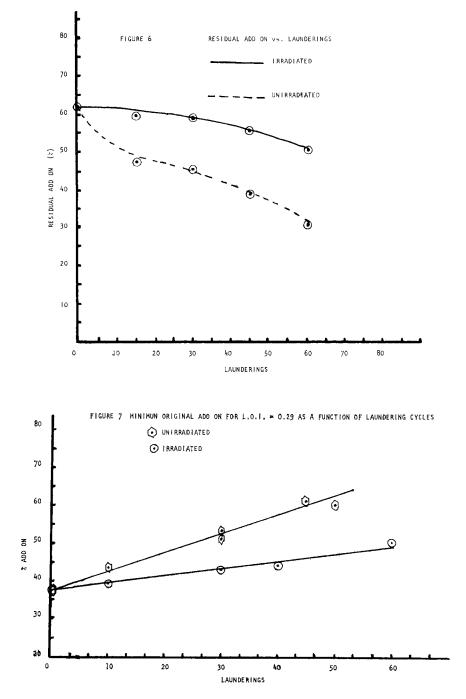


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Thus attaining an LOI value of 0.29 after 50 laundering cycles requires a minimum original add-on of 47% for the irradiated flame retarded samples versus 62% for their unirradiated analogues. Higher add-on's will of course provide greater LOI values and therefore, increased flame retardancy. Such add-on's are undesirable esthetically and also result in excessive weight, hand, etc.

As expected the low irradiation dosage employed minimized degradative effects on the strength of the cotton fabrics. The strength retentions of the irradiated fabrics generally exceeded 90%. Tests carried out with a 4 MR irradiation dosage appear to substantially enhance the durability of the finish as formulated. Strength retentions were somewhat lower at the higher dosages.

Experiments are in progress with modified flame retardant formulations of the same general type which will hopefully permit lower add-on's with at least equivalent laundering durability at acceptably high flame retardancy levels. High energy irradiation should be viewed as a potentially attractive approach for the development of improved durable flame retardancy of cotton textiles with judicious combinations of known materials and suitable co-agents.

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