

# Flame Retardation of Poly(ethylene Terephthalate) Containing Poly(4-Bromo Styrene), Poly(vinyl Bromide), and Poly(vinylidene Bromide)\*

M. DAY, T. SUPRUNCHUK, J. D. COONEY, and D. M. WILES,  
*Division of Chemistry, National Research Council of Canada,  
Ottawa, Canada K1A 0R9*

## Synopsis

The pyrolysis and gaseous combustion of poly(ethylene terephthalate) (PET) incorporating poly(4-bromostyrene), poly(vinyl bromide), and poly(vinylidene bromide) has been studied using thermogravimetry, flammability limit evaluation, and hydrogen bromide (HBr) evolution techniques. The data obtained have been compared with limiting oxygen index (LOI) flammability data to elucidate flame retardation mechanisms. All the organo bromides studied (applied either via topical treatment or radiation grafting) released HBr on pyrolysis which is capable of inhibiting the gas phase combustion reactions. Condensed phase interactions were also detected which were capable of altering the gaseous pyrolysates. Thermal stability considerations suggest that, although the aliphatic bromides are excellent sources of HBr, they are not ideal flame retardants for PET.

## INTRODUCTION

Organic bromides have long been recognized as effective flame retardants for polymeric materials in general<sup>1,2</sup> and poly(ethylene terephthalate) (PET) fabrics in particular,<sup>3,4</sup> and there is a large amount of data in the literature on the topic. Two approaches (chemical and physical) have been used to explain the action of bromine-containing chemicals; the one most widely accepted is the chemical one. The chemical mechanism assumes that the bromine-containing compounds release volatile species, mainly hydrogen bromide (HBr), into the gas phase combustion zone associated with the flame. It is in this zone that these species interfere with the free radical chain branching reactions to remove the high energy OH and H radicals and inhibit the combustion process.<sup>5</sup> The alternate physical mechanism proposed by Larsen<sup>6,7</sup> postulates that changes in mass and heat transfer are responsible for reduction in flame propagation. However, it should be noted this inhibition effect can only apply during the ignition of a polymer and will not be effective in preventing combustion under external heat flux conditions where polymer vaporization is high.

Organic bromides such as aliphatic, cycloaliphatic, and aromatic have all been utilized as flame retardants. In the case of PET, several comparative studies have been made of aromatic and aliphatic bromides as flame retardants from which it has been concluded that aromatic compounds are superior to aliphatic compounds.<sup>8,9</sup> However, studies in our laboratory indicate<sup>10</sup> that

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the suitability of an organic bromide for PET depends upon its thermal stability irrespective of being aromatic or aliphatic. The volatilization of undecomposed flame retardant chemicals is capable of causing ambiguity in data interpretation. It was therefore decided in this study to investigate polymeric species as opposed to conventional molecular compounds. It was also decided to compare polymeric bromine species incorporated into PET by topical treatment of the fabric against polymeric species grafted into the PET backbone. Using this approach, it should be possible to determine if incorporation technique plays any role in flame retardation efficiency, besides those associated with the chemical structure of the organo bromide.

The three brominated polymeric systems studied in this investigation were poly(4-bromostyrene) (PBS), poly(vinyl bromide) (PVB), and poly(vinylidene bromide) (PVDB). Radiation grafting, as a technique to achieve changes in the chemical and physical properties of textile materials, is a subject of much interest<sup>11</sup> with its use for flame-retarding materials, being the subject of two reviews<sup>12,13</sup> and a recent European study.<sup>14</sup> In the case of topical treatment with the polymers, penetration into the PET would be anticipated to be less than that of a monomer used in grafting. Consequently, comparisons of the two treatments should yield information concerning the influence of proximity of flame retardant structures to the combustibility of the basic polymer. Although studies have been made of vinyl bromide<sup>11,15</sup> and vinylidene bromide<sup>11</sup> and bromostyrene<sup>16</sup>-grafted PET, no direct comparisons have been made of their effectiveness as flame retardants. The present investigation was therefore undertaken to examine the flammability, thermal stability, and combustibility of the gaseous pyrolysates produced from a series of topically treated and radiation-grafted PET fabric samples.

## EXPERIMENTAL

### Materials

The PET fabric was a woven spun Dacron type 54 obtained from Test-fabrics Inc. (Style 767). All fabric samples were Soxhlet-extracted for 100 h with tetrahydrofuran and then vacuum dried before use.

The PBS, PVB, and PVDB were obtained from Polysciences Inc. and used as received without further purification. Chemical analysis for percentage bromine revealed the following data: PBS = 35% (expected 43.7%); PVB = 66.4% (expected 74.8%); PVDB = 76.0% (expected 85.9%).

The monomers 4-bromostyrene and vinylidene bromide were obtained from Polysciences Inc. while the vinyl bromide was obtained from Matheson. All monomers were vacuum-distilled prior to use in order to remove the inhibitor, while other solvents employed in the radiation grafting were degassed prior to addition to the fabric.

Topically treated samples were obtained by immersing the fabrics in a methylene chloride solution of the appropriate polymer. The wet fabric was then squeezed gently through a set of nip rollers and dried under ambient conditions. The percentage chemical pickup of the polymer was determined by appropriate chemical analysis for bromine.

The irradiation graftings were conducted in a  $^{60}\text{Co}$  gamma cell with a dose rate of 1.2 Mrad/h. In all cases a weighed fabric sample ( $10 \times 30$  cm) was placed in a Pyrex grafting tube on a vacuum line. The monomer or monomer plus solvent was then distilled from an adjacent tube and degassed in the grafting tube along with the fabric. The grafting tube was then sealed and irradiated for a predetermined period. The bromostyrene grafts were performed using monomer solutions of various concentrations in methyl alcohol (total weight 6 g) for a period of 30 min. The vinyl bromide and vinylidene bromide grafts were performed using neat monomer with irradiation times of 90 min. Following irradiation, all samples were removed from the grafting tubes and extracted for 100 h with tetrahydrofuran in a Soxhlet extractor to remove traces of homopolymer. The samples were then dried, and the percentage grafting was determined by chemical analysis for bromine.

### Flammability Measurements

Limiting oxygen index (LOI) determinations were made using the Textile Research Institute flammability apparatus.<sup>17</sup> Because of limitations in the size of the samples and difficulties in obtaining satisfactory data for unsupported specimens due to the melt-drip phenomenon, a special test method was developed. The LOI values were determined on  $5 \times 100$  mm strips suspended on a nonwoven fibreglass support mounted on the wheel. Measurements were made using forced bottom ignition at the 3 o'clock position on the wheel.

### Flammability Limit Measurements

These determinations were carried out according to the procedure used previously to study other flame-retarded PETs<sup>18</sup> and described in detail in an earlier publication.<sup>19</sup> Essentially, the technique involves pyrolysing 200 mg of material in a closed bomb ( $165\text{ cm}^3$ ) and exposing the pyrolysate gases to a spark discharge. Data concerning weight loss as a function of time and times required to obtain flammable pyrolysate/air mixtures enable flammability limits of the gaseous products to be determined.

### Thermal Analysis

Thermogravimetric (TG) data were obtained using a DuPont 951 thermogravimetric analyzer coupled to a DuPont 1090 thermal analyzer. Experiments were performed on  $18 \pm 2$  mg samples heated in flowing air (50 cc/min) at seven heating rates between 1 and  $50^\circ\text{C}/\text{min}$ . Kinetic data were extracted from the TG curves using the Kissinger technique<sup>20</sup> previously described in detail.<sup>21</sup>

### Thermal Release of Bromide Ions

The release of HBr under thermooxidative degradation conditions was measured using the procedure described previously.<sup>10</sup> This technique involves monitoring  $\text{Br}^-$  ions present in the exhaust gas from a Cahn balance in which 15–18 mg samples are heated rapidly with an infrared heater, similar to that employed in the flammability limit studies.

TABLE I  
Weight Loss and Bromide Ion Release for the Brominated Polymers

Polymer	PBS			PVB			PVDB		
	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>8</sup> )	% Br liberated	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>8</sup> )	% Br liberated	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>8</sup> )	% Br liberated
10	2.06	0.79	0.15	0.11	0	0	0.32	0.51	0.05
20	3.16	2.09	0.39	0.13	0	0	0.36	2.46	0.23
40	5.06	5.20	0.96	0.17	7.05	0.75	0.41	12.46	1.16
60	8.90	8.76	1.61	0.20	33.77	3.61	0.46	29.49	2.74
80	—	—	—	0.25	122.95	13.15	0.90	89.03	8.27
90	—	—	—	0.70	299.56	32.04	1.83	110.69	10.29

### Chemical Analysis

The bromine content of the treated PET samples was determined by igniting the samples in an oxygen bomb and titrating the resultant solutions potentiometrically with a standard silver nitrate solution.

## RESULTS AND DISCUSSION

### Release of HBr

An initial examination of the effectiveness of the different polymers to liberate monitorable bromide ion is presented in Table I. This data clearly indicate that, under the oxidative pyrolysis conditions employed in the bromide ion liberation experiments, the PVB yields the largest quantities of measurable bromide ion in terms of total amount and expressed as a percentage of the available bromine in the polymer. Meanwhile the PVDB, while liberating a substantial amount of measurable Br<sup>-</sup>, appears to decompose preferentially to give species other than those easily converted to bromide ion, since at the 90% weight loss just over 10% of the available bromide has been monitored as bromide ion. In the case of PBS, only a small amount of bromide ion is released under these conditions. This may be attributed in part to the greater stability of the polymer. However, when the experiment was stopped after 15 min at a percentage weight loss of 77.4%, only 2% of the available bromine in the polymer had been released as monitorable bromide ion.

The thermal stability of the chemical species responsible for the flame retardation process is obviously an important factor in the physical and chemical interactions taking place between these active species and the material being flame retarded. Thus, while the data presented in Table I provide information regarding the potential capability of these chemical species to generate monitorable bromide ions, it is essential to confirm that their release is appropriate to influence either the fuel producing reactions of the decomposing PET or be present to inhibit the combustion of the gaseous fuels. The weight loss and liberation of monitorable bromide ion from topically treated PET and grafted PET samples are presented in Tables II and III respectively, along with weight loss data for untreated PET.

TABLE II  
Weight Loss and Bromide Ion Release from Topically Treated PET Samples

Polymer	Neat	PBS			PVB			PVDB		
		at 12.9% add-on (5.6% Br)			at 5.3% add-on (3.9% Br)			at 9.4% add-on (8.1% Br)		
% Wt loss	Decomp. time (min)	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>5</sup> )	% Br liberated	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>5</sup> )	% Br liberated	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>5</sup> )	% Br liberated
10	1.72	2.52	0.20	0.28	0.57	8.95	18.33	0.21	15.06	14.86
20	1.92	3.68	0.42	0.60	0.68	9.49	19.45	0.33	21.29	21.00
40	2.14	5.21	0.73	1.04	0.81	10.05	20.60	0.46	23.91	23.58
60	2.42	5.94	0.90	1.28	0.92	10.47	21.46	0.58	25.24	24.90
80	3.18	7.83	1.71	2.43	1.55	11.58	23.73	1.15	27.46	27.09
90	8.00	—	—	—	3.40	12.95	26.53	2.19	30.14	29.73

In the case of PBS incorporated into PET, samples from both the topically treated sample and the radiation-grafted sample liberated approximately the same percentage of available bromine as was obtained from the neat PBS (i.e., between 1.5 and 2.5%). Thus the HBr liberation from this chemical appears to be unaffected by its polymeric environment, and the nature of its incorporation into the PET (i.e., topically or chemically grafted). The HBr yields being measured are also in close agreement with the values reported by Mey-Marom and Rajbenbach,<sup>16</sup> who concluded that, in an oxygen environment, the majority of the bromine is released as species other than HBr, mainly as the monomer, formed as a result of depolymerization.

The thermal degradation of PVB is known to yield almost 100% of its available bromine as HBr<sup>22</sup> in a chain-stripping reaction, before the resultant polyene undergoes subsequent degradation. In our studies, although large quantities of monitorable Br<sup>-</sup> were detected, the values were consistently in the 26–32% range irrespective of whether the species was present in the PET as a topical treatment or a chemical graft or, alternatively, decomposed separately from the PET system. Although PVB is capable of releasing large quantities of HBr on heating, the data in Tables II and III suggest that in the case of its incorporation into PET its poor thermal stability may be causing the HBr release to be premature for a combustion inhibitor.

TABLE III  
Weight Loss and Bromide Ion Release from Radiation-Grafted PET Samples

Polymer	Neat	PBS			PVB			PVDB		
		at 14.5% add-on (6.3% Br)			at 4.5% add-on (3.4% Br)			at 8.7% add-on (7.5% Br)		
% Wt loss	Decomp. time (min)	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>5</sup> )	% Br liberated	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>5</sup> )	% Br liberated	Decomp. time (min)	[Br <sup>-</sup> ] (mol/mg × 10 <sup>5</sup> )	% Br liberated
10	1.72	1.21	0.05	0.07	0.33	7.02	16.51	0.31	2.32	2.47
20	1.92	1.51	0.05	0.07	0.40	8.16	19.19	0.37	3.36	3.58
40	2.14	2.42	0.06	0.08	0.51	9.07	21.31	0.49	4.38	4.66
60	2.42	2.88	0.17	0.21	1.03	10.74	25.23	0.81	4.83	5.14
80	3.18	4.09	0.67	0.85	5.57	13.62	32.00	1.73	5.76	6.14
90	8.00	8.05	1.53	1.93	—	—	—	4.88	6.98	7.43

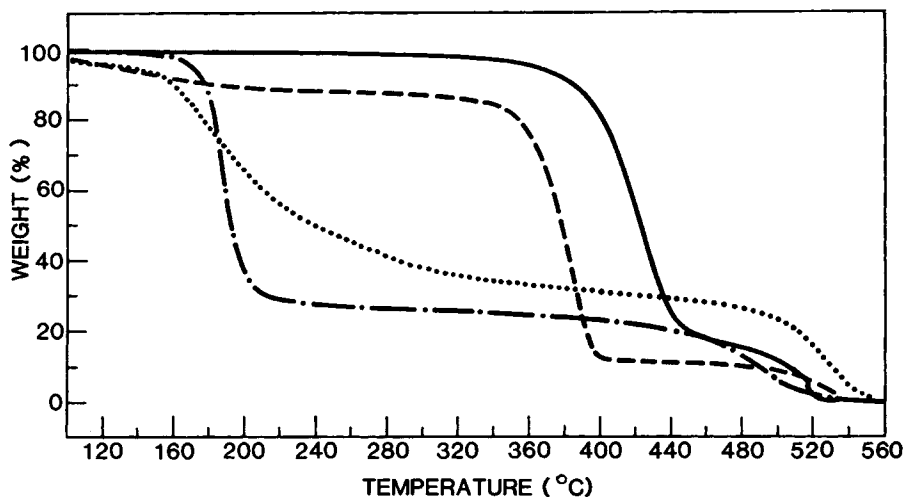


Fig. 1. Thermogravimetric weight loss curves for PET (—), PBS (---), PVB (···), and PVDB (— · —) heated at 5°C/min in air.

PVDB, like PVB, releases large amounts of HBr, although, in the case of the neat polymer and chemically grafted polymer, the amounts are slightly less than those observed with the topically treated sample. However, as was noted in the case of the PVB treated samples, the HBr release occurs very rapidly in the early stages of the thermal decomposition of the PET, and its availability in the gas phase at the appropriate time must be questioned.

The thermogravimetric weight loss curves for the bromo polymers and PET are shown in Figure 1, for the samples heated in air at a heating rate of 5°C/min.

The early weight losses corresponding to the liberation of HBr are clearly evident for the PVB and PVDB polymers. Although the onset of weight loss associated with the HBr liberation commences slightly earlier in the case of PVDB, its release rate is not as rapid as that observed in the case of PVB, which is soon completed. The thermal stability of PBS, while being more comparable to that of PET, because of the relative low release of HBr, its effectiveness as a gas phase combustion inhibitor will be correspondingly reduced.

### Flammability Limits

Investigations in this laboratory have shown that measurement of the flammability limits of gaseous pyrolysates produced by the thermal oxidative decomposition of polymer mixtures are capable of providing fundamental information on the role of a variety of chemical species on the gaseous fuel producing reactions and combustion inhibition processes.<sup>18</sup> The results of these studies for the PBS, PVB, and PVDB systems are shown in Figures 2, 3, and 4, respectively. These graphs also contain information on the untreated PET and the treated polymer as well as experiments performed in which the PET and treatment polymer were heated separately under conditions in which the pyrolysates from each were liberated without any condensed phase interaction.

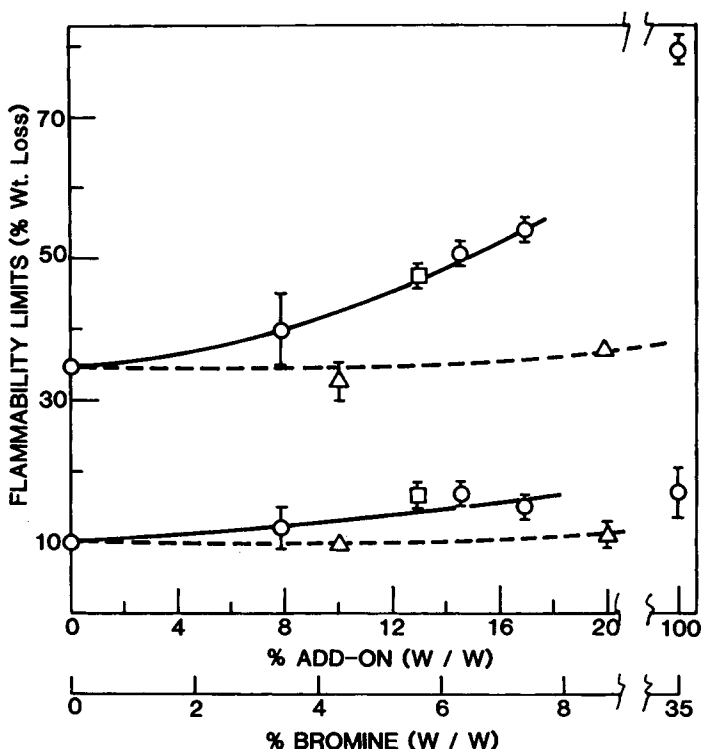


Fig. 2. Flammability limits (expressed as a function of percentage total weight loss) for PET/PBS combinations examined as a function of PBS in the system. Results for grafted samples (○), topically treated sample (□), and physically separated tests (△).

Considering first the results obtained with the PBS (Fig. 2), it will be noted that the data for the grafted PET and the topically treated PET behave in a similar manner, being responsible for an increase in both the lower and upper flammability limits of the PET material. However, the increase in the lower flammability which is taken as an indication of reduced ignitability is only slight in comparison with the increase in the upper flammability limit and expanded flammability range observed as a function of treatment level. This expansion of the flammability range suggests that the gaseous pyrolysates, produced as a result of condensed phase interaction, are more flammable than those produced from PET on its own. This adverse effect of the presence of PBS on the condensed phase degradation of PET appears to be confirmed by the experimental results obtained when the PET and PBS were heated separately in the same test, since, in these studies, the upper and lower flammability limits are very similar to those obtained for PET alone, implying that the increased flammability range is due to condensed phase activity. These data, therefore, clearly suggest that PBS is an unlikely candidate for flame retarding PET whether used as a topical treatment or as a chemical graft in the polymer. The observed wide distribution of the flammability limits of the neat PBS suggests that its gaseous pyrolysates are also highly combustible, and instead of inhibiting the chemical reactions, it is capable of contributing more fuel to the pyrolysates produced from PET.

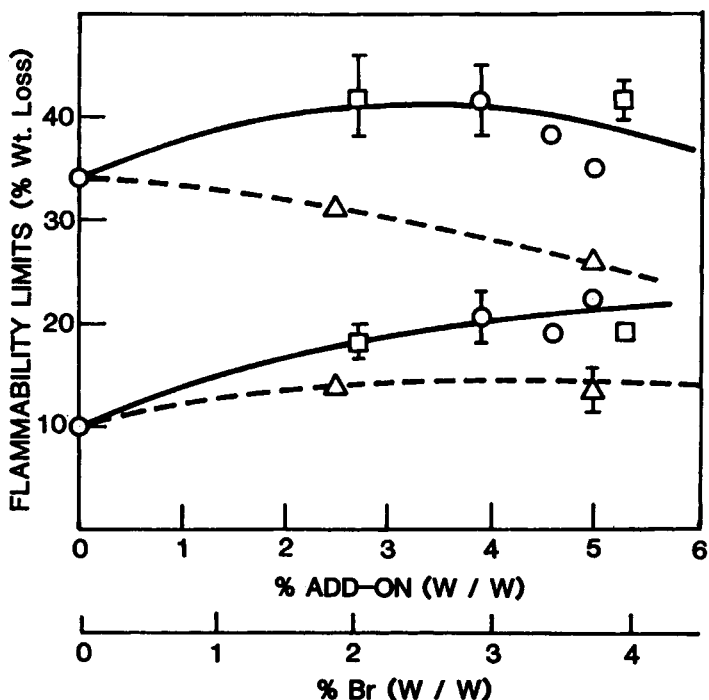


Fig. 3. Flammability limits (expressed as a function of percentage total weight loss) for PET/PVB combinations examined as a function of PVB in the system. Results for grafted samples (○), topically treated samples (□) and physically separated tests (△).

The results obtained with PVB are given in Figure 3. Examination of the gaseous pyrolysates produced from neat PVB in the absence of PET resulted in no ignition of the fuel/air mixtures. This result is not too surprising in view of the large amount of HBr known to be liberated on pyrolysis of this polymer. The interesting observation is the discrepancy in the flammability limits produced by the topically treated and grafted samples in comparison with those obtained in the experiments employing the separated system. In the case of the treated PETs (both graft and topical) the increase in the lower limit is greater than those obtained in the separated system, while in the case of the upper flammability limit, although the nature of the treatment has little effect on the limit, when tested in the separated mode (i.e., no condensed phase activity), the PVB causes a marked reduction in the upper flammability limit. Clearly some condensed phase interaction between the PVB and PET is occurring prior to the release of gaseous pyrolysates to account for these differences. Taken as a whole, therefore, PVB appears to be an effective combustion inhibitor for PET. In terms of the nature of the incorporation of PVB into PET (i.e., grafted or topically treated) it appears to have little influence on the observed flammability limits and, consequently, the condensed phase decomposition mechanism.

The neat polymer PVDB failed to give combustible fuel/air mixtures which could be ignited under our test conditions. This result, like that for the neat PVB, was anticipated in view of the major gaseous pyrolysate being HBr.



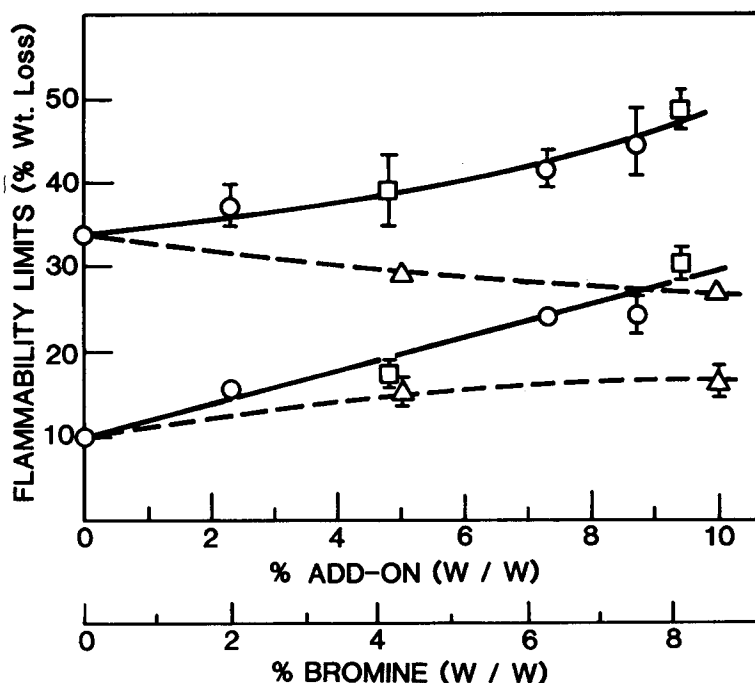


Fig. 4. Flammability limits (expressed as a function of percentage total weight loss) for PET/PVDB combinations examined as a function of PVDB in the system. Results for grafted samples (○), topically treated samples (□) and physically separated tests (△).

When tested in combination with the fuel generating PET, results for PVDB (Fig. 4) are similar to those found with PVB. The liberation of HBr and its influence on the combustion of gaseous fuel produced from PET is immediately evident from the studies employing the polymers physically separated in that both the lower and upper flammability limits are converging as the amount of PVDB in the system is increasing. However, when the PVDB is included in the PET either as a graft or topical treatment, the flammability limits are displaced to higher values, although there is still some reduction in the flammability range as the concentration of PVDB is increased. This suggests that, as in the case of PVB, the PVDB is still acting as a gas phase inhibitor and is capable of influencing the kinetics or mechanism of the fuel producing reactions of PET. The presence of the PVDB in the PET as a topical treatment or as a chemical graft once again appears to have no effect upon the condensed phase degradation and chemical inhibition processes.

#### Degradation Kinetics

Because of the apparent influence of PVB and PVDB on the condensed phase degradation processes, it was decided to analyze the kinetics of the main thermal decomposition steps of PET to determine what kind of an effect, if any, these organo bromides had on the kinetics of the weight loss process. In this investigation, it was decided to consider the main PET weight loss stage and the high temperature degradation stage only and determine the

TABLE IV  
Maximum Rate Temperatures and Calculated First-Order Activation Energies  
for Degradation of PET According to Kissinger's Method<sup>20</sup>

Chemical	Treatment	Add-On (% w/w)	% Br on fabric (% w/w)	Temp. max rate at 5°C/min (°C)		Activation energy (kJ/mol)	
				Main	High temp.	Main	High temp.
None	—	—	—	425.5	515.7	242.8 ± 6.6	146.1 ± 5.5
PBS	Graft	14.5	6.3	422.6	523.1	183.2 ± 6.5	164.3 ± 6.3
PBS	Topical	12.9	5.6	428.6	517.6	196.9 ± 3.9	155.5 ± 5.5
PVB	Graft	4.5	3.4	429.8	526.7	180.5 ± 7.5	150.3 ± 3.7
PVB	Topical	5.3	3.9	431.1	514.6	197.7 ± 5.9	171.9 ± 7.2
PVDB	Graft	8.7	7.5	431.8	520.7	184.1 ± 4.3	198.2 ± 5.6
PVDB	Topical	9.4	8.1	425.3	502.3	202.5 ± 3.5	175.5 ± 8.2

activation energy from the maximum rates of weight loss according to the technique described by Kissinger.<sup>20</sup> The results of these calculations are given in Table IV along with the actual maximum rate temperatures as determined from the experiments conducted at a heating rate of 5°C/min. The reported activation energies, meanwhile, are calculated from all seven heating rates as described previously.<sup>21</sup> From an examination of the data for the main weight loss stage of PET, it can be seen that, although there does not appear to be any major changes in the actual temperature of the maximum rates of weight loss for the treated samples in comparison with the untreated PET, in all cases the activation energies are less for the treated samples than the untreated one. In all cases, the grafted samples were also noted to have activation energies slightly less than those for the topically treated samples.

When the high temperature degradation of PET is considered, however, some different factors appear. Although there are no large differences in the actual temperatures of the maximum rates between the treated and untreated samples, the temperature of the maximum rate for the grafted samples are always higher than those for the topically treated samples. In terms of the calculated activation energies for the degradation, meanwhile, all treatments, both graft and topical, appear to be responsible for a slight increase in activation energy. However, because of the tendency of all polymers to form residues which undergo further weight losses at these temperatures (see Fig. 1), the actual number of chemical species undergoing reactions at these higher temperatures could be large, and consequently difficult to interpret.

### Flammability Data

The chemical and physical analyses outlined above indicate that grafting to or topically treating PET with brominated polymers causes changes in the pyrolysis and gas-producing reactions of degrading PET. However, the ultimate question is how effective they are in reducing the flammability characteristics of the treated fabrics. The results of the LOI determinations are presented in Figure 5. It will be noted that while all treatments caused increases in the LOI values, the actual magnitude of the increases were not exceptionally large. In terms of the HBr release measurements and flammabil-

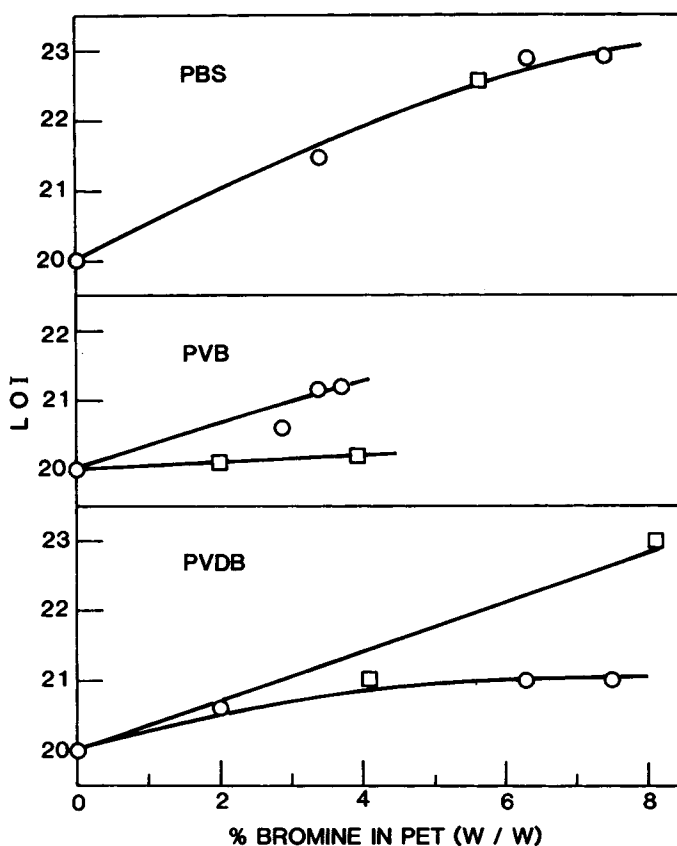


Fig. 5. Limiting oxygen indices for the grafted (○) and topically treated (□) samples of PET containing PBS, PVB, and PVDB.

ity limit determination, the behavior of the PBS in comparison to the other two polymers is rather surprising. The flammability limits of the PBS systems increased as the concentration of PBS increased (see Fig. 2), reflecting the greater flammability of PBS in comparison to PET. It would therefore have been anticipated that the LOI values may have decreased rather than increased as a function of adding PBS to the system. The release of HBr from the system, however, must be sufficient under the conditions of the LOI test to effectively cause the observed increase.

In the case of PVB and PVDB, the increases in LOI are not as great as would have been anticipated based upon the HBr release and flammability limit measurements. This, in part, may be explained by the fact that, in the case of combustion in the LOI studies, the HBr evolution occurs in advance of the gaseous pyrolysate fuel producing reactions of the PET, and as a result the normal combustion reactions are not being inhibited as efficiently as if the HBr was being released directly into the fuel.

In terms of incorporation techniques (i.e., graft compared to topical treatment), there do not appear to be any large differences based upon the oxygen index values.

## SUMMARY

The results of this study indicate that all of the organo bromo polymers studied are capable of acting as flame-retardant additives for polyester fabrics. While PVB and PVDB would appear to have the greatest merit in terms of releasing HBr to inhibit the gas phase combustion reactions, because of low HBr release temperatures their potentials are not fully realized. Flame quenching due to HBr release is, however, important in achieving reduced flammability characteristics since both the flammability limit and thermogravimetry studies suggest some condensed phase chemical interactions are occurring to influence the fuel producing reactions. In the case of PBS, flammability limit data suggest that the gaseous pyrolysate fuel being produced from the treated PET is more flammable than that obtained from untreated PET. The evolution of HBr therefore is the major contributing factor to flame retardation in the PBS/PET system, not the reduction of volatile flammable gases.

## References

1. E. R. Larsen, in *Encyclopedia of Chemical Technology*, Kirk-Othmer, 3rd ed., Wiley, New York, 1980, Vol. 10, p. 373.
2. M. M. Hirschler, *Dev. Polym. Stabilization*, **5**, 107 (1982).
3. H. E. Stepniczka, *J. Fire Retardant Chem.*, **2** (1), 30 (1975).
4. E. L. Lawton and C. J. Setzer, in *Flame-Retardant Polymeric Materials*, M. Lewin, S. M. Atlas, and E. M. Pearce, Eds., Plenum Press, New York, Vol. 1, p. 193 (1975).
5. M. C. Drake and J. W. Hastie, *Combust. Flame*, **40**, 201 (1981).
6. E. R. Larsen, *J. Fire Retardant Chem.*, **1**, 4 (1974).
7. E. R. Larsen and R. B. Ludwig, *J. Fire Flammability*, **10**, 69 (1979).
8. U. Einsele, *Melliand Textilber. Int.*, **57**, 64, (1976).
9. W. C. McNeil, M. J. Drews, and R. H. Barker, *J. Fire Retardant Chem.*, **4**, 222 (1977).
10. M. Day and D. M. Wiles, *J. Fire Sci.*, **1**, 255 (1983).
11. V. Stannett, W. K. Walsh, E. Bittencourt, R. Liepins, and J. R. Surles, *J. Appl. Polym. Sci. Symp.*, **31**, 201 (1977).
12. R. Liepins, V. Stannett, and W. K. Walsh, *Prog. Polym. Sci.*, **6**, 1 (1978).
13. A. H. Zahran, V. Stannett, R. Liepins, and N. Morosoff, *Radiat. Phys. Chem.*, **16**, 265 (1980).
14. B. J. Hill, U. Einsele, G. DiModica, and D. Wattiez, Comm. Eur Communities, [Rep], EUR Commission of the European Communities. Directorate General, Information, Market, and Innovation, Luxembourg, 1980, EUR 6718, 38 pp.
15. R. Liepins, J. R. Surles, N. Morosoff, and V. T. Stannett, *J. Appl. Polym. Sci.*, **21**, 2529 (1977).
16. A. Mey-Marom and L. A. Rajbenbach, *J. Appl. Polym. Sci.*, **28**, 2411 (1983).
17. B. Miller and C. H. Meiser, *Text. Chem. Colour.*, **3**, 118 (1971).
18. M. Day, T. Suprunchuk, and D. M. Wiles, *Fire Mater.* **7**, 150 (1983).
19. M. Day, T. Suprunchuk, and D. M. Wiles, *J. Appl. Polym. Sci.*, **28**, 3085 (1981).
20. H. E. Kissinger, *Anal. Chem.*, **21**, 1702 (1957).
21. J. D. Cooney, M. Day, and D. M. Wiles, *J. Appl. Polym. Sci.*, **28**, 2887 (1983).
22. I. C. McNeill, T. Straiton, and P. Anderson, *J. Polym. Sci., Polym. Chem. Ed.*, **18**, 2085 (1980).

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