

# Preparation of Flame-Retardant Polyethylene Foam of Open-Cell Type by Radiation Grafting of Vinyl Phosphonate Oligomer

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## SYNOPSIS

Flame-retardant polyethylene foam was successfully obtained by grafting a vinyl phosphonate oligomer onto polyethylene foam of an open-cell type using a simultaneous radiation grafting technique. The foam was impregnated with the oligomer and irradiated in a nitrogen atmosphere with an electron beam. The grafted foam thus obtained was found to pass the three most severe flammability tests that have to be cleared when the foam is to be used for materials where high flame-retardant property is required. No hydrogen cyanide was detected in burning exhaust gas of the grafted foam. © 1994 John Wiley & Sons, Inc.

## INTRODUCTION

Polyethylene foam of an open-cell type is widely used as sealing, shock absorbing, and sound-insulating materials, because of its excellent character in elasticity and chemical resistivity. However, the foam is highly flammable, because most of its volume is occupied by air, and, therefore, it becomes material that is dangerous in a fire.

The most popular material used for cushion material of seats of automobiles, passenger trains, or airplanes is polyurethane foam or flame-retardant polyurethane foam for the case where flame retardancy is highly required. However, polyurethane contains C—N bonds that are converted to a very toxic hydrogen cyanide gas during a fire depending on its burning condition, causing a serious disaster. Therefore, preparation of the foam material that does not form these toxic gases is a most important and urgent necessity for public safety. Since polyethylene does not contain such chemical bonds as C—N, nor C—Cl, which form poisonous gases on

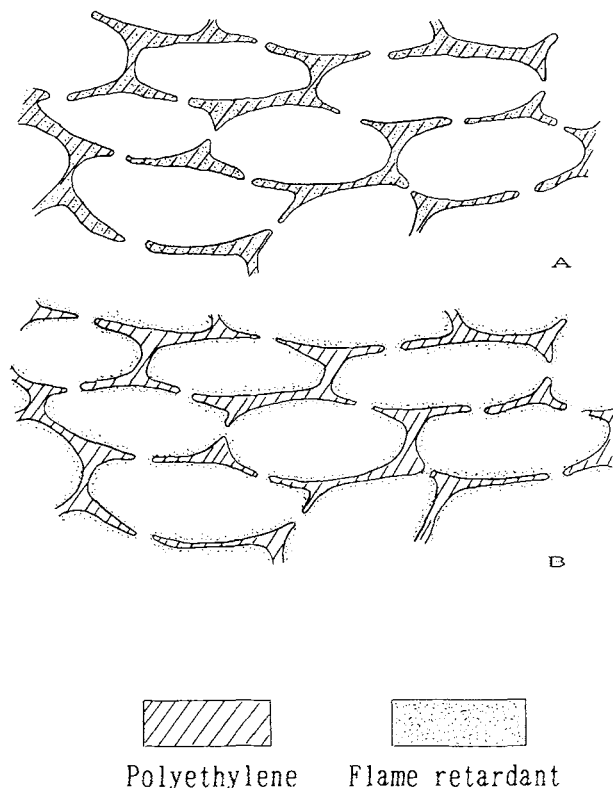
fire, modification of the polyethylene foam to give a flame-retardant property is especially suitable for this purpose.

Flame-retardant modification of plastic material has been carried out by the method in which the flame-retardant reagent is simply kneaded to the material before the molding or casting process, but this method was proved unsuccessful for polyethylene foam of an open-cell type [Fig. 1(a)], because the foaming was not possible when the least amount of the reagent required to give a flame-retardant property was kneaded into the polyethylene. One of the present authors reported that self-extinguishing polyester fabric was successfully obtained by radiation-induced grafting of a vinyl phosphonate oligomer (degree of grafting, ca. 10%) on the polyester fabric.<sup>1</sup> The vinyl phosphonate oligomer was selected because the unsaturated oligomer of high molecular weight does not diffuse into the polyester fabric but forms an apparent grafted layer of polymerized and cross-linked oligomer. By applying this technique to the polyethylene foam of an open-cell type, it is expected that a flame-retardant grafted layer can be formed on the cell wall of the foam [Fig. 1(b)].

This article describes a successful result of this technique to prepare highly flame-retardant polyethylene foam of an open-cell type by radiation grafting of vinyl phosphonate as a flame-retardant

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**Figure 1** Two methods for preparation of flame-retardant polyethylene foam of open-cell type: (A) kneading of flame retardant to polyethylene before foaming process; (B) grafting of flame-retardant reactive oligomer.

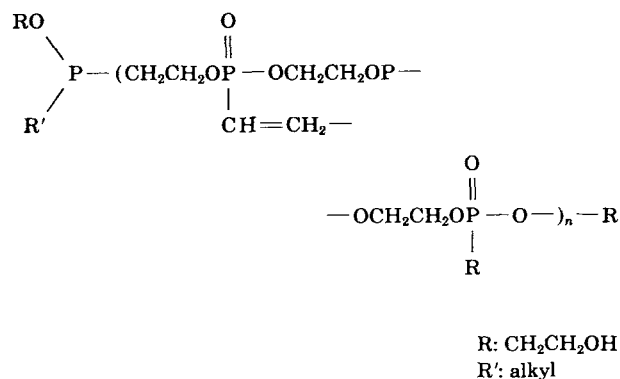
reactive oligomer covering the cavity wall in the foam materials and results of three typical flammability tests: UL 94-HF (horizontal),<sup>2</sup> Tetsu-un 81 (45°),<sup>3</sup> and FAR 25.853(b) (vertical),<sup>4</sup> which should be cleared in order that the material can be used for electronic appliances, interiors of a railway coach, and interiors of airplanes, respectively.

## EXPERIMENTAL

### Materials

The open-cell-type polyethylene foam was supplied by Sanwa Kako Co. (Type LC-300 #3, apparent density, 0.027 g/cm<sup>3</sup>, average radius of the cell, 2 mm), in a sheet form of 1 cm × 30 cm × 2–6 m. Vinyl phosphonate oligomer (VPO; Fyrol 76R; molecular weight, 500–1000; content of phosphorus, 22.5% by weight) was obtained from Akzo Chemicals Co. and used as received. The chemical formula as

given by the supplier is



### Grafting Procedure

Grafting was carried out using a “simultaneous irradiation” method. The whole procedure is illustrated schematically in Figure 2(a) and the apparatus used for the research is shown in Figure 2(b).

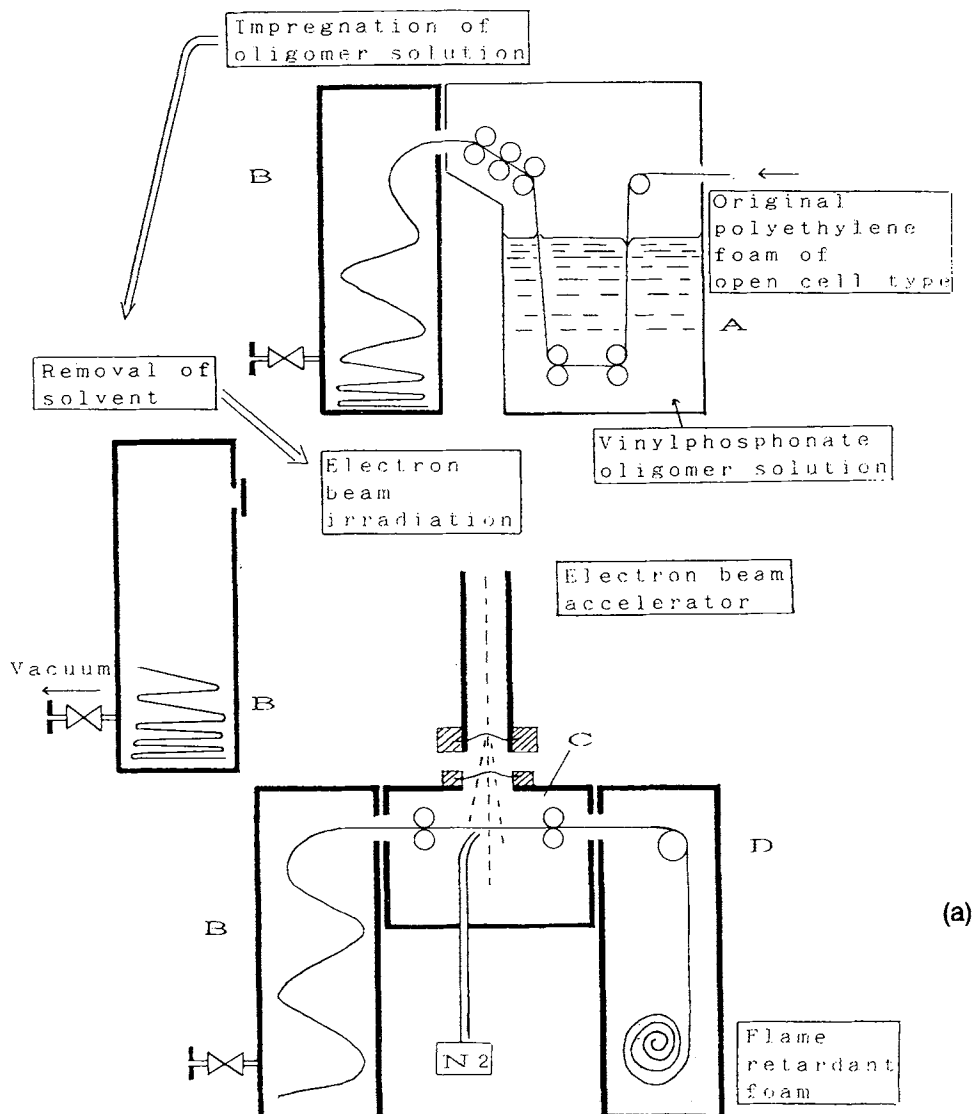
### Viscosity Measurement

The relative value of the viscosity of VPO diluted with water or methanol was measured by the Zahn cup method<sup>5</sup> as a function of concentration of the VPO solution, measuring the elution time through a 1.98 mm-diameter orifice.

### Impregnation of VPO

The foam material was immersed in a 100 L vessel containing a VPO aqueous solution or a methanol solution of different concentrations, and then the foam was squeezed by passing between a variable gap of two rollers to remove the excess amount of the oligomer solution. The semidried foam was then dried in a vacuum oven at 50°C to a constant weight to remove the solvent. The degree of impregnation was determined gravimetrically.

In the continuous impregnation for irradiation, the impregnation was carried out using the vessel shown in Figure 2, containing 36% VPO aqueous solution. Two sets of rollers squeeze and expand foam successively in the solution to impregnate the solution and simultaneously replace air contained in the foam cell with the solution. The foam then further passed through three gaps between three sets of rollers in a nitrogen atmosphere to remove the VPO solution for a desired degree of impregnation. The amount of removal of the VPO solution can be adjusted by selecting the proper gap width. The solvent was removed in a vacuum oven at 50°C for 30 min.



**Figure 2** (a) Preparation of flame-retardant polyethylene foam of open-cell type: (A) impregnation compartment; (B) carrying cassette; (C) irradiation vessel; (D) uptake role compartment of final product. (b) Photographs showing apparatus for (1) impregnation and (2) irradiation to manufacture flame-retardant-grafted polyethylene foam: (A) roll of original polyethylene foam; (B) impregnation vessel; (C) controller for foam carrying velocity; (D) rollers; (E) electron accelerator; (F) foam feeding cassette; (G) roller gap adjuster; (H) inlet of purge gas (nitrogen); (I) controller for foam carrying velocity; (J) foam take-up role.

### Irradiation

The foam containing VPO was introduced from the impregnation vessel at a constant velocity from 0.5 to 10 m/min into an irradiation chamber equipped with an irradiation window at the top (Fig. 2). Irradiation was carried out with electron beam (0.8 MeV, 2–7.5 mA) from an accelerator of a transformer type (Nissin High Voltage Co.) in a nitrogen

atmosphere on a conveyer traveling 1–15 cm below the irradiation window. The concentration of oxygen in the irradiation atmosphere was monitored by an oxygen meter (Touken model ECOA Z-CG) and was below 1.0%.

The irradiation conditions were selected so that most of the VPO impregnated in the foam is polymerized on the basis of the extraction experiment mentioned below. Therefore, the degree of apparent

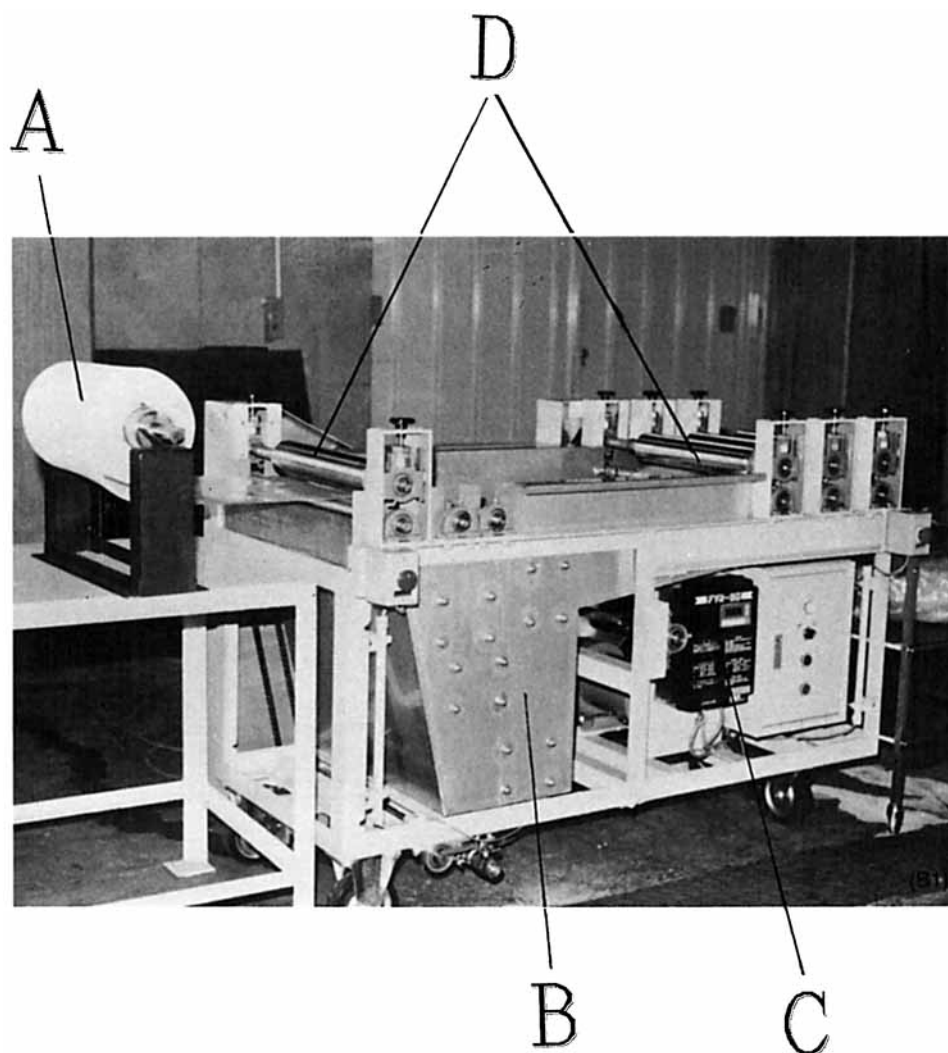


Figure 2 (Continued from the previous page)

graft is regarded to be equal to the degree of impregnation. Examples of the irradiation conditions for the complete polymerization of VPO are listed in Table I.

#### **Extraction Experiment**

Irradiated samples were subjected to Soxhlet extraction to determine the amount of polymerized VPO using acetone and water as the solvent at 80 and 100°C, respectively. The samples were dried in a vacuum oven for 30 min at 50°C. Conversion to polymer was calculated from the difference of the mass of the sample before and after the extraction procedure. Correction was made for the decrease of mass of the original foam by the same extraction procedure.

#### **FTIR Measurement**

A block of foam extracted with water was sliced to thin pieces (thickness, ca. 2 mm) that were forced on both surfaces of a 45° zinc arsenide prism for ATR-IR spectroscopy. The spectrum was recorded on a Perkin-Elmer 1720 × spectrophotometer.

#### **Tests of the Grafted Foam**

##### **Measurement of Dripping During Burning**

To estimate the amount of dripping during burning and the burning rate, a torsion balance shown in Figure 3 was used to measure the weight of sample during burning as a function of time. A piece of the sample (1 × 50 × 0.1 cm) was hung at the end of an

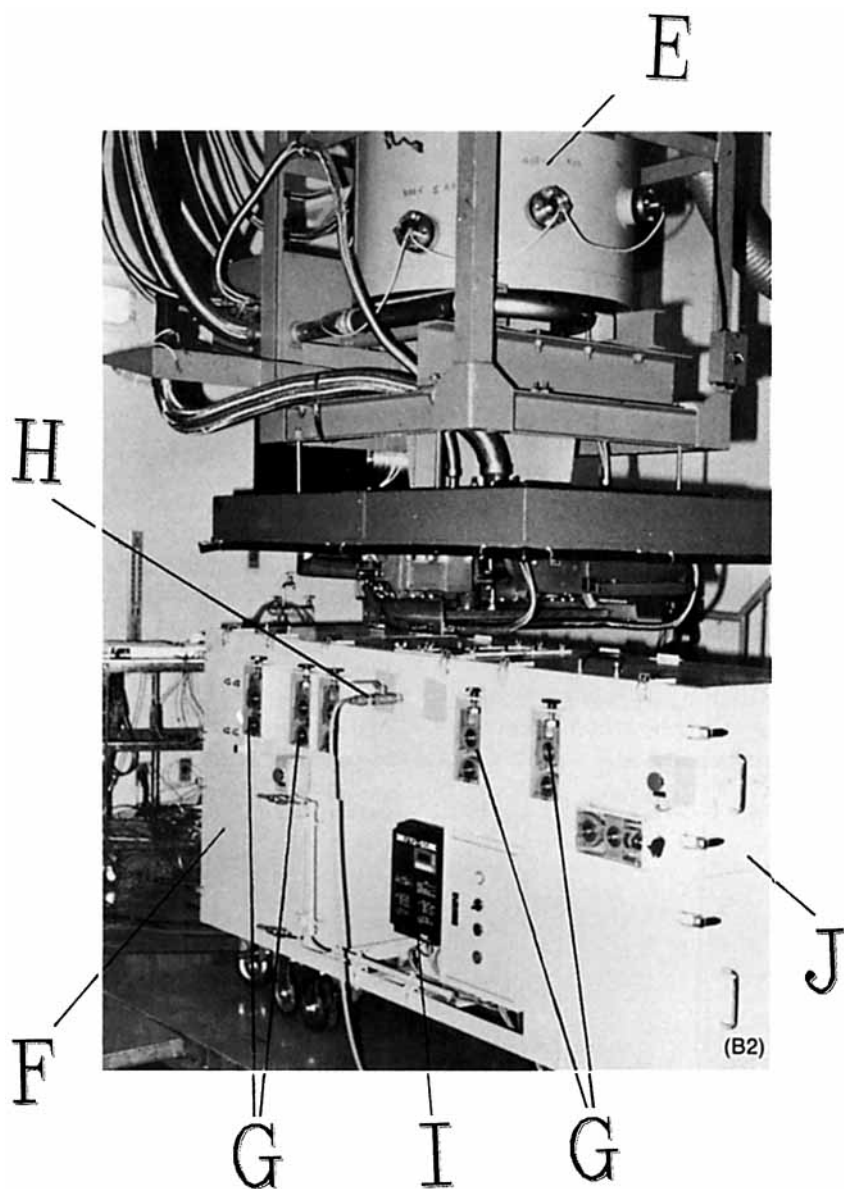


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arm of the torsion balance and the output of a linear variable differential transformer was recorded on a strip chart recorder.

#### **Test of Flammability of the Grafted Foam**

Tests of the flammability of the grafted foam were carried out by the following three standards that are illustrated in Figure 4(a)–(c). The burning time and charred distance were measured in seconds and millimeters, respectively, after the following

procedure:

- (i) (a) UL (Underwriter's Laboratories, Inc.) 94HF-1 (horizontal burning test): The specimen ( $152 \times 50.8 \times 5$  mm) was placed on a gauze (4 mesh,  $8\frac{1}{2} \times 3$  in.), and a flame of burning methane was applied to either edge of the specimen for 60 s and then removed.
- (b) UL 94VO (vertical burning test, not shown in Fig. 4): The specimen ( $127 \times 12.7 \times 10$  mm) was held vertically using a clamp, and a flame of burning methane was

**Table I Irradiation Conditions**

Beam current (mA)	4.0	7.5
Dose rate (kG/s)	23.0	44.0
Traveling velocity (m/min)	0.9	3.0
Dose (kGy)	180	100

Scanning width: 30 cm; electron accelerating voltage: 0.8 MeV.

applied to the lower edge of the specimen for 10 s and then removed. If extinguished, the flame was applied again 10 s after the extinguishment.

- (ii) Tetsu-un 81 (45° inclined burning test approved as a railway safety regulation by the Japanese Government): The specimen was held in 45° inclined by a clamp, and a fuel container filled with 0.5 mL ethyl alcohol was fixed so that the bottom of the container comes 25.4 mm below the lower edge of the specimen. The bottom of the specimen was exposed to a flame until the whole amount of ethyl alcohol was consumed.

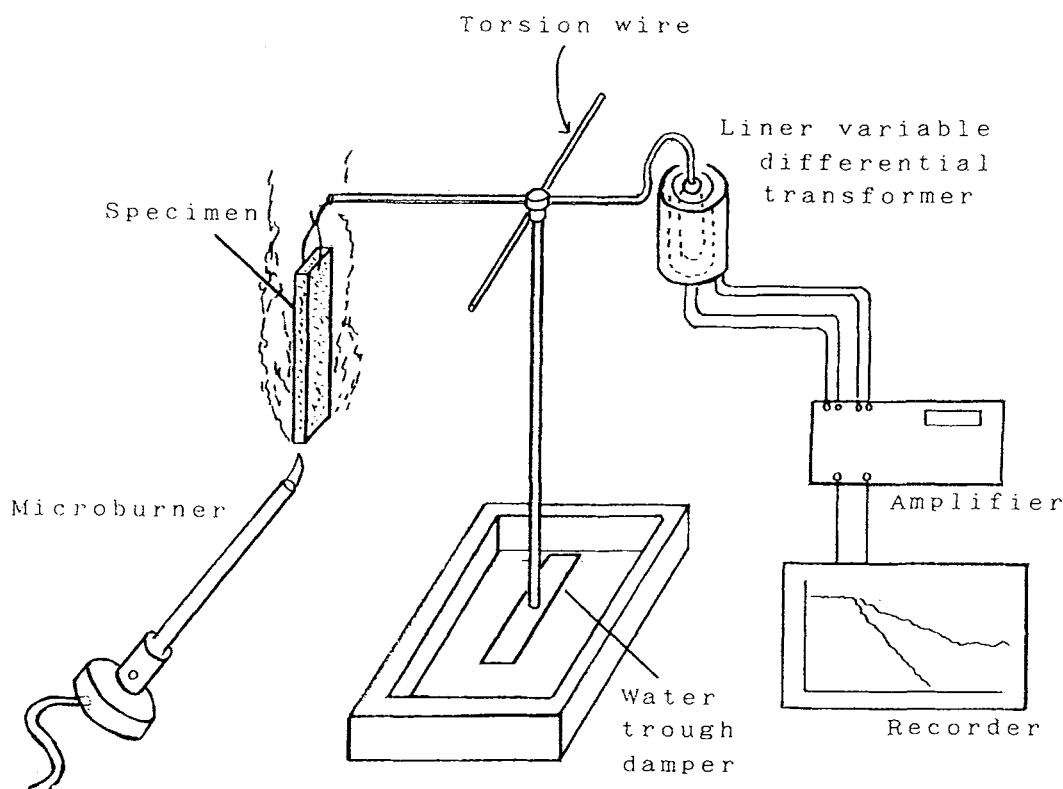
- (iii) FAR 25.853(b): The specimen was (368 × 76 × 10 mm) held vertically using a clamp and a flame of a burner was applied at the lower end of the specimen for 12 s and then the flame was removed.

### Measurement of Least Oxygen Index (LOI)

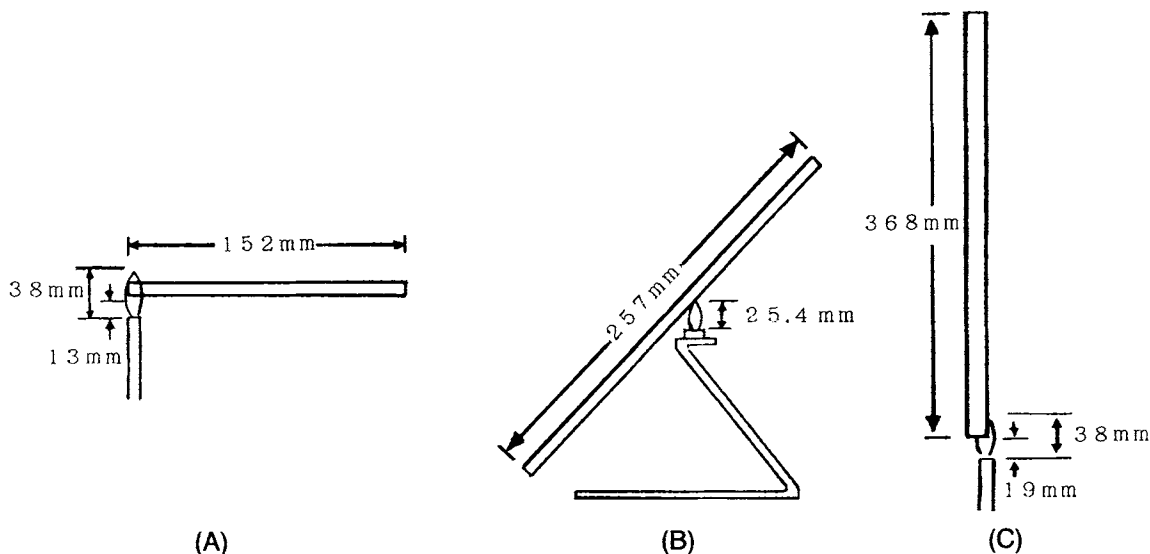
LOI was measured on the grafted foam as minimum oxygen concentration (in percent) in the atmosphere required to continue flaming of the sample as determined by JIS K-7201.

### Detection of Hydrogen Cyanide in Combustion Exhaust

Grafted foam was sent to the Japanese Association for Chemical Analysis for detection of hydrogen cyanide in the combustion exhaust by JIS K0102.<sup>6</sup> The foam, 0.3 g, was heated to 850°C in an electric heating oven under an air stream (300 mL/min) and combustion exhaust was passed through a 0.1N NaOH aqueous solution. The solution was subjected to pyridine-pyrazoron colorimetric analysis.



**Figure 3** Torsion balance for burn-dripping test.



**Figure 4** Typical flammability tests: (A) UL 94, horizontal test; (B) Tetsu-un 81, 45° test; (C) FAR 25.853(b), vertical test.

**Measurement of Elongation and Tensile Strength**

Measurements of elongation and tensile strength were carried out on dumbbell-shaped samples cut out from the original foam and the grafted foam using an Instron tensile tester on the basis of JIS K 6767. The velocity of the cramp was 500 mm/min and atmosphere of the measurements was 20°C and 65 ± 5% RH.

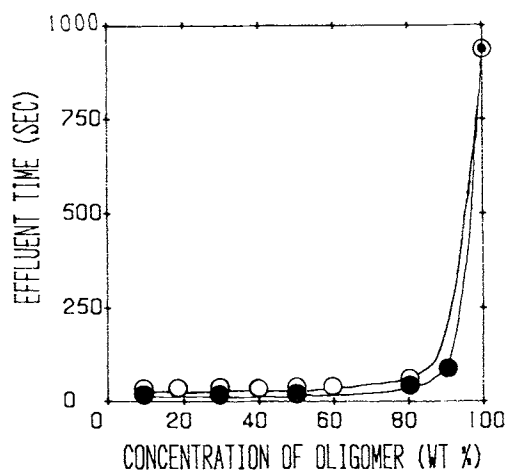
**The Degree of Impregnation**

In Figure 6, the degree of the oligomer pickup was plotted as a function of the VPO concentration of the aqueous solution for different gap widths between the rollers. The degree of impregnation increased with increasing concentration of VPO in the solution depending on the gap width. It is concluded that the amount of VPO pickup can be controlled by adjusting both the concentration of the solution

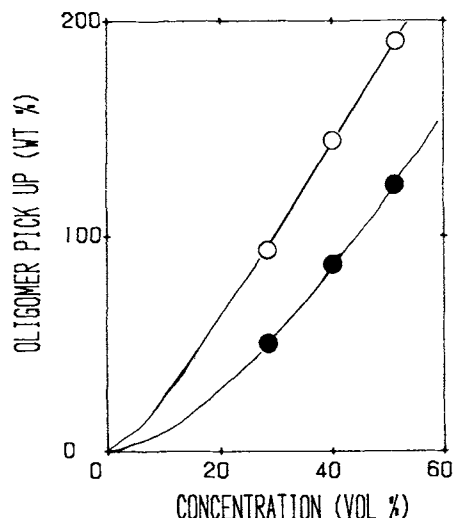
**RESULTS AND DISCUSSION**

**Elution Time of VPO Solution as a Function of Concentration**

Figure 5 shows the elution time of the VPO solution as a function of the concentration of VPO in the solution. The viscosity of the solution decreased markedly by the addition of a small amount of diluent, but the decrease of the viscosity with an increasing amount of diluent was small when the concentration of the solvent exceeded 80% by weight. The change of viscosity as a function of an added amount of methanol was almost the same as that of water. The results indicate that addition of a small amount of diluent can decrease the viscosity of the VPO solution to a practical level for the impregnation.



**Figure 5** Viscosity (Zahn cup method) as a function of concentration of oligomer solution; solvent: (○) water; (●) methanol.



**Figure 6** Degree of impregnation of VPO as a function of concentration of oligomer solution; solvent, water; gap width: (○) 1.0 mm; (●) 0.3 mm.

and the gap width of the rollers. A similar result was obtained for the experiments where methanol was used as the solvent instead of water as shown in Figure 7. The results showing that the increase of VPO pickup in the foam with increasing concentration of VPO solution is small in the lower concentration region, but becomes larger at high concentrations of VPO, indicate that more VPO remains in the foam after the squeezing by the rollers due to higher viscosity; thus, careful adjustment of the VPO concentration is necessary for the nonlinear dependence of VPO pickup on the concentration.

### Conversion of VPO to Polymer

Table II summarizes the results of the extraction experiment. About 97% of the VPO impregnated in the foam remained in the foam by the extraction, indicating that most of VPO was polymerized by 180 kGy irradiation. This was also supported by the infrared spectra shown in Figure 8.

The absorption appeared in spectrum (c) of VPO at  $1406.5\text{ cm}^{-1}$ , which is assigned to  $\delta$  (C—H) of the C=C—H group and which disappeared in spectrum (b) of the grafted foam. It is further noticed that three absorption peaks characteristic of VPO at  $1718.6$ ,  $1319.7$ , and  $1123.7\text{ cm}^{-1}$ , which are not observed in spectrum (a) of original foam, are observed in spectrum (b) of the grafted foam. These

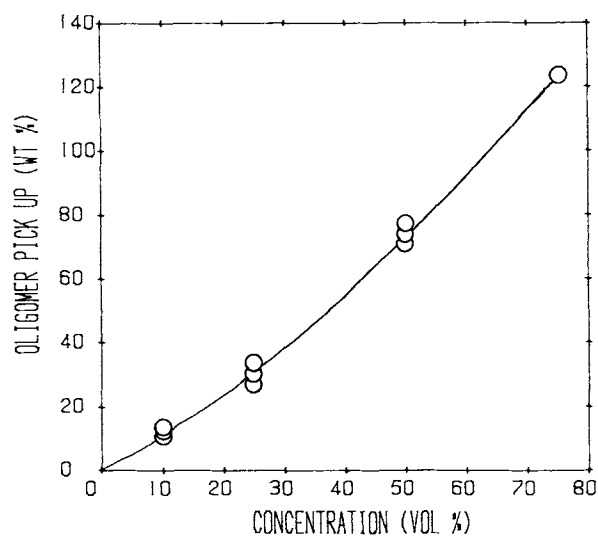
results indicate that VPO was polymerized by the irradiation in the foam.

### Dripping Test

Figure 9 shows examples of the recorder trace of the mass of the two foam pieces, one being grafted polyethylene foam, and the other, polyurethane foam with the flame-retardant treatment, as a function of time after a test flame was attached at the bottom of the sample for 5 s and then removed. Severe dripping accompanied by a sudden decrease of mass was observed for the polyurethane foam, whereas only a little decrease of mass was observed for the grafted polyethylene after the removal of the test flame.

### Flammability Test

Results of the flammability test by UL 94HF-1 are summarized in Table III (a) and (b). It is concluded that all samples grafted to a grafting degree of more than 60% cleared the flame-retardant requirements. The results of the test on polyurethane foam with a flame-retardant treatment and polyethylene foam modified with conventional flame-retardant treatments are also shown in the table for reference. The content of the flame-retardant additive in the polyethylene foam was the highest as possible, to the degree that the presence of the additives may not



**Figure 7** Degree of impregnation of VPO as a function of concentration of oligomer solution; diluent, water; gap width: 0.1 mm.



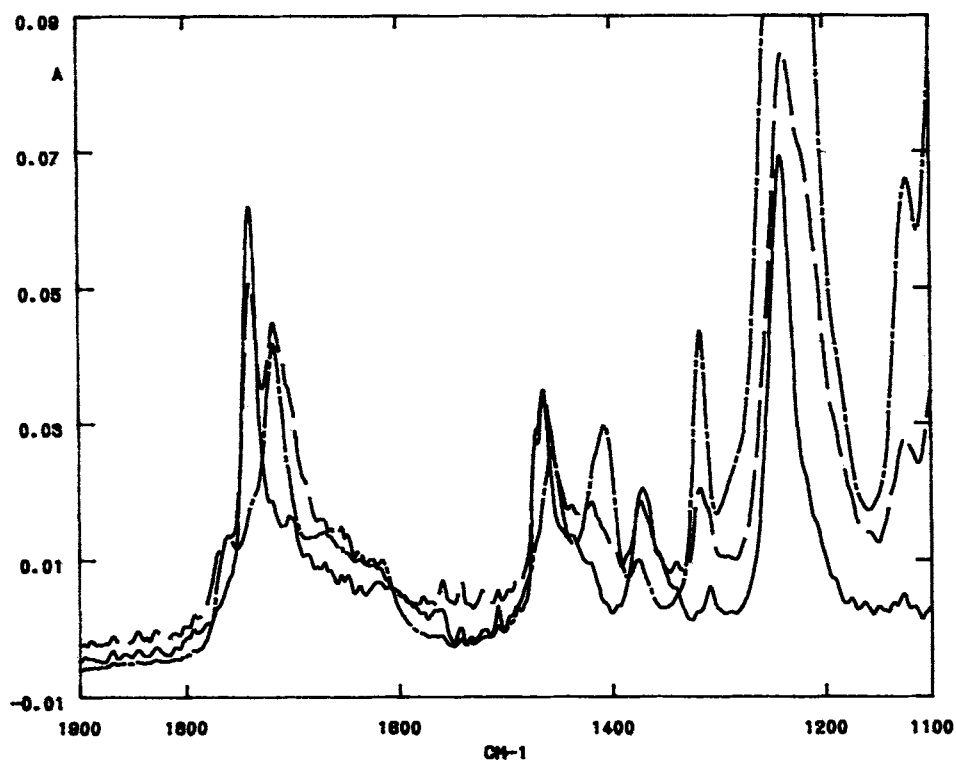
**Table II Insoluble Fraction of Foams**

	Weight of Original Foam (g)	Weight of Foam After Extraction (g)		Weight Loss A (%)
Polyethylene foam	0.2228	0.1968		11.7
	0.2196	0.1949		11.2
Average				11.5

	Before Extraction		After Extraction		Conversion F (%)	Degree of Grafting (%)
	Weight of Foam B (g)	Weight of VPO C (g)	Weight of Foam + VPO D (g)	Residual VPO <sup>a</sup> E (g)		
Polyethylene foam						
Impregnated with VPO	0.2278	0.1335	0.3613	-0.0061	—	
Dose: 0 kGy	0.2145	0.1257	0.3402	-0.0027	—	
Polyethylene foam						
Impregnated with VPO	0.2178	0.1400	0.3292	0.1365	97.5	55.4
Dose: 180 kGy	0.2246	0.1444	0.3378	0.1391	96.3	54.8
Average					96.9	55.1

<sup>a</sup> For the calculation of residual amount of VPO in the foam, weight loss of original foam during the extraction procedure is corrected by the following equations:  $E = D + B \times A/100 - B$ ;  $F = E/C \times 100$ .



**Figure 8** ATR-FTIR spectra of foam and grafted foam: (a) (—) original foam; (b) (---) grafted foam, graft percent: 44%, dose: 180 kGy; (c) (-.-) VPO.

**Table III Flammability Tests of the Flame-retardant Polyethylene (PE) Foam (UL 94 HF-1 and UL 94 V-0)**

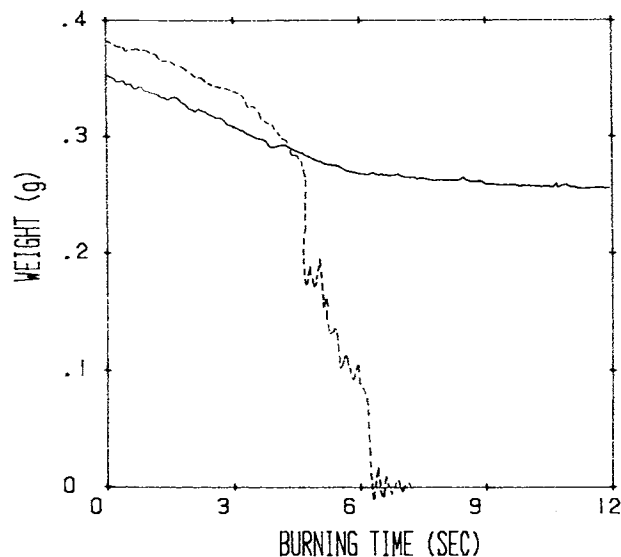
(a) (UL 94 HF-1)						
Degree of Grafting (%)	Flaming Time (s)	Charred Distance (cm)	Smoldering Time (s)	Dripping	Judgment	
94.1	0	29	0	No	Pass	
93.1	0	31	0	No	Pass	
91.0	0	45	0	No	Pass	
89.0	0	37	0	No	Pass	
85.7	0	40	0	No	Pass	
62.5	0	23	0	No	Pass	
Modified polyurethane foam	0	32	0	Yes	Fail	
Closed-cell-type PE foam (L-2500NN)	0	24	0	No	Pass	
Open-cell-type PE foam (LC-300#3)	5	36	0	Yes	Fail	
Requirement	< 2	< 57.2	< 30	No		
(b) (UL 94 V-0)						
Degree of Grafting (%)	Flaming Time (1st) (s)	Flaming Time (2nd) (s)	Smoldering Time (s)	Dripping	Judgment	
97.3	0	0	0	No	Pass	
96.6	0	0	0	No	Pass	
94.4	0	2	0	No	Pass	
94.1	0	5	0	No	Pass	
88.0	0	8	0	No	Pass	
87.5	5	7	0	No	Pass	
Modified polyurethane foam	0	0	0	Yes	Fail	
Closed-cell-type PE foam (L-2500NN)	14	—	—	Yes	Fail	
Open-cell-type PE foam (LC-300#3)	5	5	0	Yes	Fail	
Requirement	< 10	< 10	< 30	No		

\* L-2500, closed-cell-type PE foam containing  $Sb_2O_3$ , Br, and Cl. LC-300#3, open-cell-type PE foam containing  $Sb_2O_3$ , Br, and Cl, both not grafted.

disturb the satisfactory foaming of the material. Comparisons of the data indicate that the flame-retardant polyurethane satisfied most requirements except that severe dripping was observed during burning. The closed-cell-type polyethylene prepared by the conventional method passed the test, whereas the open-cell-type polyethylene foam kneaded with antimony trioxide-, bromine-, and chlorine-containing compounds before the foaming process failed to satisfy the requirements, in that the afterflaming time exceeded 5 s and dripping was observed.

Results of the flammability test by Tetsu-un 81 on the grafted foam are summarized in Table IV along with those on the other three flame-retardant foams, where it is evident that the grafted foam passed all requirements of the test, whereas the other flame-retardant foams modified by conventional methods failed the test.

In Table V, similar comparisons were made among the grafted polyethylene foam of the open-cell type, polyurethane foam with the flame-retardant treatment and the closed-cell-type polyethylene



**Figure 9** Weight of specimen as a function of time during burning and after the test flame was removed: (—) flame-retardant polyethylene foam; (---) flame-retardant polyurethane foam.

foam modified with conventional flame-retardant treatments on the results of flammability test by FAR 25.853(b). It is concluded that all samples grafted to a grafting degree of more than 88% satisfied the flame-retardant requirements with better flame-retardant data than did those obtained for conventional flame-retardant polyurethane. It is also noted that none of the conventional techniques can modify polyethylene foam of the open-cell type to give a flame-retardant property to such a degree to pass this test.

### Least Oxygen Index

The least oxygen index (LOI) of the grafted polyethylene foam was plotted as a function of grafting degree in Figure 10, where it is shown that LOI increased with increasing grafting degree, and with a degree of grafting of 100%, it reached 28, which is difficult to obtain by the conventional kneading method in which successful foaming is impossible when a sufficient amount of additives are kneaded into polyethylene to obtain such a high LOI value.

**Table IV** Flammability Test of the Flame-retardant Polyethylene (PE) Foam (Tetsu-un 81<sup>a</sup>) 45° Test

Degree of Grafting (%)	During Burning				After Burning				Judgment
	Glowing (s)	Flaming (s)	Smoke	Burning	Glowing	Flame	Char (mm)	Deform (mm)	
94.1	No	No	A little		No	No	97	97	Pass
91.0	No	No	A little		No	No	106	106	Pass
89.9	No	No	A little		No	No	125	125	Pass
88.0	No	No	A little		No	No	110	110	Pass
Modified polyurethane foam	Yes	Yes	A little	Weak	No	No	65	100	Fail
Closed-cell-type PE foam (L-2500NN) <sup>b</sup>	Yes	Yes	A little	Weak	No	No	70	—	Fail
Open-cell-type PE foam (LC-300#3) <sup>b</sup>	Yes	Yes	A little	Weak	No	No	60	75	Fail
Requirement <sup>c</sup> (a)	No	No	A little	No	No	No	* <sup>d</sup>	< 150 mm	
(b)	asap <sup>e</sup>	asap	A little	Weak	No	No	< 30 mm	< 150 mm	

<sup>a</sup> There are three categories in flame-retardant standards: (i) extremely flame retardant, (ii) highly flame retardant, and (iii) flame retardant. Materials that satisfy one of the requirements (a) and (b) are classified as highly flame retardant.

<sup>b</sup> L-2500, closed-cell-type PE foam containing Sb<sub>2</sub>O<sub>3</sub>, Br, and Cl. LC-300#3, open-cell-type PE foam containing Sb<sub>2</sub>O<sub>3</sub>, Br, and Cl.

<sup>c</sup> In case of no flaming on a test piece, requirement (a) is applied; when small flaming is observed on testing, requirement (b) should be cleared to pass the test.

<sup>d</sup> Deformation zone should not reach to the fringe of a test piece.

<sup>e</sup> As small as possible.

**Table V Flammability Test of the Flame-retardant Polyethylene (PE) Foam [FAR 25.853(b)], Vertical Flaming Test**

Degree of Grafting (%)	Extinguishing Time (s)	Burned Length (mm)	Dripping	Dripping Extinguishing Time (s)	Judgment
94.1	0	6	No	—	Pass
91.0	0	8	No	—	Pass
89.9	0	5	No	—	Pass
88.0	0	6	No	—	Pass
Modified polyurethane foam	0	55	Yes	< 1	Pass
Closed-cell-type PE foam (L-2500NN) <sup>a</sup>	1.8	106	Yes	0	Pass
Open-cell-type PE foam (LC-300#3) <sup>a</sup>	109	356	Yes	1	Fail
Requirement	< 15	< 203 mm	asap <sup>b</sup>	< 5	

<sup>a</sup> L-2500, closed-cell-type PE foam containing Sb<sub>2</sub>O<sub>3</sub>, Br, and Cl. LC-300#3, open-cell-type PE foam containing Sb<sub>2</sub>O<sub>3</sub>, Br, and Cl.

<sup>b</sup> As small as possible.

### Hydrogen Cyanide in the Combustion Exhaust

The pyridine-pyrazorone coloration test indicated that the presence of hydrogen cyanide is below the detection limit (10 μg).

### Mechanical Properties of the Grafted Polyethylene Foam

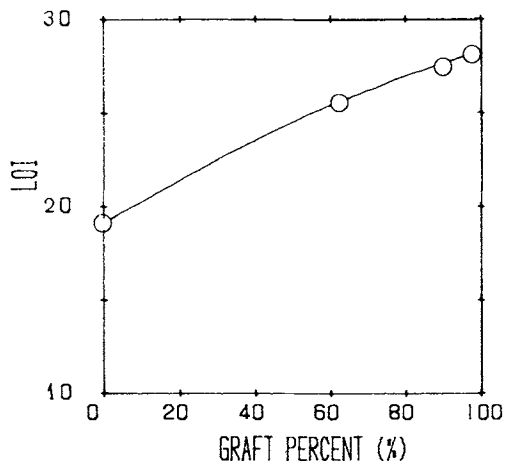
In Table VI, mechanical properties of the grafted polyethylene foam (grafting degree: 100%) are compared to those of the ungrafted original foam. Elongation was decreased by 20%, but tensile

strength and 50% compression strength increased by 10% and 30%, respectively.

### CONCLUSIONS

Highly flame-retardant polyethylene foam of an open-cell type was obtained by grafting flame-retardant oligomer onto the surface of cells, and it satisfied the flame-retardant requirements of a major standard testing material of flammability.

The grafting method has several advantages over conventional kneading methods: (i) a high-speed process is possible, since an accelerator of a very high dose rate recently developed can be used for the process. (ii) Since the oligomer impregnated in the foam can be polymerized completely, control of the amount of oligomer is simple and no additional process is necessary to remove the unreacted oligomer. (iii) Grafting of VPO at high density on the

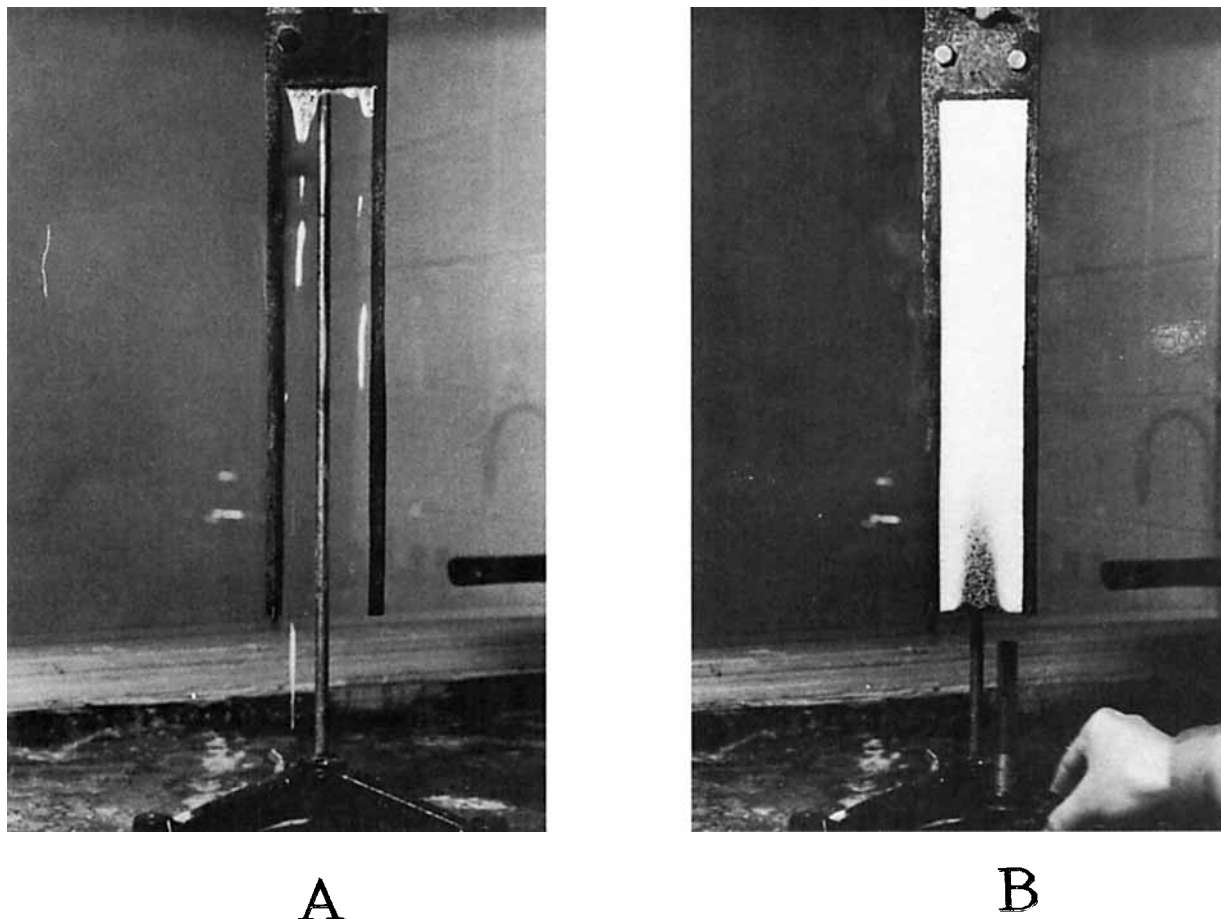


**Figure 10** LOI as a function of graft percent.

**Table VI Mechanical Properties of Flame-retardant Polyethylene Foam**

	Original Foam	Grafted Foam <sup>a</sup>
Tensile strength (kg/cm <sup>2</sup> )	0.96	1.05
Elongation at break (%)	220	180
50% compression strength (kg/cm <sup>2</sup> )	0.041	0.066

<sup>a</sup> Grafting degree: 100%.



**Figure 11** Photograph showing flammability test by FAR 25.853(b): (A) original foam; (B) grafted foam (graft percent: 100%).

inner surface of the foam cell effectively gives a foam of a higher flame-retardant property than does the conventional method. In the conventional kneading method, such a high flame retardant cannot be obtained because addition of the flame retardant to the polyethylene foam to give enough flame-retardant property sacrifices foaming quality. (iv) Only the radiation method can be applied to modify the polyethylene of low softening temperature, because the reaction was successfully carried out at room temperature.

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