# Limiting Oxygen Indices of Silicone Block Polymers

R. P. KAMBOUR, H. J. KLOPFER, and S. A. SMITH, General Electric Research and Development Center, Schnectady, New York

# Synopsis

The limiting oxygen index (LOI) has been determined for each of a large number of silicone-containing resins, principally dimethylsiloxane (DMS) block polymers varying widely in DMS content and hard block type. A synergistic enhancement in LOI of varying strength is seen in several families of resins. For the major families explored, the synergism varies with hard block type in roughly the following order: bisphenol-A carbonate and bisphenol fluorenone carbonate > styrene and 2,6diphenyl-1,4-phenylene oxide > bisphenol chloral polycarbonate > methyl methacrylate and phenolphthalein carbonate, the enhancement being nil in the last case. Strength of the synergism is discussed in terms of DMS degree of dispersion, char-forming tendencies, melt viscosity, and other factors.

#### INTRODUCTION

A synergism in the Fennimore–Martin limiting oxygen index has been found in the bisphenol fluorenone carbonate–silicone block polymer family.<sup>1</sup> Auxiliary investigations suggested that the enhancement in LOI brought about by silicone incorporation in bisphenol fluorenone carbonate resins resulted from the production of continuous layers of silica that retarded oxidation of the char.<sup>2</sup>

In the work reported here, the silicone dependence of LOI has been surveyed in each of several families of silicone copolymers in order to assess the generality of the LOI synergism. An added motive in this survey has been to help develop an understanding of the role of the nonsilicone portion of the resin in the synergism. In addition to block polymers, a few graft polymers and polymer blends have been tested to aid in assessing the importance of component dispersity.

Auxiliary studies reported in a subsequent article<sup>15</sup> have been focused principally on the BPA polycarbonate—silicone block polymer family in an attempt to explore the mechanism of LOI enhancement in depth. These latter studies reinforce the original view that the mechanism of enhancement involves char formation, though the details of the mechanism must differ substantially from those originally proposed.

Each family of resins, defined by the "hard" polymeric species involved, together with types of subfamilies tested (blocks, grafts, blends), is listed below:

(1) Bisphenol fluorenone polycarbonate: block polymers; blends of homopolymers with blocks, with silicone gum, and with silica gel.

(2) Bisphenol A polycarbonate: block polymers.

- (3) Bisphenol chloral polycarbonate: block polymers.
- (4) Phenolphthalein polycarbonate: block polymers.
- (5) Polystyrene: block polymers.
- (6) Poly(methyl methacrylate): block and graft polymers.

(7) Poly(2,6-diphenyl-1,4-phenylene oxide): block polymers; blends of homopolymer with block polymers and with silicone gum.

Journal of Applied Polymer Science, Vol. 26, 847–859 (1981) © 1981 John Wiley & Sons, Inc. CCC 0021-8995/81/030847-13\$01.30 (8) Miscellaneous: one or two block polymers of the following: polysilphenylenesiloxane, copolyether from dichlorodiphenyl sulfone and bisphenol A, 50/50 copolycarbonate from bisphenol A and tetrabromobisphenol-A.

# EXPERIMENTAL

#### Materials and Processes: General

All of the materials except for the sulfone resins were made within the General Electric Co. Synthesis of the bisphenol fluorenone (BPF) resins and of many of the bisphenol-A resins is described elsewhere.<sup>3</sup> Molecular architecture of the other resins varied considerably<sup>3</sup>: binary, ternary, and multiblock polymers were tested. Silicone block average degrees of polymerization,  $\overline{DP}_n$ , varied from two to several hundred, though not all in one family. The more important architectural details are indicated below in the context of results for each family.

Most specimens were compression molded into 1/8-in.-thick bars, and ASTM Standard Limiting Oxygen Index bars were cut from these. A few test specimens in the BPF carbonate family were cut from injection moldings. All specimens were tested in accordance with ASTM Test No. D2863-70.

#### **Bisphenol Fluorenone (BPF) Polycarbonate Family**

Bisphenol fluorenone (BPF) polycarbonate and 15 multisequence dimethylsiloxane (DMS) block polymers in this family were tested. DMS content ranged from 7 to 62 wt % and DMS  $\overline{\text{DP}}_n$  values ranged from 10 to 30. Moreover, three blends of BPF PC with the 62 wt % DMS block polymer were formulated to have 10, 20, and 30% DMS; these were codissolved in chloroform, precipitated, and dried before molding. In addition, a blend of BPF PC with SE-30 PDMS gum and was prepared in a similar fashion. Finally, a blend of BPF PC with Cabosil silica gel was prepared by solution mixing, film casting, and comminution of the film to a powder.

In Figure 1, all LOIs are plotted versus elemental silicon content in order to include the silica gel blend. A maximum in LOI is reached at roughly 8% silicon. With the exception of one block polymer, maximum LOIs were 46–51. This level was achieved over the range 4–12% elemental silicon for all materials containing dimethylsiloxane regardless of the details of its incorporation (i.e., DMS block length; pure block polymer vs. blend of homopolymer with block polymer or silicone gum). Beyond about 12% elemental silicon, a roughly linear drop in LOI with increasing silicon occurs.

One block polymer of DMS  $\overline{DP}_n = 7$  exhibited a significantly higher LOI (57-60) in repeated testing than the LOIs of other resins of similar silicone content and higher DMS block lengths. No resins of DMS block  $DP_n > 7$  were made in this silicone content range, so that conclusions about block length effects are hazardous.

Finally, the mixture containing  $SiO_2$  exhibited a LOI equal to or lower than that expected of the homopolymer.

The character of the burning and of the residue produced changed substantially with silicone content. Rather loud decrepitation occurred with all resins. ٥

50

LOI

30



20 0 10 20 30 37.8 WT % SILICON

Fig. 1. BPF carbonate family. Dependence of LOI on silicone content: [(O) block polymer; (+) BPFPC + block polymer blend;  $(\times)$  BPFPC + SiO<sub>2</sub> (upper DMS scale for all materials except SiO<sub>2</sub> mixture)].

The residue changed from a fine black friable char with the homopolymer to a more voluminous, very strong, largely black char at moderate amounts of silicone to a gray coarse weak residue at high silicone contents. By contrast, the  $SiO_{2}$ -filled homopolymer burned quietly and slowly with little or no swelling of the residue. In fact, burning of a thin surface layer only seemed to occur. The burnt layer peeled off leaving a white-surfaced core.

# **Bisphenol A Polycarbonate Family**

Twenty-four multisequence block polymers of 6-81 wt % DMS and DMS  $\overline{DP}_n$ s of 2-40 in the BPA carbonate family were tested. The LOIs obtained are plotted versus silicone content in Figure 2.

The LOI dependence on silicone in this family was very much like that in the BPF family in every respect once allowance was made for the fact that BPA polycarbonate's LOI is 25 versus 39 for BPF polycarbonate. The LOI increased 13 points again, reaching a broad maximum in this family at LOI = 38-40 and wt % DMS = 15-30. However, two resins of DMS  $\overline{DP}_n = 11$  and 5 and wt % DMS = 12 and 18, respectively, exhibiting much higher LOIs than other resins of similar silicone contents and block lengths. The value of 50 for the latter resin was reproducible. It is the largest LOI relative to that of the corresponding "hard" homopolymer of any resin tested in this work.

No clear dependence of LOI on block length at fixed silicone content was evident in these results. Nor could any marked effects of polymer molecular weight be seen. For example, two resins of DMS  $\overline{\text{DP}}_n = 20$  and wt % DMS = 25 had LOIs of 35–38 even though their intrinsic viscosities were 0.6 and 1.2 dl/g, re-



Fig. 2. BPA carbonate family. Dependence of LOI on silicone (DMS) content. Design silicone block DP:  $(\Phi)$  2;  $(\Theta)$  5;  $(\Theta)$  10 and 11;  $(\Theta)$  20, (O) 40. Line is upper bound.

spectively. (The viscosity of latter is higher only because the total number of blocks per macromolecule is greater.) None of the block polymers tested, however, was low enough in molecular weight to exhibit "runny" melt behavior.

In contrast to BPF polycarbonate, BPA polycarbonate tends to drip somewhat on burning so that the char that forms on the latter homopolymer does not form a stable protective cap. The burning characteristics and the chars produced from all the BPAC/DMS block polymers tested, however, were much like those in the BPF carbonate family: a stable char that increased in amount initially with increasing silicone content, a coarsening and weakening of the residue beyond the LOI maximum, and a residue progressively lighter in color. Other characterizations of the chars produced in this system are described in a subsequent article.<sup>15</sup>

# **Bisphenol Chloral (BPC) Polycarbonate Family**

Bisphenol chloral polycarbonate (I)(BPC),



is an amorphous tough thermoplastic with a  $T_g$  of 180°C.<sup>4</sup> Its LOI is 55. Hydrogen chloride is eliminated and large amounts of char are formed on vigorous heating; both phenomena presumably underlie the unusually high LOI. Three multisequence silicone block polymers have been made (DMS block  $\overline{DP}_n = 20$ )



Fig. 3. LOI dependence on silicone content in bisphenol chloral (O) and phenolphthalein  $(\Delta)$  polycarbonate families.

and tested here.<sup>5</sup> A small but real synergism in LOI exists at low silicone contents (Fig. 3). All block polymers charred during test.

Except for thermogravimetric results (ca. 50% char at 700°C in nitrogen for the homopolymer and 10% DMS block polymer), no other support information has been accumulated for this family.

# Phenolphthalein Polycarbonate (PhPC) Family

Phenolphthalein polycarbonate and three of its multisequence silicone block polymers (DMS  $\overline{DP}_n = 20$ ) were synthesized and tested here.<sup>5</sup> All resins char heavily on burning. No synergism in LOI is produced, however, by the incorporation of the silicone (Fig. 3). The homopolymer and the two block polymers of lowest DMS content produce ca. 50% char on TGA pyrolysis to 700°C. No other support experiments with these resins were carried out.

## **Polystyrene Family**

Ten styrene/silicone block polymers varying in number of blocks per chain from 2 to 12, DMS  $\overline{DP}_n$  from 25 to 900 and DMS wt % from 11 to 82, were tested. In some cases polymer molecular weights were low enough that the resins were like paraffin wax in consistency at room temperature. Other materials were strong solids.

All LOIs are plotted versus silicone content in Figure 4. These fall within an envelope, the lower bound of which is the interpolation line between the LOIs of the two homopolymers. The upper bound reaches a broad LOI maximum of 28 centered at 60% silicone roughly.

Homopolystyrene burns with no char formation. A "cap" of melted resin exists on top of the LOI specimen and eventually runs down its side [Fig. 5(a)].



Fig. 4. Styrene family. Dependence of LOI on DMS content. Upper and lower bounds set by resin viscosities.

Vapor bubbles are produced in the cap, the vapor presumable consisting of styrene monomer and low molecular weight oligomers.<sup>6</sup> (Polystyrene's ceiling temperature for depolymerization is well known to be ca. 300°C.) The volume of the "cap" under the flame increases very slowly, and eventually molten resin runs slowly down the side of the specimen. A stream of fine black smoke is emitted from the top of the flame.

A dramatic change in appearance is brought about by the presence of silicone in the test specimen even in small amounts. A solid residue forms on the top [(Fig. 5(b)]. At low silicone contents, the residue is black with minor areas of gray but becomes much whiter with increasing silicone content. The volume increase accompanying residue formation is much less than with either bisphenol carbonate-silicone family.

The two bounds of the envelope in Figure 4 appear to result from differences in rheological behavior of the resin. Specimens of some resins became very fluid in regions close to the flame and the molten material tended to run down the sides of these specimens; the LOIs resulting from these tests fell on the lower bound. Specimens of the other resins showed no tendency to flow in this manner; LOIs for these materials fell on the upper bound. (This difference in melt flow behavior appears primarily due to resin molecular weight: intrinsic viscosities averaged 0.50 dl/g for upper bound resins but only 0.23 for lower bound materials.)

Each material on the upper bound left a residue in the form of a roughly cylindrical shell, the wall thickness of which was estimated by eye to be 0.02 in. approximately. The lower end of the shell covered the molten resin cap; the upper end was completely empty. The resins on the lower bound left residues that tended to be fragmented.

Thus, if melt viscosity is high enough, the incorporation of silicone in sufficient amount can produce a stable char that results in a substantial increase in the LOI of styrene resins. Fractionally, the increment in LOI achieved is as great as with the polycarbonate families. The silicone content required, however, is about twice as great.



Fig. 5. (a) LOI specimen of polystyrene; magnification,  $3.25 \times$ . (b) LOI specimen of styrene/ silicone block polymer of 13% DMS; magnification,  $2.9 \times$ .

Under inert atmospheres at temperatures of  $500^{\circ}$ C and above, styrene monomer and its oligomers are known to break down to small hydrocarbon fragments (C<sub>1</sub>-C<sub>6</sub>).<sup>6</sup> Presumably the shell of silica residue acts as a red-hot screen, beneath which the atmosphere is largely inert and through which the styrene vapors must pass. During passage the temperature of the vapor must be raised to the point that these small hydrocarbon fragments are produced and some of these are carbonized.

#### **Poly(methyl Methacrylate) Family**

The high-molecular-weight MMA-silicone resins tested included two graft resins and seven binary block polymers of wt % DMS from 5 to 58. All LOIs from this family are plotted versus silicone content in Figure 6. Very little sign of LOI enhancement is seen. PMMA itself burns with a smokeless flame and no char formation. A molten cap of resin exists on top of the LOI specimen. Bubbles of vapor, believed to be pure MMA monomer originating from thermal depolymerization,<sup>6</sup> form in the cap and feed the flame.

With the block and graft polymers the flame is also smokeless. However, a gray-to-white solid residue forms slowly on top of the molten cap. The residue



Fig. 5. (Continued from previous page.)

takes the shape of a hollow cylindrical shell with a thin convoluted wall (Fig. 7). The shell is white to gray outside but usually darker inside. Its diameter and wall thickness increase strongly with DMS content. The gross form of this shell is much like that on the styrene/DMS resins.

Of all the homopolymers tested in this study, PMMA shows the least tendency



Fig. 6. Methyl methacrylate family. Dependence of LOI on DMS content:  $(\Theta)$  block polymer:  $(\Theta)$  graft polymer).



Fig. 7. LOI specimen of methyl methacrylate/silicone block polymer (20% DMS); magnification,  $3.1 \times$ .

to form char or smoke. Presumably this characteristic is related to the thermal depolymerization which occurs at low temperature and which yields only monomer, the monomer breaking down at higher temperature to smaller fragments.<sup>6</sup> In any case, the evident lack of tendency to carbonize on burning is thought to be responsible for the lack of LOI enhancement by silicone in the block and graft polymers. However, the fact that the inside surface of the residue shell is often black indicates that even MMA monomer can deposit a little carbon on a very hot substrate in an oxygen deficient atmosphere.

# Poly(2,6-diphenyl-1,4-phenylene Oxide) Family

High-molecular-weight poly(2,6-diphenyl-1,4-phenylene oxide) (P<sub>3</sub>O) is a resin with a  $T_g$  of 220°C and a  $T_m$  of 490°C.<sup>7</sup> It can be cast from chloroform solution into films that are clear, amorphous, and about as brittle as polystyrene. These films can be compression molded at temperatures up to 250°C into amorphous bars that are nearly clear. At 260°C crystallization begins; once crystallized, the polymer is intractable since degradation commences just above  $T_m$ .<sup>8</sup>

Multisequence  $P_3O/DMS$  block polymers have previously been made with a wide range of compositions and  $P_3O$  block lengths.<sup>9,10</sup> The making of these materials involved the use of the difunctional oligomers



where  $\overline{n}$  is 2 or greater. (Note, however, that these strictly are phenylene oxide oligomers only for  $\overline{n} > 2$ .) Microdomain formation occurs in all these materials with hard block domain  $T_g$ 's varying with block length, as expected.<sup>11</sup> Crystallinity is not found in resins of  $\overline{n} \leq 20$ .

The homopolymer and each of five rubbery block polymers in this family were available for testing. The latter resins had hard block  $\overline{n}$ s from 0 to 40 and wt % DMS of 66 to 83.

Three blends of  $P_3O$  homopolymer with  $P_3O/DMS$  block polymer of 82% DMS and  $\overline{n} = 0$  were made by coprecipitation of chloroform solutions in methanol followed by vacuum drying. Finally, a blend of  $P_3O$  with SE-30 silicone gum was made in similar fashion. These blends were made in light of the behavior of the BPF polycarbonate/BPFC-DMS block polymer blends and in the hope that the blends would serve as substitutes for block polymers of high  $P_3O$  content as far as LOI testing was concerned.

LOIs for this family are displayed versus silicone content in Figure 8. The block polymers showed a substantial synergism similar to that seen in the BPF carbonate family. The four blends, however, showed no LOI enhancement at all.

 $P_3O$  homopolymer burns with substantial smoke and char formation. The char is slightly larger than the specimen in cross section and consists of a strong foamy core with a thin sooty surface covering. Thus, the homopolymer behaves superficially rather like BPF polycarbonate. The LOI behavior of the block polymers and the appearance of their chars are consistent with this fact. The behavior of the blends, therefore, seems inconsistent. Lack of block polymer materials has prevented a resolution to this inconsistency.

#### Miscellaneous

A silicone block polymer from each of three other families was available in sufficient quantity for compression molding and LOI testing. These materials and the corresponding polymers with no silicone together with test results are listed in Table I. Each "family" is discussed separately below.



Fig. 8. Diphenylphenylene oxide family. Dependence of LOI on silicone content:  $(\mathbf{0}, \mathbf{0}, \mathbf{\Theta})$  block polymer of  $\overline{DP}_n$  of DMS block equal to 2, 22, and 40, respectively; ( $\emptyset$ ) blend of P<sub>3</sub>O homopolymer with P<sub>3</sub>O/DMS block polymer; ( $\emptyset$ ) blend of P<sub>3</sub>O and silicone gum.

# Polysulfone Family

Polysulfone itself burns with substantial smoke and black char production. The char is foamy and about as strong and voluminous as the char from BPF polycarbonate. The 58% silicone block polymer produced char that was visually much like that from the polycarbonate-silicone block polymers of the same DMS content: a white flaky exterior and a dark, more continuous foamy interior. A 5% DMS block polymer was previously reported to have an LOI equal to that of polysulfone itself.<sup>12</sup> The 58% DMS resin tested here yielded a LOI, at most, 4 units higher than the interpolation value (i.e., the value predicted from the LOI versus % DMS interpolation line between the homopolymers). By contrast, both the BPF and BPA polycarbonate families gave 10–12 units LOI enhancement over their interpolation lines at this DMS concentration. The source of this difference in behavior could not be sought owing to lack of other related materials.

# Poly(tetramethylsilphenylenesiloxane) (PSPS) Family

Small amounts of PSPS and one rubbery multisequence block polymer containing 59% DMS of  $\overline{\text{DP}}_n = 40$  were available. The homopolymer exhibits a  $T_m$  of 150°C and a  $T_g$  of negative 25°C.<sup>13</sup> The block polymer tested exhibits a hard domain  $T_m$  of 100°C. Both homopolymer and block polymer are opaque.

The burning of PSPS yielded a substantial amount of a thick, coarse, flaky, weak residue. Residue color was variegated—white in some places, gray in others, still black in others. A thick zone of clear melt existed below the burn front on the LOI specimen. The melt zone tended to flow and the "char" plus underlying melt tend to tip over, sag, and collapse on the lower, still solid section of bar. This collapse tended to fragment the residue and also to melt and ignite the lower bar section. The smoke also varied in character, being black, coarse, and whispy most of the time but white periodically. The block polymer burned in similar fashion in every respect except that the residue was somewhat lighter in color.

The LOI of PSPS is a very respectable 43 in spite of the unstable melt zone. The LOI of the block polymer is only four units higher than the value of 30 obtained from interpolation between the two homopolymers.

Several factors may underlie the lack of LOI enhancement in this family. The first, by analogy with behavior seen in part of the styrene family, is low melt

TABLE 1 Miscellaneous Polymers Tested			
Polymer	Found wt. % DMS	$\overline{\mathrm{DP}}_{\mathrm{n}}$	LOI
Polysulfone of BPA and dichlorodiphenyl sulfone	0		30.7 ± 1.5
Sulfone/silicone block polymer		<u>a</u>	29.5
Polytetramethylsilphenylenesiloxane (PSPS)	0		42.4
SPS/silicone block polymer	59 <sup>b</sup>	40	34.5
BPA/tetrabromo-BPA carbonate block dimethylsiloxane <sup>c</sup>	43	10	53

<sup>a</sup> Not known to us.

<sup>b</sup> Exclusive of DMS in SPS.

<sup>c</sup> Copolycarbonate block contains 26 wt % bromide.

viscosity. The second is the elemental silicon content—27 wt % in PSPS and 33 wt % in the block polymer. The Si levels are much higher than that at the LOI maximum of either polycarbonate family (7–8%). In fact, the 27 wt % silicon of PSPS is equivalent to 71% DMS in any of the silicone block polymers in which the "hard" block does not contain silicon. In none of these other systems does the LOI at 71% DMS exceed 34, a level almost ten units below the LOI of PSPS. A third factor is block chemical structure and its role in pyrolysis, a subject dealt with at greater length in the subsequent paper.

#### BPA/Tetrabromo-BPA Copolycarbonate Family

Mixtures of tetrabromo-BPA with BPA form random copolycarbonates having LOIs that rise linearly with bromine concentration.<sup>14</sup> Thus, at 17 wt % bromine the LOI is 44, an increase of 19 units over that of BPA polycarbonate.

A silicone-copolycarbonate multisequence block polymer of 43% DMS of  $\overline{DP}_n$ = 10 and 15 wt % bromine was available. The copolycarbonate block contains 26 wt % bromine (28 mole % tetrabromo-BPA carbonate). The LOI for this material was 53. The material burns much like a BPAC/DMS resin of similar DMS content, except that the char is darker and much more voluminous.

There are two ways to compare the relative effects of bromine on the LOIs of polycarbonate versus BPAC/DMS block polymer. In the first comparison, the silicone content is fixed; in the second, the mol fraction of polycarbonate is fixed. First, a BPAC/DMS resin of the same silicone content (43%) is expected to have a LOI of about 35 (Fig. 2). Thus, the bromine has raised the LOI by 18 units, which is 1.18 units per % bromine in the whole polymer or 0.68 units per % bromine in the polycarbonate block. Second, replacement of the 15% bromine with the corresponding mole % of hydrogen results in a BPAC/DMS block polymer of 51% DMS. Such a block polymer is expected to have a LOI of  $34 \pm 2$  (Fig. 2). Conversely, the substitution of bromine for hydrogen in this block polymer has raised the LOI 19 units or 1.28 units for each part of bromine in the whole polymer.

In any case, the increment in LOI per part of bromine is, if anything, slightly greater in the block polymer than in the homopolymer. This result suggests that the LOI enhancement mechanisms associated with silicone and with bromine are separate but additive.

# DISCUSSION

It is clear from the above results that LOI synergistic effects discovered initially in the BPF carbonate family are neither exclusive to that family nor universal to all silicone copolymers. Just what characteristics are required in the organic portion of the polymer for a synergism to exist is not certain.

Tentatively, it appears that the existence of a synergism is related primarily to processes in the solid rather than in surrounding flame. Specifically, conversion of organic polymer to mechanically stable char seems an important requirement: though polystyrene itself does not char, the presence of copious soot in its flame suggests the resin's proclivity to do so. On the other hand, this requirement is not sufficient by itself; otherwise the phenolphthalein polycarbonate family should show a substantial synergism. Except for the  $P_3O$  family, the synergism seems rather insensitive to the degree of dispersion of the silicone in the organic portion of the resin. Apparently, dispersity on the scale of a fine mixture (e.g., microns) is often sufficient. And the puzzling results in the so-called  $P_3O$  family perhaps arise in some way because of differences in backbone chemical structure therein: specifically, the dimeric  $P_3O$  unit is, in fact, a phenol, not an aromatic ether. The 20-unit block contains a biphenolic unit at each end (i.e., 10% of the chain linkages are of this type). The  $P_3O$  high polymer, of course, contains a negligible amount of the biphenolic linkages.

The LOI synergism in the BPA carbonate-silicone family is explored in depth in a subsequent report.<sup>15</sup>

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