Flame Retardants

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The four processes involved in the flammability of materials are described and related to the various flame retardance mechanisms that may operate. Following this the four practical approaches used in improving flame retardance of materials are described. Each approach is illustrated with a number of typical examples of flame retardants or synthetic procedures used. This overview of flammability, flame retardance, and flame retardants used is followed by a more detailed examination of most of the plastics manufactured in the United States during 1973, their consumption patterns, and the primary types of flame retardants used in the flame retardance of the most used plastics. The main types of flame retardants are illustrated with a number of typical commercial examples. Statistical data on flame retardant market size, flame retardant growth in plastics, and price ranges of common flame retardants are presented.

Flammability Process and Flame Retardance

Fundamentally, four processes are involved in polymer flammability (Fig. 1): (1) preheating, (2) decomposition, (3) ignition, and (4) combustion and propagation.

Preheating involves heating of the material by means of an external source which raises the temperature of the material at a rate dependent upon the thermal intensity of the ignition source, thermal conductivity of the material, specific heat of the material, and latent heat of fusion and vaporization of the material.

When sufficiently heated, the material begins to degrade, i.e., it loses its original properties as the weakest bonds begin to break. Gaseous combustion products may be formed with the rate dependent upon such factors as intensity of external heat, temperature required for decomposition, and rate of decomposition.

Flammable gases increase until a concentration is reached that allows sustained oxidation in the presence of the ignition source. The ignition characteristics of the gas and the availability of oxygen are two important variables in any ignition process. After ignition and removal of the ignition source, combustion becomes self-propagating if sufficient heat is generated and is radiated back to the material to continue the decomposition process. The combustion process is governed by such variables as rate of heat generation, rate of heat transfer to the surface, surface area, and rates of decomposition. Flame retardancy, therefore, can be improved (achieved) by retarding (eliminating) any one of these processes. Figure 2 illustrates how flame retardancy may be improved in the heating stage (1).

Flame retardancy is improved by flame retardants that cause the formation of a surface film of low thermal conductivity and/or high reflectivity that reduces the rate of heating (A). It is also improved by flame retardants that might serve as a heat sink by being preferentially decomposed at a low temperature (B). And finally, it is improved by flame retardant coatings that upon exposure to heat may intumesce into a foamed surface layer with low thermal conductivity properties (C).

Figure 3 illustrates how flame retardancy is improved in the decomposition stage (1). A flame retardant can promote transformation of a plastic into char and thus limit production of combustible carbon-containing gases (A). Simultaneously, the char will decrease thermal

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conductivity of the surface. Flame retardants can also chemically alter the decomposition products, resulting in a lower concentration of combustible gases (B). Reduced fuel will thus result in less heat generation by the flame and may lead to self-extinguishment.

Structural modification of the plastic, or use of a flame retardant additive, might induce decomposition or melting upon exposure to a heat source so that the material shrinks or drips away from the heat source (C). And finally, through selection of chemically stable structural components or structural modifications of a polymer, it is also possible to significantly retard the decomposition process (D).

Figure 4 illustrates flame retardancy in the ignition stage (I). In general, anything that will prevent the formation of a combustible mixture of gases will prevent ignition. In this sense, all of the previously mentioned approaches are
pertinent. However, we may also distinguish those cases in which the flame retardant or the modified polymer unit upon exposure to a heat source will form gas mixtures that will react chemically in the gas phase to inhibit ignition.

The goal of flame retardance in the combustion and propagation stage is simply to decrease the rate of heat generated or radiated back to the substrate. Any or all of the above discussed mechanisms could function to prevent a self-sustaining flame.

Practical Methods of Flame Retardation

Flame retardation is achieved in the follow-

| Class | Example |
|-------|---------|
| Nonreactive, organic | | |
| Phosphate esters | [CH₂(CH₂)₃O]₃—PO |
| Halogenated phosphate esters | (BrO₃)₃—PO |
| Halogenated phosphonate esters | (CH₂CH₂O)₃—P—CH₂CH₂Br |
| Halogenated hydrocarbons | Chlorinated paraffins | (CH₃)ₗ—(CH)ₗ Cl |
| Nonreactive, inorganic | | |
| Antimony oxide | Sb₂O₅ |
| Aluminum oxide trihydrate | Al₂O₃·3H₂O |
| Zinc borate | Zn₅B₄O₁₁ |
| Ammonium orthophosphate | (NH₄)₃PO₄ |
| Ammonium sulfamate | NH₄SO₃NH₂ |
| Reactive | | |
| Bromine and/or phosphorus-containing polyols | CH₂OOCR OH |
| | CH(OH)₂CH₂—CH—CH—CH—(CH₃) |
| | CH₂OOCR |
| | Br Br |
| Halogenated phenols | | |
| Tetrachlorophthalic anhydride | | |
| Phosphonate esters | (OCH₂CH₂)₄O₃—P—CH₃OH |
| Dibromoneopentyl alcohol | (BrCH₂)₃—C(CH₂OH)₃ |
Table 2. Typical flame retardants used in finishes.

| Structure | Reference |
|-----------|-----------|
| Tetrakis(hydroxymethyl)phosphonium chloride (THPC) | *(CH₂OH)₂P-CH₂OH | (4-7) |
| THPC–NaOH–NaCl | | (8, 9) |
| N-Methylol dimethylphosphonopropionamide | (CH₃O)₂P-CH₂CH₂C-NHCH₂OH | (10, 11) |
| Cyanamide–phosphoric acid | H₂NCN+H₃PO₄ | (12) |
| Tris(2,3-dibromopropyl) phosphate | (CH₂-CHCH₂O)₃PO | (13) |

Table 3. Typical flame retardants in coatings.

| Structure | Reference |
|-----------|-----------|
| Nonintumescent coatings | | |
| Phosphonitrilic chloride in urethane coatings | | (14) |
| Chlorinated aromatics and aliphatics (with and without antimony oxide) | Cl-OH | (15, 16) |
| Chlorinated paraffins (70% Cl) | | |
| Zinc borate in resin formulations | | (17, 18) |
| Intumescent coatings | | |
| Halogenated alkyl phosphates in resins | (CH₂CHCH₂O)₃PO | (19) |
| Ammonium polyphosphate in resins | NH₄O-P-O⁺NH₄ | (20) |
| Boric acid or borax or zinc borate in resins | H₃BO₃; Na₃B₄O₇·10H₂O | (21, 22) |
| 3ZnO·2B₂O₃ | | |
ing four ways: (1) by using flame retardants as additives prior to polymer processing; (2) by using them as a finish or a surface coating; (3) by using flame retardants as comonomers in the polymerization or grafting; (4) by synthesizing inherently flame resistant structures.

The flame retardants used as additives can be subdivided into nonreactive and reactive types, and the nonreactive type can be further divided into organic and inorganic additives. Although a very large number of flame retardants can be used as additives, at this time a small group of distinct chemical types dominates the field. Table 1 illustrates typical examples of these (2,3).

Table 2 lists the more popular flame retardants used in textile finishes (4-13). Typically, the finishing formations include one or more of such other components: urea, a triazine resin, methylol melamine, ammonia, or a halogenated alkyl phosphate. Flame retardant coatings are of two types: those that are merely flame-resistant and those that are also intumescent. When heat is applied to such a coating, combustion produces a residue which is puffed up by the escaping gases and the resulting cellular material (foam) may actually insulate and protect a flammable substrate. Table 3 illustrates examples of both types (14-22). Table 4 illustrates some of the more commonly used flame retardants as monomers in copolymerization or grafting (23-27).

Table 4. Typical flame retardants used as monomers in copolymerization or grafting.

| Structure | Reference |
|-----------|-----------|
| Vinyl chloride or bromide | \( CH_2=CHCl, Br \) | (29) |
| Vinylidene chloride | \( CH_2=CCl_2 \) | (24) |
| 2,2-Bis[3,5-dibromo-4-(2-hydroxyethoxy)phenyl]propane | | (25) |
| 2,5-Dibromoterephthalic acid | | (26) |
| Bis(2-chloroethyl) vinyl phosphonate | \( CH_2=CH-P(OCH_2CH_2C) \) | (27) |

Table 5. Polymeric structures with inherently high flame resistance.

| Structure | Reference |
|-----------|-----------|
| \( p \)-Polyphenylene | (31, 32) |
| Phenolphthalein polycarbonate | (28) |
| Oxobenz[de]imidazobenzimidazoisoquinoline (benzimidazobenzophenanthroline-type ladder polymer, BBL) | (33) |

A number of studies on new high-temperature polymers have correlated the structure of polymers with their flame resistance properties (28-30). One general conclusion can be drawn —high flame resistance can be related to the
number of moles of multiple bonded aromatic rings per gram of polymer. Table 5 illustrates some examples of such polymeric structures (39). These are the current space-age products in research or development stages. However, as a result of the research efforts of private industry and the various government agencies, a new class of high temperature resistant polymers is available commercially as textiles and/or films. For example, Table 6 lists such names as Nomex, Kynol, and PBI (polybenzimidazole), which are associated with this class of materials. These are all materials containing high aromatic ring content and intrinsically improved flame retardance properties. They are presently expensive and will receive more importance as government-regulated markets require their use during the next decade.

**Typical Flame-Retarded Polymers**

Because of the enormous amount of literature available on flame retardants and flame retarded polymers, only the most recent literature will be reviewed here. For example, in a recent book (44) more than 420 flame retardants were identified by their chemical structure and more than 170 flame retardants were listed as proprietary. According to a 1972 directory (35), some 70 major manufacturers were producing flame retardants.

It is important to briefly review the major resin types and the quantities manufactured in the United States during 1973 (Table 7) (36). Polyethylene, polystyrene and styrene copolymers, poly(vinyl chloride) and copolymers, polypropylene, phenolics, urethane foam, polyesters, urea, and melamine constitute about 89% of all the resins manufactured, and the quantity produced of each of these is more than a billion pounds per year. Table 8 illustrates the pattern of consumption for these top eight resins (36). The pattern of consumption reveals that each type of resin is used in markets that require improved flame retardant properties. Table 9 illustrates typical flame retardants used in commercial plastics (37–53). Since polyethylene and polypropylene usually respond similarly to the same flame retardants, they are considered together. The most effective and and commonly used flame retardants for these polyolefins are combinations of antimony oxide and halo-organic compounds (54–57). Phosphorus-halogen flame retardant systems have been also increasingly found to be satisfactory for polyolefins (38,58). Effective flame retardant combinations for polystyrene and its copolymers are: halogen-containing compounds with or without antimony oxide, and halogen and phosphorus combinations with or without antimony oxide. Poly(vinyl chloride) and its copolymers contain an appreciable amount of halogen (56.8% in PVC). Thus, incorporation of antimony oxide gives an effective flame retardant composition (42,59). Other very effective retardants are phosphorus-based systems which are used to balance off phthalate plasticizer effects (44). Phenolics are not especially flammable, mainly because of their high aromatic content. However, in some applications improvement in flame resistance is sought. Phosphorus and nitrogen compounds have been reported effective in improving flame resistance of phenolics (45). The use of chlorinated compounds (for example, chlorinated phenols) (60) as well as halogen- and phosphorus-containing compounds has been patented (46). The majority of polyurethane flame retardants are based on phosphorus. These may include phosphates and halogenated phosphates (61) as well as ammonium polyphosphate (47). Although halo-
Some Statistics on Flame Retardants

Flame Retardant Markets

Flame retardant markets and their size (63) estimated for 1971 and projected for 1975 are illustrated in Table 11. The figures are all based on pre-energy-shortage data. Existing

| Table 7. Resins manufactured during 1973.* |
|-------------------------------------------|
| Resin                          | Amt manufactured, lb |
|-----------------------------------|----------------------|
| Polyethylene                     | 8,619,600,000        |
| Polystyrene and styrene copolymers| 5,295,400,000        |
| Poly(vinyl chloride) and copolymers| 4,776,200,000       |
| Polypropylene                    | 2,151,600,000        |
| Phenolics                        | 1,438,800,000        |
| Urethane foam                    | 1,304,600,000        |
| Polyester                        | 1,091,200,000        |
| Urea and melamine                | 1,020,800,000        |
| Other vinyls                     | 860,200,000          |
| Alkyls                           | 752,400,000          |
| Acrylics                         | 512,260,000          |
| Coumarone-imdene and petroleum resins | 345,400,000       |
| Epoxies                          | 217,800,000          |
| Nylons                           | 176,000,000          |
| Cellulosics                      | 169,400,000          |
| Others, (polyacetals, fluoro plastics, poly-carbonates, silicones) | 26,840,000 |
| **TOTAL:**                       | **ca. 29,000,000,000** |

* Literature data (36).

Table 8. Pattern of consumption of resins.*

| Resin and form or application | Consumption, lb |
|-------------------------------|-----------------|
| Polyethylene (high and low density) |                 |
| Film                          | 3,273,600,000   |
| Injection molding             | 1,249,600,000   |
| Blow molding                  | 1,060,400,000   |
| Extrusion coating             | 519,200,000     |
| Wire and cable                | 497,200,000     |
| Pipe and conduit              | 334,400,000     |
| Polystyrene film              |                 |
| Trash bags, related products for home, industrial, institutional use | 580,800,000 |
| Miscellaneous packaging including miscellaneous food | 407,000,000 |
| Miscellaneous nonpackaging    | 275,000,000     |
| Produce packaging             | 250,000,000     |
| Industrial liners, shipping bags | 226,600,000    |
| Food and baked goods packaging | 224,400,000    |
| High density film             | 72,000,000      |
| Non-packaging uses            | 1,237,800       |
| Poly(vinyl chloride) and copolymers |                 |
| Pipe and conduit              | 1,155,000,000   |
| Flooring                      | 464,200,000     |
| Electrical wire and cable     | 426,800,000     |
| Furniture                     | 319,000,000     |
| Upholstery and seat covers    | 182,600,000     |
| Polypropylene                 |                 |
| Filament and fiber            | 660,000,000     |
| Transportation including battery cases | 308,000,000 |
| Packaging                     | 145,000,000     |
| Appliances                    | 96,000,000      |
| Phenolics                     |                 |
| Molding compounds             | 380,820,000     |
| Plywood                       | 275,000,000     |
| Insulation material           | 246,400,000     |
| Electrical and electronic switches, etc. | 134,000,000 |
| Urethane foam                 |                 |
| Furniture                     | 369,600,000     |
| Transportation                | 363,000,000     |
| Building insulation           | 143,000,000     |
| Bedding                       | 114,400,000     |
| Polyester                     |                 |
| Reinforced plastics other than sheet | 605,000,000 |
| Reinforced plastic sheet      | 287,600,000     |
| Urea and melamine             |                 |
| Bonding and adhesive resin for fibrous and granulated wood | 576,400,000 |
| Molding compounds             | 96,000,000      |
| Adhesive for plywood          | 88,000,000      |

* Literature data (36).

regulations make the carpets and rugs market the largest market for flame retardants. It is also one of the fastest growing markets because practically no flame retardants were used in this area as recently as 7-8 yr ago. Hydrated alumina, selling for under 10¢/lb, is the
Table 9. Typical flame retardants in commercial plastics.

| Plastic                        | Flame retardant                                                                                                                                                                                                 | Reference |
|-------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|
| Polyethylene, polypropylene   | $\text{Sb}_4\text{O}_6 + \{(\text{BrCH}_2\text{CCH}_3)_2\text{O}\} \quad \left[ \text{NCCH}_2\text{CH}_3\text{P} - \text{CH}_3\text{CH}_2 - \text{P} - \text{(CH}_2\text{CH}_3\text{CN})_3 \right] 2\text{Br}^+ + \text{C}_6\text{Br}_6 \quad \text{NH}_4\text{BF}_4 + \text{Sb}_4\text{O}_6$ | (37)      |
| Styrene and styrene copolymers | $\text{Sb}_4\text{O}_6 + (\text{CH}_2\text{CH}_3\text{O})_3\text{PO}$                                                                                                                                           | (40)      |
| Poly(vinyl chloride) and copolymers | $\text{Sb}_4\text{O}_6 - \text{CaCO}_3$                                                                                                                                                                | (43)      |
| Phenolics                     | $\text{POCl}_3 + \text{NH}_3$                                                                                                                                                                                 | (45)      |
| Urethane foam                 | $(\text{NH}_4\text{PO}_3)_n$                                                                                                                                                                                 | (47)      |
| Polyesters                    | $(\text{CH}_2\text{CH}_3\text{O})_3 - \text{PO}$                                                                                                                                                        | (50)      |
| Urea and melamine resins      | $\text{H}_3\text{PO}_4$                                                                                                                                                                                     | (52)      |

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dominant flame retardant in this area. Transportation and home furnishings are two areas coming most recently under legislation and are consequently projected to have the most dramatic increase in usage of fire retardants. In general, the economic factor is a tremendous force in regulating the flame retardant market volume. The volume of all flame retardants with a price tag of more than 10¢/lb is only 1/3 of the total market volume.

Flame Retardant Growth in Plastics

Table 12 illustrates the increasing utilization of flame retardants (more than 41% growth between 1972 and 1973) in commercial plastics (64). The items of special note in the nonreactive flame retardant areas are that there has been more than a 60% increase in use of halogenated phosphate esters, and more than 80% increase in various other types of nonreactive flame retardants. Thus, the trend is towards phosphorus-halogen and numerous other specialized flame retardant systems. The overall reactive flame retardant growth was comparable to that of nonreactives, with the greatest increase occurring in the polycarbonate (80%) and epoxy (58%) resin systems. These, however, are low volume resins.

Price Ranges of Common Flame Retardants

Table 13 lists some common types of flame retardants with their corresponding pre-energy-shortage price data (65). The cost can range from a few cents a pound to many dollars a pound for some flame and smoke retardants. The cost of the flame retardant may therefore rule out a plastic entirely from a certain application. On the other hand, a high concentration of low-cost flame retardant, such as hydrated alumina, may make the flame retarded formulation less expensive than the standard polymer formulation. Correspondingly, flame retardants with a price tag of less than 10¢/lb have captured about two-thirds of the total flame retardant market volume, and those with a price tag of more than 75¢/lb only a very small fraction (about 1%). It can be seen that the nondurable inorganic flame retardants constitute exclusively the lowest price (<10¢/lb) group of additives. The reactive flame retardants or those that are of most interest in

| Plastic              | Flame retardant |
|----------------------|-----------------|
| Polyethylene         | Sb₂O₃+X; P+X    |
| Polypropylene        |                 |
| Polystyrene          |                 |
| Poly(vinyl chloride) | X±Sb₂O₃; P+X±Sb₂O₃ |
| Phenolics            |                 |
| Polyurethane foam    |                 |
| Polysterers          |                 |
| Urea and melamine    |                 |

* X = halogen-containing organic compounds; P = phosphorus-containing compounds; P+X = phosphorus- and halogen-containing organic compounds; P+N = phosphorus- and nitrogen-containing organic compounds; X+Sr₂O₃ = halogen-containing compound combined with antimony oxide; ± = composition with or without the following component.

| Market               | Estimated annual consumption, lb×10⁶ |
|----------------------|--------------------------------------|
| 1971                 | 1975                                 |
| Carpets and rugs     | 100-125                              |
| Construction         | 14-22                                |
| Miscellaneous nondurables | 10-15                          |
| Electrical and electronics | 11-12                   |
| Apparel              | 8-10                                 |
| Transportation       | 4-5                                  |
| Home furnishings     | 3-4                                  |

* Industry data (63).

| Market               | Estimated annual consumption, lb×10⁶ |
|----------------------|--------------------------------------|
| 1972                 | 1973                                 |
| Nonreactive          |                                     |
| Phosphate esters     | 53,680,000                           |
| Chlorinated paraffins| 41,800,000                           |
| Phosphate esters     |                                      |
| (non-halogenated)    |                                        |
| Chlorinated paraffins| 41,800,000                           |
| Phosphate esters     | 14,980,000                           |
| (halogenated)        |                                        |
| Antimony oxide       | 17,600,000                           |
| Bromine compounds    | 7,040,000                            |
| Boron compounds      | 3,960,000                            |
| Others               | 10,120,000                           |
| Reactive             |                                      |
| Urethane intermediates| 18,040,000                       |
| Polyester intermediates| 11,440,000                        |
| Epoxy intermediates  | 5,280,000                            |
| Polystyrene intermediates| 1,100,000                    |
| Polycarbonate intermediates| 7,280,000                    |

* Industry data (64).
plastics and textile applications are those in the 25–50¢/lb and above range.

Conclusions

The involvement by government agencies, consumer groups, and industry in safety programs during the 1970's will continue. Interest in increasing the fire safety aspects of polymers will grow, leading to the use of a number of fire retardant chemical approaches. These chemical approaches will utilize large quantities of halogen, antimony, phosphorus, nitrogen, and other inorganic, and aliphatic and aromatic organic species which will be added as non-reacted or reacted components of the polymer structure.

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