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Laboratory Test Methods for the Evaluation of Flammability of Polymers

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Combustion is a complex phenomena involving a multiplicity of variables. Some important variables measured in flame tests follow [1]. In order to characterize ignition, such related parameters as ignition time, ease of ignition, flash ignition temperature, and self-ignition temperature are measured. For studying the propagation of the flame, parameters such as distance burned or charred. area of flame spread, time of flame spread, burning rate, charred or melted area, and fire endurance are measured. Smoke characteristics are studied by determining such parameters as specific optical density, maximum specific optical density, time of occurrence of the densities, maximum rate of density increase, visual obscuration time, and smoke obscuration index. In addition to the above variables, there are a number of specific properties of the combustible system which could be measured. These are soot formation. toxicity of combustion gases, heat of combustion, dripping phenomena during the burning of thermoplastics, afterglow, flame intensity, fuel contribution, visual characteristics, limiting oxygen concentration (OI), products of pyrolysis and combustion, and so forth. A multitude of flammability tests measuring one or more of these properties have been developed [2]. Admittedly, no one small scale test is adequate to mimic or assess the performance of a plastic in a real fire situation. The conditions are much too complicated [3, 4]. Some conceptual problems associated with flammability testing of polymers have been reviewed [5, 6].

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It is beyond the scope of this review to list and discuss all fire tests, as reasonable descriptions exist elsewhere [2, 5, 7-24]. Testing of polymers under flaming conditions has also been examined [24].

The existing techniques can be classified into two categories: flammability tests and fundamental research methods of investigation. The former class includes standard test methods such as ASTM (American Society for Testing and Materials) tests, BS (British Standards) tests, NBS tests, and UL tests. These are designed to serve the following functions [19]: 1) information to indicate the likely behavior of materials or products in a fire, 2) compliance to show if the functional requirements can be met, and 3) quality control to maintain a standard of manufacture so that consistent fire behavior can be expected.

In the second category we find a limited number of methods which have been devised in order to develop knowledge of the mechanism of combustion itself. The standard tests do not cover all the parameters of polymer flammability, and in general they require large samples. The nonstandard tests are primarily meant for research and developmental work. They give an insight to the combustion mechanisms and assist in evaluating structure-property relationships. Smaller samples are used in these tests.

ADVANTAGES AND DISADVANTAGES OF LABORATORY SCALE TESTS

One of the major problems facing researchers is the difficulty of finding test methods which correlate with true fire situations [25]. It is generally agreed that small-scale tests measure properties of materials rather than evaluate hazards [26, 27].

Poor reproducibility was noted on high performance (i.e., high melting) resin specimens such as polysulfones, polycarbonate, and polyphenylene oxide, which are intumescent under test conditions [25].

Isaacs [28] summarized the drawbacks of normal test methods as follows:

(1) The end point for these tests has been set at some arbitrary time or distance which was chosen without any regard for any real situation.

(2) The tests are of the "go-no-go" type which determine only whether the specimen of material will burn in air while placed on a particular test configuration, so the results are of little value in studying the material.

(3) The tests are of little use in studying very flammable and very inflammable materials.

(4) Finally, tests have little relationship to each other, and no relationship to any actual end use conditions.

Emmons [29] exemplifies the inadequacy of the presently available fire tests, which lead to a remarkable discrepancy in judgements throughout the world. A specific phenolic wall board was rated the safest by the standard test of Federal Republic of Germany, while Denmark's standard test rated it as the most flammable.

Even the most complex test represents a simplification of the fire situation with its many interdependent features, but nevertheless a test would find acceptance and have a meaning if it could be linked with the fire environment. It is unrealistic to expect that a few tests can truly answer the question of the hazards of a material, since these depend not only on the material but also on its environment.

It should be recognized that fire tests are unlikely to have a high degree of reproducibility. With the variability of materials in view, a coefficient of variation of 10% represents the best that can be expected [19]. A variety of polymers has been screened with a good degree of reproducibility [24]. The use of ambiguous classifications such as self-extinguishing, burning, and nonburning is replaced by new test designations.

Recently the potential hazards associated with the unusually high toxicity of the gaseous combustion products of some polymeric compositions and fire retardant polymer formulations [30] have created a pressing need for the development of suitable test methods for evaluating this potential fire hazard. Despite the present uncertainty, many of the ASTM test methods remain the only standards currently available [20].

COMPARISON BETWEEN LABORATORY SCALE AND LARGE SCALE TEST METHODS

The lack of correlation of most of the small-scale laboratory test methods with many real fire conditions, when large quantities of material are involved, has increased the lack of general confidence of the utility of present test methods and has resulted in pressure for the development of more large-scale tests that would give a more realistic evaluation of real fire hazards [31]. The most that laboratory tests can be expected to do is to show trends in burning behavior that will maximize the probability that a given polymer system can pass a large-scale test [32].

The methods for evaluating polymer flammability are presently in a high state of flux. Not only is the relevancy of small-scale test methods in serious question [33, 34], but the need for additional flammability information that is not evaluated by these tests, such as smoke generation [31] and the toxicity of combustion gases, has emphasized the inadequacy of some of these test methods. As a result, the importance of test procedures like ASTM D635, D757, and D1692 has decreased tremendously in the last few years. The largescale flammability tests include design and material tests like the ASTM E84 (Steiner) tunnel test and engineering or structural tests like the NBS corridor test and building burnout tests. A number of publications are available on this aspect in the literature [20, 31, 35-39]. These tests evaluate materials under representative or actual construction conditions. These tests are necessarily very specific and are large in scale. They require large samples and test equipment. Hence they are very expensive and, unfortunately, seldom reproducible. They are not included in the following discussions.

LABORATORY SCALE TEST METHODS

Because of the great variety and complexity of available test methods, this discussion is limited to the more common tests currently being used.

Ignition Tests

The most widely used test to measure auto and flash ignition temperatures is the Setchkin's test [40]. The apparatus consists of an ignition furnace with provisions to hold the sample and to pass off air or oxygen inside. Thermocouples near the sample measure the temperature with time. Ignition temperature and time can be obtained from time-temperature plots from the point where the temperature suddenly shoots up and the time starting with the introduction of the sample. In the determination of the flash ignition temperature a small burner flame fixed near the exit is made use of. A recommended test procedure and the ignition temperatures of a number of polymers are given by Patten [41]. The Setchkin ignition test is accepted as an ASTM Standard (ASTM D1929).

Baillet et al [42] used an apparatus for self-ignition study consisting of a cylindrical Pyrex reaction vessel located in an oven, and in which an atmosphere of a predefined composition may be introduced at subatomic pressures. The polymer sample is attached to a metallic tip and introduced to the reaction vessel after filling the vessel with O₂. Measurements are made with a thermocouple and a photomultiplier inserted into the reaction vessel.

Brauman et al. [43] used the furnace thermal ignition method to determine the ignitability of polymers by pyrolyzing the samples under N_2 in a tube furnace and testing the ignitability of the off gases in air with a small pilot flame.

Morimoto et al. [44] describe an ignition test apparatus for determining the limiting ignition oxygen index. The apparatus consists of a combustion unit which is essentially an open furnace and a gas supply unit. The determination is made by varying the temperature of the furnace by a trial and error method. The limiting ignition oxygen index is also determined by gradually varying the atmosphere concentration at a fixed temperature.

Ignition phenomenon is also studied by using tests in which radiative heat sources are used.

Disk Radiant Ignition-Spark Pilot [43]

In this method a spot heater is used as the radiant source. The sample is a thin circular disk placed horizontally on a small holder. A glass ring is placed around the sample to contain molten polymer that may be formed during heating. The radiant lamp is focused only over the central portion of the disk sample. The energy flux is controlled by adjusting the voltage to the lamp with a powerstat. The linear gas flow past the sample is maintained constant. The ignition times are measured as the time required to achieve sustained ignition. A spark pilot is used for ignition. The spark is generated by a tesla coil, struck to the edge of the sample holder downstream of the sample surface.

Arc Image Radiant Ignition-Unpiloted

The arc image radiant ignition apparatus [45] consists of a strong arc lamp, a refocusing mirror, a rotating disk shutter, and accessory apparatus and instrumentation.

The refocusing mirror causes an image of the electric arc to be focused within the vertical glass pipe sample enclosure. The ignition time is determined either by the rotational speed of the shutter mechanism or by measuring the time to ignition with a photocell with the shutters wide open. The driven rod configuration (which is discussed in the section on propagation tests) with superimposed radiant heating is also used [43] for measuring ignition without a pilot. The test for spontaneous ignition is initiated by starting the radiant heaters and timer simultaneously. Samples are exposed at radiant fluxes of 3 and 4 cal/cm²·s.

With radiant energy sources the samples are heated only at one surface, resulting in a steep temperature gradient. However, in a furnace the entire sample is heated by conduction, convection, and some radiation. Heat losses in the solid are unimportant and fuel production is rapid. Unlike the radiant ignition geometrics where the hot fuel is ejected into a cool oxidant, the furnace maintains the gaseous fuel at high temperature so the sample can continue to crack or even react with the gaseous components in the unit if it reaches the ignition temperature.

Ignition temperature data may be obtained by an open cup DSC or thermogravimetric technique [35]. Underwriters' Laboratories uses arc ignition (high current) and hot wire ignition tests.

Fristrom et al. [46] introduced a moving wire ignition apparatus. The apparatus consists of a polymer sample in the form of a wire passing through an ignition source which can be a flame, a jet of heated gas, or a radiation bath. The advantage claimed is that this technique converts ignition from a time-dependent to a steady-state process. Ignition occurs sharply and is reproducible for a critical residence time which depends on the oxygen concentration. A sealing factor is determined from the experiments.

ASTM D635-74 Test for Flammability of Self-Supporting Plastics

A flame is touched to a specimen of the polymer placed horizontally for 30 s. The burning rate, average burning time, and average extent of burning are determined relative to a gauge mark.

Underwriters' Laboratories has suggested a modification in which a wad of cotton is placed beneath the burning end of the specimen and checked for ignition due to dripping.

ASTM D757-74 Flammability Test for Self-Extinguishing Plastics

This test is for specifically rigid plastic sheets. The sample is ignited with an electrically heated ignition bar set at 950° C. The burning rate is determined.

Intermittent Flame Test [20]

A more severe laboratory test, often designated as HLT (Hooker Laboratory Test), is capable of distinguishing between materials rated as nonburning by all the above tests. This test is principally useful for the evaluation of highly fire retardant materials. In this test a flame is intermittently applied to the bottom of a specimen vertically suspended for increasingly longer periods. A rating from 0 to 100 can be calculated from the time of burning after each application of the flame.

ASTM D1692-74 Test for the Rate or Extent of Burning of Cellular Plastics

The burning rate or the time for extinguishment can be determined for cellular plastics. Burning characteristics such as intumescence, melting, dripping, and the presence of flaming drops are also recorded.

UL Subject 94, Standards for Tests for Flammability of Plastic Materials for Parts in Devices and Appliances [47]

This test is an important method for classifying polymeric materials to be used in electrical applications. The test specimen, supported vertically by its upper end, is ignited at the lower end for 10 s by a Bunsen burner. The duration of flaming or glowing and dripping are noted. Flammability ratings of V0, V1 and V2 are applied depending upon the specimen behavior during the test.

ASTM D1360-70 Test for Fire Retardancy of Paints: Cabinet Method

The test consists of impinging a flame under controlled conditions onto a weighed wood panel coated on one side with the paint being tested. The loss of weight and the char volume of the panel are determined.

Two Foot Tunnel Test [48]

This test is a miniature of ASTM E84 which is a large-scale test. It is reproducible and often exhibits a high degree of correlation with ASTM E84 test results [49]. The apparatus consists of a $24'' \times 4''$ specimen mounted on a steel plate at an angle inclined 28° from the horizontal. The tunnel allows a natural draft. In the test evaluation the test panel is ignited at the fire end for a period of 4 min, and the movement of the flame front down the tunnel is recorded in 15-s intervals. A flame spread rating is then calculated on an arbitrary scale where asbestos board is assigned a value of zero and red oak 100.

ASTM E162.67 Test for the Surface Flammability of Materials Using a Radiant Heat Energy Source

The method employs a radiant panel heat source on the front of which is placed a specimen of the test material on a plane 30° from the vertical plane of the panel. The ignition occurs at the upper end and the flame front progresses downward. The flame spread index is calculated as the product of a flame spread factor and a heat evolution factor which are obtained from the test data. The smoke is reported as the total weight of the solid collected on the filter of the smoke measuring device. Highly fire-retardant materials exhibit index values of 25 or less by this test method while standard red oak yields an index of 100.

<u>SRI (Stanford Research Institute) Flame Spread</u> <u>Test</u> [43]

In this test a horizontal flame spread is evaluated by employing a superimposed radiant heating to sustain combustion. Stick samples in tight-fitting aluminum foil containers are mounted horizontally on thin suspension wires to minimize conductive heat loss during burning. Radiant energy is provided by a bank of quartzline lamps. The sample is ignited at one end and the overall flame spread rate is determined for 1-cm long intervals marked off along the sample surface.

Brauman et al. [43] developed 3 different configurations to determine burning rates of polymers as a comparative measure of relative flammability in experimental research. They are the driven rod, a high thermal mass (HTM) puddle, and a low thermal mass (LTM) puddle configurations.

Driven Rod Configuration

This has proved very useful for characterizing the ignition (piloted and unpiloted) burning and extinction of rods of polymer samples [50]. A polymer rod is mounted vertically in a tight-fitting low thermal mass, quartz sleeve (etched to 0.2 mm wall thickness) centered within a chimney in a controlled atmosphere. The rod is burned or pyrolyzed from the top. The regressing surface is maintained level with the end of the sleeve by a mechanical syringe pump drive assembly. The advance of the rod compensates for polymer consumption and yields a linear regression rate for burning or pyrolysis. Temperature measurements were made by continuous measurement of the emf as a function of time of a thermocouple embedded in the center of the burning driven rod samples.

HTM and LTM Puddle Configurations

In a puddle configuration the disks of material are burned in a pan mounted inside a glass chimney on a top-loading recording balance. Rates are determined from the linear portion of the burning curve. Variations in the thermal environment of the sample are obtained by changing the construction material of the pan (glass or aluminum). The high thermal mass (HTM) puddle configuration refers to the glass dish resting on the weighing platform; the low thermal mass (LTM) configuration refers to the aluminum foil pan on the pins. The minimum O_2 concentration required to sustain combustion increases with increases in the effective thermal mass of the pan assembly [51].

Opposed Flow Diffusion Flame (OFDF) Method

Blazowski et al. [52] and Holve [53] suggested a very useful and accurate technique, known as "opposed flow diffusion flame (OFDF)," for combustion studies on polymers. OFDF is of simple geometrical configuration and is able to simulate the basic surface heat flux and condensed phase temperature characteristics of a conventional fire [54].

In the OFDF technique the oxidizer flow impinges on the end of the fuel sample, which in the steady-state burning condition produces a fuel steam opposing the oxidizer flow. Therefore, for a given burning condition the stagnation plane and the flame zone are located at a fixed distance from the fuel surface. Burning rates and extinction measurements can be done accurately, and mass transfer numbers can be calculated. Holve et al. [55] presented a simplified analytical model of OFDF. OFDF is extensively used for the accurate determination of the burning rates of polymers, the temperature profile, and the concentration profile in the flame [56].

Tewarson et al. [57] described a mass burning rate assembly and used it for the determination of combustion characteristics such as the heat of gasification.

Apart from the equipment mentioned here, a number of other instruments have been developed by many authors in their investigations of flame propagation.

Smoke Evolution Tests

Numerous techniques for the evaluation of the smoke behavior of burning polymers have been developed [58-62]. Some methods use light transmittance as the criterion; others use trapping and weighing procedures. In the first category are the NBS [63, 64] and XP-2 smoke chambers [65], ASTM E-84 tunnel test, modified oxygen index chambers [66, 67], and TGA apparatus [68]. In the latter category one finds modified oxygen index chambers and other devices which use vacuum collection of smoke particles on filter paper [69]. In the first case smoke is generated in a burning chamber and caused to flow from the chamber through an elongated enclosure in which light transmitted through the smoke in the enclosure is measured. Smoke includes opaque and light-absorbing components, thus permitting detection by observation and measurement of its lightabsorbing character. In the second case the smoke is filtered, weighed, and analyzed. These filtering tests have the drawback of being unable to test those components of smoke which pass through the filter.

NBS Smoke Chamber 63, 64

This is one of the most widely used and most reproducible devices for smoke measurement. A standard radiant heat source is used normal to the exterior surface of a specimen with or without the presence of small pilot flames applied at the base of the specimen.

Results are reported as specific optical density (D) and represent the optical density: log (100/T) measured over a unit path length (L)within a chamber of unit volume (V) produced from a specimen of unit surface area (A). The smoke measurements can be conducted under smouldering or flaming conditions. A photometer is employed to measure the light transmittance.

Rohm and Haas XP-2 Test (ASTM D2843-70)

The method measures the loss of light transmission when a standard specimen is burned in a test chamber under standardized conditions. The smoke density data is normally presented as maximum smoke density (MSD) in percentage and smoke density rating (SDR) in percentage. The SDR represents the total amount of smoke collected and is defined as 100 multiplied by the area under the curve of light absorption versus time.

Structure containing the aliphatic carbon backbone, such as polyolefins, nylons, acetal and PMMA are of low smoke density. Additives which increase oxygen index usually increase smoke.

Oxygen Index Test (ASTM D2863) [70]

One of the most versatile tests used in both industrial applications and experimental research is the oxygen index test, first proposed by Fenimore and Martin. The experimentation is simple. A 6-in, long sample is placed in the center of a glass cylinder. A mixture of O_2 and N_2 is fed into the cylinder at the base. The sample is lit by a small gas flame at the top. By trial and error the minimum O_2 concentration which will sustain continued combustion of the sample is determined. The oxygen index of a specimen is the minimum percentage of oxygen in a slowly rising O_2/N_2 atmosphere which will just sustain combustion of the specimen. OI is clearly a measure of extinction. There are abundant studies conducted on the oxygen index of polymers in the literature [32, 54, 70-88].

Calorimetric Methods

Static O_2 bomb calorimetry gives the heat of complete combustion of the sample. There are numerous references available [89-91].

A recent technique is isoperibol calorimetry [92] which gives the rate of heat release in a controlled atmosphere, an important parameter in fire retardancy.

Apart from the test methods discussed above, numerous other techniques exist which have been proposed from time to time in the literature.

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