Chapter

Cone Calorimetry in Fire-Resistant Materials

Rakesh Kumar Soni, Meenu Teotia and Aakansha Sharma

Abstract

Polymeric materials are specifically designed by compounding with additives to achieve specific properties that make them suitable for a particular application. Flame retardant materials offer fire-resistant properties to the polymers. The fire behavior of polymeric materials can be investigated with the help of various analytical techniques such as Underwriters Laboratories test standard UL-94, LOI test, Thermal gravimetric analysis (TGA) and Cone Calorimetry. Among these tests, Cone Calorimetry is the most suitable test method for predicting the real-scale fire behavior of polymeric materials and is adopted by the International Organization for standardization (ISO 5660-1). It quantifies heat generation, smoke production, mass loss and helps in the selection of polymeric materials for desired applications. In this chapter, an attempt has been made to present an overview of the thermal decomposition of polymers and the action of flame retardants. Different fire testing techniques generally used for investigations of fire characteristics of polymers are summarized.

Keywords: cone calorimetry, fire retardants, polymers, TGA, LOI

1. Introduction

Fire is a unique destructive force that thermally oxidizes polymeric materials. Fire-resistant materials can withstand high temperatures and resist burning. These may be inherently flame retardant or can be made fire resistant by adding flame retardants. Flame retardants are chemical substances that are incorporated in polymeric systems to combat fire risks hence the use of flame retardants in polymeric systems play a crucial role. Employment of flame retardants never signifies the absence of fire but flame retardants retard the ignition, growth and propagation of fire, thus minimizing fire-induced destruction [1, 2]. An increase in demands for polymers due to their wide applications in thermal, electrical and mechanical sectors has rekindled the flammability issues of flame retardants with polymers and created challenges for modern technology [3–5]. An exemplary flame-retardant system should have resistance to fire ignition, high combustion speed, smoke production and toxicity without economic penalty [2]. As polymers are organic compounds, they produce volatile combustible products when subjected to heat. Flame retardants interfere with the combustion process of polymers at various stages such as decomposition, ignition, the spread of flame and also smoke process [6]. Polymer flammability is defined by many processes and/or characteristics, including burning rates (solid degradation rate and heat release rate), spread rates

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(flame, pyrolysis, burn-out, smolder), ignition characteristics (delay time, ignition temperature, critical heat flux for ignition) [7], emission distribution (particularly toxic species emissions), smoke production. Several testing techniques are employed to analyze the fire behavior of polymers such as Underwriter's laboratories test standards (UL-94), limiting oxygen index (LOI) test, thermogravimetric analysis (TGA) and Cone calorimetry. In this chapter brief overview of the thermal decomposition of polymers and fire retardants & their mechanisms is presented under headings 2&3. Different types of flammability/fire tests used to analyze the fire characteristics of polymers are discussed under heading 4. It also covers the instrumental set up of the Cone calorimeter along with fire parameters obtained with the help of the instrument. Heading 5 concludes the advantages of Cone calorimetry over other analytical techniques used to determine fire characteristics of polymers.

2. Thermal decomposition of polymers

The physical and chemical properties of solid polymeric material change when heat is supplied to them as a result of thermal decomposition and thermal degradation. Thermal decomposition is a process in which extensive chemical changes can occur due to the application of heat. On the other hand, thermal degradation leads to loss of physical, mechanical and electrical properties of material when heat or elevated temperature is applied. To correlate the thermal properties of the material to the fire parameters, the process of thermal decomposition of the materials plays a crucial role. Thermal decomposition via chemical reactions generates gaseous flammable volatiles. When the flame is applied to polymeric substances, it tends to release flammable volatiles since polymer substances undergo pyrolysis. It releases some combustible gases, along with non-combustible gases (**Figure 1**). If the concentration of volatiles is at or above the combustible boundary then only it supports combustion otherwise flame dies out. Due to the presence of air, combustible products lead to flame and produce huge amounts of thermal energy, which in turn feed fire [8].

When thermal decomposition of deeper layers of solid polymeric material continues, the volatiles produced pass through the char layer formed to reach the surface. During this process, the char may cause secondary reactions to occur in the volatiles. Carbonaceous chars can be foamy layers, which slow down the decomposition of polymers considerably. Inorganic remains, on the other hand, can form glassy coverings that may then become impenetrable to volatiles and protect the beneath layers from any further thermal breakdown [9, 10]. Main chemical decomposition mechanisms are; (a) random-chain scission, in which chain scissions occur at random sites in the polymer backbone (b) end-chain scission, in which individual monomer units are consecutively removed at the chain end; (c) chain-stripping, in which atoms or groups not part of the polymer chain is cleaved; and cross-linking, in which bonds are created between polymer chains. Virgin polymer materials get decomposed by these processes. The reported literature indicates some type of correlation between mechanisms of polymer degradation and fire retardancy pathways. **Table 1** represents the polymer degradation mechanism of some synthetic polymers along with products evolved such as water, hydrocarbons and other volatiles.

The chemical processes involved in thermal decomposition are variably complex and the structure of polymers also influences the end consequence of decomposition. The chemical structure of repeating units and their heat-releasing capacity effects the fate of ignited polymer. Higher aliphatic molecules have a high potential for heat release which directly contributes to the thermal stability of the polymer [11].
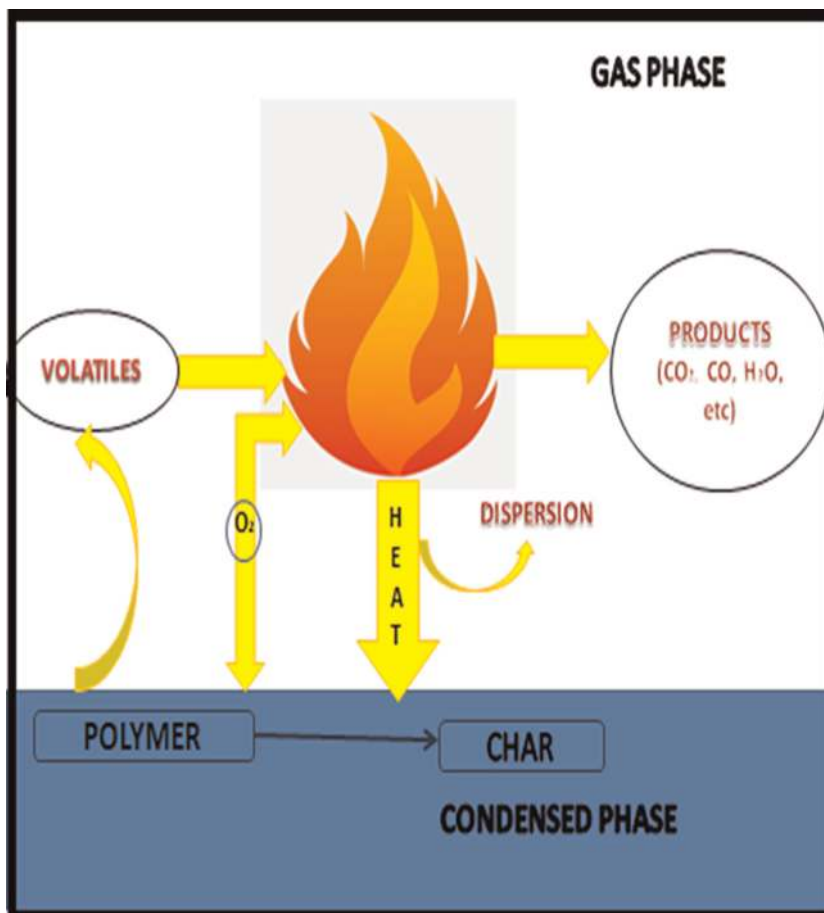
Figure 1.
Thermal decomposition of polymers.

Mechanism of polymer degradation	Examples of polymers	Products evolved	Flame retardant mechanism
Random chain scission	Polyethylene	Hydrocarbons	Vapor phase halogenated/ intumescent based
	Polypropylene	Hydrocarbons	Vapor phase halogenated/ intumescent based
	Polystyrene	Hydrocarbons	Condensed phase char formation
End chain scission	PMMA	Monomers	Halogenated FR (Free radical based)
	PFE	Monomers	Halogenated FR (Free radical based)
Chain stripping	PVC	Char, HCl, Hydrocarbons	Vapor phase brominated/ char formation
	PVA	Water, Char	Protective covering on material
Cross-linking	Polyacrylonitrile	Char and evolution of volatiles	Vapor phase / char formation
	Poly (oxy-m–xylene)	Char and evolution of volatiles	Vapor phase / char formation

Table 1.
Volatile products and flame retardant mechanism of polymers [12].

3. Fire retardants and their actions

Fire exists as a unique destructive force and results in damage to life and property if not handled carefully. Nowadays polymers are used in every walk of life and therefore their properties whether chemical or mechanical are very important parameters for their application in a particular product. In applications where fire retardance is an important requisite, polymers used must possess fire retardant ability to combat the unwanted risk of fire. Generally, polymers are made fire resistant by incorporating compounds, which are known as Flame retardants imparting flame retardance to the polymers. These can be thermoplastics, thermosets, fabrics, coating and prevent or inhibit the spread of fire. The word "flame retardant" describes a function rather than a chemical family. Flame retardants are made up of a range of compounds with various characteristics and structures, and these chemicals are frequently combined for increased efficacy [13, 14]. The action of flame retardants can be classified in three ways:

3.1 Vapor phase inhibition

When flame retardant chemicals react with the burning polymer during the radical gas phase, vapor phase inhibition occurs. These additives reduce or suppress the supply of combustible gases by interfering with the creation of free radicals, cooling the system, and lowering or suppressing the generation of free radicals. Brominated flame retardants (BFRs) are commonly utilized because of their vapor phase blocking characteristics. Before the material achieves its ignition temperature, bromine releases active bromine atoms into the gas phase. These atoms inhibit the chemical process occurring well within the flame, extinguishing or delaying its spread. This allows families or employees more time to flee the building or suppress the fire in another way.

3.2 Solid phase char formation

During a fire, solid-phase char flame retardants encourage the development of char. These additives reacted with the burning polymer, forming a carbon - containing layer on the surface of the material. This layer acts as a protective barrier, preventing the discharge of flammable gases and shielding the underneath material from the heat of such flame.

3.3 Quench and cool system

To increase flame resistance, quench and cool systems depend on hydrated materials. When hydrated minerals are exposed to fire, they emit water molecules that cool the substrate and interfere with the burning process.

4. Types of flammability/fire tests

Flammability testing is an important component of assuring safe and dependable consumer products. Textiles and consumer products, aircraft and transportation, mattresses, and furniture materials are among industries that use flammability test

procedures. Flammability testing methods determine how readily materials ignite, burn, and respond when they are ignited. These include the following fire tests [15]:

4.1 UL-94 test (underwriters laboratories test standard UL-94)

One of the most commonly used flammability tests for estimating comparative flammability for plastic materials is UL 94 (Underwriters Laboratories test standard UL 94). Under specified laboratory circumstances, it analyzes a sample capacity to extinguish a flame after ignition as well as its dripping behavior in respect to a small naked flame or radiant source of heat [15]. Depending on the specifications, the materials are arranged vertically or horizontally over a Bunsen burner. During a vertical flammability test, the length of time a material burns after the initiating flame is withdrawn, the amount of the specimen that burns and whether or not it drops flaming particles are all measured. Horizontal flammability tests, on the other hand, look to see if the material burns after the test flame has been withdrawn, and then determine the rate at which the specimen burns. This fire test is intended to provide as a preliminary indicator of a plastic's acceptability to be used as part of an equipment or appliance in terms of flammability. The UL 94 standard is used to determine the intensity and characteristics of combustion based on the standard samples. UL-94 test is broadly classified into UL-94 VB and UL-94 HB [16].

The UL94 vertical burning test is a popular fire testing method for industrial polymeric products and materials. A 1/2" × 5" specimen is used for this test, which is held vertically at one end. The open end of the specimen is exposed to a burner flame at two 10-second periods, separated by the time required for the flaring combustion to stop after the first application [17]. The specimens are divided into two groups of five each. For each specimen, the following data is kept: (1) After the initial burner flame is applied, the duration of igniting combustion is calculated, (2) After the second burner flame is applied, the duration of blazing combustion is measured, (3). After the second burner flame is applied, the duration of glowing combustion is measured, (4) If burning droplets ignite cotton is put beneath the specimen and (5). If the specimen burns up while being held in the clamp.

Horizontal UL-94 Test is typically regarded as the easiest test to pass. The sample is clamped horizontally on the U-shaped frame and ignited in the combustion chamber for 15 seconds with a specified tiny flame. The flame is lit at one end of the specimen to determine the time it takes for the flame to spread to a certain extent and the distance at which it burns. A 1/2″ × 5″ specimen is used in the test, which is held horizontally at one end with markings at 1″ and 5″ from the open end. For 30 seconds or until the flaming front reaches the 1-inch mark, a flame is applied to an open-end "a mark if the combustion persists, the timer is set between the 1″ and 5″ marks. If the combustion process comes to a halt before the 5th minute, "The damaged length between the two markers, as well as the duration of combustion, is all recorded [16].

4.2 LOI (limiting oxygen index) test

Charles P. Fennimore and Fred J. Martin proposed the Limiting Oxygen Index (LOI) for the first time in 1966 [18]. It's a term that has been used to describe the relative flammability of various materials. The LOI test is subject to an international standard that is standardized in France (NF T 51-071) and the United States (ASTM D 2863) (ISO 4589). The limiting oxygen index (LOI) is the lowest oxygen concentration that will allow a polymer to burn. It is given as a percentage. It's determined by

flowing a combination of nitrogen and oxygen over a burning material and gradually decreasing the oxygen level until the critical level is achieved. Standard tests, such as the ISO 4589 and ASTM D2863, are used to establish LOI values for various polymers. The limiting oxygen index is still the most important sorting criteria for polymer ignitability (LOI). The minimal oxygen content in the air for polymeric ignition and burning is determined by this method. As air contains 21% oxygen, polymers with LOI less than 21% are considered flammable, whereas those with LOI greater than 21% will not ignite in normal air. In reality, materials having an LOI of at least 25% should be less ignitable. Although, LOI is useful for proving polymer ignitability, it only provides limited evidence of material characteristics when exposed to heat, flames, or both [19, 20].

The test sample is vertically placed in a glass chimney, and then an oxygen/nitrogen atmosphere is created by a flow from the chimney's base. The flame is sparked at the top portion of the test sample, and thus the oxygen content in the flow is reduced until the flame can no longer be produced. The specimens are exposed to one or more specified sets of laboratory test conditions in this test technique. This is not always possible to detect changes in the fire-test-response characteristics assessed by or from this test if alternative test circumstances are substituted or the end-use conditions are modified. As an outcome, the results are only valid for the conditions indicated in the test procedure for exposure to fire [21].

4.3 TGA (thermal gravimetric analysis)

Thermo gravimetric analysis, often known as thermal gravimetric analysis (TGA), is a type of thermal analysis that measures the mass of a sample over time as the temperature varies. Physical events like as phase transitions, absorption, adsorption, and desorption, along with chemical phenomena such as chemical adsorption, thermal breakdown, and solid–gas interactions, are all revealed by this measurement (e.g., oxidation or reduction) [22]. The fundamental premise of thermo gravimetric analysis (TGA) has been that a sample's mass change may be evaluated under predetermined conditions. Absorption, adsorption, desorption, vaporization, sublimation, breakdown, oxidation, and reduction are all examples of thermal phenomena that TGA is used to explain [23]. Applications of TGA include, determination of thermal stability of materials (describes the breakdown mechanism, as well as the fingerprint materials, are used for identification and quality assurance), oxidative stability of materials, the composition of multi-component systems, estimated lifetime of a product, decomposition kinetics of materials, the effect of reactive or corrosive atmospheres on materials and moisture & volatiles content of Materials. This technique assists in investigations of fire characteristics of flame retardant polymers. Ureyen and Kayank investigated the effect of zinc borate with phosphorous-based FR finishing agents using cone calorimetry and TGA analysis [24]. TGA and DTA plots of zinc borate provided mass loss data of the samples which were further supported by heat release rate curves obtained from Cone Calorimetry. TGA measures thermal degradation and it can be related to flame resistive data of the samples.

4.4 Cone Calorimetery

The study of the thermal decomposition of polymers is important in terms of their fire performance behavior. This issue has been highlighted earlier by Van Krevelen [25], for many polymers, the limiting oxygen index [26] (primary test of

flammability) could be linearly related to char yield as measured by Thermogra-vimetric analysis under specified conditions. The authors computed the char yield of various polymers with the help of structural parameters and found general low flam-mability linearly dependent on minimum thermal decomposition. Later on, some cases were noted in which low thermal stability and low flammability were found simultaneously in the polymeric substances. This raises a big question mark on the reliability of thermal decomposition of polymers and its correlation with the LOI test for predicting fire performance of polymeric materials [27, 28]. Later on, Lyon and Walters [29] gave a pathway to the preliminary prediction of fire performance of the polymers from heat release data derived from thermoanalytical data. Heat releasing capacity and the rate of heat release can be considered as important parameters to predict the fire performance of the material. Cone calorimetric analysis is considered one of the reliable techniques to comment on fire performance [30–35].

The fire community pushed for dependable bench-scale instruments to assess material flammability depending on heat release rate in the late 1970s and early 1980s. Heat release rates were believed to be the most dependable and accurate indicator of a material's flammability. The Cone Calorimeter, developed by NIST's Fire Research Division (formerly known as the Center of Fire Research at the National Bureau of Standards), was launched in 1982 as the next-generation device for determining material flammability [36].

A cone calorimeter is a device that is used to examine the condensed phase fire behavior of tiny samples of diverse materials. In the area of fire safety engineering, it is frequently utilized [37]. It collects information on the sample's burning characteristics, such as heat release rate, ignition time, combustion products, mass loss and other factors [38]. Huggett's principle states that the gross heat of combustion of any organic substance is proportional to the quantity of oxygen required for burning. This concept is used to calculate the heat release rate [39]. The Cone is a fire-testing device that works on the concept that the amount of heat generated by a burning sample is proportional to the amount of oxygen used during combustion. The intensity of a fire, such as fire development rate, is directly proportional to the amount of heat a sub-stance generates.

4.4.1 Instrumentation setup

As it offers a lot of information with tiny samples, the cone calorimeter is the most commonly used device for studying the fire behavior of materials. ASTM E1354 and ISO 5660 have been used to standardize the procedure. The fundamental idea is to detect the decreasing oxygen content in flammable gasses of a sample ($100 \times 100 \times 4$ mm^3) exposed to a certain heat flux (10–100 kW/m^2) [40]. A cone calorimeter is made up of several components and gadgets. Several parameters such as temperature, gas flow, mass and concentration are measured, logged, set, and adjusted by these parts when used together. The sample is mounted on a metal sample holder that is mounted on the load cell. During the experiment, the load cell records the sample's weight. Sample holders can be divided into two groups. The sample holder's edges may be open or closed, depending on the sample. If the sample is intumescent (that is, it swells and develops a protective char layer), a wired grid can be used to maintain it in place as it swells. When testing horizontally and with an edged framed sample holder, the wired grid is used. Below the cone heater is a spark igniter, which is placed directly above the sample surface. When the sample is heated, this ignites the flam-mable vapors that are escaping. The igniter is switched off and shifted to the side after

the entire sample area has burned. A water-cooled heat flux measurement instrument is positioned on the sample surface level before the experiment during calibration. The temperature of the cone heater is then adjusted until the desired heat flux is achieved at the specimen's surface. The cone is usually positioned horizontally, although it can also be put vertically.

The form of the heater inspired the name of this testing device. Over 3 m long resistive heating wire packed with magnesium oxide refractory is coiled into a conical form to make the heater. The flue gases out from the flaming sample are gathered in an extraction chamber above the heater. The flow rate of combustion products is controlled by a flue gas fan installed in the flue gas line. In the flue gas line, the gas sampling ring is located before the fan. The gas sampling in the circle is first passed by two filters to remove pollutants, then via a cold trap and a drying agent to eliminate any potential water before contacting the gas analyzers. A smoke measuring system is located between both the gas sampling ring as well as the fan. A laser photometric beam detects the quantity of smoke generated [41, 42].

4.4.2 Important fire parameters determined by cone calorimeter

4.4.2.1 Heat release rate and Total heat release

Cone Calorimeter measures heat release rate at different incident heat fluxes. This measurement is based upon the principle that a nearly constant amount of heat is released per unit mass of oxygen consumed [43]. The concept of heat release rate calorimetry is simply the measurement of the rate at which a known weight (volume or area) of a carefully prepared specimen releases heat when exposed to a prescribed and controlled heating environment. Oxygen concentration in the flue gases is used to calculate the heat release rate. Oxygen consumed during combustion has been shown proportional to the heat released from a fuel [44]. Heat release rates and total heat released for a given time period are obtained from the strip chart record of changes in the fuel consumption required to maintain a constant temperature in the exhaust gases from the particular instrument. The traces record the decrease in fuel flow that is equivalent to the fuel contribution of the sample. An instantaneous heat release rate, expressed in watts/cm^2 area of the sample, is obtained by multiplying the net scale deflection at a given time by the calibration constant. The total heat release for a particular time interval was derived by graphic integration of the area under the curve between the desired time limits. The convention was established that zero time was the moment the sample "saw" the radiant flux in the exposure chamber (i.e., time was measured from the moment the door was closed). The average heat release rate for a given time period was obtained by determining the area under the curve (i.e., total heat released) and dividing by the time period of interest. In practice, this averaging is done for each successive 1-minute interval under the curve.

The heat release rate is an important parameter in determining the fire characteristics of materials. Holdsworth et al., in the year 2014 investigated different metal oxalates blended with Polyamide 6, 6 using cone Calorimetry at a heat flux of 50 kw/m^2 for peak heat release rates and found lowest PHRR (peak heat release rate) for halogenated composites showing better flammability resistance [45]. The authors further added metal complexes in Polyamide 6, 6 and obtained data for various fire parameters such as ignition time, THR, HRR and PHRR [46]. These formulations were also investigated for LOI values which were not found in agreement with Cone calorimetric data. This disagreement in LOI values with cone calorimetric data was

indicated towards cross-linking and increment in char formation. Hence LOI values cannot be considered as sufficient data to comment upon fire characteristics of the material. Ramgobin et al., used the Cone Calorimetry technique to observe metal salen complexes as a fire retardant for thermoplastic polyurethane (TPU) [47]. Salen complexes of nickel, manganese and copper were fabricated in TPU and the comparative curves of heat release rates gave an idea of the best formulation based on PHRR and THR values. The lowest is PHRR and HR, better is the flame resistance performance of the sample. The width of the peak implies the release of combustibles at a broad range of temperature. Therefore, from THR data, it was found that formulations of TPU incorporated with metal complexes have at least 15% lower HR values than neat TPU. Recently, copper salen complexes were incorporated in PVC samples to study their flame resistive effect. It was found that from the cone calorimetric data that the complexes were improving fire retardance of PVC sheets only at 1 phr concentration. A decrease of 15.41 MJ/m^2 in total heat released was observed in PBr0 and of 10.42 MJ/m^2 in PBr1 sheets for the control sample sheet [48] as represented pictorially in **Figure 2**.

4.4.2.2 Char

The properties of the char and the underlying sample determine the time at which the stresses in the char layer are relieved by cracking and spalling, with subsequent reduction in the protective effects of the char layer. The finer and more uniform the texture of the sample, the longer the char layer is expected to remain intact. Schaffer showed that the charring rates of samples generally decrease as the density increases [49]. McLean attributed this effect to the variation of thermal diffusivity with density
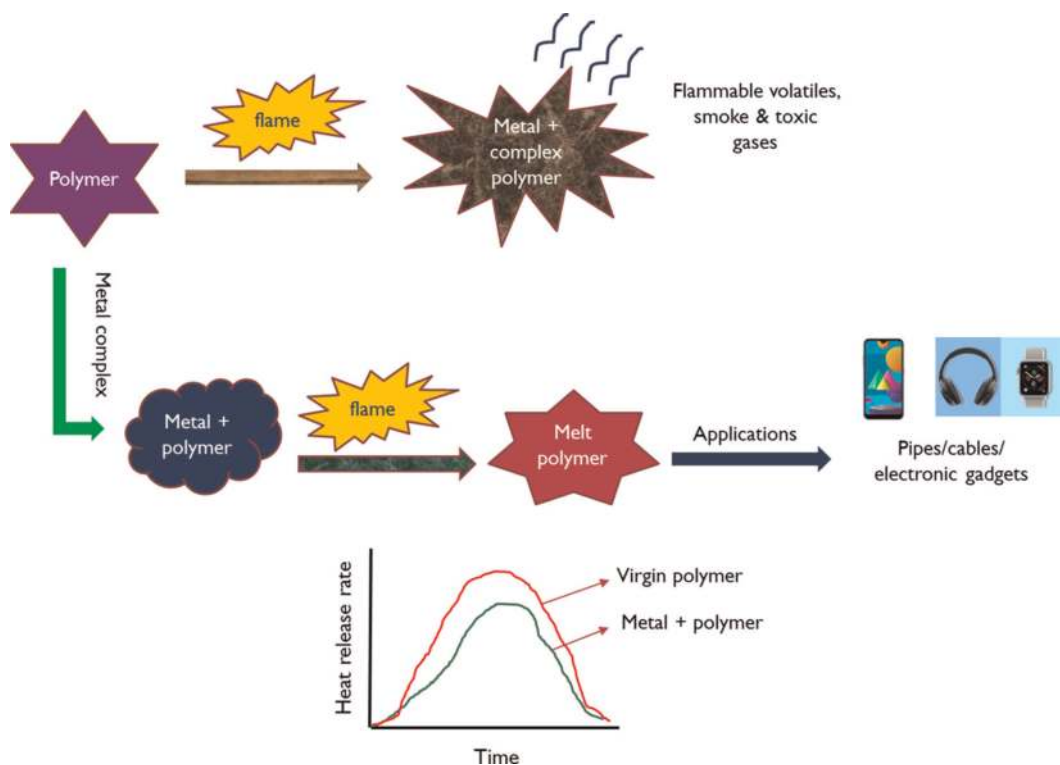
Figure 2.
Effect of fire retardants on the heat release rate.

[50]. Thermal diffusivity, a measure of how quickly a material absorbs heat from its surroundings, is defined as the ratio of thermal conductivity to the product of density and specific heat: a = (k/pC).

4.4.2.3 Mass loss rate

The rate of weight loss of a burning material has also been used as a measure of the rate of energy released during burning. The weight loss rate is directly related to the rate of heat release only in those cases where fuel composition and combustion efficiency remain constant as burning progresses [51].

4.4.2.4 Smoke release

Cone calorimeter also provides smoke production data which is often unused for predicting fire characteristics of polymers. Sonnier et al., in the year 2019 proposed new insights into the investigation of smoke production using a cone calorimeter [52]. The authors correlated smoke release to heat release using pure and flame retarded polymers, the carbon fractions and the presence of aromatic groups.

4.4.3 Interpretation of results by cone calorimeter

The cone calorimeter is an appropriate technique for evaluating the smoke suppression and fire retardance behavior of polymer samples. The cone calorimetric test is one of the most accurate methods for determining the fire resistance of polymeric materials. The overall heat release rate (HRR), peak heat release rate (PHRR), ignition duration, char yield, and smoke concentration of the sample are all determined by this test. The fire parameters can be determined by bench-scale test methods illustrated as arbitrary scale evaluation and FO- category models. The least correlation was found between these two test methods when applied on PVC panels and other polymeric materials.

Fire behavior analysis can be done through Petrella arbitrary scales, Östman and Tsantaridis model and Hansen & Hovde model [53, 54]. Two parameters, the flash-over propensity 'x' (in kW/m^{2}s) and THR 'y' (in MJ/m^2) were proposed by Petrella for studying the contribution of the materials to flashover and thermal contribution [55]. The x and y values are helpful in determining the risk factor of the materials to thermal and flashover contribution under fire hazards.

$$x = \frac{peak(HGR)}{t_{ig}} \tag{1}$$

$$y = THR = \int_0^\infty HGR(t)dt \tag{2}$$

The total heat released (gross heating value, in MJ/m^2) is calculated by Eq. (3) and average heat generation rates; T60 (over 60 s After ignition), T180 (over 180 s After ignition) and T300 ((over 300 s After ignition (kW/m^2)) are calculated using Eqs. (4)–(6) respectively.

$$THR = \int_0^\infty HGR(t)dt \tag{3}$$

$$\overline{T}_{60} = \frac{1}{60} \int_{tig}^{tig+60} HGR(t)dt \tag{4}$$

$$\overline{T}_{180} = \frac{1}{180} \int_{tig}^{tig+180} HGR(t)dt \tag{5}$$

$$\overline{T}_{300} = \frac{1}{300} \int_{tig}^{tig+300} HGR(t)dt \tag{6}$$

Östman and Tsantaridis presented a relatively simple empirical linear regression model for the prediction of time to flashover in the room corner test. The model is based on empirical data, and was found to predict time to flashover with good accuracy for several products. Cone calorimeter results from tests at incident radiation heat flux of 50 kW/m^2 are used as input data to this model, which also requires information about the mean density of the tested product.

Cone calorimeter data can be used to derive useful information on studying the fire behavior of polymers. Based on the data and rational developed models, prediction of flashover time leading to fire behavior classification is feasible. Pyrolysis dynamics will help the understanding of the fundamental thermal behavior of materials leading to macroscopic fire behavior [56].

5. Advantages of cone calorimeter

Cone calorimetric test is one of the reliable tests to comment on the fire performance of polymeric substances. This test gives precise results about total heat release rate, peak heat release rate, ignition time, char yield, the smoke concentration of the sample. The best parameter for predicting fire hazard of a polymer is HRR (Heat Release Rate) in flaming combustion, although determination of HRR is a complex task for solid samples as it depends upon the heat flux, sample thickness, sample position, ventilation, etc. The justification for the widespread use of LOI test in its present form in flammability research is questionable. Aside from convenience and precision, it has many unfavorable features. Since it is a downward-burning test, this is a fire configuration of minor importance in real fires. Moreover, in the LOI test, characteristics of the heat transfer and rate of burning are fundamentally different from those in the much more important upward burning configuration. The LO1 has the unreal feature of being run at oxygen concentrations usually above the normal oxygen content of air, conditions which are probably important only in some space vehicles and oxygen contents in hospitals. It has poor thermal coupling between flame and substrate, and it is prone to severe disturbance by melt-flow phenomena. These are the ignition conditions well defined in the ASTM D2863 version of the oxygen index test. Probably the most severe deficiency of the LOI, is the lack of correlation with heat release results, is that it does not, except in unusual cases, predict the real fire performance of materials. It is still not clear whether any of the fire tests would provide enough energy feedback to stronger fire, to be able to predict full-scale fire performance, but they might provide better guidance for research and development than the LOI [57].

The development of a fire in the flame spread and sustained burning stages is dependent upon the rate of heat release and is not inherently related to the total heat that would be released if the specimen burned to completion. An important aspect of

the flame spread and sustained burning stages is the production of smoke and toxic gases. These stages, culminating in flashover when the fire is in an enclosure, are dominant as far as life safety is concerned. The important aspect of the third stage is fire endurance, in which efforts to contain the fire and save the structure depend in part upon the rate of combustion or rate of heat release of building contents and materials of construction. Test methods to evaluate the performance of building materials for flame spread, smoke production, and fire endurance have assumed major importance in efforts to control fires. The rate of heat release is a relatively new criterion for evaluating fire behavior and is expected to assume similar importance in research, material assessment, and building regulations.

Cone Calorimetry is a reliable technique that involves the determination of one specific physical, chemical, or behavioral characteristic of a material, product, or system. A system test would characterize the overall behavioral reaction of a material, product, or system with the environmental as well as internal variables which influence its performance. A system test involves interactions between the material, product, or system with its surroundings. Heat release rate test methods are among those classified as system tests. Cone Calorimeter predicts real-scale fire behavior of materials. The obtained results are used in the ranking of products for fire performance and assist in the development of new fire-resistant materials and products [58]. The physical observations that can be visualized during the tests are surface rising, deformation owing to intumescences, residual stresses, the collapse of structures, char cracking, char development and cracking through bubbling and sparking, creation of solid crusts, tiny explosions, surface layers or bubbles, afterglow, and so on. Cone Calorimeter has been used widely for a variety of samples including ornamental plants [59], wood [40], textiles [28] and polyvinyl chloride products [48, 60]. Moisture content, physical properties and chemical composition are all variables that impact the flammability of live plants and can be analyzed with the help of Cone Calorimeter.

6. Conclusions

Polymeric materials are normally compounded with various compounds to enhance their properties such as tensile strength, flexibility, stability and fire resistance to be used in specified applications. These properties are analyzed using efficient analytical techniques to rank the polymeric materials. Fire resistance property is analyzed with the help of Underwriters Laboratories Test standard UL-94, LOI test, Thermal Gravimetric analysis and Cone calorimetry. Underwriters Laboratories Test is specially designed for the evaluation of those plastic materials that used in appliances, in response to a small, open flame or radiant heat source under controlled laboratory conditions and it cannot be used for polymeric materials used in construction, wall and floor coverings or other decorative objects. LOI test results are only valid for specified conditions indicated in the test procedure and cannot be applied to real scale conditions. Thermogravimetric analysis predicts the volatile components evolved during the decomposition of the polymeric sample along with decomposition temperature; however, the fire characteristics of the polymer can be studied with the help of Cone calorimetric analysis. Cone calorimetry is a key tool for the real scale analysis of fire-resistant polymers. The flame profile data suggests the fire hazards and is important for development of new fire-resistant materials.

Conflict of interest

The authors declare no conflict of interest.

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