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Flame Retardants for Textile Materials

By

Asim Kumar Roy Choudhury



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Preface

The words *fire* and *flame* have been curses for millions of people throughout the ages all across the Globe. Those who have lost their homes, belongings, and relatives by fire cannot forget these events for the rest of their lives. In many cases, fires are silent killers, killing people during sleep. With tremendous efforts by firemen and firefighters, fires may be extinguished in time, only leading to realization that many unfortunate individuals have lost their lives, or have been injured, by fire. The most common natural textile materials (namely, cotton, flax, and jute), wood, and many household materials are cellulosic in nature. All of them burn quickly, spread rapidly, and release toxic gases. People have realized this since ancient times, and the flame retardancy concept has been applied using borax and other flame retardant (FR) materials. With the advent of synthetic fibers and polymers, this problem was intensified due to their poor absorbency, caused by hydrophobicity. They also melt easily, and the dripping of melt drops results in severe injury to the burn victim. The period from 1960 to 1980 saw the development of many of well-established flame retardant materials. During the last few decades, the knowledge about the toxicity and environmental impact of chemicals has rapidly grown, and people have become more aware of potential dangers associated with FRs. In February 2003, the Restriction of Hazardous Substances Directive (RoHS) was adopted by the European Union. This was followed by banning many FRs, mainly halogen- and halogenated-phosphorous FRs by various countries. The researchers put their best efforts to find eco-friendly substitutes and a large number of research works came into light. A good number of books on flame retardancy have been published in the last two decades, but most of them are devoted to specific or limited fields of flame retardancy. I came across a very large number of research publications on various chemicals and substrates. Hence, I decided to write a book covering broader topics. In this book, flame and fire retardancy of textiles and various nontextile materials (e.g., plastics, resins) are discussed which may help researchers to find newer FRs for the textile materials or vice-versa.

This book consists of 10 chapters. Chapter 1 discusses the hazards caused by fire from a historical perspective. From ancient times until the present, many cities in all parts of the world are ruined by fire; fire hazards are very common in cities and thousands of people are burned and die every year. Extinguishing fires is the job of the fireman or firefighter. Most textile materials are flammable and continue burning, even if they are taken away from fire or flame. Moreover, people who are rescued from fire die because of severe burns from burned garments; inhaling toxic gases released by burning; melting and dripping of polymers; and suffocation due to oxygen shortage. Various fire-related aspects, such as combustion, ignition, charring, and flammability are discussed in this chapter.

Chapter 2 discusses thermal and flammability properties and their variations among various natural and manmade textiles. Flammability test methods measure how easily materials ignite, how quickly they burn, and how they react when

burned. A large number of flammability tests are in use, and may be classified into five groups: ignition tests (positioning test samples in vertical, horizontal, and inclined position); reaction to fire tests (how easily fire grows and spreads); application-based tests, i.e. performance of firefighters' clothing; radiant energy tests, i.e., testing on manikins in flash fire scenarios; and scientific assessment of thermal and flammability parameters such as limiting oxygen index (LOI), which measures minimum % oxygen in air required to initiate combustion, and is the most simple, effective and popular measure to express flammability. Various standard methods of flammability tests are discussed very elaborately.

Chapter 3 describes fibers and polymers that are self- or inherent flame retardants (IFR). They do not need any further treatment to protect from fire. Their fire retardancy property is durable and can prevent hazards that are caused during finishing. Wool is naturally flame retardant, while man-made fibers (including synthetics) can be made FR by adding FR chemicals during fiber spinning or by copolymerization. Most important IFR fibers are aramid fibers and polyvinyl chloride polymers.

In Chapter 4, a variety of flame retardants are described. Flame retardant finishes are chemicals which are added to combustible materials to render them resistant to ignition. Various FRs are classified according to characteristics such as chemical structure, and durability. Mineral-, halogen-, phosphorous-, nitrogen-, and silicon-based char-forming intumescent, reactive, and hybrid organic-inorganic FRs are described and their operating principles are explained.

Chapter 5 is devoted to the most economic, most popular, and at the same time most controversial halogen-based FRs. They are widely used in consumer products because of their low impact on other material properties, and the low loading levels required to meet the required flame retardancy. However, halogen-based FRs have raised concerns due to their persistency, their bio-accumulation on living organisms, and their potential toxic effects on human health. As a result, most of them are banned or awaiting substitution by more eco-friendly FRs.

Eco-friendly and versatile, phosphorus-based FRs are described in Chapter 6. Inorganic phosphorous derivatives, mostly nondurable or semidurable, entails primarily phosphoric acid and its ammonium salts. Organophosphorous FRs include aliphatic and aromatic phosphines, phosphine oxides, phosphites, phosphates, phosphinites, phosphinates, phosphonate esters, and phosphonium salts; they promote char formation and act in condensed mode. Nitrogen acts as a synergist in some cases, and some P-N-Si compounds are popular as FRs. These compounds are successfully used, both as additives and as reactive flame retardants for a wide variety of polymer-based systems, namely cotton, rayon, wool, polyester, polyamide, polyacrylic, epoxy resin, polyurethane, and polystyrene. They have also wide applications in nontextile sectors, such as resin, and plastics.

Chapter 7 is devoted to intumescent FRs (IFRs). Researchers showed that the sustainable materials obtained from natural resources can char on burning and form protective layer(s) to make a barrier between substrate and flame/

burning gases. These intumescent FRs (IFRs) are economic, efficient, and easily applicable on various substrates such as textile fibers, resins, and foam. The intumescent behavior resulting from a combination of charring and foaming of the surface of the burning polymers is being widely developed for fire retardance because it is characterized by a low environmental impact. Research work in intumescence is very active. New commercial molecules, as well as new concepts, have appeared.

Chapter 8 examines nanocomposites. The composites are made from two or more constituent materials; at least one of the phases shows dimensions in the nanometer range. These are high-performance materials that exhibit unusual property combinations and unique design possibilities, and are thought of as the materials of the 21st century. Fire retardant, carbon-based nanomaterials are made from graphene, carbon nanotubes (CNTs), and carbon black (CB). Layered aluminosilicates, also popularly described as clays, are one such type of filler; they are responsible for a revolutionary change in polymer composite synthesis, as well as for transforming polymer composites into fire retardant polymer nanocomposites.

Chapter 9 discusses flame retardancy of synthetic fibers. In the absence of functional groups, synthetic fibers are less prone to charring. Furthermore, hydrophobicity and melting are the two disadvantages of making synthetic fibers flame-resistant. To address these problems, back-coating and intumescent FRs are alternative ways to make FR synthetic fibers. The thermally-stable FRs can be added in melt or solutions of polymer before spinning, or may be applied as back-coating. Various FRs suitable for synthetic fibers and their methods of application are discussed in this chapter.

Finally, Chapter 10 explores environmental aspects of FRs. While FRs could ensure the production of fire safety products, many of them are not safe to human beings. There are more than 175 different types of FRs in the market, which contain bromine, chlorine, phosphorus, nitrogen, boron, and antimony compounds or their combinations of inorganic and organic origins. Flame retardant products do not easily obtain eco-labels. The introduction of novel, sustainable, natural-based, intumescent FR systems represents a major scientific and technological challenge. This is expected to make a breakthrough in the production of flame-retarded polymer materials that would follow the principles of *eco-designing*.

Who will read this book? Students who read portions of this book will gain a basic understanding of principles and issues related to fire retardancy, the knowledge on how FR materials and associated application methods changed with time and how their performances can be tested in different flaming environments. Researchers in one application field may find how a FR product used in other fields can be developed for their own applications. Developers, including quality assurance professionals, will find a variety of techniques which can fulfill the FR requirements of their products per specific national requirements that are dictated by prevailing national laws. Technical managers will find a coherent approach to prevent loss from burning and improve FR quality of their products. Therefore, a diverse reading audience should benefit from the contents of this book.

I would like to thank all those who helped to make this book possible. My special thanks to Professor Richard Murray, Chairman, Textile Institute Publication committee who inspired me to write a complete textbook on flame retardant applications in textile materials. I also wish to thank the reviewers who gratefully gave so much of their time to review each chapter, as well as those at CRC Press to make this project successful.

Asim Kr. Roy Choudhury

Author Biography



Dr. Asim Kumar Roy Choudhury is presently working as principal, KPS Institute of Polytechnic, Hooghly (W.B.) India. He retired from the post of Professor and HOD (Textile), Government College of Engineering and Textile Technology, Serampore (W.B.), India. He has over 35 years of experience in textile coloration and finishing in academia and in industry, he has written numerous research papers and acted as reviewer for several reputable international journals and presented papers in several international conferences. He is a fellow and silver medalist of

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Dr. Choudhury's first book, *Modern Concept of Color and Appearance*, was published in the year 2000 and the second book, *Textile Preparation and Dyeing*, was published jointly in the year 2006 by Science Publishers, New Hampshire, USA and Oxford & IBH Publishing Co. Pvt. Ltd. New Delhi. Two books—*Principles of Colour and Appearance Measurement, Volume 1: Object Appearance, Colour Perception and Instrumental Measurement* and *Volume 2: Visual Measurement of Colour, Colour Comparison and Management*—were published by Woodhead (UK) in the 2014. Dr. Choudhury also authored *Principles of Textile Finishing*, published by Elsevier in the year 2017. In addition, he has contributed chapters in 27 edited books, published by reputed publishers, such as CRC Press, Woodhead, Elsevier, Springer, Pan Stanford, Apple, and Academic Press. Future project: Principles of Textile Printing (CRC Press).



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1 Fire Hazards and Associated Terminology

1.1 INTRODUCTION

About 100,000 people around the world die in fires each year. Fires account for ~ 1% of the GDP within EU. Human beings have been afraid of fire since they started making buildings from wood rather than from stone. In fact, firing infernos have been so common throughout history that nearly every major city in the world has been largely burnt to the ground at some point. Some of these cities have been burned on multiple occasions. Constantinople burned many times between 406 and 1204 only to be, like a damaged ants' or termites' nest, rebuilt each time, thereby setting the stage for the next great inferno. During historical wars, many of these fires were man-made, but most of them were due to natural calamity combined with poor construction methods, excessive use of flammable building materials, and/or the human incapability to fight really large blazes. Some of these fire incidences will be remembered forever because of their size and their dominant roles in shaping historical events. Which fires are these? The list of the top ten most destructive, most famous, or most historically significant non-war-related infernos in history are as follows (Danelek, 2011):

1. Rome (64 AD),
2. London (1212),
3. London (1666),
4. Chicago (1871),
5. Boston (1872),
6. Peshtigo, Wisconsin (1871),
7. San Francisco (1906),
8. Halifax, Nova Scotia (1917),
9. Tokyo, Japan (1923), and
10. Texas City, Texas (1947).

Among common household materials, wood is very fire-prone; it is made of cellulose, the same component of cotton and many other textile materials. These textile materials are, therefore, also fire-prone. Recently (2012–2013), about 800 people were injured in fires in garment and textile factories in Bangladesh, most of which were unreported, according to the data compiled by international labor campaigners. The high number of casualties raises concerns about the slow pace of change in this politically unstable southern Asian state, where more than 1,130 people died in a garment factory building.

The building caught fire on November 24, 2012, and collapsed in April 2013. The tragedy was the worst industrial accident anywhere in the world for a generation (Burke, 2013).

The material mainly responsible for the development of the fire in 25% of all dwelling fires, and the item first ignited in 26% of all dwelling fires in 2017–18 was “Textiles, upholstery and furnishings” (The Home Office, 2018). The former caused 46% of all fire-related fatalities in dwellings. This proportion is normally higher (64% in 2016–17).

The particular hazard posed by burning textiles, especially those based on natural cellulosic fibers such as cotton, jute and flax (linen), was recognised during early civilisations and such salts as alum had been used because they reduce their ignitability, and thereby confer flame retardancy. A major problem arises because most of the polymers on which textile materials are based are organic and thus flammable. In the United Kingdom alone, some 800–900 deaths and roughly 15,000 injuries result from fire each year. Most of the deaths are caused by inhalation of smoke and toxic combustion gases, with carbon monoxide being the most common cause, while the injuries result from exposure to the heat that evolves from fires.

The Home Office (UK) has responsibility for fire services in England. The vast majority of statistics produced by the Home Office are for England, but some clearly marked, tables are for the United Kingdom and are separated by national data. In the past, the Department for Communities and Local Government (which previously had responsibility for fire services in England) produced releases and tables for Great Britain and at times the UK.

The annual UK Fire Statistics 2018 (The Home Office, 2018) contains some of the most comprehensive documents available and provides information that is perhaps representative of a European country with a population of about 55 million. For every million people in England, there were 6.0 fire-related fatalities in 2017–18.

Smokers’ materials (such as lighters, cigarettes, cigars, or pipe tobacco) were the source of ignition in 7% of accidental dwelling fires and in 9% of accidental dwelling fire nonfatal casualties in 2018–19. In contrast, smokers’ materials were the source of ignition in 20% of fire-related fatalities in accidental dwelling fires in 2017–18.

The most common cause of death for fire-related fatalities in 2017–18 (where the cause of death was known) was “overcome by gas or smoke”, given as 30% (99 fire-related fatalities) of fire-related fatalities. This was lower as a proportion compared with 2016–17 (38%). This was followed by “burns alone” (24%; 80 fire-related fatalities) and the combination of “burns and overcome by gas and smoke” (15%; 50 fire-related fatalities) in 2017–18.

Flame retardancy is an important characteristic of textile materials that protects consumers from unsafe apparel. Firefighters and emergency personnel require protection from flames. Floor coverings, upholstery, and draperies also need protection from fire, especially when used in public buildings. The military and airlines industries have multiple needs with respect to fire-retardancy (Schindler and Hauser, 2004).

Horrocks (2011) reviewed the state of the art for the different commercially available flame retardants (FRs) for textile materials, during the following periods.

1.1.1 1950–1980

The fire safety issue and federal regulations in European countries and in the United States dictated the development of new, effective flame retardant chemicals to reduce the fire hazards and meet the product flammability standards. The first patent on organo-phosphorus FRs for cellulosic textiles (i.e., cotton) was accepted during this period. Inherently FR synthetic fibers bearing aromatic structures were also developed during this “golden period” of flame retardant research.

1.1.2 1980–LATE 1990s

This was a lean period in research on FRs.

1.1.3 2000–ONWARD

Phosphorus-based char-former flame retardant additives were developed during this period. The efforts were made to find the possibility of replacing bromine derivatives with other less toxic and efficient products. The outstanding potential of nanotechnology for conferring flame retardant features to fibers and fabrics was discovered. The preformed nanoparticle suspensions, single nanoparticles or nanoparticle assemblies, and hybrid organic–inorganic structures were proved prospective roles in FRs (Yu et al., 2013).

In 2013, the world consumption of flame retardants was more than 2 million tonnes. The construction sector is the most commercially important application area, requiring for instance, flame retardants for pipes and cables made of plastics. In 2008, the United States, Europe and Asia consumed 1.8 million tonnes, worth US\$4.20–4.25 billion. According to Ceresana, the market for flame retardants is increasing due to rising safety standards worldwide and the increased use of flame retardants. It is expected that the global flame retardant market will generate US\$5.8 billion. In 2010, the Asia–Pacific region was the largest market for flame retardants, accounting for approximately 41% of global demand, followed by North America and Western Europe (Ceresana, 2019).

1.2 FIRE HAZARDS IN TEXTILE INDUSTRY

The textile industry produces materials made of various natural and artificial fibers. It is one of the oldest and most important branches of industry. However, textile materials themselves are flammable. In addition, the textile industry deals with numerous flammable materials and chemicals. The chances of catching fire are very high along the entire textile production chain and easily cause fires and dust explosions.

A fire may break out at any place including houses, residential and commercial buildings, restaurants, cinemas, sports stadiums, jungles, industries, and mills (particularly those like textile mills and industries where raw material or finished products are combustible). In the case of a fire, there may be several reasons related to its initiation and propagation.

Statistics have clearly indicated that textile products have been significantly involved in fire hazards involving human lives. Such products include clothing (including oversuits, undergarments, work wear, and suiting), bedsheets, floor coverings, upholstered textiles in seats, bedding, and home furnishings. Human beings are always in close contact with these textile materials.

Since textiles made from natural fibers are flammable or combustible, they can provide a means of initiating fire. Actual fire cases had shown that textile and clothing were the main items causing injury and death to human lives.

Public concern over the fire-retarding textiles for the protection of human lives and property appeared in the form of legislation. An early example is seen in the United States. The Flammable Fabric Act, 1953, was the first major piece of such legislation.

In the 1970s and 1980s, there were significant discussions on textile flammability legislations in the United States and Western Europe. These were directed at control of fire hazards to lives and other valuables.

In 1988, there were 15,080 textile-implicated fires in the United Kingdom, and textile-related fires caused 4,000 casualties and 495 deaths. The United Kingdom upholstery furniture safety regulations were also introduced in 1988 to exercise a control for reducing textile flammability hazards. The legislations employed in the United Kingdom covers nightwear, upholstered furniture, and toys, and specify how products are to be tested for safety purposes and for evaluating performance, labeling for safe use, and the nature of materials used. An equally important subject, along with the enforcements of legislation is the standard testing procedure designed for evaluating the degree or level of flame retardancy offered by the product for general and specific purposes.

A standard testing procedure usually incorporates the overall conditions that would be experienced by the textile item in an actual fire environment. Presently, several textile flammability testing procedures are available that can be used for a variety of textile products under specified application conditions. These products are apparel, upholstery, building materials, plastic toys, folding portable cots and, car racing suits.

British standard flammability tests are available for products such as curtain, carpets, and bedding, but statutory legislation does not exist for the use of these products in the domestic market.

It is a conventional practice for a retailer to specify a particular test to fulfill the requirements of safety and protection. The Consumer Protection Act 1975, the United Kingdom requires that all products sold in the United Kingdom must be fit for their purpose. In the United States, the Consumer Product Safety Act, 1972 is designed to protect the public from hazardous products.

The Consumer Safety Commission (CPSC), in conjunction with industry, has power to produce standards for product testing to protect masses from hazards.

This commission has recently amended the standard for the flammability of clothing textiles as originally issued in 1953. This is a voluntary standard for assessing the flammability risk of clothing textiles in terms of the ease of ignition and the speed of flame spread. This standard is aimed to reduce danger of injury and loss of life by introducing on-national-basis methods of testing and rating the flammability of textile products used for clothing (Uddin, 2019).

In the textile industry, nearly all materials being used are flammable to some degree. Some fire-prone substances are listed next (HSE, 2019):

- Loose materials, e.g., fabric offcuts or open layers of wadding—low density fibers burn very easily.
- Deposits of fluff and dust (fly)—dust on light fittings is a particular risk. Cotton fly is very hazardous when it is on fire.
- Oily fibers, such as contaminated wool or cotton; oil results from the spinning process.
- Rough, raw edges on rolls or bales—bales tend to burn on the surface and smoulder underneath—deep-seated smouldering in bales is almost impossible to put out from the outside.
- High piles of stock, especially if close together, can increase the speed at which a fire spreads.
- Traditional textile mills, constructed using a high amount of wood and with the presence of fly, means that a fire can spread rapidly.
- Flammable liquids that ignite easily or oxidizing agents that may make an existing fire more intense by fueling it with oxygen.

Some suggested precautionary steps to be followed to avoid fire hazards are listed next (HSE, 2019):

- Good housekeeping—cleaning up fluff and dust regularly, especially high ledges;
- Keeping offcuts in bins, preferably metal;
- Minimum storage in workrooms;
- Indirect heating in workrooms;
- Restricting smoking areas;
- Controlling heated work areas; and
- Storing raw materials and finished goods systematically with proper spacing—not randomly on the floor.

Some fire-prone areas in textile production units are discussed next (HSE, 2019).

1.2.1 CARPET MAKING

This involves the manufacture and storage of latex foam and rubber underlay and foam carpets. These can burn to produce enough smoke to classify the material as a highly flammable solid.

- A high level of sprinkler protection is needed where foam-backed carpets are stored.
- Traditional wool or nylon hessian-backed carpet is not particularly flammable.

1.2.2 SPINNING

During opening and carding, foreign bodies in fibers and baled raw material can come into contact with rotating metal parts of machinery and produce sparks or frictional heat. Natural fibers are more likely than synthetic fibers to contain foreign bodies.

Opening rags is a vigorous process and it is highly likely that they will contain foreign bodies, such as coins and metal buttons, that may cause a spark.

The spread of fire from opening machinery through ducting can be high; the spread of fire through the fiber delivery and trash recovery systems is also a fairly high risk. Automatic fire detection in a ducted system is essential.

Traditional spinning causes deposits of fly, and, if contaminated by oil, can be particularly flammable

1.2.3 WEAVING

The main hazard is ignition of fly by electrical faults, usually insulation failures caused by mechanical vibration. Modern looms are less susceptible to vibration.

Effective controls include good housekeeping and good maintenance of electrical systems and machinery.

1.2.4 FINISHING PROCESSES

These are processes that alter the physical characteristics of the cloth, either by

- Physical means, e.g., raising or milling, or
- Chemical means, e.g., crease resistance.

Processes involving a naked flame, e.g., flame bonding can cause smoldering.

The stenters used for thermal bonding are a common source of fires—smoldering in the finished reel of material can develop into a fire later. Also, if the material stops in the stenter, it is important for the heat supply to cut off automatically. Thermostats can also fail causing overheating.

Gas singeing, i.e., burning of projected fibers from the fabric surface by open flame may cause fire hazard.

Some causes for explosion are:

- Wool spinning: wool dust can cause explosions. Good housekeeping is essential and the dust in the carding machines should be controlled.
- Flocking: Ground flock (rather than precision cut) from mainly cotton, acrylic and nylon fibers, gives a higher risk of explosion. If

dispersed into the atmosphere, e.g., when cleaning down, it can cause an explosion and/or fire.

Burn injuries continue to be one of the leading causes of unintentional death and injury in the United States. Between 2011 and 2015, approximately 486,000 fire or burn injuries were seen at Emergency Departments. In 2016 alone, there were 3,390 civilian deaths from fires, which includes 2,800 deaths from residential structure fires, 150 deaths from non-residential structure fires 355 from vehicle fires, and 85 from outside and unclassified fires other than structure or vehicle fires. One civilian fire death occurs every 2 hours 35 minutes. The lifetime odds of a U.S. resident dying from exposure to fire, flames or smoke are 1 in 1,498.

The primary causes of burn injury include fire-flame, scalds, contact with hot object, electrical and chemicals.

(ABA, 2002)

1.2.5 BURN INJURY

A burn is an injury to the skin or other organic tissue primarily caused by heat radiation, radioactivity, electricity, friction, or contact with chemicals. Thermal (heat) burns occur when some or all of the cells in the skin or other tissues are destroyed by:

- Hot liquids (scalds),
- Hot solids (contact burns), or
- Flames (flame burns).

Burns are a global public health problem, accounting for an estimated 180,000 deaths annually. The majority of these occur in low- and middle-income countries and almost two thirds occur in Africa and Southeast Asia.

In many high-income countries, burn death rates have been decreasing, and the rate of child deaths from burns is currently over seventimes higher in low- and middle-income countries than in high-income countries.

Nonfatal burns are a leading cause of morbidity, including prolonged hospitalization, disfigurement, and disability, often with resulting stigma and rejection.

- Burns are among the leading causes of disability-adjusted life-years (DALYs) lost in low- and middle-income countries.
- In 2004, nearly 11 million people worldwide were burned severely enough to require medical attention.

The burn statistics of some countries are as follows (WHO, 2018):

- In India, over 1,000,000 people are moderately or severely burned every year.
- Nearly 173,000 Bangladeshi children are moderately or severely burned every year.

- In Bangladesh, Colombia, Egypt, and Pakistan, 17% of children with burns have a temporary disability and 18% have a permanent disability.
- Burns are the second most common injury in rural Nepal, accounting for 5% of disabilities.
- In 2008, over 410,000 burn injuries occurred in the United States, with approximately 40,000 requiring hospitalization.

1.3 FIRE

A fire is defined as any combustion that is not under control. The development of fire can be subdivided into four different phases: ignition, propagation, development, and decline. Since the flames are still contained during the first two phases and the ambient temperatures are changing, the risk of damage can be relatively limited. The limit of this risk is tied to the duration of these two phases, which is determined by the geometry and ventilation of the area, and the amount of contact between the combustible source, the oxygen in the air, and the ignition. Up to the point when the flashover is reached, the mix of inflammable gases propagates the flames very quickly. The average temperature rises (over 1,200°C) and all combustible material burns and the fire increases. The decline or extinguishing phase begins after the maximum temperature is reached. The fire is considered extinguished when the ambient temperature drops around 300°C.

Fire is the rapid oxidation of a material in the exothermic chemical process of combustion, releasing heat, light, and various reaction products (NWCG, 2009). Slower oxidative processes, such as rusting or digestion, are not included by this definition.

Fire generates heat because the conversion of the weak double bond in molecular oxygen, O₂ to the stronger bonds in the combustion products carbon dioxide and water releases energy (418 kJ per 32 g of O₂); the bond energies of the fuel play only a minor role here (Schmidt-Rohr, 2015). At a certain point in the combustion reaction, called the ignition point, flames are produced. The flame is the visible portion of the fire. Flames consist primarily of carbon dioxide, water vapor, oxygen and nitrogen. If a large quantity of heat is generated, the gases may become ionized to produce plasma (Helmenstine, 2009). The color of the flame and the fire's intensity depend on the substances alight, and any impurities outside.

Fire is an important process that affects ecological systems around the globe. The positive effects of fire include stimulating growth and maintaining various ecological systems.

Fire has several negative effects, such as hazard to life and property, atmospheric pollution, and water contamination. If fire removes protective vegetation, heavy rainfall may lead to an increase in soil erosion by water (Morris and Moses, 1987). Also, when vegetation is burned, its nitrogen is released into the atmosphere, unlike elements such as potassium and phosphorus, which remain in the ash and are quickly recycled into the soil. This loss of nitrogen caused by a fire produces a long-term reduction in the fertility of the soil, which only

slowly recovers as nitrogen is fixed from the atmosphere by lightning and by leguminous plants such as clover.

Fire has been used by humans in rituals; in agriculture, for clearing land; for cooking, generating heat and light, signaling, propulsion purposes, smelting, forging, incineration of waste, and cremation; and as a weapon or mode of destruction.

1.4 BURNING PROCESS

Fire initiation and propagation in textiles are mainly due to the formation of various gases and liquids during burning. The flame and heat resistance of textiles is concerned with the flammability of such materials, i.e., whether flammable or nonflammable, and the ability of these materials to reduce the transfer of heat from a high-temperature source, either by direct contact (conduction/convection) or via radiation. The required flame and heat resistance of a textile product depend on its end-uses in particular applications within a given textiles sector.

The burning process of textiles involves the release of heat, decomposition of the material, combustion, and propagation of the flame. The decomposition of the material is explained as the breakdown of the hydrogen bonds that make up the composition of the fabric. The fabric is broken down into gaseous liquid and solid composites, which further fuel the combustion process.

The burning of material is a complex phenomenon. It involves processes such as heat transfer and thermal decomposition. For synthetic fibers, the thermoplastic behavior adds to the effect. While burning of the cellulosic textile materials, the combustible vapor is generated, and char is formed.

Carbon and oxygen react to form carbon monoxide (CO); it is an exothermic reaction and energy liberated is 26.4 kcal. The char becomes ash in an afterglow process by conversion of CO to CO₂ in the presence of excess oxygen. This reaction is also an exothermic reaction and the energy involved is 94.3 kcal, almost four times than that involved in CO formation. Because ignition, shrinkage, melting, dripping, and afterglow are involved, high energy and large amounts of heat evolve during burning.

When the textile material is heated, chemical and physical changes occur, depending on the temperature and chemical composition of the material. Thermoplastic fibers soften at glass transition temperature (T_g) and subsequently melt at melting temperature (T_m). At some higher temperature called pyrolysis temperature (T_p), both thermoplastic and nonthermoplastic materials chemically decompose or pyrolyze into lower molecular weight fragments and continue through the combustion temperature (T_c). For thermoplastic fibers, T_g and/or T_m are lower than T_p and/or T_c , while for non-thermoplastic fibers, T_g and/or T_m are higher than T_p and/or T_c .

In case of nonthermoplastic natural fibers, pyrolysis and combustion start before softening and melting. Thermoplastic synthetic fibers melt and drip away from the flame before pyrolysis and combustion temperatures are reached. However, if the melt does not shrink away from the flame front, pyrolysis and

combustion temperatures are eventually reached and ignition occurs. FR cotton and inherent FR synthetic fibers (e.g., Nomex, Kevlar, PBI) can offer protection to the wearer because they do not shrink away from the flame. Thermoplastic fibers pass the ignition test by shrinking away from the flame. In reality, however, the wearer is exposed to direct heat and thereby suffers burning by contact of the body with the molten mass (Tomasino, 1992).

The thermal processes and combustion products of organic products occur in a progressive and definable cycle as shown in Figure 1.1. When heat is applied, the temperature of the fiber increases until the pyrolysis temperature (T_p) is reached. At this temperature the fiber undergoes irreversible chemical changes, producing nonflammable gases (carbon dioxide, water vapor, and higher oxides of nitrogen and sulfur), flammable gases (carbon monoxide, hydrogen and many oxidizable organic molecules), tars (liquid condensates) and carbonaceous char. As the temperature continues to rise, the tars also pyrolyze, producing more flammable and nonflammable gases and char. When combustion temperature (T_c) is reached, the flammable gases combine with oxygen in the process called combustion, which is a series of gas-phase free radical reactions. These highly exothermic reactions generate large amounts of heat and light. The generated heat provides additional thermal heat for the pyrolysis process to continue. More and more flammable gases and consequently, higher and higher amounts of heat are generated causing devastating effects. In the case of burning of textiles, the speed or rate of heat release is more important than the amount of generated heat (Schindler and Hauser, 2004). An important factor in combustion is the Limiting Oxygen Index

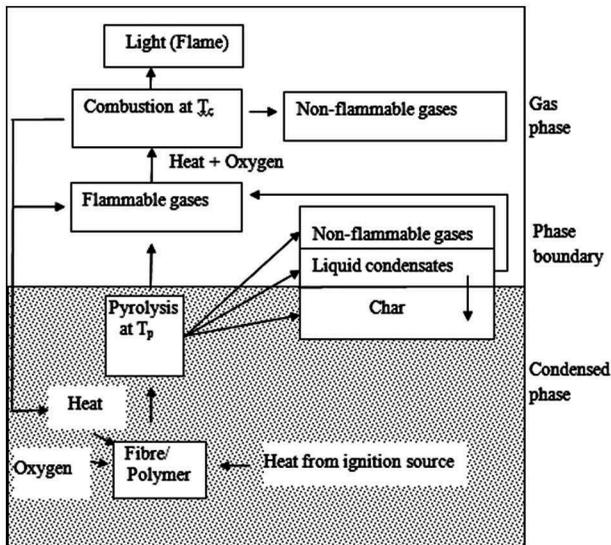


FIGURE 1.1 Combustion cycle for fibres and polymers.

(LOI), which is the percentage of oxygen in the fuel mix needed to support combustion. The higher this number is, the more difficult the combustion is.

Thermal decomposition precedes combustion and ignition of the material. Combustion is an exothermic process that requires three components, namely heat, oxygen, and fuel. When left unchecked, combustion becomes self-catalyzing and will continue until the oxygen, the fuel supply, or excess heat is depleted.

After combustion, the polymer may degrade without flame, burst into flame, or change physically by melting, shrinking, or charring. The combustion products may further be decomposed and ignited. In other words, on heating, a polymer may be liquefied with or without decomposition, be converted into carbon-residues (chars), or release combustible or noncombustible gases. The thermal decomposition products determine the flammability of polymers. The role of flame retardants (FRs) is to inhibit the formation of combustible products and/or to alter the normal distribution of decomposition products originating from the original material.

1.5 PYROLYSIS

The decomposition of materials due to fire is called pyrolysis or thermolysis. All textile fibers in their natural form are inherently fire retardants. Almost all known fibers have a high flash point or melting point. However, when the surrounding temperature reaches the flash point temperature of fibers, they catch fire. Cellulose such as cotton is solid and has an appreciably low vapor pressure. They do not burn but decompose into flammable fragments, which generate heat. This heat further decomposes the cellulose to carry on the decomposition process. Thermal decomposition of cellulose leads to the formation of products such as liquids, tar, and solid materials. Bond rupture, bond reformation, volatilization, and many exothermic reactions occur simultaneously.

Pyrolysis is the thermal decomposition of materials at elevated temperatures in an inert atmosphere (IUPAC, 2009). It involves the change of chemical composition and is irreversible. The word is coined from the Greek-derived elements *pyro* (fire) and *lysis* (separating).

Pyrolysis is most commonly used to the treatment of organic materials. It is one of the processes involved in charring wood (InnoFireWood's website, 2019). In general, pyrolysis of organic substances produces volatile products and leaves a solid residue enriched in carbon char. Extreme pyrolysis, which leaves mostly carbon as the residue, is called carbonization.

The aforementioned process is used heavily in the chemical industry, for example, to produce ethylene, many forms of carbon, and other chemicals from petroleum, coal, and even wood, to produce coke from coal. Inspirational applications of pyrolysis would convert biomass into syngas and biochar, waste plastics back into usable oil, or waste into safely disposable substances.

Pyrolysis differs from other processes such as combustion and hydrolysis in that it usually does not involve the addition of other reagents such as oxygen (O_2 , in combustion) or water (in hydrolysis) (Cory et al., 2009). In practice, it is often not practical to achieve completely oxygen- or water-free conditions,

especially as pyrolysis is often conducted on complex mixtures. This term has also been applied to the decomposition of organic material in the presence of superheated water or steam (hydrous pyrolysis), for example, in the steam cracking of oil. Pyrolysis has been assumed to take place during catagenesis, the conversion of buried organic matter to fossil fuels.

The possibility of extinguishing a polymer flame depends on the mechanism of thermal decomposition of the polymer. Whereas ignition of a polymer correlates primarily with the initial temperature of decomposition, steady combustion is related to the tendency of the polymer to yield a char, which is produced at the expense of combustible volatile fragments. Therefore, the dependence of steady combustion on the amount of char seems to be simple, and in an early study, it was established that the oxygen index shows a very good correlation with the char yield (Van Kleeven, 1975). In reality, char also serves as a physical barrier for heat flux from the flame to the polymer surface, as well as a diffusion barrier for gas transport to the flame (Levchik and Wilkie, 2000). Therefore, the contribution of the char can be more significant than is expected from a simple reduction in combustible gases.

Four general mechanisms are important for thermal decomposition of polymers (Hirschler, 2000):

1. Random chain scission, in which the polymer backbone is randomly split into smaller fragments;
2. Chain-end scission, in which the polymer depolymerizes from the chain ends;
3. Elimination of pendant groups without breaking of the backbone; and
4. Cross-linking.

Only a few polymers decompose predominantly through one mechanism; in many cases, a combination of two or more mechanisms is in effect. For example, polyethylene and polypropylene tend primarily to decompose via random chain scission, which in the case of polyethylene, is also accompanied by some cross-linking. Poly(methyl methacrylate) and polystyrene tend to depolymerize, poly(vinyl chloride) primarily undergoes elimination of pendant groups (dehydrochlorination), and polyacrylonitrile crosslinks.

In terms of flammability, random scission and depolymerization polymers are usually more flammable than polymers that cross-link or remove pendant groups. Cross-linking (Wilkie et al., 2001) leads to precursors of char and as a result, to lower flammability. Elimination of pendant groups results in double bonds, which can also give cross-links or lead to aromatization.

In general, polymers with aromatic or heterocyclic groups in the main chain are less combustible than polymers with an aliphatic backbone (Aseeva and Zaikov, 1986). Polymers with short flexible linkages between aromatic rings tend to cross-link and char. These polymers are thermally stable and show relatively good flame retardancy. For example, bisphenol A-based polycarbonate, phenol formaldehyde resins, and polyimides are self-extinguishing and show either a V-2 or V-1 rating in the UL-94 test. On the other hand,

polymers with relatively long flexible (aliphatic) linkages are still relatively combustible despite aromatics in the backbone. Examples of these polymers are poly(ethylene terephthalate), poly(butylene terephthalate), polyurethanes, and bisphenol A-based epoxy resin.

1.6 COMBUSTION

Combustion, or burning, is a high-temperature exothermic redox chemical reaction between a fuel (the reductant) and an oxidant, usually atmospheric oxygen that produces oxidized, often gaseous products, in a mixture termed smoke. In thermodynamics, the term *exothermic process* (exo-: “outside”) describes a process or reaction that releases energy from the system to its surroundings, usually in the form of heat, but also in a form of light (e.g., a spark, flame, or flash), electricity (e.g., a battery), or sound (e.g., explosion heard when burning hydrogen).

In complete combustion, the reactant burns in oxygen, and produces a limited number of products. When a hydrocarbon burns in oxygen, the reaction primarily yields carbon dioxide and water. When elements are burned, the products are primarily the most common oxides. Carbon yields carbon dioxide, sulfur yields sulfur dioxide, and iron yields iron (III) oxide. The combustion of methane, a hydrocarbon, is as in Equation 1.1.



Combustion in a fire produces a flame, and the heat produced can make combustion self-sustaining. Combustion is often a complicated sequence of elementary radical reactions. Solid fuels, such as wood and coal, first undergo endothermic pyrolysis to produce gaseous fuels; their combustion then supplies the heat required to produce more of them. Combustion is often hot enough that either incandescent light glows or a flame is produced.

Combustion is a type of chemical process in which a substance reacts rapidly with oxygen releasing heat. The original substance is called the fuel, and the source of oxygen is called the oxidizer. The fuel can be a solid, a liquid, or a gas, e.g., for aeroplane propulsion the fuel is usually a liquid.

Burning or combustion is a chemical process that occurs when oxygen combines/reacts with a substance producing sufficient heat and light (exothermic reaction) to cause ignition. The chemical process is called oxidation. The materials are oxidized continuously until they are exposed to an oxidizing agent (e.g., air) or directly to oxygen. At normal temperatures, the rate at which oxidation occurs is slow, and the heat generated is negligibly small, and is naturally conducted away from the material by the immediate environment. The oxidation rate increases with the increase of temperature, more and more heat releases, and pyrolysis takes place at a temperature specific to the material, i.e., the material decomposes by the action of heat.

Combustion means burning. It is an exothermic process that requires three components to start a chemical chain reaction, namely:

1. heat,
2. oxygen, and
3. a suitable fuel.

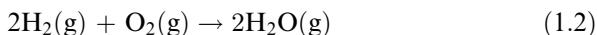
The combustion is self-catalyzing and unless controlled, combustion continues as long as the oxygen, the fuel or the excess heat remain. For the combustion process to take place, fuel, oxygen, and an ignition heat source are required. For example, in a campfire, wood is the fuel, the surrounding air provides the oxygen, and a match or lighter is the ignition heat source. Increasing any of these elements increases the fire's intensity, while eliminating any one of them causes the process to stop. If the campfire is smothered with water or dirt, for example, the oxygen can no longer get to the heat and fuel, and it goes out.

1.6.1 FUEL

Fuel is the substance that burns during the combustion process. All chemical fuels contain potential energy; this is the amount of energy that is released during a chemical reaction. The quantity of energy released by a substance during burning is known as the heat of combustion. Each fuel has a specific energy density, or megajoules (MJ) of energy produced per kilogram (kg) of the substance; methane, for example, has an energy density of 55.5 MJ/kg, meaning that it can supply more energy than sulfur, having an energy density of 9.16 MJ/kg.

A wide variety of substances may be used as fuels, but hydrocarbons are among the most common. Some examples of fuels are methane, propane, gasoline, and jet fuel. All fossil fuels, including coal and natural gas, are hydrocarbons. Other substances that are commonly used as fuels include hydrogen, alcohol, and biofuels such as wood.

Combustion, or burning, is a redox chemical reaction that releases heat. A fuel (the reductant) reacts with an oxidant (e.g., atmospheric oxygen), thereby producing mixed oxidized gaseous products called smoke. Combustion in a fire produces a flame, and the heat produced can make combustion self-sustaining. Combustion is often a complicated sequence of elementary radicals. Wood and some other solid fuels first undergo endothermic pyrolysis producing gaseous fuels, which on combustion, supply the heat required to produce more fuels. Combustion is often hot enough that either light glows or a flame is produced. A simple example can be seen in the combustion of hydrogen and oxygen into water vapor. The reaction is commonly used for fuelling rocket engines. This reaction releases 242 kJ/mol of heat and reduces the enthalpy accordingly at constant temperature and pressure (Equation 1.2).



Combustion of an organic fuel in the air always releases heat because the double bond in O_2 is much weaker than other double bonds or pairs of single bonds, and therefore, the formation of the stronger bonds in the combustion

products of CO_2 and H_2O results in the release of energy. The bond energies in the fuel are not significant, as they are similar to those in the combustion products; for example, the sum of the bond energies of CH_4 is nearly the same as that of CO_2 . The heat of combustion is about -418 kJ per mole of O_2 consumed in a combustion reaction and can be estimated from the elemental composition of the fuel (Schmidt-Rohr, 2015).

In order for a fire to start or be sustained, a fuel, an oxidizer, and an ignition source must be present. If one of the three components is eliminated, then there cannot be a fire (or explosion).

Fuel, a flammable or combustible material, in combination with a sufficient quantity of an oxidizer such as oxygen gas or another oxygen-rich compound (though nonoxygen oxidizers exist), is exposed to a source of heat or ambient temperature above the flash point for the fuel–oxidizer mix. The fire tetrahedron or fire pyramid (Figure 1.2) adds a fourth component—chemical chain reaction—as a necessity in the prevention and control of fires. The free radicals formed during combustion are important intermediates in the initiation and propagation of the combustion reaction. Fire suppression materials that scavenge these free radicals are able to sustain a rate of rapid oxidation that produces a chain reaction. Fire cannot exist without all of these elements in place and in the right proportions. For example, a flammable liquid would start burning only if the fuel and oxygen are in the right proportions. Some fuel-

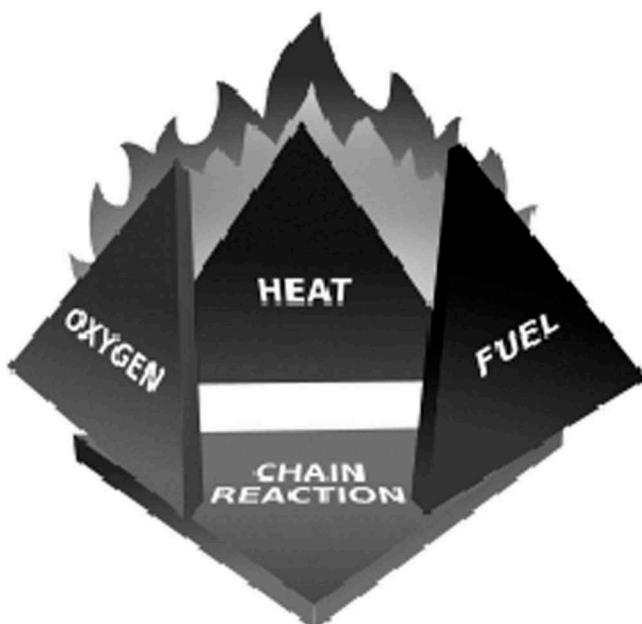


FIGURE 1.2 Fire Tetrahedron (https://en.wikipedia.org/wiki/File:Fire_tetrahedron.svg, (free to use)).

oxygen mixes may require a catalyst, a substance that is not consumed when added, in any chemical reaction during combustion, but which enables the reactants to combust more readily.

Once ignited, a chain reaction must take place whereby fires can sustain their own heat by the further release of heat energy in the process of combustion and may propagate, provided there is a continuous supply of an oxidizer and fuel.

1.7 COMBUSTION PRODUCTS

The gaseous products released during combustion of polymers and fibers are shown in Table 1.1 (Lewin and Sello, 1975).

1.8 IGNITION

During burning, when the flashpoint is reached, runaway exothermic reactions are triggered. This is accompanied by the appearance of a flame or glowing zone. This phenomenon is known as ignition. The time interval between the onset of heating and ignition is called ignition time.

Ignition of fabrics (such as apparel, upholstery, and bedding materials) subjected to open flames is a topic of much relevance in understanding and controlling the initiation of unwanted fires. The ignitability of materials is of basic importance when fire initiation and developments are analyzed. For example, in order to predict the burning behavior of fabrics, it is crucial to understand the role played by various physical and chemical properties in determining:

- Whether ignition would occur, and
- If it does occur, the duration of exposure to accomplish it.

TABLE 1.1

Gaseous products released during combustion of organic polymers

Polymer/Fibers	Gases
All organic polymers	CO, CO ₂
Nitrogen-containing polymers (wool, silk, acrylic, polyurethanes, amino resins etc.)	NO, NO ₂ , NH ₃ , HCN
Wool, vulcanised rubber, sulphur containing polymers	SO ₂ , H ₂ S, COS, CS ₂
Cellulosic fibers	Formic and acetic acid
Wood, cotton and paper	Acrolein
Polyolefin and others	Alkanes, alkenes
Polystyrene, PVC, polyesters	Benzene
Wood, cotton, paper, phenolic resins	Aldehydes
Phenolic resins	Phenol, formaldehyde
PVC, PTEE, and other halogenated flame retardants	HCl, HF, HBr

Fabrics that are considered inherently noncombustible are made from what are termed high-performance fibers (HPFs), either inorganic fibers such as glass or ceramic fibers, or fibers spun from thermally resistant synthetic polymers, typically fibers such as Kevlar and Nomex, that are made from aramid polymers. HPFs have very strong bonds and require high-heat energy to break them.

One must understand the mechanisms and conditions that lead to a sustained appearance of a flame in the gas phase when a combustible solid is heated by an external source. Two types of ignition are possible under these conditions: spontaneous (auto) and piloted (forced). This depends on whether the ignition occurs with or without the aid of an external pilot such as a spark or a flame. From the fire research perspective, piloted ignition is more important because:

- It occurs at a lower threshold;
- It is the mechanism responsible for fire growth; and
- In practice, it is usually impossible to exclude all possible external pilot sources.

1.8.1 AUTOIGNITION

As bonds break, the bond-fragments can form combustible substances liberated as a gas, depending on the initial chemical composition of the base material. The amount of gas liberated increases with temperature, and when its ignition temperature is reached (forced ignition or auto-ignition) burning occurs. However, there must be sufficient oxygen present to combine with the gas molecules to generate the amount of heat that will raise the temperature to the point of ignition.

The lowest temperature at which a substance spontaneously (in the absence of external ignition sources such as flame or spark) ignites in a normal atmosphere is known as the autoignition temperature or kindling point of the particular substance. This temperature is required to supply the activation energy needed for combustion. The temperature at which a chemical ignites decreases as the pressure increases or oxygen concentration increases. This is applicable to a combustible fuel mixture. If the heat released is sufficient to sustain or increase the oxidation rate, then burning continues until the material is consumed. While burning continuously, more and more heat releases; the temperature may reach a level causing neighboring flammable materials to ignite; and flash-over may occur.

1.8.3 PILOTED IGNITION

The earliest known scientific investigation into piloted ignition of wood was carried out by Bamford et al. (1946). They measured the time in which flaming would persist upon removal of the pilot heat source called the *ignition threshold*. They postulated a critical lower limit pyrolysate mass flow rate criterion for sustained ignition. The sustained ignition is possible if the pyrolysate mass flux at the fuel surface is less than $2.5 \times 10^{-4} \text{ g/cm}^2/\text{s}$.

A comprehensive experimental and theoretical works of spontaneous and piloted ignition of cellulosic solids was reported by Akita (1959). He presented evidence that the ignition occurs due to some thermal phenomenon directly pertaining to the exposed surface itself. Formation of a combustible gas mixture in the proximity of the exposed surface in itself is a necessary and sufficient condition for piloted ignition (since an external heat source already exists). Such a condition is necessary but not sufficient to ensure spontaneous ignition. This sufficiency is fulfilled by a thermal condition at the attainment of a temperature above 500°C by the exposed surface.

Martin (1964) published his work on ignition based on the following:

- While the internal temperature profile is considerably influenced by pyrolysis, a critical exposed surface temperature criterion describes the onset of ignition.
- The persistence of ignition depends not on any unique composition of such a pyrolysis product mixture, but upon the continued outflow of flammable pyrolysis products.
- The exposed surface is completely pyrolyzed long before ignition.

Thomas and Dry Dale (1987) defined critical ignition temperature as the surface temperature of material at which ignition occurs. Furthermore, the piloted ignition temperature can be defined as the lowest temperature at which the ignition of the decomposition products gives rise to sustained burning at the surface. It is similar to the fire point of a combustible liquid, but differs in what is referred to as a surface temperature, rather than as a bulk temperature.

1.8.2 PROCESS OF IGNITION

The ability to control fire dramatically changed the habits of early humans. Making fire to generate heat and light made it possible for people to cook food, simultaneously increasing the variety and availability of nutrients, and reducing disease by killing organisms in the food. The heat produced also helped people stay warm in cold weather, enabling them to live in cooler climates. In addition, fire kept nocturnal predators at bay. Evidence of cooked food is found from 1.9 million years ago (Bowman et al., 2009), although there is a theory that fire could have been used in a controlled fashion about 1 million years ago. Early humans harnessed fire as early as 1 million years ago, much earlier than previously thought, which suggests evidence unearthed in a cave in South Africa (Krajick, 2011). Evidence became widespread around 50,000 to 100,000 years ago, suggesting regular use from this time; resistance to air pollution started to evolve in human populations at a similar point in time. The use of fire became progressively more sophisticated, for example, to create charcoal and to control wildlife from tens of thousands of years ago.

Fire has also been used for centuries as a method of torture and execution, as evidenced by deaths by burning, as well as by torture devices such as the

iron boot, which could be filled with water, oil, or even lead, and then be heated over an open fire to the agony of the wearer.

Setting fuel a flame releases usable energy. Wood was a prehistoric fuel, and is still viable today. In power plants, the use of fossil fuels, such as petroleum, natural gas, and coal, supplies the vast majority of the world's electricity today.

1.9 LIMITING OXYGEN INDEX (LOI)

Minimum percent of oxygen in the environment that sustains burning under specified test conditions. In other terms, it is the content of oxygen in an oxygen-nitrogen mixture that keeps the sample at the limit of burning (Equation 1.3).

$$\text{LOI} = \frac{\text{O}_2}{\text{H}_2 + \text{O}_2} \times 100 \quad (1.3)$$

Tesoro (1978) defined several terms relating to flammability, a few are discussed next.

1.10 CHARRING

Charring is a chemical process of incomplete combustion of certain solids when subjected to high heat. The resulting residue matter is called char. By the action of heat, charring removes hydrogen and oxygen from the solid, so that the remaining substance is composed primarily of carbon. Polymers such as thermoset, as well as most solid organic compounds such as wood or biological tissue, exhibit charring behavior (Chylek et al., 2015).

Charring means partially burning to blacken the surface. Charring can result from naturally occurring processes such as fire; it is also a deliberate and controlled reaction used in the manufacturing of certain products. The mechanism of charring is a part of the normal burning of certain solid fuels such as wood. During normal combustion, the volatile compounds created by charring are consumed at the flames within the fire or released to the atmosphere, while combustion of char can be seen as glowing red coals or embers that burn without the presence of flames.

Coke and charcoal are both produced by charring, whether on an industrial scale or through normal combustion of coal or wood. Normal combustion consumes the char, as well as the gases produced in its creation, while industrial processes seek to recover the purified char with minimal loss to combustion. This is accomplished by either burning the parent fuel (wood or coal) in a low-oxygen environment or by heating it to a high temperature without allowing combustion to occur. In industrial production of coke and charcoal, the volatile compounds that are driven off during charring are often captured for use in other chemical processes.

Charring is an important process in the combustion ignition of solid fuels and in smouldering. In construction of heavytimbered wood buildings, the predictable formation of char is used to determine the fire rating of

supporting timbers and is an important consideration in fire protection engineering.

Charring of organic materials starts at temperatures considerably lower than that of soot formation. Burning of food during cooking (e.g., the production of nicely black toast) is an example of low-temperature charring. At temperatures above about 300°C, most of the organic materials undergo a slight thermal decomposition; hydrogen and other noncarbon elements are stripped from carbon chains and rings and the carbon condenses into a graphite like structure. The density of black porous residuum depends on the mass ratio of carbon to other elements in the original material.

Charring of polymers proceeds through various stages (Levchik and Wilkie, 2000):

1. Cross-linking,
2. Aromatization,
3. Fusion of aromatics, and
4. Graphitization.

The ability of a polymer to perform in one or several of these stages leading to char formation depends primarily on the polymer structure. However, this performance can be improved significantly by the use of flame retardants. Although many polymers tend to cross-link at early stages of thermal decomposition, this does not necessarily result in char formation. Char is formed only if the cross-linked polymer contains aromatic fragments and/or conjugated double bonds and is prone to aromatization during thermal decomposition (Wilkie et al., 2001). Fused aromatic rings in the char tend to assemble into small stacks, which are precursors of graphite. These pregraphitic domains are embedded in the amorphous char. This type of char, called turbostratic char, is usually formed at 600°C to 900°C, temperatures typically found on the surface of burning polymers. Char that contains more pregraphitic domains is more stable to thermal oxidation and therefore less likely to burn away and expose the polymer surface to the heat of the flame. On the other hand, highly graphitized chars are rigid and may have cracks, which do not retard diffusion of combustible materials to the flame. The best-performing char would be amorphous uncracked char with requisite pregraphitic domain content (Levchik, 2007).

1.11 SMOLDERING

Combustion occurs without flame and without prior flaming combustion, but usually with incandescence and smoke. Smoldering is the slow, low-temperature, flameless form of combustion, sustained by the heat that evolves when oxygen directly attacks the surface of a condensed-phase fuel. Many solid materials can sustain a smoldering reaction, including coal, cellulose, wood, tobacco, synthetic foams, charring polymers (including polyurethane foams), and some types of dust. Common examples of smoldering phenomena are the initiation of

residential fires on upholstered furniture by weak heat sources and the persistent combustion of biomass behind the flaming front of wildfires.

The fundamental difference between smoldering and flaming combustion is that smoldering occurs on the surface of the solid, rather than in the gas phase. Smoldering is a surface phenomenon but can propagate to the interior of a porous fuel if it is permeable to flow. The characteristic temperature and heat released during smoldering are low compared to those in the flaming combustion. Smoldering propagates in a creeping fashion, around 0.1 mm/s (0.0039 in/s), which is about ten times slower than flames spread over a solid. In spite of its weak combustion characteristics, smoldering is a significant fire hazard. Smoldering emits toxic gases (e.g., carbon monoxide) at a higher yield than flaming fires and leaves behind a significant amount of solid residue. The emitted gases are flammable and could later be ignited in the gas phase, triggering the transition to flaming combustion.

1.12 AFTERGLOW

Glowing combustion in a material after cessation (natural or induced) of flame. The behavior of thermoplastic material towards the flame/heating has a different story. Fabrics made of synthetic materials that exhibit melting and surface involvement in afterglow is different from those exhibiting the same in flaming. Prevention method of afterglow is also different. Afterglow is mainly due to the burning of remnant char, which forms as a result of lack of oxygen in surrounding atmosphere. Carbon and oxygen react to form carbon monoxide (CO); this is an exothermic reaction, and energy liberated is 26.4 kcal. The char becomes ash in afterglow process by conversion of CO to CO₂ in the presence of excess oxygen.

1.13 SELF-EXTINGUISHING MATERIALS

Self-extinguishing materials are incapable of sustained combustion in air under the specified test conditions after removal of the external source of heat.

Hensel (2011) said flame retardant is good, but self-extinguishing is better for electrical installations. Only *self-extinguishing* material provides added safety with respect to fire protection. All Hensel products made from thermoplastic are flame retardant and self-extinguishing and fulfill the glow wire tests at least at 750°C or even at 960°C. The proof of self-extinguishing characteristics should be carried out using a glow wire test at a temperature of 750°C. The glow wire test at 750°C should prove that a flame would extinguish itself within 30 s of the energy source being switched off, i.e., it does not continue burning or catch fire.

In the Russian aviation industry, in flame resistance assessment, the determining indices (according to the industry standard OST 1090094-79) are the duration of combustion and the length of the burned part of the polymer specimen (measuring 50 x 290 mm) during heating by a gas burner with a flame temperature of 840°C. According to the given indices, materials are classified

into low-burning, self-extinguishing, slow-burning, and burning (combustion time >15 s, length of burnt part of specimen >170 mm) (Petrova et al., 2014).

The airplane trims should satisfy the following requirements: as a minimum they should be self-extinguishing (after exposure to a gas burner flame for 12 s (60 s), the duration of residual burning should not exceed 15 s, and the length of burn-through should not exceed 152 mm); they should also have limited smoke formation (the specific optical density of smoke within 4 min should be no more than 200 units) (Barbot'ko, 2010; Mikhailin, 2011).

1.14 SMOKE

Smoke is a fine dispersion of carbon, other solids, and liquids resulting from incomplete combustion; particles are not individually visible, but cause opacity due to scattering and/or absorption of visible light.

Smoke is a collection of airborne solids, liquid particulates, and gases that are emitted when a material undergoes combustion or pyrolysis, together with the quantity of air that is entrained or otherwise mixed into the mass. It is commonly an unwanted by-product of fires (including stoves, candles, oil lamps, and fireplaces), but may also be used for pest control (fumigation), communication (smoke signals), defensive and offensive capabilities in the military (smoke screen), cooking, and smoking (tobacco, marijuana (drug)). It is used in rituals in which incense, sage, or resin is burned to produce a smell for spiritual purposes. Smoke is sometimes used as a flavoring agent, and as a preservative for various foodstuffs. Smoke is also a component of internal combustion engine exhaust gases, particularly diesel exhaust.

Smoke inhalation is the primary cause of death in victims of indoor fires. The smoke kills by a combination of thermal damage, poisoning and lung irritation caused by carbon monoxide, hydrogen cyanide, and other combustion products.

Smoke is an aerosol (or mist) of solid particles and liquid droplets that are close to the ideal range of sizes for Mie scattering of visible light. A smoke cloud does not obstruct an image, but thoroughly scrambles it.

Polymers are a significant source of smoke. Aromatic side groups, e.g., in polystyrene, enhance generation of smoke. Aromatic groups integrated in the polymer backbone produce less smoke, likely due to significant charring. Aliphatic polymers tend to generate the least smoke, and are non-self-extinguishing. However presence of additives can significantly increase smoke formation. Phosphorus-based and halogen-based flame retardants decrease production of smoke. Higher degree of cross-linking between the polymer chains has such an effect as well (Van Krevelen and Nijenhuis, 2009).

1.15 FLAME

A flame (from Latin *flamma*) is the visible, gaseous part of a fire. It is caused by a highly exothermic reaction taking place in a thin zone. Very hot flames are hot enough to have ionized gaseous components of sufficient density to be considered plasma.

There are different methods of distributing the required components of combustion to a flame. In a diffusion flame, oxygen and fuel diffuse into each other; the flame occurs where they meet. As a result, the flame speed is limited by the rate of diffusion. In a diffusion flame, combustion takes place at the flame surface only, where the fuel meets oxygen in the right concentration; the interior of the flame contains unburnt fuel. This is opposite to combustion in a premixed flame.

In a premixed flame, the oxygen and fuel are premixed beforehand, which results in a different type of flame. Candle flames (diffusion flames) operate through evaporation of the fuel, which rises in a laminar flow of hot gas that then mixes with surrounding oxygen and combusts.

A flame is a mixture of reacting gases and solids emitting visible, infrared, and sometimes ultraviolet light, the frequency spectrum of which depends on the chemical composition of the burning material and intermediate reaction products. In many cases, such as the burning of organic matter, for example wood, or the incomplete combustion of gas, incandescent solid particles called soot produce the familiar red-orange glow of *fire*. This light has a continuous spectrum. Complete combustion of gas has a dim blue color due to the emission of single-wavelength radiation from various electron transitions in the excited molecules formed in the flame. Usually oxygen is involved, but hydrogen burning in chlorine also produces a flame, producing hydrogen chloride (HCl). Among many other possible combinations producing flames are fluorine and hydrogen, and hydrazine and nitrogen tetroxide. Hydrogen and hydrazine/unsymmetrical dimethylhydrazine (UDMH) flames are similarly pale blue, while burning boron and its compounds, evaluated in the mid-20th century as a high-energy fuel for jet and rocket engines, emits intense green flame, leading to its informal nickname of “Green Dragon”.

The chemical kinetics occurring in the flame are very complex and typically involve a large number of chemical reactions and intermediate species, most of them radicals. For instance, a well-known chemical kinetics scheme, GRI-Mech (2007) uses 53 species and 325 elementary reactions to describe combustion of biogases.

The glow of a flame is complex. Black-body radiation is emitted from soot, gas, and fuel particles, though the soot particles are too small to behave like perfect blackbodies. There is also photon emission by de-excited atoms and molecules in the gases. Much of the radiation is emitted in the visible and infrared bands. Flame color depends on several factors, the most important typically being black-body radiation and spectral band emission, with both spectral line emission and spectral line absorption playing smaller roles. In hydrocarbon flames, the most common type of flame, the most important factor determining color is oxygen supply and the extent of fuel-oxygen pre-mixing, which determines the rate of combustion, and thus the temperature and reaction paths, thereby producing different color hues.

The dominant color in a flame changes with temperature. The photo of the forest fire in Canada is an excellent example of this variation. Near the ground, where most burning occurs, the fire is either white, the hottest color possible for organic material in general, or yellow. Above the yellow region,

the color changes to orange, which is cooler, then to red, which is cooler still. Above the red region, combustion no longer occurs, and the uncombusted carbon particles are visible as black smoke.

The common distribution of a flame under normal gravity conditions depends on convection, as soot tends to rise to the top of a general flame, as in a candle in normal gravity conditions, making it yellow. In microgravity or zero gravity (NASA, 2010), such as an environment in outer space, convection no longer occurs, and the flame becomes spherical, with a tendency to become more blue and more efficient (although it may go out if not moved steadily, because the CO_2 from combustion does not disperse as readily in microgravity, and tends to smother the flame). There are several possible explanations for this difference, of which the most likely is that the temperature is sufficiently evenly distributed so that soot is not formed and complete combustion occurs (NASA, 2007). Experiments by NASA reveal that diffusion flames in microgravity allow more soot to be completely oxidized after they are produced than diffusion flames on Earth, because of a series of mechanisms that behave differently in microgravity when compared to normal gravity conditions (www.deros.com, 2019). These discoveries have potential applications in applied science and industry, especially those concerning fuel efficiency.

In combustion engines, various steps are taken to eliminate a flame. The method depends mainly on whether the fuel is oil, wood, or a high-energy fuel such as jet fuel.

1.15.1 FLAME TEMPERATURES

It is true that objects at specific temperatures do radiate visible light. Objects whose surface is at a temperature above approximately 400°C (752°F) glow, emitting light at a color that indicates the temperature of that surface. It is a misconception that one can judge the temperature of a fire by the color of its flames or the sparks in the flames. For many reasons, chemically and optically, these colors may not match the red/orange/yellow/white heat temperatures on the chart.

1.15.2 ADIABATIC FLAME TEMPERATURE

Adiabatic means no loss of heat to the atmosphere. An adiabatic process occurs without transfer of heat or mass of substances between a thermodynamic system and its surroundings. In the study of combustion, there are two types of adiabatic flame temperature, depending on how the process is completed. These are constant volume and constant pressure, describing the temperature that the combustion products theoretically reach if no energy is lost to the outside environment. The constant volume adiabatic flame temperature is the temperature that results from a complete combustion process that occurs without any work, heat transfer or changes in kinetic or potential energy. Its temperature is higher than the constant pressure process because none of the energy is utilized to change the volume of the system (i.e., generate work).

TABLE 1.2**Adiabatic flame temperature of some fuel and oxidizer pairs**

Fuel and oxidizer pairs	Adiabatic flame temperature
Oxy-dicyanoacetylene	4,990°C (9,000°F)
Oxy-acetylene	3,480°C (6,300 °F)
Oxyhydrogen	2,800°C (5,100 °F)
Air-acetylene	2,534°C (4,600 °F)
Bunsen burner (air-natural gas)	1,300 to 1,600°C (2,400 to 2,900°F) (Begon et al., 1996)
Candle (air-paraffin)	1,000°C (1,800°F)

The *adiabatic flame temperature* of a given fuel and oxidizer pair (Table 1.2) (Fire Wikipedia, accessed 9.2.2019) indicates the temperature at which the gases achieve stable combustion. This is the maximum temperature that can be achieved for given reactants. Heat transfer, incomplete combustion, and dissociation all result in lower temperature. The maximum adiabatic flame temperature for a given fuel and oxidizer combination occurs with a stoichiometric mixture (correct proportions such that all fuel and all oxidizer are consumed).

A fire-resistance rating typically means the duration for which a passive fire protection system can withstand a standard fire resistance test. This can be quantified simply as a measure of time, or it may entail a host of other criteria, involving other evidence of functionality or fitness for purpose.

1.16 FLAMMABILITY

Flammability is the tendency of a material to burn with a flame. Flammable materials are those that ignite more easily than other materials, whereas those that are harder to ignite or burn less vigorously are combustible.

Lewin (1985) explains flammability as the tendency of a material to burn with a flame. Indeed the flammability of textiles is a measurement of the ease with which fabric is able to be ignited and how effectively it burns. Kasem and Rouette (1972) stated that the ignitability of the fabric as well as the combustibility are the indicators of fabric flammability characteristics. The combustibility of the fabric is stated as the rate at which the flame (or the afterglow) is able to propagate. Ignition of the fabric is described as a more complex phenomenon by Backer et al. (1976). Ignition involves the transfer of heat with thermal decomposition governed by fluid mechanics and chemical kinetics. Exothermic reactions are triggered as the ignition temperature of the fabric is reached. The reactions accompanied by a flame or glowing of any sort is termed ignition.

The degree of flammability or combustibility in air depends largely upon the chemical composition of the subject material, as well as the ratio of mass to surface area. For example, finely divided wood dust can undergo explosive

combustion and produce a blast wave. A piece of paper (made from wood) catches on fire quite easily. A heavy oak desk is much harder to ignite, even though the wood fiber is the same in all three materials.

Common sense (and indeed scientific consensus until the mid-1700s) would seem to suggest that material *disappears* when burned, as only the ash is left. In fact, there is an increase in weight because the combustible material reacts (or combines) chemically with oxygen, which also has mass. The original mass of combustible material and the mass of the oxygen required for combustion equals the mass of the combustion products (ash, water, carbon dioxide, and other gases). Antoine Lavoisier, “father of modern chemistry” (August 26, 1743–May 8, 1794), a French nobleman and one of the pioneer chemists in these early insights, stated that nothing is lost, nothing is created, everything is transformed, which would later be known as the law of conservation of mass. Lavoisier used the experimental fact that some metals gained mass when they burned to support his ideas.

Historically, flammable, inflammable and combustible meant capable of burning. The word “inflammable” came through French from the Latin *inflammāre* = “to set fire to”, where the Latin preposition “in-” means “in” as in “indoctrinate”, rather than “not” as in “invisible” and “ineligible”.

The word “inflammable” may be erroneously thought to mean “nonflammable”. The erroneous usage of the word “inflammable” is a significant safety hazard. Therefore, since the 1950s, efforts to put forward the use of “flammable” in place of “inflammable” have been accepted by linguists, and it is now the accepted standard in American English and British English. Antonyms of “flammable/inflammable” include: nonflammable, noninflammable, incombustible, noncombustible, not flammable, and fireproof.

Flammable applies to materials that ignite more easily than other materials, and thus, are more dangerous and more highly regulated. Less easily ignited less-vigorously burning materials are combustible. For example, in the United States, flammable liquids, by definition, have a flash point below 100°F (38°C), whereas combustible liquids have a flash point above 100°F (38°C). Flammable solids are solids that are readily combustible, or may cause or contribute to fire through friction. Readily combustible solids are powdered, granular, or pasty substances that easily ignite by brief contact with an ignition source, such as a burning match, and spread flame rapidly. The technical definitions vary between countries so the United Nations created the Globally Harmonized System of Classification and Labeling of Chemicals, which defines the flash point temperature of flammable liquids as between 0° and 140°F (−17.8° and 60°C) and combustible liquids between 140°F (60°C) and 200 F (93°C) (United Nations, 2011).

Flammability is the ability of a substance to burn or ignite, causing fire or combustion. The degree of difficulty required to cause the combustion of a substance is quantified through fire testing. Internationally, a variety of test protocols exist to quantify flammability. The ratings achieved are used in building codes, insurance requirements, fire codes and other regulations governing the use of building materials as well as the storage and handling of highly flammable substances inside and outside of structures and in surface and air

transportation. For instance, changing occupancy by altering the flammability of the contents requires the owner of a building to apply for a building permit to make sure that the overall fire protection design basis of the facility can take the change into account.

The factors affecting the flammability of apparel are:

- Fiber content,
- Fiber construction,
- Fabric weight,
- Fabric construction,
- Fabric surface texture,
- Moisture content,
- Presence of additives or contaminants in the fabric,
- Garment design,
- Effect of components used in apparel, and
- Laundering of the apparel after use.

1.17 FLAME-RESISTANT VS. FLAME-RETARDANT

Various terms are used to express the way a fabric reacts when in contact with a flame. If negligibly affected it is said to be flame-proof or fire-proof; if it ignites but self-extinguishes on removal from the flame, it is called fire-resistant–difficult to burn; if the material does not burn but can melt and/or decompose at high temperatures, it is referred to as flame retardant, noncombustible or incombustible – not capable of igniting and burning. A very important aspect of flameproof/fireproof textiles is that they are thermally stable; they do not readily burn or shrink when exposed to a flame or intense heat (heatproof/heat-resistant).

Flame-resistant fabrics are made from materials that are inherently nonflammable—the materials have flame resistance built into their chemical structures. Fabrics made with these types of materials are designed to prevent the spread of fire and do not melt or drip when in close proximity to a flame. Because flame-resistant fabrics are not usually made from 100% flame-resistant materials, they burn, but do so very, very slowly, and are often self-extinguishing. The most important function of these materials and fabrics is to prevent the further spread of fire.

Fire-resistant is a synonym for flame-resistant. It means exactly the same thing, and it is correct to use them interchangeably.

With fire-resistant clothing, the promise is not that the garments will never catch fire. They are designed to resist igniting, and generally fulfill this purpose in all but the most extreme situations. The great strength of flame-resistant garments, however, is that they prevent fires from spreading. Even if the garments do catch fire, they almost always extinguish themselves quickly.

Fire-retardant or flame retardant fabrics are those that have undergone chemical treatment to acquire some of the same properties that flame-resistant fabrics inherently have. As a result of these chemical procedures, flame retardant fabrics

become self-extinguishing and slow-burning. Any type of fabric may be used, but must undergo this treatment before it can be considered flame retardant.

If an employee works in environments where heat, fire, or electrical injuries are a real possibility, the odds are good that this employee should be wearing flame-resistant clothing.

Not all fire-resistant clothing is made from the same fabrics. There are multiple different choices available, and no choice is perfect. Each comes with different benefits and hazards. Each organization is best served by choosing the fabric that will be most suited to its needs and working environment. There are three broad categories of workers who should wear flame-resistant clothing for protection, based on the type of hazard to which the workers would be exposed while completing their work. The three primary hazards are (Ashley, 2017):

- Electric arc: People who are exposed to this hazard include electricians, as well as certain utility workers and others.
- Flash fire: This category includes pharmaceutical and chemical workers, as well as those who work in refineries and other industries.
- Combustible dust: This category includes workers in food processing plants, and in the paper and pulp industry, among others.

Here are a few of the common fibers with inherent flame-resistant qualities commonly used to create FR clothing (Ashley, 2017).

- Modacrylic: These are the most popular and common option available today. These fibers are often used as part of a blend to create several different flame-resistant fabrics. These various combinations of fibers work together to create fabrics that can easily stand up to several types of standards and regulations.
- Nomex: This is another type of fiber that has inherent flame-resistant qualities. Unlike modacrylic fibers, Nomex can create FR garments on its own. It doesn't have to be a standalone, however. It can also be combined with other materials such as Kevlar.
- Kevlar: These fibers are certainly flame-resistant, but have many other additional properties such as high strength. Kevlar can create flame-resistant clothing, as well as many other different items. When used to make FR clothing, Kevlar is often combined with Nomex.

Each type of flame-resistant fabric will come with its own pros and cons. Kevlar, for instance, is extremely heavy-duty, but consequently comes with a high price tag. There are no specific flame-resistant clothing dangers, however, and all are designed to protect the wearer from hazardous heat-based conditions.

Fire-resistant clothing is also referred to as FRC by industry people. The garments underneath the flame-resistant clothing have a significant impact on the safety and the effectiveness of the FRC clothing. Whenever someone wear these garments, he or she should always take care to wear only nonmelting garments underneath them.

There are two primary reasons for this caution. The first is that by doing this, essentially a second layer of FR protection is added. Even if the first layer of outerwear gets damaged or burned, a second layer would protect the wearer. The layer of air insulation between the two layers also helps keep the wearer safe.

Flame retardant fabrics are chemically treated to be slow-burning or self-extinguishing when exposed to an open flame. These fabrics can be made from any material, but they must be treated with special chemicals to qualify as flame retardant.

The term flame retardants (FRs) subsume a diverse group of chemicals which are added to manufacture materials, such as plastics and textiles, and surface finishes and coatings. Flame retardants are activated by the presence of an ignition source and are intended to prevent or slow the further development of ignition by a variety of different physical and chemical methods. They may be added as a copolymer during the polymerization of a polymer, mixed with polymers at an molding or extrusion process or, particularly for textiles, applied as a topical finish (EPA, 2005). Mineral flame retardants are typically additive while organohalogen and organophosphorus compounds can be either reactive or additive.

The most important difference between flame-resistant and flame retardant fabrics lies in how each is made. Without a special chemical application, a fabric does not qualify as flame retardant. Similarly, without being made of certain nonflammable fibers, a fabric will not qualify as fire resistant.

The special protective wear is called fire-resistant clothing/fire-resistant garments/fire-resistant apparel, or even personal protective equipment which also includes further accessories such as gloves, helmets, or boots. *FR* refers to the flame-resistant or fire-resistant and heat-resistant properties of the clothing, by virtue of which a fabric is able to resist burning or melting and even self-extinguish once the source of ignition or fire is removed from it.

Fire-resistant clothing is basically made from two kinds of fabric that are differentiated as *treated* and *inherent* FR fabrics. The former type quiet simply includes the natural or synthetic fabrics that are later treated with a combination of flame retardant chemicals to give them the flame-resistant properties. However, in the case of inherent fabrics, the fabric itself is made from fibers that have flame-resistant qualities and this resistance is ingrained into the molecular structure of the fiber by engineering retardant compounds into a permanent chemical change inside the hollow core of a fiber, creating an all-new fabric with FR characteristics (Islam, 2018).

Inherently flame retardant (FR) fibers may be of three types, namely:

1. Inherent thermally stable chemical structure (e.g., the polyaramids or other aromatic structures),
2. Flame-retardant additives incorporated during the production of manmade fibers (e.g., FR viscose), or
3. Produced by the synthesis of conventional fiber forming polymers with flame retardant comonomers (e.g., FR polyester).

The word *inherent* was not originally a textile or FR term. Its definition varies slightly from source to source, but the common thrust is “by its very nature, built-in, implicit,” while “treated” is usually defined as chemical engineering to impart properties not previously present. Nature provides very few FR fibers; the most well-known of these is asbestos, which is obviously not in common use in protective apparel.

Conversely, all flame-resistant fibers in common use today for industrial protective apparel are engineered by humans, using chemistry, to be flame-resistant. What is important is not how the engineering was accomplished; what matters is that the engineering was accomplished, correctly and consistently, so that a garment is flame-resistant weeks later, months later, and years later, regardless of how many times it is laundered (Margolin, 2012).

1.18 FLAME RETARDANTS (FRS)

Normal textile materials are finished with flame retardant agents in the form of surface treatments, coatings, or functional finishes which become an integral part of the fiber structure.

Flame retardant materials were first produced around 400 BC, but the need for them was not realized much until the 17th century. In 1638, the idea of reducing the risk of fire in theaters originated in Paris with fireproofing of plaster and clay, thus beginning the process of creating flame-resistant materials. If humans intervene with chemistry to treat naturally flammable fibers, they could prevent potential harm. Therefore, the process of making things flame-resistant became a priority from this point on.

In 1735, Jonathan Wyld of England patented a flame-retarding mixture of alum, ferrous sulfate, and borax. The first systematic attempt to make textiles flame-resistant was made in 1821 by the eminent chemist Gay Lussac, who developed a flame-retarding finish for hemp and linen fabrics that contained various ammonium salts, with or without borax. The salts first broke down into a nonflammable vapor when they were heated up, while borax was low-melting and formed a glassy layer on fabrics. This was yet another step toward making today's textiles flame-resistant. By the 20th century, other scientists perfected the same method by incorporating stannic oxide into fabrics to make them flame retardant. Stannic oxide, also known as tin oxide, is an off-white, powdery product that is produced thermally from high-grade tin metal.

These techniques were used to make natural fibers fire-resistant. Once synthetic materials started dominating the market, cotton producers needed to come up with a better way to promote their products, or they would not have been able to survive. The Army Quartermaster Corps' were in demand for flame-resistant clothing and the research regarding fire-resistant fabrics increased to a large extent. Advanced technologies of the 20th century allowed scientists to start the process of chemically modifying the cellulose molecules on both the surfaces and within cotton fibers. To keep this special process commercially viable, the scientists needed to work hard to find a chemical combination that still kept the cotton's strength and durability while keeping the cost competitive (Caitlin, 2015).

TABLE 1.3**Early historical developments of flame retardants (WHO, 1997)**

Alum used to reduce the flammability of wood by the Egyptians.	About 450 BC
The Romans used a mixture of alum and vinegar on wood.	About 200 BC
Mixture of clay and gypsum used to reduce flammability of theater curtains	1638
Mixture of alum, ferrous sulfate, and borax used on wood and textiles by Jonathan Wyld in Britain	1735
Alum used to reduce flammability of balloons	1783
GayLussac reported a mixture of $(\text{NH}_4)_3\text{PO}_4$, NH_4Cl and borax to be effective on linen and hemp	1821
Perkin described a flame retardant treatment for cotton using a mixture of sodium stannate and ammonium sulfate	1912

The chemical nature of textile substrates is highly diversified. Hence, the field of flame retardancy is multidisciplinary and complex as well. The flame-retarding chemicals and formulations are also numerous and include halogen, phosphorous, nitrogen, antimony, sulphur, boron, and other elements in many forms and combinations. Flame-retarding treatments require the application of a relatively large quantity of chemicals; e.g., 10%–30% of the weight of the material. Hence, the aesthetic properties (softness, stiffness, luster, handle, drape etc.), physical properties (washability, soil repellence and soil release, static charge accumulation), tensile properties, creasing, and pilling properties of textile materials may change.

A brief historical development of FRs through the ages is listed in Table 1.3.

1.19 CLASSIFICATION OF FLAME RETARDANTS

Flame retardants (FRs) are mainly of two types:

1. Additive flame retardants, and
2. Reactive flame retardants.

They can be further separated into several different classes (Van der Veen et al., 2012):

- a. Minerals such as:
 - Aluminum hydroxide (ATH),
 - Magnesium hydroxide (MDH),
 - Huntite and hydromagnesite,
 - Various hydrates,
 - Red phosphorus, and
 - Boron compounds, mostly borates.

- b. Organohalogen compounds. This class includes organochlorines such as chlorendic acid derivatives chlorinated paraffins and organobromines such as:
- Decabromodiphenyl ether (decabde),
 - Decabromodiphenyl ethane (a replacement for decabde),
 - Polymeric brominated compounds such as brominated polystyrenes, brominated carbonate oligomers (BCOs), brominated epoxy oligomers (BEOs),
 - Tetrabromophthalic anhydride,
 - Tetrabromobisphenol A (TBBPA), and
 - Hexabromocyclododecane (HBCD).
- c. Organophosphorus compounds. This class includes organophosphates such as:
- Triphenyl phosphate (TPP),
 - Resorcinol bis(diphenylphosphate) (RDP),
 - Bisphenol A diphenyl phosphate (BADP), and
 - Tricresyl phosphate (TCP),
 - Phosphonates such as dimethyl methylphosphonate (DMMP), and
 - Phosphinates such as aluminium diethyl phosphinate.
- d. Halogenated phosphorus compounds:
- Brominated: tris(2,3-dibromopropyl) phosphate (tris), and
 - Chlorinated: tris(1,3-dichloro-2-propyl) phosphate (TDCPP) and tetrakis(2-Chlorethyl)dichloroisopentyl diphosphate (V6) (Weil and Levchik, 2015).

Most but not all halogenated flame retardants are used in conjunction with a synergist to enhance their efficiency. Antimony trioxide is widely used, but other forms of antimony, such as the pentoxide and sodium antimonate are also used.

The mineral flame retardants mainly act as additive flame retardants and do not become chemically attached to the surrounding system. Most of the organohalogen and organophosphate compounds also do not react permanently to attach themselves into their surroundings, but further work is now underway to graft further chemical groups onto these materials to enable them to become integrated without losing their retardant efficiency. This will also make these materials nonemissive into the environment. Certain new nonhalogenated products with these reactive and nonemissive characteristics have been coming onto the market since 2010, because of the public debate about flame retardant emissions. Some of these new reactive materials have even received US-EPA approval for their low environmental impacts.

1.20 SYNERGISM AND ANTAGONISTIC

Synergism and antagonism are the interactions of two or more substances or agents to produce a combined effect greater or smaller than the sum of their separate respective effects.

Synergistic effects are observed in the vapor phase, as well as in condensed-phase, active FR systems. Their modes of action in the various systems are different and involve a wide variety of interactions. They involve chemical interactions between the FR additive and the synergist, between the synergist and the polymer or between all three: polymer, FR additive and synergist, between two synergists and the FR additive, and even between two polymers in a polymer blend. In some cases, the synergist itself is not a flame-retarding agent and becomes active only in the presence of an FR additive. This is the case with halogen-based additives and antimony trioxide. In other cases such as the bromine-phosphorus synergism, both additives are active flame retardants. In certain cases the interaction between the ingredients in a formulation brings about a decrease in the flame retardancy parameters and is thus antagonistic. This is encountered in the case of the application of phosphorus derivatives together with nitriles (Khanna and Pearce, 1978).

Lewin (1999) stated that the term synergism, in the FR terminology is a poorly defined term. Strictly speaking, it refers to the combined effect of two or more additives, which is greater than that predicted on the basis of the additivity of the effect of the components. The term *synergistic effectivity* (SE) (Lewin and Sello, 1975) is meant to serve as a general tool for characterizing and comparing synergistic systems. It is defined as the ratio of the FR effectivity (EFF) of the flame retardant additive plus the synergist to the EFF of the additive without synergist. EFF is defined as the increment in oxygen index (OI) for 1% of the flame retardant element. The differentiation between the synergistic systems and catalytic phenomena observed in FR technology is not straightforward. Some synergistic systems could easily be classified as catalytic and vice versa. The catalyst is highly effective at a low concentration in the formulation. The number of FR catalytic systems reported in the literature is, until now relatively small, but the interest in catalytic approaches appears to be growing.

1.21 FUTURE TRENDS

The Center of Fire Statistics (CFS) of International Association of Fire and Rescue Services (CTIF) presents its latest report №23 (CTIF, 2018), containing fire statistics of 27–57 countries during the years 1993–2016 representing 0.9–3.8 billion inhabitants of the Earth, depending on the year of reporting. In these countries 2.5–4.5 million fires and 21–62 thousand fire deaths were reported to fire services annually, depending on the year. The statistics clearly shows that fire hazards are not being abated in spite of all human efforts. Fire not only cause loss of human lives and our valuable wealth, but also causes tremendous air pollution. The biggest health threat from smoke is from fine

particles. These microscopic particles can get into the eyes and respiratory system, where they can cause health problems such as burning eyes, runny nose, and illnesses such as bronchitis. Fine particles also can aggravate chronic heart and lung diseases. In addition, burning of various materials may generate toxic gases. To make textile and other substances less prone to burning, various flame retardant substances are used. In recent years, however, it has been observed that many of them, especially halogens, are very efficient, but not environment friendly.

It is a true challenge to find suitable and efficient substitutes at cheaper prices. The methods of application of flame retardants has also been revolutionized in recent years.

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