



Environmentally Friendly Inorganic Materials for Anti-flammable Cotton Fabrics

Marina Y. Soliman ^a and Ahmed G. Hassabo ^{b*}

^a*Textile Printing, Dyeing and Finishing Department, Faculty of Applied Arts, Benha University, Benha, Egypt.*

^b*National Research Centre (Scopus affiliation ID 60014618), Textile Industries Research Division, Pre-treatment, and Finishing of Cellulose-based Textiles Department, 33 El-Behouth St. (former El-Tahrir str.), Dokki, P.O. 12622, Giza, Egypt.*

THIS SUBJECT describes three main topics: (i) cellulosic materials' physical and chemical structure; (ii) cellulosic cure finishing and flame retardancy; and (iii) cellulosic and its composite cure of flame delay and fire. The first topic details various sources of cellulose and their chemical and physical structure. The second subject includes the application of flame retardant textile materials and describes the difference in the definition of retardant/resistant terms, and explains in more detail the theory of combustion or burning, and fire and flame retardant mechanism. Different phosphorous flame retardant synergistic were also mentioned, including the types and classifications of flame retardant finishes based on durability and nature. At the end of this section, various textile inflammability tests are described. The last part of the chapter shows the preparation by layer by layer of branched polyethyleneimine of A flame retardant surface Kaolin, urea and Phosphate (DAP) (BPEI).

Keywords: Cotton fabric, Flame-Retardant, Layering, Flammability Test.

Introduction

Cotton fibres are widely utilised for the production of clothes, home furnishings and various industrial products, including medical, industrial and tarpaulins. Because cotton is abundant, low cost and an adaptable starting material for developing a new product, the use of cotton is encouraged on a variety of consumer markets. While cotton is known for its softness, its breathability and moisture absorption, it is more fuel-efficient than most synthetic fibres. [1-5]

A significant number of textile flame retardants have developed in the second half of the last century to comply with the fire safety regulations and to expand the use of cotton in textile applications requiring flame resistance. Many studies have shown the presence of nitrogen enhances the actions of flame retardants from phosphorus. In the majority of these flame retardant treatments four distinct groups can be classified: inorganic, organic halogenated,

organophosphorus, and nitrogen-based. [6] Prior work has shown that such mergers have produced a greater flame resistance to phosphorus in cotton textiles than when phosphorus was used alone. [7]

Unlike halogen-based compounds that produce or can produce toxic gases, smoke or harmful substances, flame-based phosphoric agents are known as phosphoric acid transforms into phosphoric acid during combustion or heat degradation. This also leads to the formation of non-volatile polyphosphoric acid, which by dehydration and esterification can react with the decomposing polymer to promote carbohydrate. [8]

The char residue can act as a barrier against oxygen and radiant heat attacking the underlying polymer and thereby prevent the fire from being extinguished. Recently, nano coating has been layer-by-layer (LBL) assembled into fabrics, as an easy and cost-effective way of transmitting particular

chemical endings with natural and synthetic textiles. Decherd is based on an alternation of loaded anionic and cationic polyelectrolytes to create thin, multi-layered nanometre coatings of a material surface via electrostatic interactions.

A broad range of functional molecules including nanoparticles, dyes and proteins can be incorporated into coatings to change the physical and chemical characteristics of the surface of the materials. Although LBL depends on parameters such as polyelectrolyte, temperature and pH chemistry, the procedure is not limited to material surface size, shape and topography, making the technique suitable for customising the surface properties of nonplanar textile fabrics. [9-15]

The LBL deposition of clay and nanoparticles with polyethylene (BPEI) branched to cotton fabrics by Grunlan and co-workers has thoroughly studied to produce flammable multi-layer coatings of environmentally friendly, all-polymer nanocoordination that can extinct the fires of cotton materials. [16]

The use of LBL technology to alter the surfaces of textile fabrics and fibres has been widely studied in recent years but there is still little understanding of the process as a whole. Nano cutting of fire retardation is a relatively new field. Previous research with LBL flame retardant primarily focused on developing and characterizing thin, simple clay/polyelectrolyte films without adding additional small flame retardant molecules. Current LBL techniques that utilize multiple immersion/rinsing cycles to develop multi-layer coatings can also be laborious and time-consuming that frequently limit the use of the coating method for commercial use.

The current study aims to improve the flame retardancy of four cotton fabrics: print cloth, mercerized print cloth, twill, and fleece cotton fabrics through the deposition by using the newly-developed LBL self-assisted process of multilayer films containing BPEI, DAP, urea and kaolin nanoparticles. Thermogravimetric analysis (TGA), oxygen limit index (LOI, ASTM D 2863-09) [17] and vertical inflammation testing were performed on the flame retardance of cotton fabrics with deposited clay nanolayers (ASTM D 6413-11). [18] Measurement was carried out to verify the presence of deposited nanolayers and to study the morphology of the different nanocoated.

Cotton Fibres

Natural fibres are primarily made up of pectin, water-soluble compounds, waxes and inorganic

substances, non-flammable and generally called ash, which make a balance of cellulose, hemicellulose and lignin. Cellulose decomposition between 260 and 350 °C results in the development, for example, of flammable volatile and gases, non-combustible gases, tars and certain char. [19]

The flammability of the fibre is increasing with a high cellulose content. Hemicellulose falls between 200 and 260 °C but forms more gas than cellulose and less tar. In addition to cellulose or Hemi cellulation, lignin contributes more to char formation. Therefore, coirs with low cellulose and high lignin content are considerably less inflammable than, for example, cotton with a high level of cellulose, low cellulose, high lignin and low hemicellulose content based on the chemical constituents alone. Lignin begins to decompose and continues to decompose. Relatively slight bonds break at low temperatures, but cleavage of bonds occurs with higher temperatures in the aromatic lignin rings. [16]

Higher concentrations of levoglucosan give higher levels of crystallinity during pyrolysis for cellulosic fibres in particular. However, a higher ignition temperature also results in increased crystallinity, as a decomposition of the crystalline structure requires more energy.

Furthermore, the higher the orientation, the lower the fibre's oxygen permeability. Therefore, the best choice for use as a composite strengthening would be a fibre with low crystallinity and a high level of polymerisation and orientation, from the flammable aspect.

Cotton is a natural vegetable single elongated fibre, developed from an epidermal cell of the cottonseed. The main producing countries of cotton fibre are the United States, Egypt and India. The highest quality cotton, which has very long and fine fibres, is growing in Egypt.

Distinctive Features Morphology of Cotton
Primary layer holds up to 30% cellulose and non-cellulosic materials. The secondary wall is rich in cellulose of higher weight with DP of 14,000. More detailed the cotton fibre has the following composition: Cellulose: 88–96%, it is the main component of cotton fibre and the secondary wall possess the highest percentage of the total cellulose. Waxes: 0.3–1.00%, they are composed of higher monovalent alcohol-tractional, palmitic, oleic acid, glycerine. Toxin evolved from bacterial cells (0.017–100 g per bale of mass 218 kg.

Physical and Chemical Structural of Cellulosic Materials

Sources for Cellulose

Cellulosic fibre, like cotton, linen, jute, ramie, hessian and sisal is a natural polymer of vegetable origin. Cellulose is also found in plants as a microfibril (2-20 nm diameter and 100-40000 nm long). It is therefore useful to study the chemical and physical structure of these natural polymers. These form the structurally strong cell wall framework. Most of the cellulose is made of pulp. Certain bacteria (for example, *Acetobacter xylinum*) also produce cellulose in a highly hydrated form.

Chemical Structure of Cellulose

In the plant kingdom, cellulose is an important structural material. It is the main construction material of the plant cell walls that are made out of it. Native cellulose occurs in plant fibres such as cotton and wool and combination with lignin and homo-cellulose.

Haworth has proposed to write the structural sugar formulation in approximately 3D prescriptive

form, which gives the formula when applied to cellulose. It is also a polysaccharide consisting of glucose (+) β -D residues. These molecules were condensed and connected linearly with β -glucosidic 1-4 connections. [20]

Each has three reactive hydroxyl groups, thus polyhydroxy alcohols, each unit with re-occurring anhydroglucose. The secondary hydroxyl groups are two of these groups in positions 2 and 3, while the last in position 6 is the primary hydroxyl group. Because cellulose molecules are more reactive than the secondary hydroxyl group, they can form numerous bindings with the hydroxyl primary group. The primary hydroxyl group is more acidic than the secondary hydroxyl group. Steric impediment, especially for bulky reacting species, is an important consideration; position six is least sterically hindered. When the distance of different cellulose atoms from oxygen and hydrogen is 3 Å or smaller it interacts to form intermolecular hydrogen, links interact with each other. (see Fig. 1) [21]

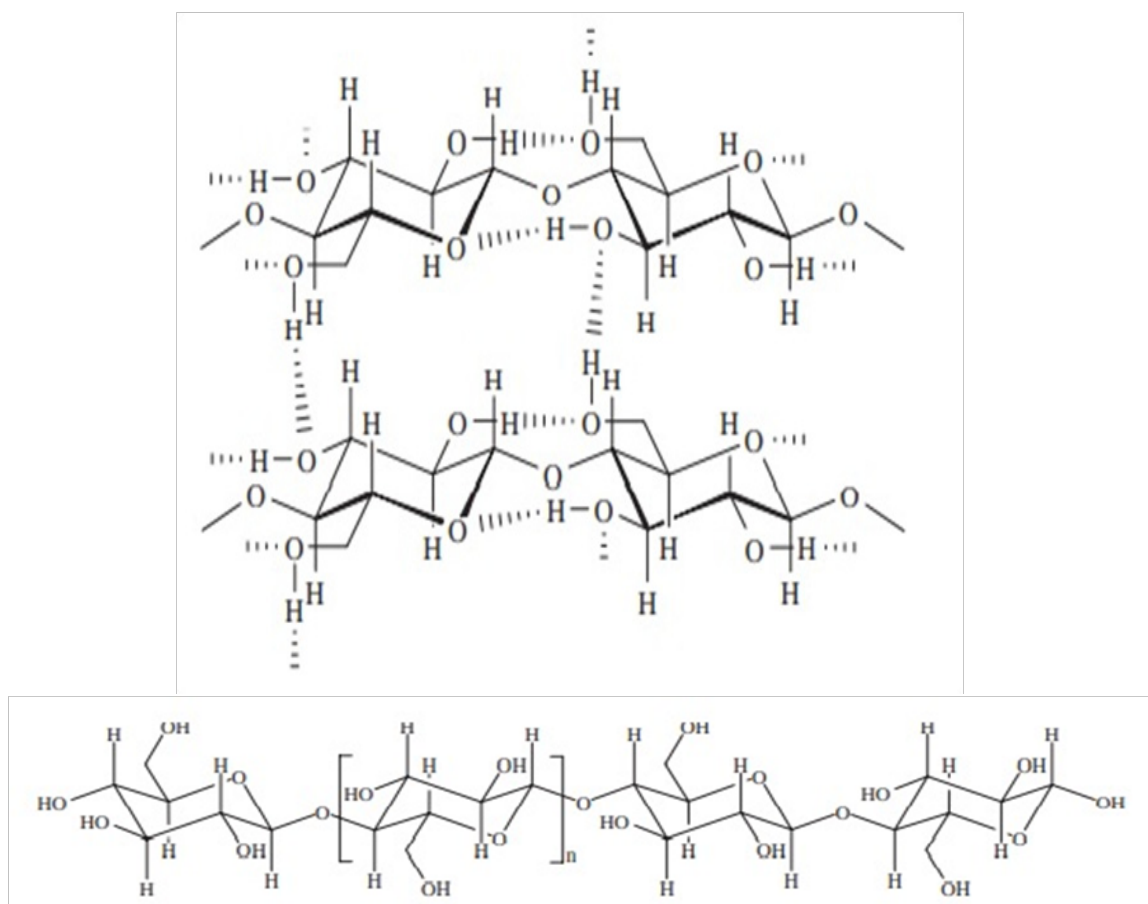


Fig. 1. Chemical Structure of Cellulose and Unit cell of the crystal lattice of cellulose in the cotton fibre.

Physical Structure of Cellulose

Crystalline and Amorphous Regions Cellulose can form many hydrogen bonds with three-hydroxyl groups along the length of the polymer chain. These bonds bind together with the other (mainly the Van der Waals attraction), and they extend into random conditions (amorphous area) to perfect geometrical packing of the crystal lattice (crystalline region). [21]

Cellulose Accessibility and Reactivity The ease with which cellulose can react was described by reactivity and accessibility. More correctly, the chain molecules' ability to react with other molecules is the cellulose reactivity, whereas the cellulose accessibility determines the ease with which a reactant molecule is reached by the functional groups of the cellulose chain

The approach for cellulose was proposed mainly depends on the number and size of the pores of the cellulose structure; on solvent or reagent dimensions and types; on the inner surface that is accessible, as determined in terms of fibrils or fibril aggregates; and on cellulose molecules structure that determines which hydroxyl groups are available. The pores must therefore be opened and both fibril aggregates and highly ordered regions must be altered to increase the accessibility of cellulose.

Fire and Flame Retardancy Finishing of Cellulosic Material

One of the most antiquated forms for textile processing was the fire-retardant treatment; as early as the 4th century Bc, risks associated with the ready combustibility of cellulose materials have been recognized. The concept of burning protection of textiles goes back to 1821 when Gay Laussac was treated in France with a blend of ammonium-phosphate, ammonium chloride and borax for the treatment of hemp, jute and linen. [22-24] The first successful, launder-resistant, flame retardant fabric finish was built on the work of Perkin, the stannic oxide precipitated in the fibre. This tissue was flame resistant but was heavy and persistent enough to consume the tissue completely. [24, 25]

Uses of Flame Retardant Fabrics

a) Clothing and clothes, sleepwear, nightwear, kids wear, loose robes, sarees, shawls, wear of the kitchen etc., where the possibility of unintentional contact with flame is present.

b) Fire uniforms - combat staff.

- c) Training clothing, boiler suits and protective clothing for working man in many industries such as petroleum, oil, paint and paint varnish, mining, steel and iron, explosives, cooking gas storage and distribution, electricity generated and distributed, foundry, welding, petrol and diesel pumping, etc.
- d) Homemade decorative - curtains, clothes, tapestries, bedding, coatings, wall coverings, etc.
- e) Carpets and tapestries.
- f) Brate cloth for coal mines, backing fabrics for carpets, barrier fabrics such as motor coats and overlays, motor vehicle underlays, wall clothing, ornamental fabrics etc.
- g) Theatres, cinema halls, religious worship and congregation halls, marriage halls, hospitals, schools, colleges etc., are located in the centre of a city of Mosque and other parish areas of the country.
- h) Aircraft defence and firefighting crew clothes, overalls, parachutes, awning, planches, skip-inclusive canvas (overhead support kit). Protection of paramilitary cladding and shelters, etc.
- i) Export clothing, ribbons, wall coverings, bed coverings, mattresses and quilts, Furnishings of airlines, fabrics of automobiles.

Flame Retardant Cellulosic Fabric

Low fire resistance to cellulosic materials. It consists of carbon and hydrogen (fuel) and oxygen (fuel or combustion supplier) and is easy to burn. The cellulosic materials burning process is an oxidation process. The process may be accompanied by a flame or glow. After the flame has been extinguished, most organic fibres are glowing. The glow can damage a lot like the flames themselves since they can consume a textile altogether. Flaming and glowing processes at different temperatures are distinctly different. [24, 25] However, before going into details of the fire and flame retardant textile finishing it is useful to specify a few terms.

Fire-Proof Textile

The term applies to textiles that are largely uninfluenced by fire. This means that they don't sustain fire (flame or glow) and that when the textile is exposed to flames there is little or no chemical or physical change. Few categories of

textiles. The most common use today are carbon, asbestos, basalt and glass fibres. [22-24].

Flame Resistant Textile

The fabric not supporting the flame is said to be flame resistant after the ignition source is removed. The glow mechanism can continue to burn a flame resistance material although there is no flaming. Flameproof fabrics can also be charred or melted. [22-24].

A Glow Resistant Textile

Once the source of ignition is removed the fabric will not continue to burn with the glow mechanisms. The glow-proof fabric could carve or melt the fabric. [22-24].

Fire Resistant Textile

The fabric is resistant not only to flame but also to glow. Therefore, once the ignition source is removed, flaming and glowing cease. The physical and chemical characteristics will be changed, the textile will be able to characterise or melt. [24] [22-24].

Theory of Combustion

If solid materials are heated, physical and chemical changes occur depending on the chemical composition of the solid at a specific temperature. At a glass transition temperature (T_g), thermoplastic polymers soften and then melted (T_m) at a higher temperature (T_p), both thermoplastic and non-thermoplastic solids chemically decompose (pyrolysis) into lower molecular fragments of weight. Chemical change begins at (T_p) and continues through the combustion temperature (T_c). When considering the flame resistance of fibres, these four temperatures are very important. The limiting oxygen index is a further important combustion factor (LOI).

This is the quantity of oxygen needed for combustion in the fuel mix. The higher the number, the harder the combustion is. (T_p) or (T_c) are less than (T_g) and/or (T_m) in non-thermoplastic faces, but in thermoplastic fibres (T_p) and/or (T_c) they are more than (T_g) and/ or (T_c), or (T_m). Therefore, when the natural fibre is not thermoplastic, pyrolysis and combustion temperatures occur before temperature smoothing or melting reaches and finally ignites.

Natural fibres, like some synthetic fibres, can be made flame retardant. Because of the lack of a flame, Nomex, Kevlar and PBI offer protection

to the wearer. Thermoplastic fibres may seem to be protective because they pass the ignition test by shrinking away from the flame, but they are exposed to direct heat and burns caused by contact between the body and the molten mass. [26]

Mechanism of Fire and Flame Retardant Burning of Cellulose

It is useful to know what chemical reactions arise when cellulose burns and the effect of the reaction when the substances are processed with a flame retardant in the developing of better flame retardants. Cellulose's burning properties are significantly affected by the chemical and thermal characteristics of anhydroglucose systems, by the available oxygen and by the nature of noncellulosic materials that may also exist. The burning characteristics of Cellulose also have an impact on fibre, such as ignition temperature, combustion rates, heat and moisture content.

Cotton is fuel material. The presence of oxygen and a high temperature can cause cotton to burn or molten. can be combusted (smoulder combustion). For cotton, intrinsic inflammation times at the auto-ignition index decrease.

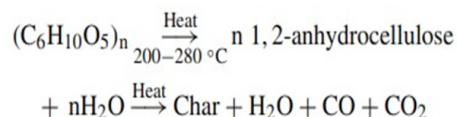
Burns cotton ready to reach a maximum temperature and char formation afterglow, and cotton have an ignition temperature.

The combustion process is ignited; the propagation and after-glow temperature within the combustion of cellulose. Flaming and after glow. Ignition is controlled by several factors including heat transfer from ignition source into cotton, cotton thermal decomposition, and the environmental oxygen reactions of decomposition products. A large number of fuel gases that can sustain ignition by and above a certain concentration with air oxygen are released through the thermal decomposition of cotton. A combustible material that generates more heat requires less fire energy to carry on burning.

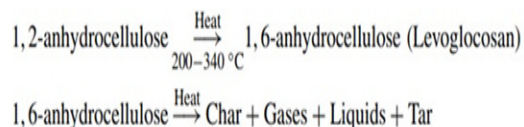
Therefore, the effect of heat on cellulose varies greatly and changes the property. The rapid reaction of Polymerisation has already been observed, and chain breaks have been suggested.

Points at the amorphous-crystalline boundaries. The number of groups of hydroxyl decreases while the group of carboxyl increases. The accessibility of cellulose to H_2O and dye molecules decreases even short heating times and significantly increased its hydrolysis rate. There has been an increase in the distance between

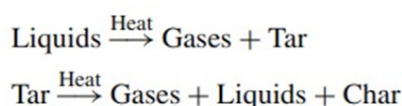
the crystalline lattice while the direction of the chain was reportedly not affected. It found that the controlled temperature occurred in two decomposition reactions. Water is lost and anhydrocellulose formed in the first reaction. [24]



The anhydrocellulose further decomposes to different volatile products at high temperatures, including alcohols, aldehyde and alkanes, flame-increasing gases, carbon monoxide, ethylene, methane and non-flammable gases, carbon dioxide, vapour and char. At temperatures above 280°C, there is a second reaction that involves the thermal splitting of glucosidic bonds and levoglucosan formation.



- Gases: combustible (methane, ethane, carbon monoxide) and non—combustible (formaldehyde, carbon dioxide et)
- Liquids: Water, Alcohols, Aldehydes, Ketones, Organic acids. [27]



The major product formed by the cellulose pyrolysis is levoglucosan, which is produced as a cyclic if the β -1,4-glucoside bond is split and the water molecular between the hydroxyls C(1) and C(6) of anhydroglucoside is lost.

Cellulose burning is carried out in two different ways: flaming and Both processes come from a hot or open flame or an overhead surface. The thermal decay of cellulose results in gaseous, liquid, tarry, and solid products during the flaming process.

The liquids are volatilized as the flammable
J. Text. Color. Polym. Sci. **Vol. 18**, No. 2 (2021)

gases burn and some of these volatile fractions burn where, as with others, the carbonised residue is not readily burned. This process goes on until it is just carbonated, starts. It glows, oxidises, and continuously glows until all organic matter is essentially consumed and only smooth ash is left.

At flammable decline in cellulose temperature, first, levoglucosan is formed by dividing the cellulose glucoside (1-4) and then intermolecular fragment rearrangement. Dehydration and polymerisation of levoglucosan are thought to form char.

The reaction rate control in thermal cellulose decomposition Includes levoglucosan, 1,6 anhydroglucopyranose chemical. The major pyrolytic product in air pyrolysis remains Levoglucosan. Even traces of impurities present in or added to the cellulose have great effects on levoglucosan yields. The percentage of levoglucosan in tar, depending on concentration, can be increased or decreased by acidic impurities; while basic impurities can decrease levoglucosan formation on all levels

Cellulose flame retardance appears to vary with levoglucosan formation within narrow limits. The increased flame retardation of cellulosic textiles is explained by these factors.

Treatment with low flame retardant concentrations. Other volatile compounds are due to their constant inflammability in the presence of enough flame retardant for depressing or removing levoglucosan. The interconnection with formaldehyde affects both the yield and flammability of Levoglucosa. With an increase of the formaldehyde reacted with cotton, levoglucosan content decreases. This decrease is due to the lower tendency to undergo the unzipping reaction for the crystalline faces (C2 and C6) substituted. The crosslinks may lead to another route of pyrolysis, different from levoglucosan and requiring greater energization. In the case of radius, with a cross-line connection the yield of levoglucosan increases.

Mechanism of Retardant Action on Cellulosic Materials

Cellulose flame retardants can function through one of the two basic mechanisms, solid (condensed) or flame-retardant vapour phase (CFP).

Halogens are known as efficient cellulose

flame retardants and Free radical inhibition of the other polymers and acts mainly through the vapour phase. But the combustion process radically pairs with the free radicals and ends the response.

The effect of solid (Condensed) active phase flame retardant exercises on cellulose was characterised by numerous researches.

Example cellulose depolymerization. The more effective flame retardant finishes for cotton are based on compounds of organophosphorus. Compared with many other types of the organic compound, these phosphorus compounds are relatively expensive and contribute to high flame retardant costs. One means of reducing costs is to replace low-cost nitrogen compounds with phosphorus in flame retardants.

The chemical, spectroscopic and thermal analysis of bromine pyrolysis was examined in detail and it was established the effect of bromine on cellulose and bromine distribution in pyrolysis products.

Several investigators have identified the effect on the cellulose of solid (condensed) active flame retardants. Their findings show that the most important effect of retarding is the reduction in tar formation and increased carbohydrate yield. The thermal degradation reacts primarily to cellulose (pyrolysis).

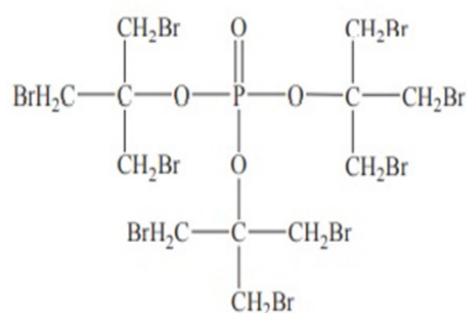
Depolymerization unzipping, that produces volatile and flammable levoglucosan and side products. In all pyrolysis temperatures, the reactions of this other set appear to have significantly lower activation energies and are at the same slow rate.

These reactions lead mostly to inflammable gases and gases such as water and carbon dioxide. The condensation and/or inhibition of the latter reaction set could result in a condensed phase flame retardant action on cellulose.

A dehydration flame retardant based on phosphorus, sulphur, boron and other acidic materials produces water and char to the detriment of the inflammable tars. Cellulose treatments are mostly based on phosphorus and, consequently, the mechanism and efficiency of flame retardancy have been widely studied for structural effect on phosphorus-based materials. Phosphorylation, presumable in the C-6 anhydroglucose unit, acts on acidic phosphates.

Phosphorous Flame Retardant Synergism Halogen-Phosphorus Synergism

The analogy with the powerful and well-established halogen antimony synergy often confuses the halogen phosphorous synergism. Contrary to the synergism of antimony halogen, phosphorous halogen is not a general synergy. There is absolutely no experimental support for the postulated formation of phosphorous oxyhalides. But, with combinations of halogen- and phosphoric flame retardants, good additive results are often obtained. In the structure of the following brominated phosphate ester, bromine and phosphorous synergism are found: [28]



Antimony-Phosphorus Synergism

Several published formulations demonstrate the attempted use of Antimony oxide combined with flame retardants of phosphorous and halogen. Results sometimes appear to be beneficial, but quantitative studies show that antagonism between antimony and phosphorus is convincing. The one element eliminates the effect from the other in the most serious case, and the effects are less than an additive in other cases. A detailed study of polyvinyl chloride (PVC) triaryl phosphate and antimony oxide showed that this antagonism occurred only in a part of the composition range. The antagonist effect is probably caused by the development of highly stable, practically inert fillers of antimony phosphates. [29, 30]

Nitrogen-Phosphorus Synergism

Fire retardant systems are like many other multi-component products. Produced regularly just through the mixture of two or more ingredients. The synergy between formulant components can have a significant impact on the efficacy of fire retardants. Depending on the proportion of each part used, the properties of the final product or system will depend. The phenomenon of synergy is that a compound formulation (a combination of

two or more components) produces a desirable property that is better than the property produced by the ratio of each compound. Unfortunately, however, such a combination of components can also cause unwanted effects. Antagonism is called a decrease of the desired property due to the combination. Possible reactions to a two-component system. [31-33]

Compounds containing nitrogen and phosphorus, such as phosphorous acid and urea, tetrakis hydroxymethyl phosphonium chloride (THPC), urea and trimethylol melamine, have been used for a long time as fire retardants. The inclusion of N-containing compounds with P-containing compounds in the finishing formula increased the fire retardance properties of the finished textiles. The role of nitrogen is expected to influence the majority of the above cases.

It has reported that phosphorus cellulose derivatives are very high. Much susceptible to dehydration that leads to char formation. The efficiency of phosphorus-based flame retardants by nitrogen-containing compounds has been well documented to increase synergies. It should be noted, however, that not all N-containing compounds are effective, but that they are only using ingredients that contain nucleophilic nitrogen atoms such as amines and amides.

Adding nitrogen to the phosphorous compound-contains compounds with the benefit of reducing acidic cellulose degradation through the release of phosphoric acid and improving flame delays. There are many benefits, low toxicity and their use of nitrogen compounds. Solid condition and absence and their low evolution of smoke of dioxins and halogen acids in the case of fire. [34, 35]

Their efficiency is between aluminium trihydrate and magnesium hydroxide halogen compounds. their efficiency. The Skinny Hydroxide divides the water and is environmentally compatible, but its low activity requires a high concentration, which changes the mechanical properties of the matrix to which it is used. Nitrogen-based flame retardants are eco-friendly because the existing polymers do not contain any new element. Finally, their modest price is the best argument.

A smaller add-on-a phosphorus/nitrogen mixture than phosphorus or nitrogen alone-can be achieved by using a comparable level of flame retardant. The effects of the flake-retardant

cotton fabric between phosphorous and nitrogen are synergistic. Triazine formaldehyde and trimethylol melamine were reported to be used.

The synergistic effect was shown to depend on the type of nitrogen present, aluminophosphate - cotton treated. Reeves et al. suggested the general increase in amide and amine nitrogen flame retardancy whereas nitrile nitrogen can lead to decreases. It has been suggested to generally increase the level of flame retardance with amide and amine, while nitrile nitrogen may decrease.

The typical formulation of cellulosic textile illustrates the curing of the resin secondary nitrogen content and therefore no synergistic tendency will be demonstrated. The observed urea antagonism-formaldehyde nitrogen should be linked to its high nitrogen content based on Weil's hypothesis. Cellulose phosphate formation steps appear to be catalysed by nitrogen compounds like amides or amines and strongly synergize phosphorous action in cellulose systems.

Synergistic interaction between nitrogen and phosphorus has been examined Different finishes for the assessment using the Limiting Oxygen Index (LOI). This is the case. The increase in P% (LOI) or the increase of N% (LOI) was reported with P%. The LOI increased with N% with N% with P%.

Types of Flame-Resistant Finishes

Flaming retardants can be separated into reactive flame retardants and additives. Reactive flame retardants are chemically integrated reactive components in a polymer molecule. The polymer has additives of flame retardants either before, during or (most often) after polymerization. [35, 36]

Classification of flame retardants

Fire retardants were classified into four based on different conditions Classes are (a) Flame-resistant non-durable finishes, (b) semi-durable flame-resistant finishes, (c) durable flameproof finishes, and (d) the durability of weatherproof flame retardants, nature of compounds, and host polymer incorporation. [37, 38]

The nature of the compound's divides flames retardant chemicals into five families. These are inorganic, halogenated, organophosphates, halogenated phosphates and flame retardants based on nitrogen.

Flame-resistant non-durable finishes

Available for use on cellulosic materials are nondurable flame retardants. When exposed to weather, chemicals were easily removed, leached or washed away from textiles. After every laundering, it should be treated again. Even when not washed, it should retreat every six months because many non-sustainable types of retardants crystallize from inside the fibre on the surface of a tissue. [39, 40]

This group has the most important examples; Borax (Sodium Borate $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) with Boric Acid or Diammonium Phosphate $(\text{NH}_4)_2\text{HPO}_4$. fabric treated with this retardant have flame resistance but can persist afterglow from approximately 30 s to a few minutes.

This flame retardant wording was used to dissolve cotton textiles equally. Borax parts of hydrogen in water and diammonium phosphate. [41]

Semi-durable flame-resistant finishes

Semidurable flame retardants may be defined as one or more cycles of washing, with an upper limit of approximately 15 mild cycles of laundry. The main benefit over the long-lasting type is lower costs. The phosphorus compounds that dissolve gradually and become resistant to both leaching and a few mild launderings are examples of this class. The most obvious way of achieving half-lasting fire resistance is insoluble salt application. The flame retardants effect of simple inorganic salts must be taken into account because they can decompose in the heat and release strong acid or alkali that are responsible for the reduction of flame spread. [42]

This group has the most important examples:

a) Cellulose Phosphates

A solution was padded to the fabric of urea and diammonium phosphate and the fabric was dried. The technique allows for per cent esterification easily, while severe tendering of cellulosic fibres. The improved tensile properties are achieved by very careful curing conditions and formulations that fix phosphorus in the fabric. [43, 44]

b) Cyanamide-Phosphoric Acid Process

An ammonium phosphate or phosphoric acid mixture of dimethylol dicyanamide or phosphoric acid will polymerize into an insoluble resin in cellulosic fibres. The additional modifications included the substitution of urea with a mixture of methylol melamine or diguanidine thiosulfate.

Durable flame resistance can be insolubilized in a somewhat associated process by pre-condensate urea and ammonium phosphate using CH_2O . During phosphorous oxychloride reaction ammonia could be obtained semi-durable flammable finishes closely resembling those made from urea and H_3PO_4 . [45]

Weather-Resistant Flame Retardants For many materials, weatherproof and flameproof finishes are extensively used. Water-soluble flameproof finishes are not weatherproof because it is readily removed from rain and humid atmospheres. The weatherproof and flameproof finish may be divided into two large groups; (a) inorganic oxide, usually of good resistance with sunlight degradation; however, it is poorly flameproof and must be used in combination with certain other materials, such as 1paraffin chlorine and (b) organic phosphorous compounds, which are usually ultraviolet light and must be protected.

Durable flame-resistant finishes

Durable Flame Retardants Sustainable ends to multiple washing processes are durable. These are harder to apply and more complex. While several treatments have been mentioned in the literature, only a small number have survived the time test. The treatment of ammonia by thermal heat treatments includes phosphorus-oriented compounds, pre-condensates and fibre responsive compounds which require multiple steps in the process. [46, 47]

Some of the more important requirements for ideal durable retardant is the following

- Capability to impart resistance to fire, that is to say, resistance to flame and glow
- Application for all weight textiles and preferably water solutions
- Easy to apply on existing finishing equipment, preferably without danger.
- Sufficient laundering and dry-cleaning resistance
- Failure to act physiologically
- Hard char production if exposed to fire or powerful radiation
- Adverse effect on the physical properties of the fabric is little
- Reasonable cost resistance to blueberries, especially hypochlorite.

The surface of fibres may be fitted with a durable flame retardant and/or penetrate the fibres. The retardant materials can be a polymer or a surface bonded with a polymer to be durable on the surface. Thermoplastic polymers provide the best results from surface deposits. In general, this retardant gives rigidity or adversely affects certain other physical characteristics of cellulosic materials since the weight addition required by surface deposition is up to about 8 percent.

in-site polymerization techniques

Flame retardants can be produced using on-site polymerization techniques. To be fixed without adverse effects of strength in cellulosic fibres.

Therefore, a typical flame retardant formula can be concluded Include:

- Reactive Phosphorous Compound
- Catalyst and/or buffer and softener and wetting agent

In recent years, significant efforts have been made to develop a flammable retardant lasting press (FR-DP) finish for lightweight fabrics. Incorporating the cross-linking agents N-methylol with N-P synergism has enhanced phosphorus flame retardant compounds' efficacy

The most important examples of this group are:

1. Tetrakis(Hydroxymethyl)Phosphonium Derivatives
2. Phosphate Esters with Bromine as Durable Flame Retardants
3. Aziridinyl Compounds
4. Phosphonates and Phosphine Oxides as Flame Retardants
5. N-Methyloldimethyl Phosphonopropioamide (PYROVATEX CP)
6. Phosphono Acetamide

Inorganic Flame Retardants materials

Multiple inorganic flammable systems have been discussed. During the decomposition of inorganic systems, metal oxide residues are formed. [48]

At the beginning of the 1970s, alumina trihydrate (ATH), as well as zinc borate and antimony compounds, were the major inorganic flame retardants used. Although antimony was one of the best-used flame retardant compounds in the years, it was costly and thus used sparingly. [49]

Kaolin–BPEI–DAP–urea coatings on printing

towel have been used with a modified laboratory pad-steam unit (Mathis, model PSA-HTF, Continuous layer by layer process of mercerized print cloth, twill cotton fabrics and fleece.

The pad-steam unit was switched from the steam range and the fabrics were loaded directly into the rink section.

Two separate chambers for rinsing. The textiles have been cut to inches in width. The rinse cabinets were sewn together to make an ongoing textile for the processing and hand-fed through each roller before the ends. aqueous BPEI (cationic) solution, containing urea and diammonium phosphate, was filled in the first rinsing chamber (DAP). The second chamber was filled with aqueous kaolin (anionic) solution with NaOH. Kaolin nanoparticles were scattered with a scraper in deionized water, and a stable colloidal solution was found. The multilayer continuous process of deposition consisted of two stages, which were diverted into a BPEI solution by cotton through a roller and the immersion in the kaolin, which also passes through a roller, of the fabrics immediately followed. The two steps were repeated constantly for each formulation without rinsing, with a rolling speed and a pad pressure.

After the deposition was completed, the pad-steam unit rinsing chambers were drained and the material removed. The cotton fabrics were dried with a constant dryer (Mathis, KTF-S model) at a speed. and were cured. The textile was immediately put out of the heating oven in a desiccator to cool down to room temperature and obtained its weight from the cooling process. Weighed all of the samples before and after the treatment. The values for add-on percentage have been fitted to the equation. or add-on levels. [50]

Flammability Tests for Textile

Develop standard tests for flammability assessment and other polymers combustion properties. The norms and codes for the classification of substances in most countries vary greatly from one test to another in terms of nature, the shape and the dimensions of the polymeric material to be tested; however, the experimental setup used for existing standard tests varies significantly ASTM E 84 [51] Steiner Tunnel, and the National Standards Bureau (NBS) smoke chamber ASTM E 662 [52] are the most common fire tests. These tests can, however, only be used as guides and have precise and reproductive problems.

Most of the fire tests used to relate to the assessment of the following material's fire properties. The facility of ignition—how the material ignites readily. Fire is spreading—how quickly fire is spreading over a surface. Heat release rate - how much heat is released and how soon.

A TGA Q500 thermal gravitational analyser was used to measure TGA (TA Instruments). At a heating rate under nitrogen with a flow rate, a sample was heated from room temperature. Average thermal decomposition parameters were obtained in three runs. Vertical flammability tests (Govmark Organization, Inc.) and LOI (Bynisco Polymer) were performed on all twill control and all the treated fabrics. For vertical flammability, the specimen sizes of these tests.

The vertical flame test placed the specimen upwards of the controlled fire, exposed it for a certain period and measured a post-glow time. Char length has been measured with a specified force, and evidence has been noted of melting or dropping. The LOI test supported a small sample of fabric vertically in a combination of oxygen and nitrogen which flowed up a clear chimney.

The following burning behaviour of the sample was observed when the upper end of the sample was ignited to record the period during which burning continued, or the sample length burned. A series of tests were carried out in different oxygen for the minimum concentration of oxygen levels.

Limiting oxygen index the LOI indicates the minimum oxygen required for candle-like flame in an atmosphere of oxygen and nitrogen when a sample is burned. Textiles are considered to be flammable when their LOI values are in nitrogen-oxygen and when LOI values, they are considered fire retardant. For these samples of flame retarded test fabric, open fire tests should be carried out horizontally and vertically in these LOI values. The sample did not glow after the fire was extinguished by itself after a very short after fire time and had a char length that did not equal the length of the test sample when it passed an open fire test.

Summary

The self-assembly has succeeded with the layer-by-layer application of a wide range of functional nanocoatings to textiles, but often takes time and is labour-intensive. deposition processes This work involved developing a continuous LBL self-assisted deposition process, which was applied to four different cotton fabrics with flame retardant nanocoatings. The four

different fabrics of aqueous solution were used by a modified pad-steam unit to provide a total alternating laies bilayers of kaolin nanoparticles and urea and DAP bPEI. The SEM images of the treated fabrics revealed that even coatings that were completely covered by the continuous LBL deposition process produced. In a variety of flammability testing methods, the flame retardant properties of coated materials were characterised and found to be considerably larger than the uncoated materials. The print cloth, mercerized printing cloth, twill, and fleece fabrics passed the vertical flame test when add-on value. In all cases, the length of the wood of the treated textiles was less. the time after and after flame. These initial results form the basis for the development and further development of commercial-grade systems, which can continuously apply functional nano-coatings to various surfaces and substrates.

Reference

1. Nishiyama, Y., "Structure and Properties of the Cellulose Microfibril". *Journal of Wood Science*, **55**(4), 241-249 (2009).
2. Ebrahim, S.A., Hassabo, A.G. and Othman, H., "Natural Thickener in Textile Printing". *Journal of Textiles, Coloration and Polymer Science*, - (2021).
3. El-Sayed, E. and Hassabo, A.G., "Recent Advances in the Application of Plasma in Textile Finishing". *Journal of Textiles, Coloration and Polymer Science* (2021).
4. Hamdy, D.M., Hassabo, A.G. and Othman, H., "Recent Use of Natural Thickeners in the Printing Process". *Journal of Textiles, Coloration and Polymer Science*, (2021).
5. Ragab, M.M., Hassabo, A.G. and Othman, H., "Recent Use of Synthetic Thickeners in Textile Printing Process". *Journal of Textiles, Coloration and Polymer Science*, (2021).
6. Agoudjil, N., Sicard, C., Jaouen, V., Garnier, C., Bonnin, E., Steunou, N. and Coradin, T., "Design and Properties of Biopolymer--Silica Hybrid Materials: The Example of Pectin-Based Biodegradable Hydrogels". *Pure & Applied Chemistry*, **84**(12), 2521 (2012).
7. Möller, M. and Popescu, C., "Natural Fibres", in *Sustainable Solutions for Modern Economies*, R. Hofer, Editor. p. 368-393 (2009).
8. Moran-Mirabal, J.M., "Advanced-Microscopy Techniques for the Characterization of Cellulose Structure and Cellulose-Cellulose Interactions", in *Cellulose - Fundamental Aspects*, T.v.d. Ven and L. Godbout, Editors Intech (2013).

9. Hassabo, A.G., "Synthesis and Deposition of Functional Nano-Materials on Natural Fibres", RWTH Aachen University, Germany, **154** (2011).
10. Hassabo, A.G., "New Approaches to Improving Thermal Regulating Property of Cellulosic Fabric". *Carbohydrate Polymers*, **101**(0), 912-919 (2014).
11. Hassabo, A.G., Mendrek, A., Popescu, C., Keul, H. and Möller, M., "Deposition of Functionalized Polyethylenimine-Dye onto Cotton and Wool Fibres". *Research Journal of Textile and Apparel*, **18**(1), 36-49 (2014).
12. Hassabo, A.G., Schachschal, S., Cheng, C., Pich, A., Popescu, C. and Möller, M., "Poly (Vinylcaprolactam)-Based Microgels to Improve Gloss Properties of Different Natural Fibres". *Research Journal of Textile and Apparel*, **18**(1), 50-63 (2014).
13. Hassabo, A.G., Erberich, M., Popescu, C. and Keul, H., "Functional Polyethers for Fixing Pigments on Cotton and Wool Fibres". *Research & Reviews in Polymer*, **6**(3), 118-131 (2015).
14. Hassabo, A.G., Mohamed, A.L., Wang, H., Popescu, C. and Moller, M., "Metal Salts Rented in Silica Microcapsules as Inorganic Phase Change Materials for Textile Usage". *Inorganic Chemistry: An Indian Journal*, **10**(2), 59-65 (2015).
15. Hassabo, A.G., Nada, A.A., Ibrahim, H.M. and Abou-Zeid, N.Y., "Impregnation of Silver Nanoparticles into Polysaccharide Substrates and Their Properties". *Carbohydrate Polymers*, **122** 343-350 (2015).
16. Ferdous, D., Dalai, A.K., Bej, S.K. and Thring, R.W., "Pyrolysis of Lignins: Experimental and Kinetics Studies". *Energy & Fuels*, **16**(6), 1405-1412 (2002).
17. ASTM Standard Test Method (D2863 – 17), "Standard Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)", ASTM International: West Conshohocken, PA (2018).
18. ASTM Standard Test Method (D6413–99), "Standard Test Method for Flame Resistance of Textiles (Vertical Test)", (2017).
19. Chapple, S. and Anandjiwala, R., "Flammability of Natural Fiber-Reinforced Composites and Strategies for Fire Retardancy: A Review". **23**(6) 871-893 (2010).
20. Cheng, T., Lin, T., Brady, R. and Wang, X., "Fast Response Photochromic Textiles from Hybrid Silica Surface Coating". *Fibers and Polymers*, **9**(3), 301-306 (2008).
21. Berendjchi, A., Khajavi, R. and Yazdanshenas, M.E., "Fabrication of Superhydrophobic and Antibacterial Surface on Cotton Fabric by Doped Silica- Based Sols with Nanoparticles of Copper". *Nanoscale Research Letters*, **6**(1), 594 (2011).
22. Kartick, K.S., Santanu, B. and Chattopadhyay, S.K., "Sustainable Flame-Retardant Finishing of Textiles Advancement in Technology", in *Handbook of Sustainable Apparel Production* CRC Press (2015).
23. Pal, A., Samanta, A.K., Bagchi, A., Samanta, P. and Kar, T.R., "A Review on Fire Protective Functional Finishing of Natural Fibre Based Textiles: Present Perspective". *Current Trends in Fashion Technology & Textile Engineering*, **7**(1), 11-30 (2020).
24. Mohamed, A.L. and Hassabo, A.G., "Flame Retardant of Cellulosic Materials and Their Composites", in *Flame Retardants*, P.M. Visakh and Y. Arao, Editors Springer International Publishing. p. 247-314 (2015).
25. Mohamed, A.L., El-Sheikh, M.A. and Waly, A.I., "Enhancement of Flame Retardancy and Water Repellency Properties of Cotton Fabrics Using Silanol Based Nano Composites". *Carbohydrate Polymers*, **102**, 727-737 (2014).
26. Hebeish, A., Zahran, A.H., Rabie, A.M. and El-Naggar, A.M.K., "Modification of Partially Carboxymethylated Cotton Via Crafting with Acrylic Acid and Styrene Using Gamma Radiation". *Journal of Applied Polymer Science*, **32**(8), 6237-6257 (1986).
27. Horrocks, A.R., Anand, S.C. and Sanderson, D., "Complex Char Formation in Flame Retarded Fibre-Intumescent Combinations: 1. Scanning Electron Microscopic Studies". *Polymer*, **37**(15), 3197-3206 (1996).
28. Posner, S., "9 - Developments in Flame Retardants for Interior Materials and Textiles", in *Interior Textiles*, T. Rowe, Editor Woodhead Publishing. p. 211-228 (2009).
29. Huang, X., Tian, Z., Zhang, D., Jing, Q. and Li, J., "The Synergetic Effect of Antimony (Sb₂O₃) and Melamine Cyanurate (Mca) on the Flame-Retardant Behavior of Silicon Rubber". *Polymer Bulletin*, **78**(1), 185-202 (2021).

30. LEWIN, M., "Synergistic and Catalytic Effects in Flame Retardancy of Polymeric Materials; an Overview". *Journal of Fire Sciences*, **17**(1-2) (1999).
31. Bakoš, D., Košík, M., Antoš, K., Karolyová, M. and Vyskočil, I., "The Role of Nitrogen in Nitrogen-Phosphorus Synergism". **6**(1), 10-12 (1982).
32. Rietra, R.P.J.J., Heinen, M., Dimkpa, C.O. and Bindraban, P.S., "Effects of Nutrient Antagonism and Synergism on Yield and Fertilizer Use Efficiency". *Communications in Soil Science and Plant Analysis*, **48**(16), 1895-1920 (2017).
33. Zhang, P., Fan, H., Tian, S., Chen, Y. and Yan, J., "Synergistic Effect of Phosphorus-Nitrogen and Silicon-Containing Chain Extenders on the Mechanical Properties, Flame Retardancy and Thermal Degradation Behavior of Waterborne Polyurethane". *RSC Advances*, **6**(76), 72409-72422 (2016).
34. El-Sabour, M.A., Mohamed, A.L., El-Meligy, M.G. and Al-Shemy, M.T., "Characterization of Recycled Waste Papers Treated with Starch/Organophosphorus-Silane Biocomposite Flame Retardant". *Nordic Pulp & Paper Research Journal*, **36**(1), 108-124 (2021).
35. Mohamed, A.L. and Hassabo, A.G., "Review of Silicon-Based Materials for Cellulosic Fabrics with Functional Applications". *Journal of Textiles, Coloration and Polymer Science*, **16**(2), 139-157 (2019).
36. Hassabo, A.G. and Mohamed, A.L., "Novel Flame Retardant and Antibacterial Agent Containing Mgo Nps, Phosphorus, Nitrogen and Silicon Units for Functionalise Cotton Fabrics". *Biointerface Research in Applied Chemistry*, **9**(5), 4272 - 4278 (2019).
37. Banks, M., Ebdon, J.R. and Johnson, M., "The Flame-Retardant Effect of Diethyl Vinyl Phosphonate in Copolymers with Styrene, Methyl Methacrylate, Acrylonitrile and Acrylamide". *Polymer*, **35**(16), 3470-3473 (1994).
38. Horrocks, A.R., "Flame-Retardant Finishing of Textiles". *Coloration Technology*, **16**(1), 62-101 (2008).
39. Ozcan, G., Dayioglu, H. and Candan, C., "Application of Flame Retardant Products to Knitted Fabric". *Indian Journal of Fibre & Textile Research*, **31**(June) 330-334 (2006).
40. Salmeia, K.A., Gaan, S. and Malucelli, G., "Recent Advances for Flame Retardancy of Textiles Based on Phosphorus Chemistry". *Polymers (Basel)*, **8**(9), (2016).
41. Ömeroğulları Basyigit, Z. and Kut, D., "Formaldehyde-Free and Halogen-Free Flame Retardant Finishing on Cotton Fabric". *Tekstil Ve Konfeksiyon*, **28**(2018).
42. Mercimek, H., "Effect of Chemicals and Binders on the Durability of Flame Retardant Treated Cotton Nonwovens", University of Tennessee: Knoxville (2010).
43. Reid, J.D. and Mazzeno, L.W., "Preparation and Properties of Cellulose Phosphates". *Industrial & Engineering Chemistry*, **41**(12), 2828-2831 (1949).
44. Granja, P.L., Pouységu, L., Pétraud, M., De Jéso, B., Baquey, C. and Barbosa, M.A., "Cellulose Phosphates as Biomaterials. I. Synthesis and Characterization of Highly Phosphorylated Cellulose Gels". *Journal of Applied Polymer Science*, **82**(13), 3341-3353 (2001).
45. Cobb, A.W. and Walton, J.H., "The Reaction of Hydrogen Cyanide with Sulfuric and Phosphoric Acids". *The Journal of Physical Chemistry*, **41**(3), 351-363 (1937).
46. Alongi, J., Carosio, F. and Kiekens, P., "Recent Advances in the Design of Water Based-Flame Retardant Coatings for Polyester and Polyester-Cotton Blends". *Polymers (Basel)*, **8**(10)(2016).
47. Mayer-Gall, T., Plohl, D., Derksen, L., Lauer, D., Neldner, P., Ali, W., Fuchs, S., Gutmann, J.S. and Opwis, K., "A Green Water-Soluble Cyclophosphazene as a Flame Retardant Finish for Textiles". *Molecules*, **24**(17), 3100 (2019).
48. Weil, E. and Levchik, S., "Flame Retardants, Phosphorus", (2017)
49. Mishra, R., Eren, T. and Wang, D.-Y., "Inorganic Polymers as Flame-Retardant Materials: Synthesis, Properties, and Emerging Applications in Materials and Life Sciences". p. 197-241 (2019).
50. Lvov, Y., Decher, G. and Moehwald, H., "Assembly, Structural Characterization, and Thermal Behavior of Layer-by-Layer Deposited Ultrathin Films of Poly(Vinyl Sulfate) and Poly(Allylamine)". *Langmuir*, **9**(2), 481-486 (1993).
51. ASTM Standard Test Method (E84-13A), "Standard Test Method for Surface Burning Characteristics of Building Materials", ASTM International: West Conshohocken, PA (2001).
52. ASTM Standard Test Method (E662), "Standard Test Methods for Optical Density of Smoke Generated by Solid Materials", ASTM International: West Conshohocken, PA (2011).

مواد غير عضوية صديقة للبيئة للأقمشة القطنية المضادة للاشتعال

مارينا يوسف سليمان^١ و احمد جمعه حسبو^٢

^١ قسم طباعة المنسوجات والصبغة والتجهيز - كلية الفنون التطبيقية - جامعة بنها - بنها - مصر.

^٢ المركز القومي للبحوث - شعبة بحوث الصناعات النسيجية - قسم التحضيرات والتجهيزات للألياف السليلوزية.

٣٣ شارع البحوث (شارع التحرير سابقاً) - الدقي - ص. ١٢٦٢٢ - الجيزة - مصر.

يتضمن هذا المرجع ثلاثة موضوعات رئيسية: (١) التركيب الفيزيائي والكيميائي للمواد السليلوزية. (٢) التجهيز للمواد السليلوزية ومثبطات اللهب ؛ و (٣) السليلوز ومعالجته لتأخير اللهب والنار. يتناول الموضوع الأول بالتفصيل المصادر المختلفة للسليلوز وهيكلها الكيميائي والفيزيائي. يتضمن الموضوع الثاني تطبيق المواد النسيجية المثبطة للهب ويصف الاختلاف في تعريف المصطلحات المثبطة / المقاومة ، ويشرح بمزيد من التفصيل نظرية الاحتراق أو الاحتراق ، وآلية مثبطات اللهب. كما تم ذكر أنواع مختلفة من مثبطات اللهب الفوسفورية ، بما في ذلك أنواع وتصنيفات التشطيبات المثبطة للهب على أساس المتانة والطبيعة. في نهاية هذا القسم ، تم وصف العديد من اختبارات قابلية النسيج للاشتعال. يوضح الجزء الأخير من الفصل تحضير البولي إيثيلين أمين المتفرعة طبقاً طبقة تلو الأخرى لسطح مثبط للهب من الكاولين واليوربا والفوسفات (DAP) (BPEI).