



# A Review on Boron and Its Derivatives Used in Flame Retardant (FR) Applications for Cotton Fiber or Various Cotton Textile Fabric Forms

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## Abstract

In this compilation study, the application of boron and its derivatives used in flame retardant (FR) applications to the cotton fiber and its textile fabric forms were examined. The main purpose of this compilation study is which boron compounds are used and the optimization of the process parameters required for their successful FR application to cotton fiber or its various textile forms. Firstly; organoborons, borates, boric acids, and boron-phosphorus compounds can be successfully applied to cotton fiber or its textile fabric forms by paying attention to the process parameters. Boron and its derivatives can be successfully applied to cotton fiber or its textile fabric forms for FR applications. The general results for the successful application of Boron or its compounds to cotton fiber or various fabric forms are that the cotton fiber must have a plasma or chemical (bleaching or mercerization) pre-treatment process. Pyrene-1-boronic acid is the boron compound that is the most effective FR chemical. It should be used at a 30% concentration in boric acid applications. It should be applied with the bath (flote) ratio of 1:20, at temperatures between 40 °C and 80 °C for between 0.5 hours and 1 hour, especially with the use of high-concentration sodium hypophosphite (SHP) salt as a catalyst in the impregnation process. It should be washed in distilled water with soap at room conditions for between 5 minutes and 10 minutes and rinsed in the washing and rinsing processes. It should be dried and fixed for 3 minutes to 5 minutes at temperatures between 100 °C and 170 °C in the drying and fixing processes. (Specifically; 120 °C and 3 minutes). pH should be alkaline environment (especially pH 10). Thus, maximum FR effectiveness will be achieved in cotton fiber or various fabric forms of cotton fiber. Moreover, thermal degradation temperature (approximately 280 °C) and tensile strength values (approximately 30%) will decrease. Fiber breaks will be observed in its surface morphology. New bonds such as P=N, P=O, and P-O-C will be observed in FT-IR chemical analysis. Its LOI values will increase from 18 to 41.

**Keywords:** Boron; Boron compounds; Flame retardant; Cotton fibers

## Introduction

The aim of this review study includes the optimization of pre-treatment processes, FR chemicals, and FR process parameters based on the results of various experimental studies of FR applications of boron and its derivatives applied to a cotton fiber or various fabric forms.

### Boron and its compounds

Boron and its compounds have been discovered many years

ago and are still used today. They determined that 4000 years ago, Babylon imported boron from the Far East for the gold industry, and in ancient Egypt, borax was used in mummification, treatment, and metallurgical applications. Later, Saudi Arabia imported boron from the Far East for its gold industry. It was transferred from the cities of Mecca and Medina in Arabia to the USA, France, and Italy for various purposes over the years, starting under the leadership of Marco Polo during the times of geographical discoveries [1].

Boron element is the 5th element in the periodic table and is a hard semimetal with a brittle structure [7,9]. It is group IIIA and its atomic number is 10 in the periodic table [8,9]. The density of boron is in the range of 2.34 g/cm<sup>3</sup> to 2.52 g/cm<sup>3</sup>. Boron has a melting temperature of above 2000 °C temperature. Its elasticity modulus is between 185 GPa and 227 GPa and is found in nature in the form of compounds in minerals [9]. The element boron is present in nature in 230 variations in the form of compounds in minerals containing sodium, calcium, and magnesium oxides containing crystal water [1,7,8]. Boron are found in forms such as boron nitride (BN), boron carbide (B<sub>4</sub>C), borates (sodium borate, zinc borate (ZnB), calcium borate (CaB), ammonium borate (AB), organo boron compounds (borate esters, boroxine), boron-phosphorus compounds, boron-halogen compounds, boron-sulfur compounds, boron-nitrogen compounds, borax (BX), boric acid (BA), boron oxide (BO), boron phosphate (BP), boranes, tincal (Na<sub>4</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>2</sub>O), kernite (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·4H<sub>2</sub>O), colemanite (Ca<sub>2</sub>B<sub>6</sub>O<sub>11</sub>·5H<sub>2</sub>O) and ulexite (NaCaB<sub>5</sub>O<sub>9</sub>·8H<sub>2</sub>O) [1,4-10]. Essentially, bioactive molecules containing boron are of two types. One type of molecule contains a single boron atom, while the other is in the form of a boron cluster. Boron can instantly transform from a trigonal plane (sp<sup>2</sup>-hybridized). It transforms from the hybridized form, which is a neutral form, to an anionic tetrahedral (sp<sup>3</sup>-hybridized) form. It forms compounds containing a single boron atom when used under physiological conditions [5]. The icosahedral cage structure of boron has 12 corners and five-fold rotational symmetry (Ih) and also has a 20-face regular lattice structure consisting of twelve boron atoms at corner positions [9]. It is known that n varies from B<sub>3</sub> to B<sub>100</sub> atoms in B<sub>n</sub> structures, which are boron clusters that are actively investigated. As n increases, the number of isomers of boron also increases and thus various isomeric structures are formed in terms of B-B bonds [3,6,9]. Due to the isoelectronic nature of C=C and B-N, as the bonding capacity increases, the use of organic boron increases [7]. As n changes, the cage number increases and also changes. Generally, cage structures are such as fullerene, buckyball, volleyball, icosahedral, tetrahedral, octahedral, rhombohedral, endohedral, nanoribbons, 1D sheets, and 2D sheets [3,4,9,10]. n is in an extremely stable phase in the atomic number range of 13 to 24 and thus dimensionally It is stable. Moreover, its mechanical properties are very high [4]. Temperature has an extremely high effect on boron's lattice structures, dimensions, geometry, and mechanical properties. Moreover, the electron band gap also changes [1-10]. It contains the boron element in its various compound forms and is the richest deposit in the world for Turkey at 72.8% [1,8]. Its areas of use are health, textile, metallurgy, nuclear reactors, glass, ceramics, agriculture, energy, hydrogen storage, battery, electronics, and cleaning sectors [1,2,5-10].

## Flame retardant (FR)

Flame retardant (FR) applications in textiles for their FR values vary depending on the process parameters, such as concentration, temperature, and time, the applied process types, and the chemical types and concentrations used [8,11-29]. While in the gas phase, organoboron compounds release some free radicals, giving textile products FR properties [8,11]. The logic of FR applications is the

creation of a combustion reaction in the gas phase on the textile surface with the effects of fuel, oxygen, and heat, and the detection of pyrolysis behavior on textile surfaces [13,16,24]. In other words, flaming combustion is an oxidative process in the gas phase that requires oxygen or air from an atmosphere [14]. Moreover, it is to minimize the risk by preventing or reducing the possibility of the material igniting against the speed of flame spread [14]. At the same time, it is the measurement of the minimum oxygen consumption required for combustion [11]. The combustion reaction is exothermic. As a result of this situation, some of the heat reaches the surface of the material. It is transferred and causes both material degradation and a self-combustion cycle in the material [14]. The combustion process takes place in 3 stages. These are condensed, mesophase, and gas, respectively.[19,20,24]. There are generally 3 basic components in FR applications. These are a source of acid, a carbonizing agent, and a blowing agent [18]. FR should provide FR efficiency, low smoke and toxicity, biocompatibility, cost efficiency, washing durability, and ease of scaling for FR applications [11]. LOI, vertical combustion test, MCC, and CC methods and tools are used to analyze flammability behavior. LOI is the most used method [11,14-18]. LOI test standards are ISO 4589 and ASTM D2863 [16]. LOI < 21.0 (of air natural oxygen content) is quite high and is flammable. LOI is moderately flammable between 21.0 and 25.0. If LOI > 25.0, it begins to pass various national and international standards for textile fibers in FR applications [14,16]. The LOI value of cotton fiber is 18 [30]. ASTM D6413-08 is used as the vertical burning test standard for FR applications. Characteristic parameters for FR applications are ignition possibility, rate and extent, flame spread, flame spread time, heat release, burning speed, combustion heat, burning time, coal length, and coal residue [11,15-19,26]. FR applications are considered successful applications to be counted in textile materials. When the flame is applied to the textile material, it must have a burning time longer than 12 seconds. Moreover, when the burning speed (mm/s) is applied low, the thermal degradation of the textile material decreases and thus the textile material maintains its dimensional stability [16]. Auxiliary chemicals are used at 50% concentration for successful FR applications [19]. Textile fibers have different burning behaviors because they have different chemical structures [11-20]. Chain branching, double bonds, or oxygen in the polymer weaken the polymer stability throughout the polymer chain. On the other hand, aromatic rings, high molecular weight, and cross-linking increase polymer stability [14]. The synthetic-based fibers such as meta-aramid, para-aramid, PBI, PBO, PMMA, PET, PAN, PVC, PA 6, PA 6.6, PEO, PTFE, PU, PC, PS, PE, PP, and protein-based fibers such as silk and wool and also cellulosic based fibers such as cotton, linen, jute, and viscose can be used as textile raw materials in the textile industry [8,11,12,14-20,21,23-30]. The first studies have been carried out from the early 1950s to the 1980s. Experimental studies in this field have increased significantly since the late 1990s [8,15,16,20]. It has been determined that the compound type of boron, its concentration, the type of fiber used, process temperature, process time, process liquor ratio, and process pH affect the LOI value, which is the FR index. Some chemicals are generally used in FR applications such as proban®

CC (tetrakis (hydroxymethyl) phosphonium salt), pyrovatex® CP (N-hydroxymethyl-3-(dimethoxy-phosphine acyl) propionamide), chlorine, halogen, nitrogen, silicon, sulfur, formaldehyde, melamine, aluminum hydroxides, iron hydroxides, metal hydroxides, biomacromolecules such as proteins, deoxyribonucleic acids, ammonium salt, pentaerythritol, ammonium polyphosphate, phosphorus compounds, urea's, halides, silica, zinc acetate dihydrate, zinc oxide, phytic acid, triazine, phosphazene, boric acids, pyrene-1-boronic acids, borates such as ammonium pentaborate, sodium metaborate, barium metaborate, and zinc borates [8,11-13,15-18,21,24,25,27-30]. FR chemical compounds containing boron ensure the release of water during heating as a result of a gradual endothermic reaction in FR applications. The mechanism by which water is released is that the mixture first dissolves within itself. The water of hydration then swells to form a foamy substance and thus loses water by boiling. Boron salts are easily soluble in water and are used as FR chemicals, especially for cellulosic-based textile fibers. FR mechanism is primarily physical and forms a glassy coating that prevents air passage. It traps the volatile pyrolysis products in the cellulosic-based textile fiber thanks to this coating. Thus, it prevents oxygen diffusion from the inside of the textile fiber to the outside. This means preventing the exothermic combustion reaction from spreading [24,30]. They should be non-toxic, harmless to health and the environment, easy to apply, able to maintain their structures for many fastness tests after application, maintain dimensional stability in textile materials, be low-cost, and not disrupt the aesthetic structure for their FR applications [15,16,24]. The processes generally applied in FR applications are such as coating methods, UV grafting, ultrasound, sol-gel, impregnation, dipping, and padding processes. Various plasma or chemical-based pre-treatment processes are generally applied before FR applications when natural fibers are used [11,12,15-18,21,24,25,27-29].

### FR applications of boron and its derivatives on cotton fiber or various cotton textile fabrics

Cotton, a naturally occurring form of cellulose, accounts for approximately between 48% and 50% of the world textile market [14,21]. It has been reported that the annual production capacity of cotton fiber in the world is estimated to be between  $10^{11}$  and  $10^{12}$  tons [23]. Cotton fiber is a cellulosic-based natural textile fiber. The chemical structure of cotton fiber consists of  $\beta(1-4)$ -D-glucose monomers, which are linear and homopolysaccharides [21,23,30]. The degree of polymerization consists of several hundred to more than ten thousand monomer units [21]. The chemical structure of cotton fiber contains between 88% and 96% cellulose [21,23,30]. The dimensional stability of cotton fiber depends on the amount of cellulose, processing conditions, and pre-treatment processes [23]. Cotton fiber can be used in applications such as home textiles, defense textiles, medical textiles, composite materials, and textile sensors [21-23,27-30]. It can be produced in woven or knitted fabric forms [21,22]. Before applying various finishing processes such as dyeing, printing, and finishing, it is generally subjected to pre-treatment processes using NaOH or KOH chemicals for cotton fiber [22]. Finishing processes applied to cotton fibers generally are

antimicrobial, antistatic, protection against ultraviolet radiation, flame retardancy, and self-cleaning [23-30]. Oxygen plays a dominant role in the breakdown and pyrolysis of cellulose, faster than an inert gas in an oxidative atmosphere at lower temperatures. In addition, to catalyzing the formation of volatile substances, oxygen creates reactions that promote coal [14]. Some chemicals such as proban® CC (tetrakis (hydroxymethyl) phosphonium salt), pyrovatex® CP (N-hydroxymethyl-3-(dimethoxy-phosphine acyl) propionamide), phosphorous compounds, polycarboxylic acids, pyrene-1-boronic acids, boric acids, triazine, and halogen are effective for its FR applications but also they are unhealthy due to the release of formaldehyde thanks to the P-N bonds in their chemical structures [11,16,21-23,26-30]. Moreover, it is effective in FR applications for cotton fibers due to the formation of C-O-C covalent bonds through nucleophilic substitution of halogen compounds or triazine chlorides with the -OH group in cellulose [11,15]. FR chemicals are generally used in cotton fibers at concentrations between 4% and 40%. As the concentration increases, the FR effect (LOI) increases. Process methods such as plasma treatment and mercerization (pre-treatment), nanocoatings, sol-gel, impregnation, dip coating, dipping, padding, and UV grafting processes, the temperature range from 40 °C to 80 °C and for between 0.5 and 1 hour as time. As the temperature increases, the LOI value increases, but it also causes degradation of the cotton fiber [11,12,17,21-23,26-30]. Pad-dry, pad-cure, or pad-steam methods are applied as post-processes [11-13,15-17,21-23,27-30]. The FR feature is generally given on cotton fiber or cotton textile fabrics by using fixing at temperatures ranging from 100 °C to 170 °C and for periods ranging from 3 minutes to 5 minutes. Its LOI values reach over 25. Moreover, a soaping process is applied before the dipping and padding processes to maintain high LOI values after washing. Cross-linking chemical aids can also be used sometimes. Washing repetitions vary between 15 and 50 and LOI values vary between 25 and 46 [11,15,17,27]. Thermal degradation of cotton fiber in the air environment occurs in 3 stages. In the first stage, at temperatures between 300 °C and 400 °C, it causes the formation of both aliphatic coal and volatile compounds. In the second stage, at temperatures between 400 °C and 800 °C, some of the aliphatic coal is aromatic, and simultaneous carbonization and coal oxidation produce CO and CO<sub>2</sub>. In the third and final stage, coal is oxidized mostly to CO and CO<sub>2</sub> at a temperature of approximately 800 °C. In particular, it has 2 significant thermal degradation peaks at temperatures between 343 °C and 489 °C [15]. Boron compounds containing phosphorus, silicon, nitrogen, and carbon increase the FR index (LOI), especially in cellulosic-based fibers. Moreover, it resulted in a decrease in pHRR and an increase in washing durability. FR feature is given to cotton fiber thanks to the glassy layer formed as a result of the reaction between the -OH group attached to the 6th carbon of cellulosic-based fibers such as cotton and boron compounds [8]. In an experimental FR study conducted on cotton woven fabric with glycosyl cross-linked boric acid and ammonium salt of phytic acid included phytic acid at a concentration of 20 moles was mixed and dissolved with boric acid at a concentration of 40 mmol at 60 °C for 2 hours. Then, it was added with 500 mmol concentration of urea and 360 mmol concentration of distilled

water and continued to be dissolved at 90 °C for 8 hours. Afterward, it was subjected to the FR finishing process, thanks to the ultrasound process, at a liquor ratio of 1:20, 100% pick-up amount, at a temperature of 50 °C, and for 0.5 hours. As post-processes, it was dried at 80 °C for 0.5 hours and immediately fixed at 170 °C for 10 minutes. (In the fixing process, it was applied 3 times repeatedly and for 10 minutes in the 1st pass, 7.5 minutes in the 2nd pass, and 5 minutes in the 3rd pass, respectively). In conclusion; LOI values decreased from 26 to 22. From the 0th to the 40th after washing weight gain decreased from 18.5% to 14% for washing fastness. As the concentration increased from 0 g/l to 250 g/l, the whiteness index decreased from 61 to 20.2 for values after 30 washes. Tensile strength decreased from 496 MPa to 353 MPa. While the thermal degradation of raw cotton fabric was a maximum of 360 °C, the thermal degradation of finished cotton fabric was a maximum of 275 °C [25]. An experimental FR study conducted on cotton woven fabric with boric acid included boric acid along with various auxiliary chemicals. It was applied to cotton woven fabrics at concentrations of 10 g/l, 20 g/l, and 30 g/l, respectively, boric acids. It was dried at 85 °C and fixed. FR test was applied. Washing fastness was applied at 40 °C for 0.5 hours using detergent at a concentration of 4 g/l. Afterward, the tensile strength test was applied according to the DIN 54335 standard. In conclusion; in the FR test, flame propagation times (s) were in warp and weft directions respectively for the raw sample. They were 2.5 and 2.4. Moreover, 3.5; 3.4, 3.8; 3.6, 4.2; and 3.9, respectively with the addition of boric acid. It had a breaking force of 1524.3 and 752.95 N and a percent elongation at a break of 44.692 and 16.310% for raw samples, respectively for tensile strength. It had a breaking force of 1406.8 and 638.58 N and a percent elongation at break of 42.157 and 16.256% respectively, for 10 g/l addition of boric acid. It had a breaking force of 1323.7 and 612.98 N and a percent elongation at break of 42.082 and 15.663%, respectively, for 20 g/l addition of boric acid. It had a breaking force of 1125.9 and 610.52 N and a percent elongation at break of 41.571 and 14.680%, respectively, for 30 g/l addition of boric acid. Briefly; in the FR test, flame propagation time increased in both warp and weft directions. In the tensile strength test, both breaking force (N) and percent breaking elongation (%) decreased in both warp and weft directions [26]. Padding, drying, and fixation processes are applied for FR processes applied to cotton fabrics. In addition to FR chemicals such as ammonium phosphate, ammonium chloride, and borax, auxiliary chemicals such as cross-linker, catalyst (salt), wetting agent, and defoamer are used in various concentrations, temperatures, and durations [27]. In a general review of boric acid and its derivatives, cotton fiber is to form strong covalent bonds with boronate esters. Boronic acid and pyrene-1-boronic acid are extremely effective in FR applications on cotton fabrics. Pyrene-1-boronic acid is the boron compound that is the most effective FR chemical on cotton fabrics. It begins to degrade at temperatures between 100 °C and 255 °C. The main thermal degradation range is between 86 °C and 422 °C. It loses 50% of its mass at temperatures between 119 °C and 515 °C. Temperature ranges depend on the type and concentration of the boron compound.[28]. In an experimental FR application on cotton woven fabrics included the cotton woven fabric was washed with

25% concentrated aqueous soap in distilled water. It was rinsed for 24 hours. It was dried at 60 °C for 40 minutes. PHMGP and SPB were impregnated at 0.5% concentration for 5 minutes. Afterward, BL chemical was absorbed for 1 minute at concentrations of 5%, 10%, and 20%, respectively. Then, it was again dried at 60 °C for 40 minutes. In conclusion, when the concentration increased from 5% to 20% in the FR test, LOI values increased from 24.5 to 41.0. In FT-IR chemical analysis, new bonds such as P=O and P-O-C were formed. In thermal analysis, the initial temperature of thermal degradation for the raw sample is 326 °C. Temperatures of thermal degradation 288 °C, 282 °C, and 284 °C were measured for 5%, 10%, and 20% concentrations of BL, respectively. That was, the thermal degradation temperature decreased for cotton woven fabrics after BL addition [29].

## Conclusion

Boron is a semi-metal element found in various minerals in nature. 72.8% of the world's boron reserves are in Turkey. Boron compounds (especially salts) used as FR chemicals form a glassy solid layer on the textile material and increase FR values (LOI) by preventing the passage of oxygen or air from inside the textile material to the outside. Characteristic parameters for FR applications are ignition possibility, rate and extent, flame spread, flame spread time, heat release, burning speed, combustion heat, burning time, coal length, and coal residue. The flame propagation process is the most important parameter and should be as long as possible. Various FR chemicals used based on boron compounds are such as organoborons, borates, boric acids, pyrene-1-boronic acid, and boron-phosphorus compounds. These FR chemicals can be successfully applied to cotton fiber and its various fabric forms by paying attention to the process parameters for FR applications. Plasma or chemical (bleaching or mercerization) pre-treatment process must be applied to the cotton fiber or its textile fabric forms to provide LOI > 25.0. Pyrene-1-boronic acid is the boron compound that is the most effective FR chemical. It should be used at a 30% concentration in boric acid applications. The impregnation process should be applied with a bath (flote) ratio of 1:20, at temperatures between 40 °C and 80 °C for between 0.5 hours and 1 hour, especially by using high-concentration sodium hypophosphite (SHP) salt as a catalyst in impregnation process. It should be washed in distilled water with soap and rinsed at room conditions for between 5 minutes and 10 minutes in the washing and rinsing processes. It should be dried and fixed for between 3 minutes and 5 minutes at temperatures between 100 °C and 170 °C in the drying and fixing processes. (Specific parameters are 120 °C and 3 minutes). pH should be alkaline environment. (especially pH is 10). Thus, maximum FR effectiveness will be achieved in cotton fiber or various fabric forms of cotton fiber. Moreover, its thermal degradation temperature (about 280 °C) and its tensile strength values (about 30%) will decrease. Fiber breaks will be observed in its surface morphology. Its LOI values will increase from 18 to 41. In future studies, optimization results of this compilation study should be taken as a basis. New experimental studies can be carried out by using new boron compounds, or by changing their process parameters such as pH, concentration, temperature, and time with

those boron compounds with added auxiliary chemical.

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## Conflict of Interest

The author declares that there is no conflict of interest.

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