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Article

Enhancement of Flame Resistance of Cotton Fabrics Using Multilayer Biomaterial Coatings of Chitosan and Sodium Alginate

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Abstract

Cotton fabrics are widely used in textiles due to their comfort and breathability, but their high flammability (limiting oxygen index (LOI) $\leq$ 18%) poses serious safety risks. While conventional flame-retardant treatments often rely on synthetic chemicals or toxic additives, biobased alternatives remain underdeveloped. The flame resistance of cotton fabrics may be enhanced using multilayer biocoatings of chitosan and sodium alginate applied via layer-by-layer (LBL) assembly—a sustainable and scalable approach. Cotton samples were coated with chitosan and sodium alginate bilayers (1, 2, 5, and 10 layers) using the LBL method. Flame resistance was evaluated using vertical flame tests and limiting oxygen index (LOI) testing according to ASTM D2863-09. The sample coated with 10 bilayers significantly outperformed uncoated cotton and lower-layer samples. With a char length of 9.72 cm (68% reduction), no dripping was observed in the vertical flame tests, and the LOI value was 23.47% compared to uncoated cotton (LOI = 18.04%). These improvements were attributed to the formation of a cohesive and protective carbon layer, which is likely capable of inhibiting the formation of flammable gases. Biomaterial multilayer coatings made from biomaterials, such as chitosan and sodium alginate, represent a promising and environmentally friendly alternative to traditional methods in improving cotton's flame resistance. The development of this technology points to potential applications in protective textiles and industrial safety clothing. Notably, chitosan and sodium alginate coatings are biocompatible. The term "biomaterials" refers to materials intended for interaction with biological systems, particularly for biomedical-related applications. The term "biobased materials" is used exclusively to describe materials derived from renewable biological sources.

Keywords: flame retardancy; biomaterial coatings; layer-by-layer assembly; protective textiles; biodegradable polymers; fire resistance testing

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1. Introduction

Polymeric materials play a fundamental role in modern society, as they are used in numerous industrial, environmental, and medical fields, in addition to serving as substitutes for many other materials, such as wood and metals [1]. It is worth noting that their widespread use stems from their many advantages, including their high resistance to

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chemical environments, their function as electrical and thermal insulators, and their light weight—they are tens of times lighter than metals [2]. However, polymeric materials also have some drawbacks that limit their use, with the most prominent being their inability to withstand high temperatures. Therefore, the improvement of these materials to achieve more sustainable properties and increased flame resistance is necessary [3]. Flame resistance is crucial in textiles, preventing rapid ignition and protecting against sudden fires and heat hazards. Without it, textiles could ignite and spread flames, leading to significant property damage and loss of life. Cotton combustion also presents a major social challenge due to its serious impacts on worker safety. Furthermore, cotton burning releases pollutants, affecting air quality and potentially harming public health. Technically, cotton is highly flammable, burning easily and making its use in high-temperature environments dangerous. The scaffolding effect is also a significant concern. In cotton blends, cotton can char and promote the melting of synthetic fibers. For example, in a cotton–polyester blend, cotton forms char when exposed to high temperatures, while polyester melts above its melting point (260 °C) and tends to flow onto the charred cotton. Thus, the charred cotton may facilitate the burning of the molten polyester [4]. The properties of cotton have been broadly evaluated due to its widespread and varied applications in the clothing and textile industry. This may be attributed to its distinctive properties, including its moisture absorption, breathability, elasticity, and comfort. Despite its widespread use, cotton suffers from some drawbacks—most notably its high flammability, which may pose a serious threat to life and property. Its limiting oxygen index (LOI) for combustion ranges from 16% to 18%. Among the most prominent cotton-related incidents is a fire that caused burns among six workers at a cotton spinning mill in India, as well as a fire at the Nile Company's cotton gin in Egypt in 2013, which resulted in the burning of 19 bales of cotton [5]. The advantages of the use of cotton outweigh its disadvantages, and surface coating with a specific number of bilayers of an appropriate material may enhance its flame resistance. A multilayer coating composed of chatoyant and phosphocellulose (PCL) was deposited onto a cotton surface using a layer-by-layer application method. Cotton fabric coated with 20 bilayers, reflecting a PCL concentration of 2% by weight, did not sustain flame growth. However, the absence of information on whether dripping had been eliminated, the lack of a limiting oxygen index evaluation, and the lack of determination of the after-flame time limit the usefulness of these observations [6]. The flame resistance of cotton fabrics has also been enhanced by coating with a flame-retardant system consisting of chitosan and lignosulfonates using a layer-by-layer method. The flame resistance of this fabric increased with increasing chitosan content. Whether dripping occurred, or whether the cotton beneath the sample during testing was ignited, was not clarified [7]. Polyammonium phosphate and chitosan have been deposited on cotton fabrics; the improved behavior of the flame retardant was due to the formation of thermally stable residual carbon and the release of more non-combustible gases. The limiting oxygen index was not determined, and it was unclear whether the cotton beneath the sample during testing was ignited [8]. Fire-resistant and antibacterial cotton fabrics have been prepared by precipitating chitosan and ammonium phytate on the surface using a layer-by-layer method. A vertical flame test and limiting oxygen index testing demonstrated that, with an 8% increase in the weight of the CS/AP coatings, the coated cotton fabrics self-extinguished, with a limiting oxygen index for combustion of 27%. The presence of burning droplets during testing was not taken into account [9]. Flame-resistant cotton fabrics can be obtained by using an environmentally friendly flame-resistant coating consisting of biochitosan, phytic acid, and divalent metal ions. A horizontal flame test and the limiting oxygen index suggest that, with increasing proportions of coatings, flame resistance increases. The addition of metal ions apparently promotes the formation of char, which inhibits heat feedback from the combustion zone and hinders the formation of combustible gases, thus reducing the rate of flame spread on the treated cotton fabrics; this is reflected in an increase in the limiting oxygen index to 22% [10]. Dripping from a coated fabric during testing was reduced using a chitosan/phytic acid-based biocoating applied layer by layer. The polyester/cotton fabric coated with 20 bilayers displayed the best flame retardancy and least dripping during testing. It was unclear whether the cotton beneath the sample was ignited during testing [11]. Chitosan has been utilized as a key component in creating multifunctional fire-resistant and antibacterial coatings for cotton fabrics, where phosphite-fused chitosan (PCS) brought about a significant increase to 80.7%, and vertical burning was strongly reduced [12]. While previous studies have documented the combustion behavior of coated materials and flame resistance improvements for cotton fabrics, many lack comprehensive and systematic evaluations of all critical flammability parameters included in international standards—particularly the after-flame time, after-glow time, char length, dripping behavior during testing, and, critically, whether burning droplets or molten material ignite the cotton beneath the test sample. These parameters are essential in evaluating true fire safety performance according to the principles of ASTM D6413. Additionally, no work has systematically explored the minimum number of bilayers required to achieve complete flame test passage. Here, chitosan/sodium alginate multilayer coatings for cotton fabrics have been evaluated, with a particular focus on three objectives: (1) determining the minimum number of bilayers required for complete flame test passage according to ASTM D6413 principles; (2) comprehensively evaluating all vertical flame test parameters, including the after-flame time, after-glow time, char length, dripping behavior, and cotton ignition beneath the test sample—parameters often previously overlooked; and (3) validating and quantifying self-extinguishing behavior through limiting oxygen index (LOI) determination according to ASTM D2863-09. By addressing these previously unexplored parameters and using a simpler, more sustainable biocoating system, critical data for advancing flame-resistant cotton textiles, with practical applicability to industrial safety garments and protective textiles, have been obtained.

2. Materials and Methods

2.1. Materials

The cotton samples used in the experimental study were 100% woven turbine cotton, as shown in Figure 1. The cotton fabric was obtained from the Lattakia Textile Company (Lattakia, Syrian Arab Republic). Cotton is primarily composed of cellulose, with some other impurities (non-cellulosic materials) also found in its composition [13].

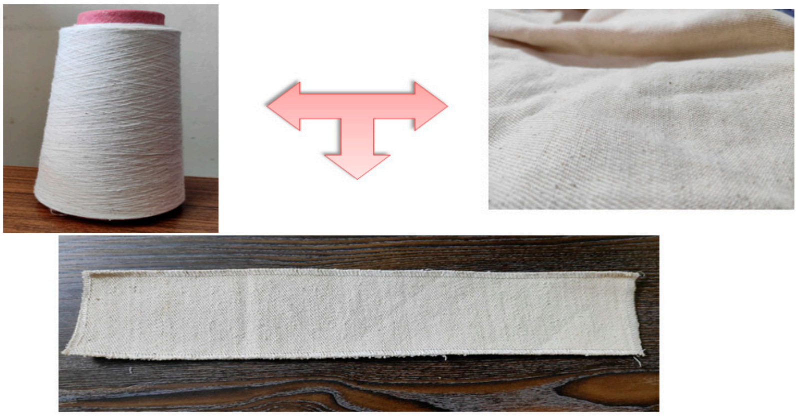

Figure 1. Cotton sample.

Chitosan, which is available in powder form, has a deacetylation rate of approximately 90%, and its chemical formula is shown in Figure 2. Chitosan is a versatile compound, having been used in flame extinguishers, food industries, medical applications, and more. This is due to its advantages of providing strong biocompatibility and biodegradability, non-toxicity, strong mechanical properties, a low cost, biosafety, and antibacterial properties [14].

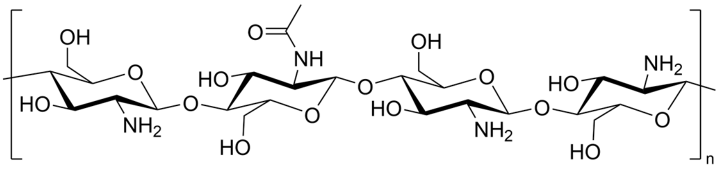

Figure 2. Chemical formula of chitosan.

Sodium alginate, which comes in powder form, has a molecular weight ranging from 32 to 400 g/mol. This substance has gained widespread popularity due to its biocompatibility and biodegradability properties, in addition to being a non-toxic substance [15]. The chemical formula is shown in Figure 3.

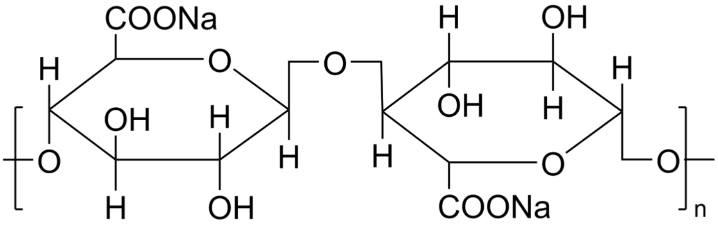

Figure 3. Chemical formula of sodium alginate.

Here, 100% pure, concentrated acetic acid was used. It is a carboxylic acid that is widely used in many industrial fields. Acetic acid is a clear, colorless liquid with a pungent odor. It affects the mucous membranes and can therefore cause difficulties in breathing [16].

Deionized water was used to prepare chitosan and sodium alginate solutions. As is well known, deionized water is free of any impurities or minerals, such as calcium, magnesium, iron, and others. Table 1 reports some of the properties of the materials used.

Table 1. Specifications of materials used.

Material	Purity	Trade Name/Supplier	Specifications
Cotton fabric	100% woven	Lattakia Textile Company	Turbine-woven
Acetic acid	100%	Acetic acid (glacial)	Concentrated, reagent-grade
Deionized water	100%	Rahmoun/Solar Power	DI water, mineral-free
Chitosan	~90%	Commercial powder	Deacetylation rate ~90%
Sodium alginate	Commercial	Powder form	MW: 32–400 g/mol

2.2. Equipment

Several devices were used to conduct the experimental study, which involved preparing sample solutions. A vertical flame tester was also designed to evaluate the combustibility of cotton samples coated with paint layers, while a limiting oxygen index tester was used to evaluate the relative flammability of the cotton sample.

The electronic balance used in the laboratories of the Faculty of Technical Engineering at Tartous University, was manufactured by the German company Sartorius (Göttingen, Lower Saxony, Germany). This digital balance has accuracy of approximately 0.0004–0.0001 g, and it can measure weights ranging from 50 g to a maximum of 1000 g.

An electric mixer was used to prepare the sample solutions. The minimum rotational speed was 40 rpm, while the maximum rotational speed was 2000 rpm. This mixer was located in the laboratory of the Faculty of Technical Engineering at Tartous University and was manufactured by the German company Heidolph (Schwabach, Bavaria, Germany).

The vertical flame test is one of the tests listed in ASTM D6413, which is used as a standard for measuring the flammability of polymeric materials. The vertical flame test is used to assess initial ignition, as it is one of the fastest and simplest tests, with a flame applied once for a continuous period of 12 s [17]. Several important parameters can be obtained from the vertical flame test, such as the after-flame time, after-glow time, char length, dripping, and cotton ignition [18]. The vertical flame tester was designed according to specific and carefully considered dimensions.

The vertical flame test was conducted in the laboratory of the Faculty of Engineering and Technology at Tartous University (Tartous, Syrian Arab Republic). Cotton samples coated with the required number of double layers were obtained, based on ASTM D6413 specimen dimensions and a 12 s exposure time, with an open-chamber configuration. The principles of ASTM D6413-11 were adhered to in the following ways: a flame exposure time of 12 s, the use of fabric as the testing material, and the vertical positioning of the sample during testing. Specific sample dimensions were also adhered to, which were 300×76 mm^2. However, the design differed from the standard due to using an open test chamber instead of a closed one. The following steps were taken: first, the sample was placed vertically in the vertical flame test apparatus; then, the butane gas valve and the air valve were adjusted to produce a blue flame that was 38 mm high at a pressure of 17.2 kPa. The flame was then continuously directed at all samples for up to 12 s.

The limiting oxygen index (LOI) represents the minimum amount of oxygen in a nitrogen–oxygen mixture necessary to sustain the combustion of a material. The limiting oxygen index test is used to compare and classify the relative performance of different materials and samples and is therefore used to examine the relative flammability of materials [18]. Materials with LOI values less than 21% are classified as combustible, but those with LOI values greater than 21 are classified as self-extinguishing, as their combustion cannot continue at ambient temperature without an external energy input [18]. The limiting oxygen index tester consisted of several basic components [19].

The limiting oxygen index test was carried out in the laboratory of Damascus University (Damascus, Syrian Arab Republic) according to ASTM D2863-09 via the following steps: (1) the sample to be tested was placed vertically in the center of a glass tube; (2) the flow valves were then adjusted to introduce oxygen and nitrogen gases at a constant pressure of 0.2 MPa and a constant flow rate of 10 L/min; glass beads helped to ensure the even mixing and distribution of these two gases; (3) the top of the sample was ignited with a flame until it began to burn; (4) the oxygen concentration was then gradually reduced until the sample stopped burning—the oxygen concentration at which combustion stops is known as the limiting oxygen index (LOI) [20].

2.3. Methods

The chitosan solution was prepared by dissolving 10 g of chitosan in a glass container containing 1 L of deionized water (DI) and 1 L of acetic acid. The solution was stirred at room temperature at a speed of 187 rpm for two continuous hours. The pH of the solution was measured using pH paper and found to be 3.5. The resulting chitosan solution had a pungent odor and was transparent with a yellowish tint.

The sodium alginate solution was obtained by dissolving 5 g of sodium alginate in 1 L of deionized (DI) water. A rotation speed of 286 rpm was used for 1.5 h at room temperature. The resulting sodium alginate solution was characterized as an odorless, milky white gel.

The samples used in the experimental study were prepared and coated using the layer-by-layer (LBL) method, as shown in Figure 4. This technique is considered a simple, versatile, and economical method, allowing for control of the porosity, mass, and thickness. The principle of the layer-by-layer method is based on the sequential adsorption of oppositely charged materials onto the sample surface [21]. The LBL technique has been widely applied in many fields, such as pharmaceutical and biomedical applications, the production of drug delivery systems and bandages, cell regeneration, and others [22].

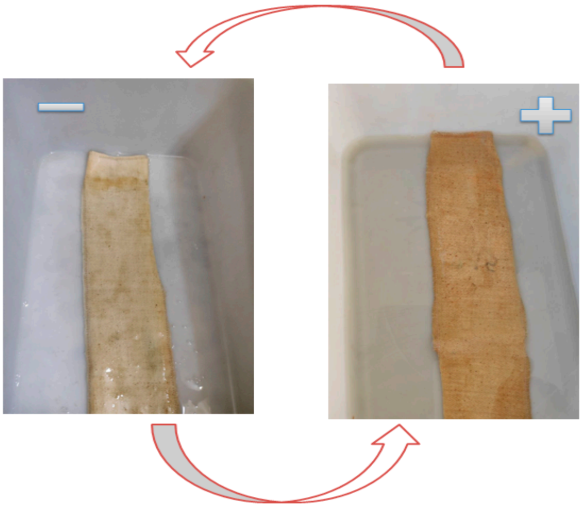

Figure 4. Layer-by-layer method, where (+) indicates the positive charge of the solution and (−) indicates the negative charge of the solution, while the two alternating arrows represent the periodic and repeated immersion of the sample in the coating solutions.

Five samples were prepared, differing from each other in the number of bilayers deposited on their surfaces. The samples were first washed with deionized water for one minute and then dried overnight at room temperature. The samples were next immersed in a chitosan solution for five minutes and then removed and dried for half an hour at 80 °C. Then, they were immersed in the sodium alginate solution for five minutes (as shown in Figure 5), removed again, and dried using a sample drying device. Then, the previous process was repeated until the desired number of double coating layers was obtained.

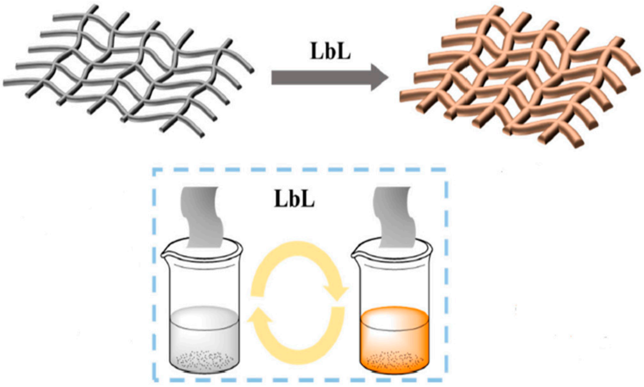

Figure 5. Samples immersed for five minutes.

Before immersing the samples in the chitosan and sodium alginate solutions, they were washed with deionized water for one minute and then dried overnight at room temperature. Five samples were prepared, differing from each other in terms of the number of bilayer coatings to be applied.

The first sample was called the reference sample (control sample). This sample was not coated; it was simply washed with deionized water. The purpose of this sample was to observe its behavior during the flame tests in its natural state.

The second sample was prepared by first immersing it in a chitosan solution for five minutes (as shown in Figure 6) and then removing and drying it. It was then immersed in a sodium alginate solution for another five minutes, removed again, and dried using a sample drying apparatus. The purpose of maintaining the same immersion time in both solutions was to obtain uniform precipitation on the surface of the sample.

Figure 6. Method of dipping the second sample into the chitosan solution.

To prepare the third sample, coated with two layers of chitosan solution and sodium alginate solution, it was first immersed in the chitosan solution for 5 min and then removed and dried. Next, it was immersed again in the sodium alginate solution, removed, and dried. Finally, the sample was immersed for a second time in the chitosan solution, removed, and then immersed once more in the sodium alginate solution. It was then dried for half an hour at 80 °C. This drying step was applied after each immersion cycle to ensure proper coating formation and sufficient adhesion between successive layers, as skipping this step would result in incomplete coating deposition and poor coating uniformity. Sample 4 was coated with five bilayers. This involved immersing the sample five times in the chitosan solution and five times in the sodium alginate solution, as shown in Figure 7. During each immersion, the sample was removed from the solution and allowed to drain. Drying was also performed after each immersion.

The fifth sample was coated with ten bilayers consisting of chitosan and sodium alginate solutions. It was essential to ensure that the sample surface was clean before coating and to dry the sample at 80 °C between each immersion step.

The amount of coating added to the cotton sample was calculated using a highly accurate electronic scale. The sample was weighed both before coating (after rinsing with deionized water) and after coating. It is worth noting that the amount of coating added varied depending on the number of bilayers used.

The amount of coating added to the sample was determined based on the initial and final weights of the cotton fabrics, using the following equation:

$$add - on\ \% = \frac{w_1 - w}{w} \times 100 \quad (1)$$

where

w_1 is the weight of the cotton sample after coating it with a specified number of double layers;

w is the weight of the cotton sample (reference/control sample) before the coating process.

Figure 7. Immersion of the fourth sample in the sodium alginate solution.

3. Results

3.1. Vertical Flame Test

The vertical flame test can provide several important parameters that can be used to evaluate the flammability of cotton samples, as well as to identify polymer materials that pass the vertical flame test.

3.1.1. After-Flame Time in the Vertical Flame Test

The results regarding the after-flame time in the vertical flame test were obtained by recording the time in seconds from the moment that the flame source was removed until the start of twilight or the moment that the flame was extinguished. Table 2 shows the results regarding the after-flame time.

Table 2. Results of the vertical flame test.

Sample	After-Flame Time (s)	After-Glow Time (s)	Char Length (cm)	Dripping
Uncoated	9 ± 0.16	6.06 ± 0.09	30 ± 0	Light
Sample coated with one bilayer	15 ± 0.16	15.12 ± 0.13	30 ± 0	None
Sample coated with two bilayers	11.4 ± 0.16	0 ± 0	30 ± 0	None
Sample coated with five bilayers	9.12 ± 0.18	0 ± 0	30 ± 0	None
Sample coated with ten bilayers	6.9 ± 0.15	0 ± 0	9.72 ± 0.08	None

The after-flame time for the reference sample (uncoated sample, number of coats = 0) was 9 s. This was due to the absence of a coating on the cotton sample surface. When the sample was coated with a single bilayer, the combustion time increased by 67% to 15 s. This increase in after-flame time is attributed to the possibility that the coating altered the combustion process, producing charcoal. This resulting charcoal is unstable and accelerates the combustion process; this has been inferred from the results of other studies [11]. When the sample was coated with two layers of paint, the time decreased to 11.4 s. This is because the coating begins to inhibit the flame by releasing non-flammable gases, such as carbon dioxide. When the sample was coated with five bilayers, the after-flame time decreased to 9.12 s, but it remained slightly higher than that of the reference sample. This indicates that, with each successive bilayer, the resulting carbon layer becomes more stable, and the release of flammable gases is reduced. The fifth sample, coated with ten bilayers, showed the shortest flame ignition time [9]. This indicates that the coating effectively suppressed the flame by forming a stable carbon layer, preventing further heat and oxygen transfer to the cotton fibers, which may contribute to reducing the release of flammable gases [23]. The vertical flame test was performed on each sample at least five times. The arithmetic mean and standard deviation were then calculated and are listed in the table below.

3.1.2. After-Glow Time in the Vertical Flame Test

The after-glow time can be obtained by recording the time in seconds from the moment that the flame is extinguished until the sample's glow diminishes. Table 2 shows the obtained after-glow time values.

The after-glow time for the uncoated cotton sample was 6.06 s, which was due to the absence of a coating layer protecting the sample from combustion. However, when the sample was coated with a single bilayer of chitosan and sodium alginate, this time increased to 15.12 s. This was due to the instability of the combustion process of the cotton sample; therefore, the resulting char was unstable, thus accelerating the combustion rate of the sample—this has been inferred from the results of other studies [11]. When the sample was coated with two and five bilayers, the after-glow time was eliminated. This demonstrates the coating's effectiveness in flame inhibition and increasing the sample's resistance to combustion [9]. Furthermore, in the ten-layer coated sample, the after-glow disappeared completely. This is attributed to the coating's ability to enhance the cotton sample's flame resistance by forming an effective carbon barrier, which may help to reduce the emission of flammable gases [23]. The vertical flame test was performed on each sample at least five times. The arithmetic mean and standard deviation were then calculated and are listed in the table below.

3.1.3. Char Length in the Vertical Flame Test

After completing the vertical flame test, the length of the damaged part of the cotton sample was measured, and the values taken were recorded, as shown in Table 2.

The char length was measured using a metal ruler and by measuring the affected area of the cotton sample. The uncoated cotton sample burned completely, leaving only some char. This is due to the absence of a coating that could increase its flammability [7]. When the cotton sample was coated with one or two layers, it burned completely, although other parameters were improved significantly. The resulting char was more stable than that of the control sample. The sample also burned completely when coated with five layers, resulting in a char length of 30 cm. However, the resulting char layer was more stable and continuous. When the sample was coated with ten bilayers, the length of the char became 9.72 cm, which is 68% smaller than in previous cases [9]. This is due to the important role of the coating in flame inhibition, especially in the condensed phase, as a stable and cohesive carbon layer was formed. This layer is expected to have played a key role in reducing the emission of flammable gases and thus reducing heat transfer [23]. We conclude from the above that the sample coated with ten bilayers of chitosan and sodium alginate solutions successfully passed the vertical flame test, as the length of the char in this sample was 9.72 cm, while the char length suggested in previous studies to pass this test is 15.24 cm (6 inches) [24]. The vertical flame test was performed on each sample at least five times.

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The arithmetic mean and standard deviation were then calculated and are listed in the table below.

3.1.4. Dripping in the Vertical Flame Test

From the vertical flame test, the fourth parameter could also be obtained, which was the phenomenon of dripping, and the results obtained are shown in Table 2.

A small amount of burning droplets fell from the uncoated cotton sample due to the absence of a protective coating. However, when the sample was coated with one layer and then with ten layers, no burning droplets were observed [11]. Thus, the phenomenon of dripping was eliminated thanks to the flame-resistant properties of the coating, which were achieved through the formation of a stable carbon layer; this may have contributed to preventing the transfer of flammable gases [23].

3.1.5. Cotton Ignition in the Vertical Flame Test

The results regarding cotton ignition are related to the dripping phenomenon, which causes the shedding of flammable droplets from the cotton sample as it burns. This may cause the cotton beneath the sample to ignite. Table 3 lists the cotton ignition results; when the uncoated sample burned, some flammable droplets fell, causing the cotton beneath it to ignite. However, when the sample was coated with one layer and then with ten layers, no ignition of the cotton was observed, as the dripping phenomenon disappeared [11].

Table 3. Results regarding cotton ignition in the vertical flame test.

Sample	Cotton Ignition
Uncoated	Yes
Sample coated with one bilayer	No
Sample coated with two bilayers	No
Sample coated with five bilayers	No
Sample coated with ten bilayers	No

3.2. Limiting Oxygen Index (LOI) Test

The results of the LOI test are shown in Table 4 and Figure 8. For the uncoated cotton sample, the LOI value is 18.04% (LOI = 18.04%), which is considered a low value at which the cotton ignited easily due to the absence of any flame-retardant coating. When the sample was coated with a single bilayer, its value increased to 19.594%. This is attributed to the coating deposited on the surface of the cotton sample beginning to inhibit flames. When the number of bilayers increased to two, the LOI value increased to 19.87% [7]. When the sample was coated with five bilayers, its value increased to 21.83%, i.e., an increase of 10% compared to the third sample. This increase is attributed to the formation of layers of stabilized carbon, which play a positive role in enhancing flame retardancy by reducing the volatilization of flammable gases. The limiting oxygen index (LOI) value reached 23.47% when the sample was coated with ten double layers [9]. This significant increase in the LOI value, from 18.04% to 23.47%, is attributed to the effectiveness of the coating used and its role in increasing the flame resistance of the coated cotton sample by isolating the fabric from atmospheric oxygen and increasing the resistance to oxygen penetration into the cotton fibers. Furthermore, the coating prevents the rapid thermal decomposition of cotton, which contributes to the emission of non-flammable gases and reduces the amount of flammable gases emitted [23]. The limiting oxygen index test was performed on each sample at least five times, and the arithmetic mean and standard deviation values were calculated, which are listed in the table below. RS represents the reference sample plot, and a trend line has been added to the graph. We observe that the relationship between the number of layers and the limiting oxygen index is linear, meaning that, as the number of layers increases, the value of the LOI increases proportionally.

Table 4. LOI test results.

Sample	Limiting Oxygen Index %
Uncoated	18.04 ± 0.114
Sample coated with one bilayer	19.594 ± 0.053
Sample coated with two bilayers	19.87 ± 0.37
Sample coated with five bilayers	21.83 ± 0.24
Sample coated with ten bilayers	23.47 ± 0.30

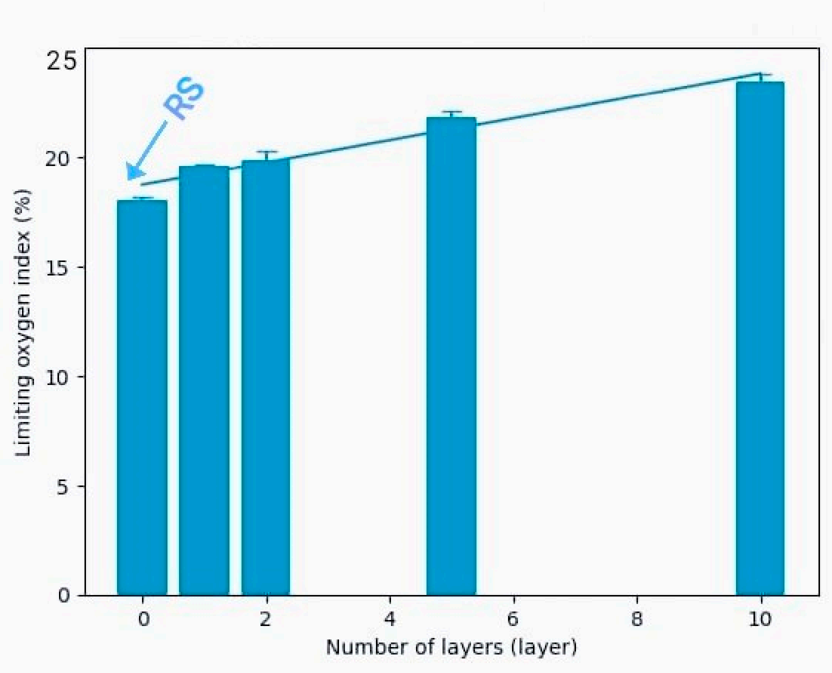

Figure 8. Limiting oxygen index test results.

Relationship Between Number of Layers and Limiting Oxygen Index

To determine the relationship between the number of layers and the limiting oxygen index, the correlation coefficient was calculated using Excel via the CORREL function. The correlation coefficient was 0.99309379—a very high value. This indicates a strong positive relationship between the two variables, with the LOI value increasing proportionally with the number of layers.

3.3. Amount of Coating Added to Sample

The results regarding the amount of paint added to the sample are shown in Table 5 and Figure 9. A certain amount of paint was added to each sample at least five times, and the mean and standard deviation were calculated. Initially, when the sample was coated with a single bilayer, the amount of coating added to the sample was 0.846%, due to the deposition of one layer of chitosan and one layer of sodium alginate on the surface of the cotton sample. When the sample was coated with two bilayers, this value increased to 1.3% due to the increased amount of coating deposited on the surface of the sample. The amount of coating added to the sample increased to 3.88% when the sample was coated with five bilayers. This increase in the amount of coating added to the sample was attributed to the ten layers of coating equally divided between the chitosan solution and the sodium alginate solution. When the sample was coated with ten bilayers, we saw a significant increase in the amount of coating added to the sample, reaching 13.98% [7]. This was due to the deposition of a large number of coating layers, reaching ten layers of the chitosan solution and ten layers of the sodium alginate solution on the surface of the cotton sample. The results regarding the amount of coating added to the cotton sample show that this amount increases with an increase in the number of double coating layers [11]. A certain quantity of coating was added to each sample at least five times, and the arithmetic mean and standard deviation values were calculated, which are given in the table below.

Table 5. Results regarding the amount of coating added to the sample.

Sample	Addition Amount %
Uncoated	-
Sample coated with one bilayer	0.846 ± 0.040%
Sample coated with two bilayers	1.3 ± 0.16%
Sample coated with five bilayers	3.88 ± 0.16%
Sample coated with ten bilayers	13.98 ± 0.16%

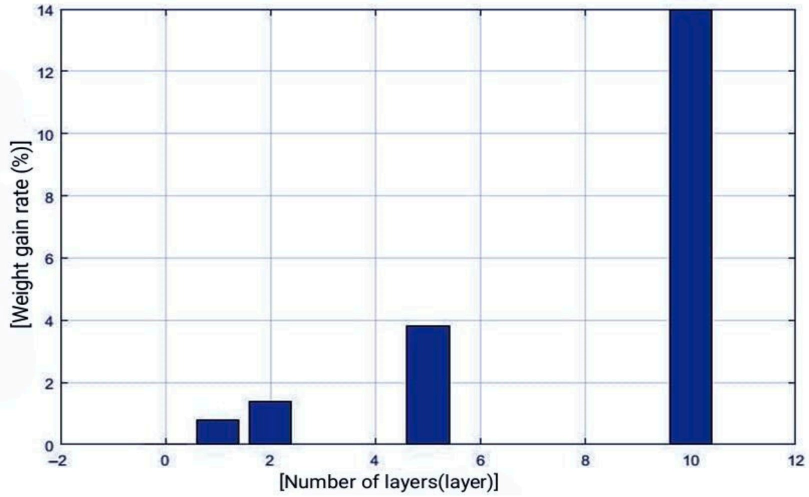

Figure 9. Results regarding the amount of coating added to the sample.

4. Limitations of the Study

Although this study demonstrates the effectiveness of chitosan and sodium alginate coatings applied via the layer-by-layer (LBL) technique in enhancing the flame retardancy of cotton fabrics, several limitations should be acknowledged. The scope of the work was limiting to evaluating the initial flame-retardant performance under laboratory conditions, without investigating the long-term durability of the treated fabrics under real service conditions, due to time constraints and limited resources for extended aging tests. Furthermore, wash resistance was not examined; therefore, the stability of the deposited multilayers after repeated laundering cycles remains unverified, but this is an important factor in assessing industrial applicability. In addition, the resistance of the coated fabrics to flexural stress and abrasion was not evaluated, as specialized mechanical testing equipment was not available within the framework of the present study.

5. Discussion

It is observed that the after-flame time for the reference sample was nine seconds. With the application of a single two-layer coating, this time increased to 15 s. This is attributed to the unstable char formed during combustion. This, in turn, accelerated the combustion process, rather than slowing it down. With successive layers of coating, we observed that this time decreased, indicating the effectiveness of the coating in inhibiting the flame by forming cohesive char layers and reducing the amounts of flammable gases released. Furthermore, the after-flame time results are consistent with [11], as the time started at a certain value and then increased and subsequently decreased. However, they contradict the results in [8], where the time was initially high and then decreased. The explanation for the increased after-glow time with the single two-layer coating of the sample also lies in the presence of unstable char, which played a negative role by accelerating the combustion process. The results for the after-glow time are consistent with [9], as this time was eliminated with successive layers of double coating applied, but they contradict the results in [8]. The results regarding the char length are also consistent with all reference studies, particularly [7,9]. The initial length of the charcoal was 30 cm; it then decreased with increasing coating layers, indicating slower combustion due to the formation of more compact charcoal. This may contribute to an increase in the amount of non-combustible gases emitted. The results for dripping and cotton ignition are consistent with [11], where dripping was initially low and then became negligible.

The results for the limiting oxygen index are also consistent with all reference studies, especially [7,9,11], where the LOI value increased with the number of coating layers applied. Finally, the results for the amount of coating applied to the sample are consistent with the results of [7,11], where the value increased with the amount of coating applied to the cotton fabric.

Achieving compliance with the ASTM D6413 principles using biodegradable, commercially accessible materials addresses a critical gap in sustainable protective textiles. The 13.98% weight gain preserves cotton's superior moisture absorption and breathability—essential for occupational comfort during extended wear. The LBL assembly method's simplicity and scalability enable implementation in resource-limited manufacturing environments, providing an alternative to complex chemical synthesis approaches.

In summary, multilayer chitosan/sodium alginate coatings effectively enhance cotton's flame resistance to achieve complete vertical flame test passage (char length: 9.72 cm, 37% below ASTM D6413 threshold) and self-extinguishing classification (LOI = 23.47%). This comprehensive evaluation of all ASTM D6413 parameters, with an explicit focus on secondary ignition prevention, provides the most complete safety assessment reported for biobased multilayer coating systems. The accessibility and biodegradability of the constituent materials position this coating as a practical alternative to synthetic flame retardants, warranting further investigation of its durability, substrate diversity, and industrial scale-up feasibility.

6. Conclusions

This study demonstrates that multilayer chitosan/sodium alginate biocoatings effectively enhance cotton fabrics' flame resistance, achieving vertical flame test passage with a 10-bilayer system (char length: 9.72 cm, 68% reduction; LOI: 23.47%). This systematic evaluation reveals that progressive bilayer deposition progressively improves all flame retardancy parameters, with critical transitions at two bilayers (complete after-glow elimination) and five bilayers (self-extinguishing threshold, LOI $\geq$ 21%). The condensed-phase charring mechanism—involving the formation of thermally stable protective carbon layers with controlled oxygen diffusion barriers—explains the observed performance. The accessibility, biodegradability, and commercial availability of these bioderived materials, combined with the simplicity and scalability of layer-by-layer assembly, position this coating system as a practical, sustainable alternative to synthetic flame retardants. Future research should optimize the coating parameters to reduce the bilayer number while maintaining performance and extend the system to alternative substrates (particularly cotton–polyester blends) based on the flame behavior and LOI trends so as to elucidate degradation pathways and inform industrial scale-up feasibility.

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