



## Research article

# Analysis of the flame retardancy effect of boron-containing compound on polyester-cotton blended fabric

Mohammad Naim Hassan<sup>\*</sup>, Tahrir Sadman Abdullah, Mehrin Beg Mou, Hasin Raihan Towsif

Department of Textile Engineering, Khulna University of Engineering & Technology, Khulna - 9203, Bangladesh

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## ABSTRACT

Flame-retardant finishing of textile materials is crucial for ensuring human safety and mitigating fire hazards. Though various textile fibers have inherent flame-resistant properties, cotton fiber has a higher affinity to burn. This research focused on developing non-durable FR treatments for cotton-rich polyester-cotton (T/C) blended products economically, using boron-containing compounds. Because of the high melting point use of borax on T/C fabric reduces the fabric's flammability. Boric acid was also used as an auxiliary substrate and Di-sodium hydrogen phosphate dihydrate was used for its cleaning and softening properties. Borax and boric acid create a layer of char when burned and stop the flame. We used the impregnation method for this finishing process. After the chemical finish on different types of T/C fabric, we completed different types of tests like 45° flame retardant, LOI, SEM, breaking strength, drapability, crease recovery, and water vapor transmission tests, and found the desired properties. It increased the flame retardancy and crease recovery properties but the slight reduction of the fabric strength was noticed in case of excessive coating. Water vapor transmission property also reduced gradually with the increase of chemical concentration. Since the chemicals are available in the local market and lower in cost than common FR chemicals, it is more economical.

## 1. Introduction

Cotton is the most essential natural fiber in textile manufacturing due to its biodegradable, eco-friendly, moisture absorbency, and lightweight properties [1,2]. Cotton fibers can offer both comfort and aesthetics. However, the flammability of cotton products is one of the problems posed to the textile industry [3–5]. On the other hand, polyester, a synthetic fiber, is used in wearable textiles due to its high durability and large staple length [6–8]. However, this low flame-resistant fabric has a meager moisture absorption capacity reducing its comfort level [9]. Fire hazards have a great socioeconomic effect on society. Many textiles and indoor decorations including carpets, curtains, wallpaper, and so on, are all easy to burn [10]. Readily burnable textile materials can serve as one of the ingredients in a fire and pose a serious risk to human life and property in fire accidents [11–14]. The amount and severity of burn injuries are substantially higher when regular cotton or polyester work clothes are burnt, which seriously reduces the chances of survival [15–17]. Government regulations and voluntary standards dictate the growing use of flame retardant (FR) textiles mainly in work clothing, firefighter clothing, transportation, institutional mats, bedding, and military clothes [18]. The demand for

<sup>\*</sup> Corresponding author.

E-mail address: [naimhassan375@gmail.com](mailto:naimhassan375@gmail.com) (M.N. Hassan).

flame-retardant textile fabrics has been growing over the past decades [19–24]. Despite significant consumer demand for pure cotton products, no commercial FR treatment is available that alleviates these concerns and allows for keeping the aesthetics of the clothing [25]. Thus, there is a need to develop new environment-friendly and durable means of producing FR garments [26–28].

Methods to increase the flame retardancy of consumer goods have been developed to provide additional safety from fires and to increase escape time when a fire occurs [29–31]. They usually either lower ignition susceptibility or lower flame spread once ignition has occurred [32,33]. To reduce the flammability inherently flame-resistant fibers such as aramids, PBI, polyamide-imides, melamine, and PPS can be used in mattresses with cotton [34,35]. However, the drawback of using these fibers is the high cost and the inequality between cost and product properties. To prevent fabric from burning, the flame retardant cure is one of the most effective methods, which improves the thermal resistance to ignition, reduces flame propagation rate, elevates ignition temperature, and prevents continuous burning. Fire retardant chemicals are classified as halogen, phosphorus, nitrogen, and inorganic compounds [36–41]. Halogen and halogen antimony systems tend to be flame inhibitors. Phosphorus and boron enhance the charring and formation of surface barrier layers, and metal hydroxides tend to be endothermic water-releasing systems. To reduce flammability halogen-based flame retardants are one of the most effective ways of reducing fire risk [42–47]. However, they have such noticeable disadvantages as the discharge of toxic and corrosive gases during thermal degradation, such as HBr and HCl [48,49]. Various efforts have been made to develop halogen-free flame retardants in recent years [50–52]. Some compounds, containing phosphorus, silicon, boron, nitrogen, and other miscellaneous elements, have gained much attention as flame retardants in the polymer materials to substitute halogen-containing flame retardants [53,54]. Boric acid and borate salts have been used as effective flame-retardant additives, but they have been less studied than halogen, phosphorus, and other compounds [55–58]. The use of borates in enhancing the flame retardancy of polymeric materials was reported earlier in the 20th century [59–61]. Borates are effective flame retardants because impenetrable glass coatings form when they thermally degrade and can contribute to the intumescent effect because they exclude oxygen and prevent further propagation of combustion [62,63]. The water of hydration is lost by endothermic decomposition and therefore both dilutes and cools, by absorbing the thermal energy from the flame [64]. Boron has high oxygen affinity and establishes a strong covalent bond with oxygen [65,66]. It is easily dissolved in water. Boron is highly resistant to fire as it is an oxide, and has a huge melting point above 2000 °C [67]. Because of heterogeneous reaction kinetics associated with the oxidation and combustion of boron, it is used as a flame-retardant substance or mixed into such substances in varying ratios [68,69]. Moreover, Di-sodium hydrogen phosphate dihydrate also forms covalent bonds with cotton fiber and boric acid [70].

In this research work, we have used 65 % cotton and 35 % polyester to make the T/C blend fabric [71–73]. But flame resistance property of the T/C blend fabric is poor due to its fiber composition [74,75]. When T/C blend fabrics are subjected to flame for several seconds, they burn and leave behind small ash [76]. The flame-resistant and thermal properties of the T/C blend fabric were analyzed by applying new flame-retardant agents [77]. Borax, boric acid, and Di-sodium hydrogen phosphate dihydrate were selected in this study because of their high flame retardancy effects and fewer environmental effects. The objective of this study was to examine the feasibility of imparting flame-retarding functions to different textile materials through the application of borax, boric acid, and Di-sodium hydrogen phosphate dihydrate with impregnation methods. Various concentrations of borax and boric acid nanoparticles were examined to enhance the flame-retarding functions [78–80]. The drape ability, strength, crease recovery, and water vapor transmission were also tested to assist the mechanical properties of the T/C blend fabric.

## 2. Materials and methodology

### 2.1. Materials

Three types of fabric samples: Bleached 65 % Cotton, 35 % Polyester Single Jersey Fabric (Fabric GSM is 156; sample size 9"×4"), Bleached 65 % Cotton, 35 % Polyester Woven Fabric (Fabric GSM is 15; sample size 9"×4"); Combination of Bleached 63 % Cotton, 35 % Polyester and 2 % Lycra Woven fabric (Fabric GSM is 239; sample sizes are 24"×24" and 9"×4") that were used in the experiment work collected from TEXEUROP BD LTD. Then Boric Acid ( $H_3BO_3$ ), Borax ( $Na_2[B_4O_5(OH)_4] \cdot 8H_2O$ ), Di-sodium hydrogen phosphate dihydrate ( $Na_2HPO_4 \cdot 2H_2O$ ), and Distilled water were purchased from the chemical shop.

### 2.2. Methodology

#### 2.2.1. Sample fabric size preparation

At first, collected fabric samples were cut using a measurement ruler, marker, and scissors according to the sample size. Then the samples were cut by using a GSM cutter in a round shape to measure the sample fabric GSM which was calculated by multiplying the obtained fabric weight by 100; using an electric balance to measure the weight of the fabric. Table 1 shows the overview of the sample-

**Table 1**  
Sample fabric size with required weight and GSM.

Sample Fabric Name	Sample Size 24" x 24"	Sample Size 9" x 4"	Fabric Weight (g)	Fabric GSM (g/m <sup>2</sup> )
65 % Cotton, 35 % polyester Single Jersey Fabric (Knit)	N/A	2	1.561	156
65 % Cotton, 35 % Polyester Fabric (woven)	N/A	2	1.502	150
63 % Cotton, 35 % polyester and 2 % Lycra (woven)	2	2	1.823	182

sized fabric weight and GSM.

### 2.2.2. FR solution preparation

The main FR solution is prepared by mixing borax, boric acid, Di-sodium hydrogen phosphate dihydrate, binder & distilled water according to the amount stated in Table 2. The chemicals were weighed by using an electric balance as per proper measurement. Then both Type-I and Type-II solutions were prepared making the stock solution of chemicals as per Table 2. A magnetic stirrer was used for proper mixing (30 min for each solution); with a temperature of 50 °C and at RPM rate 600. Table 2 shows the total overview of the required chemicals for both Type-I and Type-II solutions.

### 2.2.3. Variation in the amount of the FR solution

Here, three fabric samples, in their required sample size, were immersed in both Type-I and Type-II solutions at a constant material-liquor ratio of 1:20. Then the chemical take-up percentage was calculated by following the formula.

$$\text{Chemical Take Up} = \frac{\text{Fabric weight after treated} - \text{Fabric weight before treated}}{\text{Fabric weight before treated}} \times 100\%$$

Table 3 gives an overview of the needed chemicals for both Type-I and Type-II solutions and the calculated chemical take-up percentage for the fabric samples.

### 2.2.4. Applying the FR solution

To apply chemicals on fabric we have used the pad-dry-cure method. Pad-Dry-Cure is a finishing process applied to textiles to impart different finishing treatments, such as waterproofing, softening, antibacterial or anti-odor finishes. The textile substrate is passed through a water-based solution bath containing the finishing chemicals.

The textile substrate is then dried and cured using heat/pressure. This process is the parent process for several different finishing treatments. It is assumed that multiple finishes may be applied in the solution bath; therefore, the impacts of this stage are the same regardless of the number of finishing treatments applied. The process includes water, chemical, and energy inputs. Impregnation is achieved by passing the fabric at a constant speed through a padding mangle, which comprises a trough containing the dyes and chemicals, and nip rollers that squeeze the fabric at constant pressure to give a desired concentration of liquor on the fabric.

### 2.2.5. 45° Angle tester (ASTM D1230-94 standard)

The 45° angle tester which was manufactured by the authors, has been shown in Fig. 1, is like a flame retardancy tester. However, the texture of the tester is mounted in a casing and held at an angle of 45°. The fire is applied to the base of the texture for a specific measure of time. The fire proliferates up to the length of the texture. The time required for fire to engender through the texture length, as well as how easily it ignites are estimated and recorded.

The 45° angle tester can assess most materials, except youngsters' rest wear, defensive garments, footwear, caps, and gloves. The experimental sample size is 9' x 4'; the specimen holder interior area is 152 mm x 38 mm; the bottom-to-cord yarn length is 127 mm.

Samples were mounted in a frame and held in a special apparatus at a 45° angle. A standardized flame is applied to the surface near the lower end for a specific period. The flame travels up the length of the fabric to a trigger string, which drops a weight to stop the timer when burned. The time required for the flame to travel the length of the fabric and break the trigger string is recorded, as well as the fabric's physical reactions at the ignition point. The sample was tested in the lab also following ASTM D1230:2022 which is shown in Fig. 2.

### 2.2.6. Limiting oxygen index (LOI) test

For the Limiting Oxygen Index (LOI) test for fabric, the ASTM D2863 standard was followed which is mentioned in Fig. 3. It measures the minimum oxygen concentration that supports combustion. The sample was cut in 150 mm x 70 mm size and mounted vertically in the column. The flow meter was set as 21 % oxygen and 79 % nitrogen. The top edge of the fabric was ignited, and its burning behavior was observed.

### 2.2.7. Crease recovery test

The Shirley Crease Recovery Tester was used for this test according to AATCC TM 66. A specimen was cut from the fabrics using a template 2 inches long by 1 inch wide. It was carefully creased by folding it in half, placing it between two glass plates, and adding a weight of 0.5 kg. After 1 min, the weight was removed, and the specimen was transferred to the fabric clamp on the instrument and

**Table 2**  
FR solution preparation.

Chemicals	Type- 1 Solution (Temp. 50 °C)		Type- 2 Solution (Temp. 50 °C)	
	Quantity	Percentage	Quantity	Percentage
<b>Borax</b>	12.5 g	2.5 %	25 g	5 %
<b>Boric acid</b>	5 g	1 %	10 g	2 %
<b>Di-sodium hydrogen phosphate dihydrate (DSHPDH)</b>	5 g	1 %	5 g	1 %
<b>Distilled Water</b>	477.5 ml	95.5 %	460 ml	92 %

**Table 3**

Data sheet for required solution and chemical take-up percentage.

Fabric Sample	Sample Size	Solution Type	Material Liquor Ratio	Fabric Weight before treated	Required Solution	Fabric Weight after treated	Chemical take up
65 % Cotton, 35 % Polyester Single Jersey Fabric (Knit)	9" × 4"	Type 1	1:20	4.37 g	87.4 ml	4.62 g	5.72 %
		Type 2		4.25 g	85 ml	4.57 g	7.5 %
65 % Cotton, 35 % Polyester Fabric (woven)	9" × 4"	Type 1	1:20	3.89 g	77.8 ml	4.09g	5.14 %
		Type 2		3.93 g	78.6 ml	4.23g	7.6 %
63 % Cotton, 35 % Polyester and 2 % Lycra (woven)	9" × 4"	Type 1	1:20	5.48 g	109.6 ml	5.80g	5.84 %
		Type 2		5.52 g	110.4 ml	5.97 g	8.1 %
	24" × 24"	Type 1	1:20	85.83 g	1716.6 ml	90.54 g	5.48 %
		Type 2		86.46 g	1729.5 ml	93.28g	7.9 %



Fig. 1. 45° Angle flammability tester (manufactured by authors).



Fig. 2. 45° Angle flammability tester (testing lab).

allowed to recover from the crease. As it recovered, the instrument dial was rotated to keep the free edge of the specimen in line with the knife edge. At the end of the period allowed for recovery, usually 1 min, the recovery angle in degrees was read on the engraved scale. Warp and weft way recovery were reported separately to the nearest degree from the mean values of ten tests in each direction.

#### 2.2.8. Scanning Electron Microscope (SEM)

The ZEISS Gemini Sigma 300 machine was used to perform the SEM test according to ASTM F1372-93.



Fig. 3. LOI test of Type II solution treated sample, 63 % cotton, 35 % Polyester & 2 % Lycra woven fabric.

#### 2.2.9. Breaking strength and drape coefficient test

For the breaking strength test, the ASTM D5034 standard was followed. The Fabric sample was cut to 4 × 6 inch. The gauge length was set to 3 in. A constant rate of extension was applied and the maximum force (breaking strength) at which the fabric sample breaks was noted.

To determine the static drape coefficient of a fabric, which quantifies the drape quality of the fabric when it is in a steady state under its weight. Here ISO 9073-9 standard was followed. The fabric was cut into a circular shape with a diameter of 30 cm and conditioned in a standard atmosphere ( $20 \pm 2^\circ\text{C}$ ,  $65 \pm 2\% \text{RH}$ ) for 24 h before testing. The supporting disk was placed to ensure the fabric was draped naturally over the disk without any wrinkles or creases, and the shadow was projected onto the projection screen. The static drape coefficient was computed after measuring the area of the shadow and the supporting disk. The supporting disk was attached to the dynamic drape tester containing a rotating arm at 20–30 rpm to find the dynamic drape coefficient test.

#### 2.2.10. Water vapor transmission test

The ASTM E96 standard was followed for the water vapor transmission test. A cup was filled with distilled water, leaving a small gap (0.75" to 0.25") between the specimen and the water. The cup was sealed to prevent vapor loss, except through the test sample. An initial weight was taken of the apparatus and then periodically weighed over time until the results became linear. A 4 × 4 inch specimen was used for this. Here the thickness was less than  $1\frac{1}{4}$  inch.

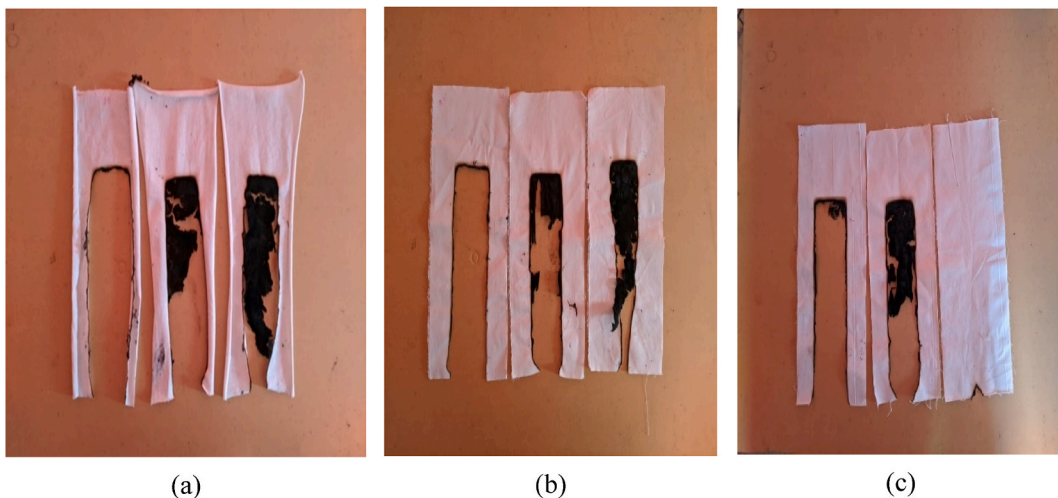


Fig. 4. 45-degree FR test result of (a) 65 % cotton, 35 % Polyester knit fabric; (b) 65 % cotton, 35 % Polyester woven fabric; (c) 63 % cotton, 35 % Polyester & 2 % Lycra woven fabric.



### 3. Result and discussion

#### 3.1. FR 45° angle test result

The flame retardancy test of all three types of treated, with both Type-I and Type-II solutions and untreated fabric samples, was carried out using a 45° angle tester according to ASTM Standard D1230-94 which is shown in Fig. 4. Among all the samples, the Type-II solution-treated blended (63 % Cotton + 35 % Polyester + 2 % Lycra) woven fabric sample gave the best result. An overview of the total test results is shown in Table 4.

Fig. 4 shows the actual picture of the fabric samples during the test period. It can be seen that the third fabric sample in picture 4(c), treated with type-II solution, did not ignite in the experiment period. For this reason, afterward, all the tests were carried out following 98 % cotton and 2 % lycra woven fabric samples.

#### 3.2. Flammability Test Report

Fig. 5 shows the report from a reputed testing company in Bangladesh for 63 % cotton, 35 % polyester, and 2 % Lycra woven fabric treated with the type-II solution. The report shows that this fabric burns at the impingement only. That means fabric is considered as “did not ignite”.

#### 3.3. LOI test result

Fig. 6 shows that the limiting oxygen index (LOI) of sample II treated T/C blend (63 % cotton, 35 % polyester, and 2 % Lycra) woven fabric increased from 19.5 % to 28.3 % which means this treated T/C blend fabric has excellent self-extinguishing property.

#### 3.4. SEM analysis

The SEM analysis determined the shape, structure, and amount of crystalline region of both untreated and Type-II solution-treated blended (63 % cotton, 35 % Polyester & 2 % lycra) woven fabric samples. Fig. 7 shows the surface of untreated and treated fiber in this Scanning Electron Microscope image. On the surface of untreated fibers, we found numerous diffraction lines that make the surface rough or fuzzy. But from the images of treated fibers, we found a smooth surface, like a glass coating, of boron instead of fuzzy lines. Here the red circles show the coating particles.

#### 3.5. Breaking strength & drape ability

Breaking strength was used to determine the tear force of the samples, before and after treatment with Type-I and Type-II solutions. According to the breaking strength test results in Fig. 8(b), the treated samples showed moderate breaking strength compared to the untreated samples. One limitation of this experiment is that while the samples treated with Type-II FR solution showed the best results for the flame resistance test, remaining unignited throughout the test duration, they exhibited moderate strength in the breaking strength assessment.

The drape test assessed the fabric samples' ability to drape, before and after treatment. In Fig. 8(a) the test result showed that the untreated fabric sample possesses better static and dynamic drape co-efficient rather than the fabric sample treated with a Type-II solution.

The authors will try to overcome the above problems in future research endeavors.

#### 3.6. Water vapor transmission

Water vapor transmission indicates the amount of water that can pass through the sample fabrics. Water vapor transmission is important for the users' comfort. If the water vapor transmission of a fabric is very low, then the moisture cannot get outside to the environment and it will get trapped inside of the clothing. This will decrease the comfort of the fabric by creating a damp feeling for the user. So it is important to maintain a good water vapor transmission of the sample fabrics even after using the flame retardant finish. From Table 5 and Fig. 9, we can see the water vapor transmission of the untreated sample is 0.75013 g/hr-ft<sup>2</sup>. The water vapor

**Table 4**  
FR test before and after result.

Fabric Sample	Condition	Ignition Time (second)	Fire Spreading Time (second)	Immersed in Solution	Ignition Time (second)	Fire Spreading Time (second)
65 % Cotton, 35 % polyester Single Jersey Fabric (Knit)	Untreated	5 s	13.87s	Type 1	8 s	16.92s
			7.84s	Type 2		18.57s
65 % Cotton, 35 % Polyester Fabric (woven)			14.10s	Type 1		10.19s
			15.5s	Type 2		12.05s
63 % Cotton, 35 % polyester and 2 % Lycra (woven)			13.87s	Type 1		12.12s
			7.84s	Type 2		Did Not Ignite

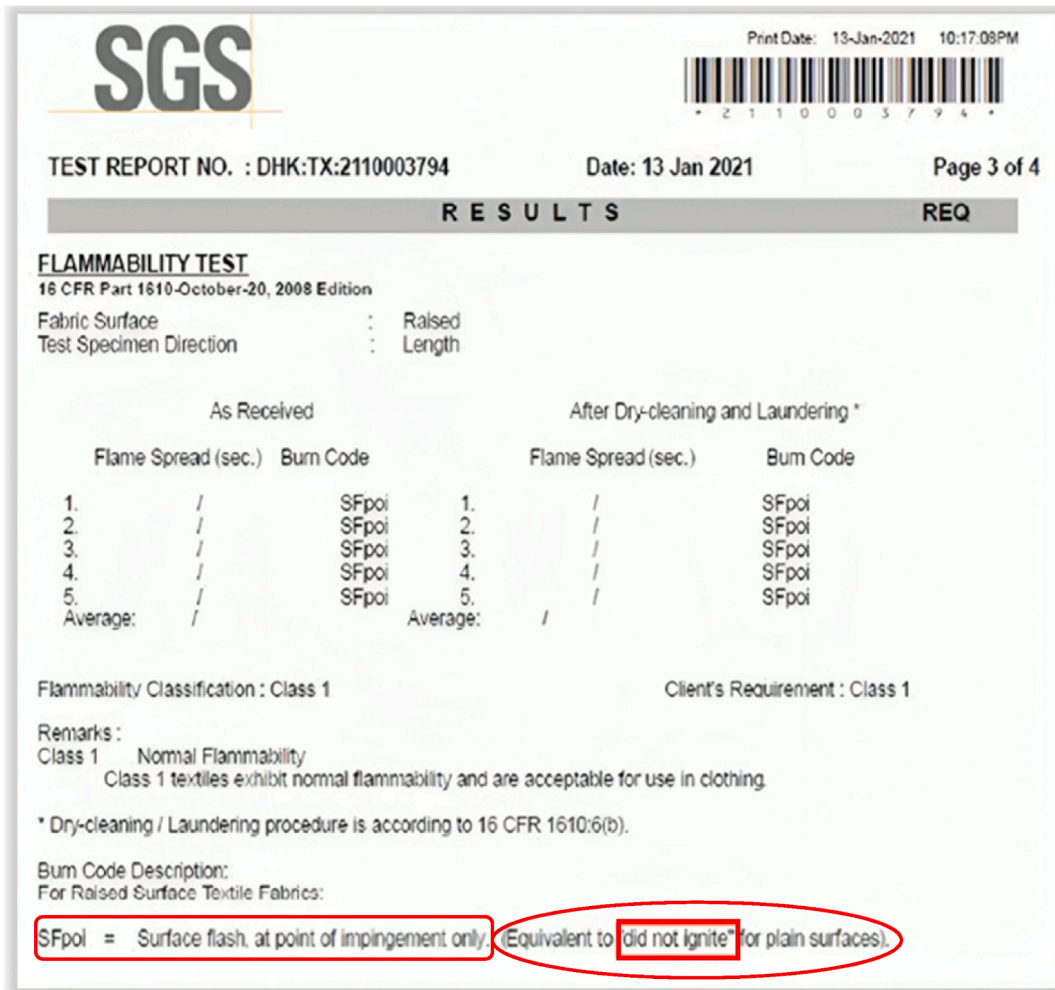


Fig. 5. Flammability test report (by SGS).

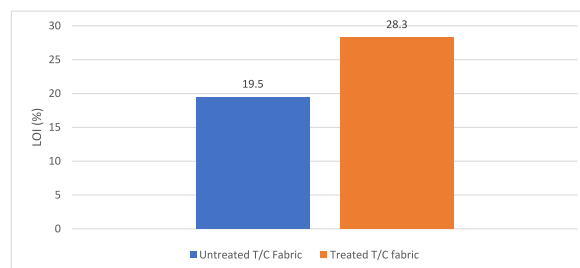


Fig. 6. LOI test result of treated and untreated fabric.

Transmission of the untreated sample is much higher. The sample, treated with the Type-1 solution, has a water vapor transmission of 0.40916 g/hr-ft<sup>2</sup>. On the other hand, the sample that was treated with the Type-2 solution has a water vapor Transmission of 0.21822 g/hr-ft<sup>2</sup>. So it indicates that the water vapor transmission of the sample fabric decreases after using the flame retardant finish. However, the Type-2 solution decreases the water vapor Transmission of the fabric more than the Type-1 solution.

Fig. 9 represents the water vapor transmission rate of fabric.

### 3.7. Crease recovery test

Crease Recovery denotes the fabric's ability to return to its original position after creasing. A crease recovery test is done to measure

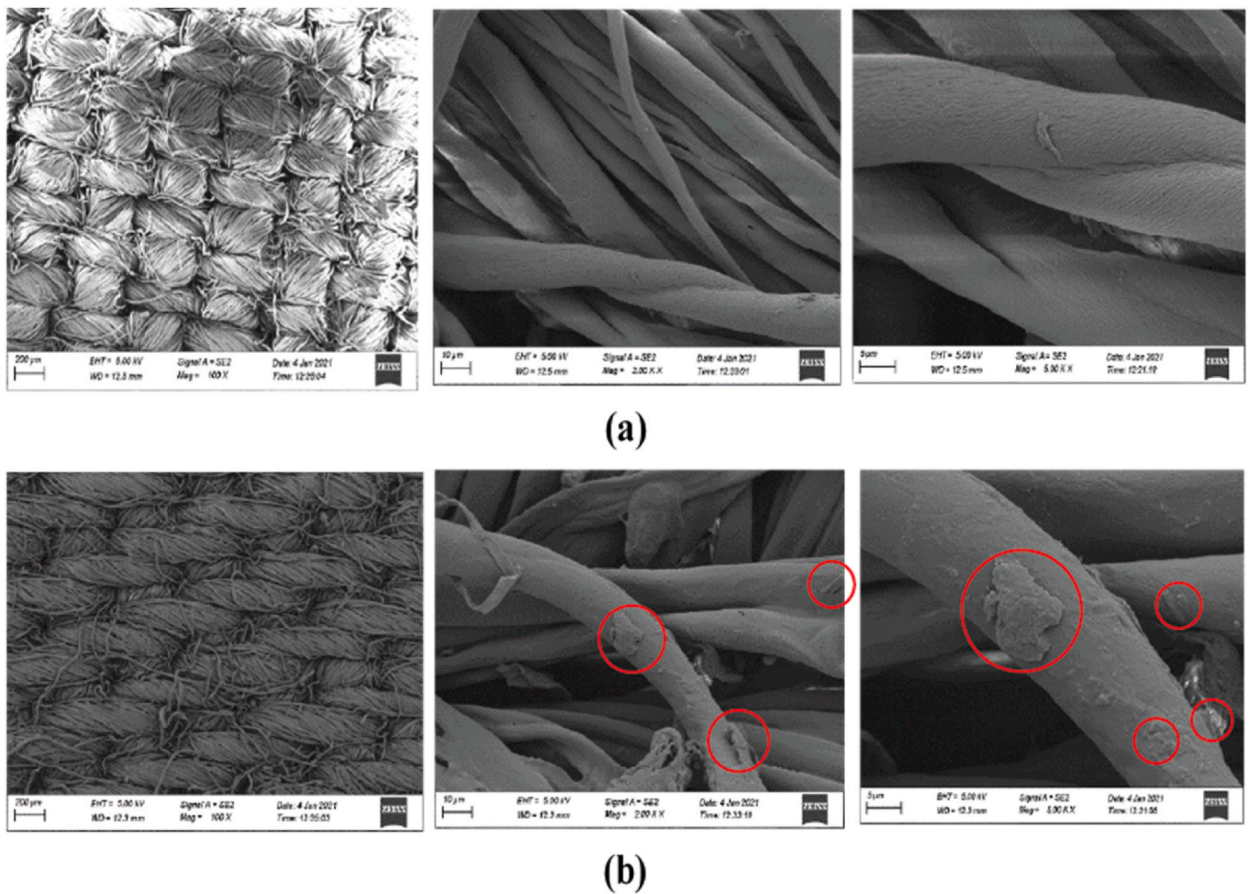


Fig. 7. SEM Test Report of (a) untreated; (b) treated with type-II solution 63 % Cotton, 35 % Polyester & 2 % Lycra blended woven fabric samples.

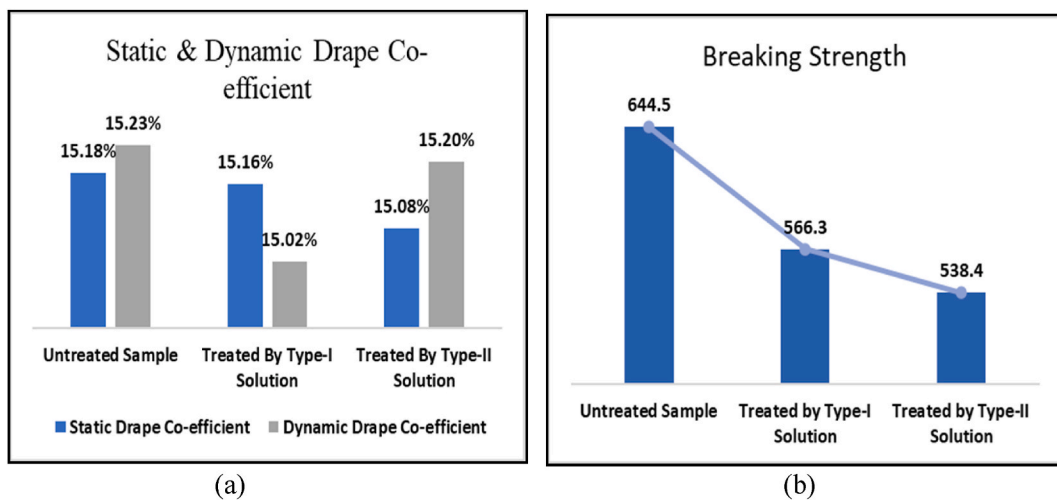


Fig. 8. (a) Drape and (b) Breaking strength test results of the Untreated and Treated samples.

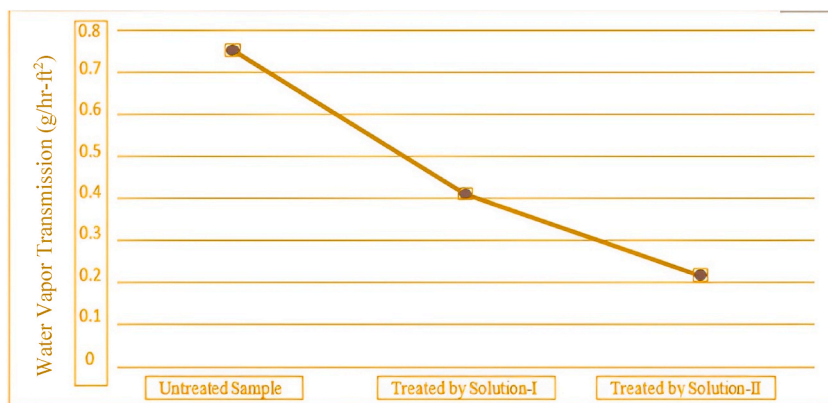
this ability of the fabric. The test method is AATCC 66. In this test, we put our fabric sample (63 % cotton, 35 % polyester, and 2 % lycra) under a 500g load for 5 min s. From Table 6 and Fig. 10, the crease recovery angle of the untreated sample is 80°. The sample, treated with type-1 solution, has a crease recovery angle of 85°. Again, the sample treated with a type-2 solution had the highest crease recovered angle, which is 95°.



**Table 5**

Water vapor transmission.

Fabric Sample (98 % Cotton & 2 % Lycra)	Sample Area	Weight of the fabric + water + pot (Before)	Weight of the fabric + water + pot (After)	Difference of Weight	Machine Run Time	Water Vapor Transmission (g/hr-ft <sup>2</sup> )
Untreated Sample	0.4 square feet	185.455 g	185.390 g	0.055 g	11 min (0.1833 h)	0.75013
Treated by Type 1 Solution		188.130g	188.100g	0.030g		0.40916
Treated by Type 2 Solution		190.111g	190.095g	0.016g		0.21822

**Fig. 9.** Water vapor transmission graph.

#### 4. Conclusion

High melting point products are essential in day-to-day life. Textile products exist in every sector from household products to industrial uses. So, as a safety precaution, these products have to be flame-retardant. For the research, boron-containing compounds have been applied to different types of cotton-blended fabric at various concentrations. FR test results indicated that 63 % cotton +35 % polyester +2 % Lycra blended woven treated fabric showed the best performance in flame resistance tests. SEM analysis revealed the smoother surface of the treated fabric compared to the untreated fabric due to the coating of boron. With increasing concentration, the flame retardancy enhanced proportionately as expected, but unwanted reduction of a few properties like strength, crease recovery, and water vapor transmission were also noticed. The flame retardant finish on the fabrics was non-durable, indicating a potential decrease in flame retardancy after several washes. Although there are some limitations, the fabric is economical and eco-friendly giving acceptable and satisfactory results for non-durable fabrics as the samples were tested in SGS and the result was overwhelming. Even the treated T/C blend showed an 8.8 % increase in the LOI value compared to the untreated fabric. The findings of this study indicate the effectiveness of boron-containing compounds and Di-sodium hydrogen phosphate dihydrate in improving the flame retardancy of T/C blended fabrics to ensure their optimal performance in various applications from protective clothing for firefighters, industrial workers, and military personnel to healthcare textiles.

#### Data availability section

- Has data associated with your study been deposited into a publicly available repository?
  - No. Data included in article/supp. Material/referenced in the article.

**Table 6**

Crease recovery test.

Sample (63 % Cotton, 35 % Polyester & 2 % Lycra)	Sample Size	Time	Load	Crease recovery angle
Untreated	(40 × 15) square mm	5 min	500 g	80°
Treated by Type-1 Solution				85°
Treated by Type-2 Solution				95°

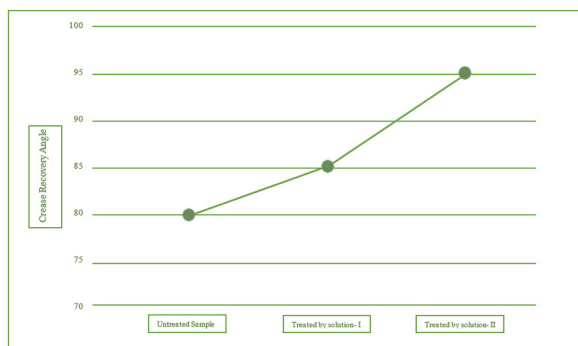


Fig. 10. Crease recovery angle.

### Ethics declaration statement

- Review and/or approval by an ethics committee was not needed for this study because the research did not involve human or animal subjects and solely involved material science experimentation on fabrics, hence, did not fall under the purview of ethical review.
- Informed consent was not required for this study because the research did not involve human participants or any personal or sensitive data collection.

### CRediT authorship contribution statement

**Mohammad Naim Hassan:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Tahrim Sadman Abdullah:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Formal analysis, Data curation, Conceptualization. **Mehrin Beg Mou:** Writing – review & editing, Writing – original draft, Software, Resources, Methodology, Formal analysis, Data curation. **Hasin Raihan Towsif:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Resources, Project administration, Methodology, Formal analysis, Data curation, Conceptualization.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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