

ИЗСЛЕДВАНЕ НА ПРИЛОЖЕНИЕТО НА DBD ПЛАЗМА ЗА ОБРАБОТКА ЗА ЗАБАВЯНЕ НА ГОРЕНЕ НА БАГРЕНИ ПАМУЧНИ ТЪКАНИ

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STUDY ON THE APPLICATION OF DBD PLASMA IN FLAME RETARDANT TREATMENT FOR DYED COTTON FABRICS

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Abstract: This article presents the results of research on flame retardant treatment for dyed cotton fabric using two procedures (with and without pretreatment with DBD plasma). One bath pad-dry-cure method was used for the flame-retardant finishing. Pyrovatex CP new (PCN), Knittex FFRC (K-FFRC) were used as flame retardant and crosslinking agents. The research results show that, with the fire retardant solution including PCN 450/l and K-FFRC 107 g/l, and the curing temperature at 180°C for 150 seconds, dyed cotton fabric after fire retardant treatment will be durable flame-retardant fabric (LOI of the flame-retardant fabric $\geq 25\%$ even after 5 washing cycles). However, if the dyed cotton fabric is pre-treated with DBD plasma with a power of 1 w/cm² for 30 seconds, the curing time can be reduced to 120 seconds, the flame retardant treated fabric will be still a durable flame-retardant fabric. At these conditions, the colour of the dyed cotton fabric is not changed by plasma treatment. However, flame retardant treatment changes the colour of dyed cotton fabrics at a medium ($2.0 \leq DE_{CIELab} \leq 3.5$).

Keywords. Dyed cotton fabric, DBD plasma, Flame retardant, Pyrovatex CP new, Knittex FFRC

Introduction

Cotton is one of the most used textile fibres. However, it is a combustible material with a low LOI of 18.4% [1]. Therefore, flame retardant (FR) treatment for cotton fabrics, especially durable flame retardant, is an important requirement for many areas of use. However, no optimal treatment process for cotton exists which produce flame protected textiles that meet all the requirements with respect to flame retardancy, toxicity, environmental compatibility, the application method, mechanical and chemical durability without the loss of vital intrinsic textile properties [2]. The most common classes of flame retardants are brominated, phosphorus, nitrogen, chlorinated, and inorganic [3]. Halogen-containing FR systems have come under scrutiny due to health and environmental concerns and have recently been restricted in many communities. Recent developments of FR molecules have focused on organophosphorus compounds [4]. Phosphorus-based FR has become a popular alternative to halogen compounds because of their environmentally friendly by-products and their low toxicity. Also, their low production of smoke in fire furthers their appeal. It is a highly effective flame retardant for cellulose and cellulose derivatives. These compounds promote dehydration and char formation [5]. One of the most commercially successful agents is N-methylol dimethylphosphonpropionamide, known under the trade name of Pyrovatex CP New (PCN). Many studies have shown that Pyrovatex CP New (PCN) combining crosslinking agents such as trimethylolmelamine (TMM) or 1,3-dimethylol-4,5-dihydroxyethylene urea (DMDHEU) is a very good choice to create durable flame-retardant cotton fabrics. However, they release formaldehyde, and their mechanical strength is reduced compared with pre-treatments because the crosslinking reaction occurs at high temperature and requires the use of phosphoric acid as a catalyst [3-5]. In our recent studies, a new formaldehyde-free crosslinking agent (Knittex FFRC (K-FFRC)) was used as a replacement for the old one as a solution to reduce

the release of free formaldehyde from fabrics [6, 7]. However, our research has shown that when treating cotton fabric with PCN and K-FFRC, for the treated fabric to be fire resistant (limiting oxygen index (LOI) > 25), the curing temperature must be greater than 170°C and the time curing must also be longer than 120 seconds. It is this condition that reduces the mechanical strength of cotton fabric [8]. To solve this problem, adding a plasma activation process to the fabric is a solution used by many studies [2, 9, 10]. In our previous study, the cotton fabric was preactivated with Dielectric-barrier discharge (DBD) plasma at a power of 1w/cm² for 90 seconds. Plasma treated fabric then treated with PCN and K-FFRC. The results show that the fabric has been pre-treated with plasma, during the flame-retardant treatment it only needs to be cured at 160°C for 90 seconds to have a LOI > 25 [11]. To achieve this value, the normal cotton fabric must be cured at 180°C for 120s during flame retardant treatment [8]. This study also shows that too strong plasma treatment conditions (high plasma power, prolonged exposure time) also adversely affect the mechanical performance of cotton fabrics [11]. However, our previous studies are all performed on white cotton fabrics after pre-treatment (undyed fabric), while functional finishing is usually carried out on dyed fabrics, the dyeing process is one of the most important but as well most complex processes of the whole textile finishing. Prinz, K [2] has studied the possibility of combining the Plasma Induced Graft Polymerisation (PIGP) and dyeing methods, various investigative processes have been conducted. The results show that FR treated cotton was dyed with common cellulose dyestuff and possible changes in colour determined by spectrophotometric measurements. Although the exhaustion rate of dyeing on FR treated cotton textiles is reduced compared to untreated cotton, reactive and direct dyeing resulted in excellent colour shade and hue. Finally, pre-dyeing of cotton textiles was performed followed by the FR treatment. It was found that the Degree Of Grafting (DOG) depends on the dyeing hue. This indicates that the hydroxyl groups of the cellulose

influence the affinity of the flame-retardant monomers towards the cotton fibres. Hence, FR treatment via PIGP was investigated on post- or pre-dyed fabrics and showed promising results in terms of flame retardancy, DOG and chromaticity. Ha-Thanh Ngo et al [12] also used DBD plasma to activate dyed polyester fabric before flame-retardant treatment of the fabric. The results showed that samples pretreated with plasma, after flame retardant treatment had LOI values equivalent to those of the sample was not plasma treated, but their fire resistance after 5 washing cycles was much higher than that of the sample which had not been pretreated with plasma [12]. This study also showed that plasma treatment also changed the colour of the dyed fabric more or less depending on the plasma treatment conditions. In the study [12], the plasma power was 1w/cm^2 , when the plasma treatment time was shorter than 60 seconds the colour difference between plasma treated sample and the reference sample is small, obvious only to the trained eye. However, when the plasma treatment time reached 90 seconds, the colour difference between sample plasma treated sample and control sample is medium, also obvious to trained eye. Therefore, in this study, DBD plasma was applied on undyed and dyed cotton fabrics before they were flame-retardant treated with PCN and K-FFRC. The objective of this study was to clarify the effect of DBD plasma treatment on flame retardant treatment for the undyed and dyed cotton fabric.

Experimental

Materials:

· Fabric:

Fabric: Dyed woven cotton fabric with weight of 242 g/m^2 was supplied by NASILKMEX, Vietnam. The colour parameters of the dyed fabric are presented in Table 1.

Table 1. The colour parameters of the dyed fabric

Colour of fabric	Colour measurement conditions	Colour parameters of the dyed fabric				
		L	a	b	c	h
Green	D65/10	34.76	-37.53	-1.23	37.55	242

· Chemicals: Pyrovatex CP New (PCN), Knittex FFRC (K-FFRC), Invadine PBN were supplied by Huntsman. Pyrovatex CP New (PCN) is a N-methylol dimethylphosphonpropionamide, in this study, it was used as flame retardant agent. Knittex FFRC is a modified dihydroxy ethylene urea, it was used as crosslinking and Invadine PBN as tenside agents.

Procedure:

The dyed cotton fabric was flame-retardant treated according to 2 procedures (with and without pre-activation with DBD plasma)

- Procedure 1: The flame-retardant treatment process for dyed cotton fabric without plasma pre-activation is shown in Figure 1

- Procedure 2: The flame-retardant treatment process for dyed cotton fabric with plasma pre-activation is described in Figure 2

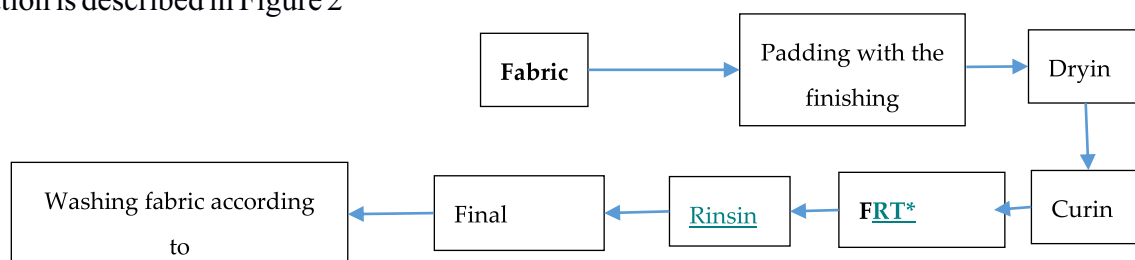


Figure 1. Procedure 1 - Flame-retardant treatment process for cotton fabric without DBD plasma pre-activation

FRT* - Flame retardant treated

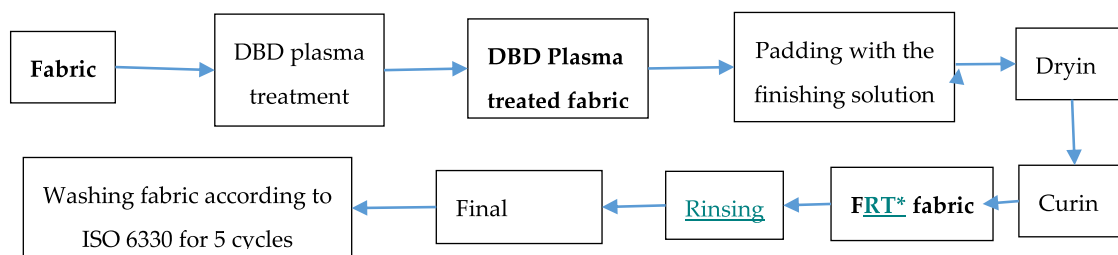


Figure 2. Procedure 2 - Flame-retardant treatment process for cotton fabric with DBD plasma pre-activation

· DBD plasma treatment for fabric

Laboratory roll-to-roll DBD plasma equipment with a width of 50 cm developed by School of Engineering Physics (SEP) of Hanoi University of Science and Technology (HUST) as part of the KC.02.13/16-20 project was used in this study. The DBD plasma environment was produced between two parallel electrodes, the length and width of the electrodes are 8 cm and 50 cm, respectively. The cotton fabric with a width of 35 cm can move continuously between the top and bottom electrodes. The tension rollers are placed before and after the electrodes to keep the fabric in uniform tension. The movement speed of the fabric can be controlled by a motor to control the fabric plasma exposure time. In this study, the distance between the electrodes (discharge gap) was 3 mm, the plasma treatment power was 400 W (1 W/cm²), an air atmosphere was employed. The plasma exposure time was 30 seconds.

· Flame-Retardant Treatment for untreated and DBD Plasma Treated Cotton Fabric

One bath pad-dry-cure method was used for the flame-retardant finishing.

To compare the flame retardant treatment conditions of dyed cotton fabric with undyed cotton fabric, in this study, the flame retardant solution composition and technological parameters of the padding and drying steps were kept the same as in our previous research (flame retardant treatment for undyed cotton [8])

Flame retardant solution included 450g/l Pyrovatex CP New, 107 g/l Knittex FFRC (DHEU) and 5g/l Invadine PBN.

First, the plasma treated fabric was impregnated in finishing solution, then padded

with the pick-up of approximately 80% by padder SDL D394A. The padded samples were dried at 120°C for 3 minutes. After that these samples were cured. The Stenter SDL D398 was used for drying and curing steps. The temperature and curing time have been changed so that the fabrics after flame-retardant treatment are a durable flame-retardant fabric (after 5 washing cycles, the LOI of the fabric must be ≥ 25 % (According to the Flame Retardant Fabric Handbook [1], the LOI of a fabric must be ≥ 25 % for the fabric to be classified as a flame retardant fabric)).

· Rinsing

After curing, the FRT samples were rinsed under the running water for 5 minutes to remove all the residual FRs on the fabric surface (unable to react with celluloses) and neutralize the treated fabric. Then, the fabric was dried in the stenter at 110°C for 3 minutes. After that, the treated samples were stored in the polyethylene bags at the standard laboratory conditions for 24 hours before any further analysis.

· Washing for FRT fabric samples

To determine the flame-retardant durability of the FRT fabric, the FRT samples were washed in accordance with ISO-6330 standard clause 6A but without added detergent. The Electrolux EW 1290W front load washing machine was used. Washed samples were stored after 5 washing cycles to test the flammability of samples.

Characterization of the Control and Treated Cotton Fabric

· Measurement of flammability for the samples

- Measurement of flammability of fabric by ASTM D 6413 -2015, the flammability of fabrics is presented by 3 parameters: (1) Char-length (mm)
- The shorter the char length, the higher the

fabric's fire resistance; (2) Afterflame time (s) - The shorter the after flame-time, the higher the fabric's fire-resistance; (3) Afterglow time (s) - The shorter the after glow-time, the higher the fabric's fire-resistance. According to ISO 11612 (The international standard EN ISO 11612 specifies the performance requirements for protective clothing made from flexible materials, which are designed to protect the wearer's body against heat and/or flame)[13], the afterflame time and afterglow time must be ≤ 2 s.

- The LOI of fabric samples were measured by ASTM D 2863 -97 standard before and after flame-retardant treatment and after washing. The LOI of the fabric is as high as possible and must be ≥ 25 % for the fabric to be classified as a flame retardant fabric[1].

• Color Measurement

The colors of the control, plasma-treated samples and FRT samples were measured by Ci4200 Spectro-Photometer, produced by X-Rite Pantone-Michigan-USA (STLF, Hanoi, Vietnam) with D65 illuminant and 10° standard observer. The measurement was repeated 3 times for each sample at 3 different positions. If the color difference between them is less than 0.35, the mean of the 3 measurements will be accepted as the color of the sample. The effect of any treatment on the fabric colour is expressed as the colour difference (DE) between the control and the treated sample. It can be expressed as $DE_{CIE\text{Lab}}$ or DE_{CMC} values [14]. Measurement of color of fabric before, after DBD plasma treatment, flame retardant treatment, and after washing cycles.

All the above experiments have been implemented at HUST laboratories.

Results and discussion

Research results on fire retardant treatment for dyed cotton fabric according to procedure 1 (without plasma pre-activation)

In our previous research (flame retardant treatment for undyed cotton [8]), the optimal curing condition for the undyed cotton fabric after flame retardant treatment to be a durable flame retardant fabric was at a temperature of 180°C for 114 seconds. Under this condition, undyed cotton fabrics that have been treated with flame retardants have a LOI $> 25\%$ and its flammability parameters consistent with the flame retardant fabric group. Moreover, the flammability parameters of the FRT samples did not change after 5, 10, 20 or even 30 washing cycles. In this study, for dyed cotton fabric, the first curing condition was selected as the optimal condition of undyed fabric (dyed sample FRT1 (Table 2)). In the second condition, the curing temperature was kept at 180°C but the curing time was extended to 150 seconds (dyed sample FRT2 (Table 2)).

- Evaluation of the flame retardancy of the dyed cotton fabric treated according to procedure 1

The results of the flammability testing of the dyed cotton fabric after flame retardant treatment according to procedure 1 are presented in Table 2.

Table 2. Flammability parameters of dyed FRT fabric samples

Fabric samples	Curing conditions		Flammability parameters of fabric samples			
	Temperature (°C)	Time (s)	Afterflame time (s)	Afterglow time (s)	Char-length (mm)	LOI (%)
Control*	-	-	24.41	90.48	-	18.1
Dyed FRT1	180	114	0	0	91	28.3
Dyed FRT2	180	150	0	0	50	28.9

Control* - Dyed fabric before any treatment

Table 2 results show that the control fabric sample (dyed fabric without flame retardant treatment) is very flammable, and the fabric has a low LOI (18%). After removing the flame, the fabric continued to burn for up to 24 seconds and then glowed for up to 90 seconds. After completely burning, no char was formed (Figure 3). Meanwhile, the two dyed fabric samples, that have been treated with fire retardants, both have very good fire resistance (their afterflame time and afterglow time are both zero seconds). However, there are still differences between them in accordance with the curing time. The LOI of the sample cured in 150 seconds is 28.9%, while that of the sample cured in 114 seconds is only 28.3%. Likewise, their char-length values are 50 and 91 mm, respectively.

- *Evaluation of the durability of the fire retardant properties of dyed cotton fabric treated according to procedure 1*

To evaluate the durability of the fabric's flame retardancy, the dyed cotton fabric, after being treated with flame retardants according to procedure 1, was washed for 5 cycles as described in part 2. These samples have been named FRT-W5, and have been evaluated for its flammability according to ASTM D 6413 -2015 and ASTM D 2863 -97 standards. The results of this test are presented in Table 3.

The photographs of the samples after vertical flammability test according to ASTM D 6413, are presented in Figure 3.

Table 3. Flammability parameters of dyed FRT fabric samples after 5 washing cycles

Fabric samples	Curing conditions		Number of washes	Flamability parameters of fabric samples			
	Temperature (°C)	Time (s)		Afterflame time (s)	Afterglow time (s)	Char length (mm)	LOI (%)
Dyed FRT1-W5	180	114	5	21.52	0	101	23
Dyed FRT2-W5	180	150	5	0	0	98	25.8

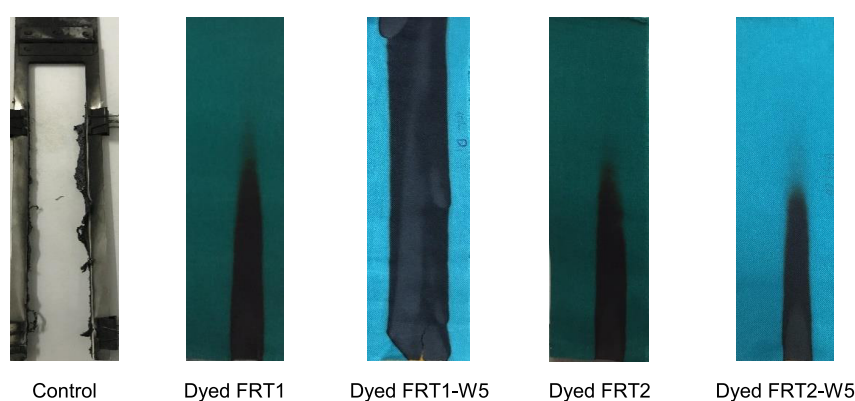


Figure 3. Photographs of the samples after vertical flammability test according to ASTM D 6413

Table 3 and Figure 3 show that, after 5 washing cycles, the dyed FRT1-W5 sample (cured in 114 seconds as for the undyed cotton fabric) has reduced flame retardancy compared to before washing (dyed FRT1). After removing the flame, the sample continued to burn for another 22 seconds until the end of the sample. However, after burning, the char was formed, the LOI was only 23%. Meanwhile, the dyed sample FRT2-W5 (curing in 150 seconds) has an LOI of 25.8% greater than 25, meeting the standard for being a flame retardant fabric [1]. Thus, in order for the dyed cotton fabric after flame-retardant treatment

to be a durable flame retardant fabric, the flame retardant treatment process can remain the same as for undyed fabric except that the curing time must last longer than 35 seconds.

The difference in flame retardant treatment conditions of dyed cotton fabrics compared with undyed cotton fabrics may be due to the fact that both dye molecules and flame-retardant molecules bind to cellulose through their active hydroxyl groups. Maybe, in dyed cotton fabric, some of the active hydroxyl groups of cellulose have been occupied by dye molecules, so dyed cotton fabric needs more time than undyed cotton fabric to have enough bond between cellulose and Flame-retardant molecules make it a durable flame retardant fabric.

- *Effect of flame-retardant treatment on fabric colour*

The colours of the fabric samples were measured as described in section 2 and their colour parameters are presented in Table 4

Table 4. Colour parameters of the sample after flame-retardant treatment and after 5 washing cycles

Fabric Samples	Measurement condition	Color parameters				
		L	a*	b	c	h
Control	D65/10°	34.76	-37.53	-1.23	37.55	242
Dyed FRT1		36.48	-39.80	-2.02	39.85	
Dyed FRT1-W5		39.29	-40.73	-1.50	40.76	
Dyed FRT2		36.83	-39.47	-1.71	39.51	
Dyed FRT2-W5		38.33	-39.16	-1.16	39.18	

From the results of Table 4, the colour difference DE_{CIELab} between samples is calculated according to Equation 3 [14], the results are shown in Table 5.

$$DE_{CIELab} = \sqrt{DL^2 + Da^2 + Db^2} \quad (3)$$

Table 5. Colour difference DE_{CIELab} between samples

Sample	Colour difference DE_{CIELab} between samples		
	Control	Dyed FRT1	Dyed FRT2
Dyed FRT1	2.90	0.00	-
Dyed FRT1-W5	-	3.00	-
Dyed FRT2	2.87	-	0.00
Dyed FRT2-W5	-	-	1.56

Table 5 shows that the colour differences between the dyed FRT1 sample and the dyed FRT2 sample with the control sample are 2.90 and 2.87, respectively. According to assessment of Mokrzycki [14] and of Ngo, H.-T [12], these differences are medium ($2.0 \leq DE \leq 3.5$), and obvious only to trained eye

Research results on fire retardant treatment for dyed cotton fabric according to procedure 2 (with DBD plasma pre-activation)

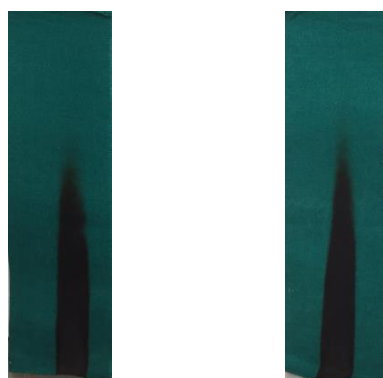
The dyed fabric was treated with flame retardant according to procedure 2 with a plasma treatment time of 30 seconds.

- First, the dyed cotton fabric was treated with DBD plasma as described in section 2 for 30 seconds. Then, the plasma treated samples were treated with flame retardants. Curing conditions were kept the same as Table 2. However, for experimental convenience, the curing time of condition 1 was rounded from 114 seconds to 120 seconds and is called condition 3. The results of testing the flammability of the sample after fire retardant treatment (P30-dyed FRT3, P30-dyed FRT2) and after 5 washing cycles (P30-dyed FRT3-W5, P30-dyed FRT2-W5) are presented in Table 6.

The photographs of the washed samples after vertical flammability test according to ASTM D 6413, are presented in Figure 4

Table 6. Flammability parameters of dyed fabric treated with flame retardants according to procedure 2 after treatment and after 5 washing cycles

Fabric samples	Plasma exposure time (s)	Curing conditions		Number of washes	Flammability parameters of fabric samples			
		Temp. (°C)	Time (s)		Afterflame time (s)	Afterglow time (s)	Char length (mm)	LOI (%)
P30-dyed FRT3	30	180	120	0	0	0	-	29.1
P30-dyed FRT3-W5				5	0	0	108	25.6
P30-dyed FRT2		180	150	0	0	0	-	28.8
P30-dyed FRT2-W5				5	0	0	110	26.2



P30-dyed FRT3-W5 P30-dyed FRT2-W5

Figure 4. Photographs of the washed samples after vertical flammability test according to ASTM D 6413

The results of Table 6 show that, with the support of plasma pretreatment for 30 seconds, the two experimental options allow the dyed cotton fabric after flame retardant treatment to be a durable flame retardant fabric [1]. It also meets ISO standards for making protective clothing [13]. Comparing the data in Table 6 with Tables 2 and 3 shows that when the fabric was pre-treated with plasma (even though the curing conditions did not change), the fire resistance of the fabric was still improved. In particular, the durability in flame retardancy of the fabric has been increased significantly. It is assumed that pre-treatment with plasma made the fabric more activated, so that the reaction between the flame retardant

and the fabric occurred more easily, which in turn helped increase the durability of the fabric's flame-retardant properties.

Colour measurement results show that the colour difference ($DE_{CIE\text{Lab}}$) between the plasma-treated sample and the dyed sample is only 0.26, showing that plasma treatment for 30 seconds does not affect the colour of the dyed fabric.

The colour difference ($DE_{CIE\text{Lab}}$) between the flame retardant treated sample and the dyed sample was 2.21, showing that the flame-retardant treatment changed the colour of the dyed fabric. Thus, it is the same as in the case of samples processed according to procedure 1 (Table 5), this difference is medium ($2.0 \leq DE \leq 3.5$), and obvious only to trained.

Conclusion

From the above results, some conclusions can be drawn as follows:

Pyrovatex CP New (MDPA) and Knittex FFRC (DHEU) are an effective combination for durable flame-retardant treatment of cotton fabrics including dyed cotton fabric.

The durable flame-retardant treatment process for dyed cotton fabric compared to that for undyed may have the same curing temperature but the curing time needs to be extended.

Plasma pretreatment with the power of $1\text{W}/\text{cm}^2$ for 30 seconds of dyed cotton fabric before the flame retardant treatment can help increase the flame retardancy of FRT cotton fabric. In particular, the durability in flame retardancy of the FRT fabric has been increased significantly.

Plasma pretreatment at $1\text{ W}/\text{cm}^2$ for 30 seconds of dyed cotton fabric did not change the colour of the fabric. But flame-retardant treatment may change the colour of dyed cotton fabrics with the medium level ($2.0 \leq DE \leq 3.5$). This colour difference is only observable to a trained human eye.

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