The Effect of Treatment with Fire Retardant on the Dyeing Process

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Abstract. This article studies the scientific and practical impact of flame retardant treatment of cotton fiber materials on dyeing technology. In the experiment, dyed cotton fiber fabric samples were given flame retardant properties and the changes in the quality indicators of dyed cotton fiber fabric samples were analyzed. The results of the flame retardant properties, physical and mechanical properties, color intensity and IQ indicators of the samples are presented. The article also reveals the problems arising from the interaction between flame retardant and dye and ways to eliminate them.

Keywords: flame retardant, dyeing, fire resistance, dye, material surface properties, chemical treatment.

INTRODUCTION

Nowadays, in textile and building materials technology, it is important to give various functional properties to products [1]. These include properties such as fire resistance (flame resistance) [2], moisture resistance (hydrophobicity) [3], and resistance to microorganisms (antibacterial) [4]. Treatment with flame retardants is carried out precisely to increase the fire resistance of materials [5]. However, once flame retardants are absorbed into the surface of the material, they have a certain effect on other processes, in particular the dyeing process [6].

A flame retardant is a chemical substance used to reduce the flammability of materials [7]. Flame retardants slow down or completely stop the combustion process under the influence of high temperatures [8]. They are mainly applied to wood, fabric, paper, plastic and other flammable materials [9],[10]. As a result, they increase the combustion temperature of materials, slow down the ignition process, reduce smoke emission, and increase the overall fire safety of the material [11].

Flame retardants may contain phosphorus, nitrogen, bromine, and other chemical compounds [11], [12]. It is these structural elements that are the main factors affecting the dyeing process.

Dyeing is the process of applying or adhering dyestuffs to the surface of a material to impart color.[13] The dyeing process typically involves the following steps:

Material surface preparation (cleaning, degreasing, drying)

Priming (if necessary)

Applying the base coat

Drying and hardening

Each of these stages depends on the chemical and physical properties of the material surface [14]. In materials treated with flame retardants, these properties change to some extent [15].

Flame retardants affect the structure and chemical activity of the material surface. As a result, the following conditions may be observed during the dyeing process:

1. Decreased dye absorption

2. Uneven color spread

3. Decreased dye adhesion

4. Dye color change

5. Extended drying time

To reduce negative effects, it is important to ensure compatibility between the flame retardant and the dye.

Although flame retardants are important in protecting materials from fire, they can also have a negative impact on the dyeing process. However, these problems can be minimized through the use of appropriate materials and experimental testing.

METHODS

The object was selected as a dyed yarn made of 100% cotton fiber. The physical and mechanical properties of the dyed fabric are presented in the table below.

TABLE 1. Physical and mechanical properties of dyed fabric

|  |  |
| --- | --- |
| **Indicators** | **Yarn - fabric** |
| **Surface density, g/m²** | 2,93 |
| **Breaking load, N:**  **- according to the warp, N**  **- according to the weft, N** | 571,8  126,2 |
| **Elongation at relative break, *Ɛр*, %:**  **- according to the warp, %**  **- according to the weft, %** | 46,57  20,91 |
| **Air permeability, dm³/m²∙s** | 10,039 |

Antiperine - the substance offered by Setas Color Center - Setaflam PNF was used as a substance. Fire-resistant antiperine - Setaflam PNF - is transparent in color, halogen-free, odorless. It is mainly used for cellulose fiber fabrics.

The experiment used chemicals such as a 3% solution of the active dye, a surfactant offered by the “Pulcra” company, soap solution, sodium carbonate, sodium hydroxide, sodium silicate, and hydrogen peroxide.

Hydrogen peroxide – 2.0

Sodium hydroxide – 3.0

Sodium silicate (d=1.44) – 15.0

Saturator SFM - 0.5

Then the samples are washed in boiling and cold water.

Samples of bleached cotton fiber fabric were continuously dyed with an active dye in a solution with the following composition, g/l:

Colourist material – 5.0

Sodium bicarbonate- 10.0

Urea – 200.0

Saturator SFM – 1.0

dyed and the dyeing process technology was carried out as follows:

Soaking-compression-drying-heat treatment. Then, the fire-resistant property was given. The fire-resistant property was given based on the following sequence.

Soaking-compression-drying-heat treatment. The fire resistance properties of the obtained samples were tested and their physicochemical properties were studied.

To give the samples fire-resistant properties, 5x20 cm long samples were prepared. The samples were soaked in a solution with a modulus of 30 (relative to dry mass) for 3 minutes. They were dried in a drying cabinet for 10 minutes at a temperature of 90-100 0С. After the drying process, thermal treatment was carried out at a temperature of 140 0С for 2 minutes.

*Determination of the fire resistance of samples.* 5 elementary samples measuring 5x20 cm are cut. Elementary samples are kept for 24 hours in standard atmospheric conditions. The level of alcohol in the laboratory alcohol burner is checked. The flame height should be 40-50 mm. The upper narrow end (edge) of the elementary sample is clamped with tweezers. The lower end of the sample is held 20 mm in the flame and at the same time the stopwatch is started. The sample is kept in the flame for 20 seconds. After the specified exposure time in the flame, the sample is removed from the fire zone. After the sample is removed from the open flame, the residual combustion and burning duration are determined. The following results are recorded in the test report:

- residual burning time of the sample, s;

- length of the burned area of the sample, cm;

- other characteristics of the test process (hole formation, melting of the sample material, etc.).

*Determination of color quality indicators of dyed samples.* The color quality indicators of dyed samples were determined in the scientific laboratory of “Kor-Uz Textile Technopark” using an X-Rite Ci 7800 laboratory spectrophotometer (Korea) under D65 standard radiation according to the method presented in.

IQ spectrometer analysis of flame retardant-treated samples was measured on a Nicolet iS50 far-infrared sbiopolymerometer in the range of 450–4000 sm-1.

*Determination of physical and mechanical properties of fibrous materials.* Samples measuring 300x50 mm are prepared for testing. Tensile strength and elongation at break are determined on the “AG-1” tensile tester at 25±30 and 60±5% humidity. The tester is calibrated before testing the sample.

EXPERIMENTAL PART AND ANALYSIS OF RESULTS

A 100% cotton raw yarn was bleached and dyed continuously with an active dye using the above dye solution and the technological sequence. The dyed samples were treated with a 50% flame retardant solution (prepared by diluting in distilled water at room temperature) to impart flame retardant properties. The color intensity, flame retardancy, and quality parameters of the obtained samples were determined.

The color intensity of samples dyed in a continuous process and given fire-resistant properties is presented in the table below.

TABLE 2. Color quality indicators of samples

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Name** | **Samples** | **Color characteristics, K/S** | | | | | |
| **R** | **L\*** | **a\*** | **b\*** | **C\*** | **h\*** |
| Sample 1 |  | **6.12** | | | | | |
| 7.06 | 44.63 | -1.91 | -33.80 | 33.85 | 266.76 |
| Sample 2 |  | **1.92** | | | | | |
| 17.65 | 59.80 | -3.70 | -31.78 | 32.00 | 263.36 |
| Sample 3 |  | **4.36** | | | | | |
| 9.41 | 45.98 | -4.33 | -24.77 | 25.15 | 260.09 |
| Sample 4 |  | **2.35** | | | | | |
| 15.29 | 55.12 | -3.70 | -24.63 | 24.91 | 261.46 |

1 - sample dyed with an active dye in a continuous method, 2 - sample dyed with an active dye in a continuous method and washed in method No. 1, 3 - sample dyed with an active dye in a continuous method and treated with a flame retardant solution, 4 - sample dyed with an active dye in a continuous method and washed in method No. 1 after treatment with a flame retardant solution.

The results of spectrocolorimetric analysis show that unsaturated (C\*) colors were formed in sample 3 treated with antiperine solution. From the results, we can see that C\* - color is unsaturated and h\* - color does not reach high values in sample -, while in sample 1 these indicators are high. The chemicals contained in the flame retardant react with the dye molecules, causing the color to fade. As a result, the color changes, becomes dull. However, when samples washed by method No. 1, that is, samples 2 and 4, are compared, the k/s indicators in sample 4 treated with antiperine are 45% higher.

The fire resistance properties of samples 3 and 4 treated with flame retardant were tested and the results obtained are presented in the table below.

TABLE 3. Fire resistance properties of flame retardant treated samples

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Name | Flame retardant concentration, % | Fire resistance indicators | | | |
| The time of holding the samples in the fire, sec. | residual burning time of the sample, s | length of the burned area of the sample, cm; | other features of the testing process |
| Sample 3 | 50 | 20 | 0 | 2,5 | - |
| Sample 4 | 50 | 20 | 0 | 5 | - |

The data in the table above show that the samples have high fire resistance properties. In sample 3, the burned area of the sample was 2.5 cm, and in sample 4, it was 5 cm. Considering that sample 4 was dyed with an active dye in a continuous method and washed in method No. 1 after treatment in a flame retardant solution, the sample retained its fire resistance properties even after washing. This indicator complies with the requirements of GOST 11209-2014.

Below, Figure 1 shows the results obtained after testing the fire resistance properties of flame retardant-treated samples.

|  |  |
| --- | --- |
| **a)** | **b)** |

FIGURE 1. a) Sample 3 - Continuously dyed sample treated with flame retardant solution, b) Sample 4 - Continuously dyed sample treated with flame retardant solution and washed using method No. 1

In the next step, the physical and mechanical properties of the samples were tested, and the results are presented in the table below.

TABLE 4. Physical and mechanical properties of samples

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Name | Breaking load, N | | Elongation at break, Ɛр, % | |
| By length | By width | By length | By width |
| Sample 1 | 571,8 | 126,2 | 46,57 | 20,91 |
| Sample 2 | 319,6 | 257,8 | 46,25 | 25,91 |
| Sample 3 | 148,6 | 70,7 | 20,93 | 12,89 |
| Sample 4 | 256,8 | 127,5 | 50,58 | 21,41 |

According to the results of the table above, the values of the breaking load and elongation at break in the samples treated with flame retardant decreased. This is explained by the fact that the flame retardant formed a film on the surface of the fabric. Also, these values were slightly higher in the washed samples. This is because the chemicals in the flame retardant that give it flame retardant properties indicate partial stability to washing.

To investigate the cause of the interaction between the dye and flame retardant in the painted and flame-retardant samples, IQ spectroscopic analysis of samples 1 and 3 was performed (see Figure 2).

Figure 2 (a) Spectra typical of the sample – dyed cotton fiber fabric. In the spectrum (a), the main peaks characteristic of cellulose were observed: a broad peak in the range of 3300–3500 cm⁻¹ is the stretching vibration of the O–H bonds in cellulose. A peak at ~2900 cm⁻¹ is the vibration of the C–H bond. The range of 1000–1200 cm⁻¹ is the vibrations of the –C–O–C and C–O bonds, which are characteristic of the polysaccharide structure of the cellulose molecule. An intense peak at ~899 cm⁻¹ confirms the presence of a β-glycosidic bond in cellulose. Also, the observation of aromatic ring vibrations of the active dye in the range of 1600–1500 cm⁻¹ indicates the successful attachment of the dye molecules to the fiber.

Figure 2 (b) Spectra typical of sample – dye + flame retardant treated cotton fiber. In spectrum (b), although the above peaks are preserved, a number of new intense peaks have appeared and the existing ones have been significantly enhanced. This indicates that the flame retardant has chemically bonded to the fiber.

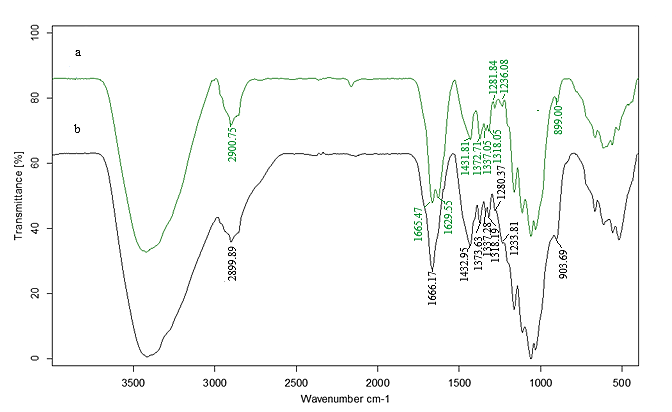


FIGURE 2. IQ spectra of the samples. a) Sample 1 was dyed with a continuous active dye, b) Sample 3 was dyed with a continuous active dye and treated with a flame retardant solution.

Main changes: The range of 3300–3500 cm⁻¹ has expanded. This is due to the additional hydroxyl or phosphorus groups in the flame retardant. A strong peak around 1660–1670 cm⁻¹. This region corresponds to the vibration of the P=O bond. This peak is clearly visible when using phosphorus flame retardants. Setaflam PNF is a phosphorus (p), nitrogen N-containing, halogen-free flame retardant. It is chemically and physically bonded to cellulose. Therefore, new P-O-C, P=O, P-O-H, N-H vibrations have appeared in the FTIR spectrum. This is explained by the intense peaks in the range of 1200–1000 cm⁻¹. Vibrations of the P–O–C, P–O, P=O and N-H bonds are observed in this region. The increase in the peaks indicates that the flame retardant is bound to cellulose through covalent or hydrogen bonds. The decrease in the peak at 899–903 cm⁻¹ indicates that the β-glycosidic bonds of cellulose have been partially changed. This is explained by the fact that the flame retardant has reacted with some parts of the cellulose chain. The appearance of new phosphorus vibrational peaks in the flame retardant-treated sample, as well as the decrease in the intensity of some chemical bonds of cellulose, indicates that a chemical interaction has occurred between the flame retardant and cellulose. Also, the aromatic dye peaks are preserved, indicating that the flame retardant has not damaged the dyed fiber.

Phosphorus-nitrogen compounds (PNF) are used as fire retardants that are part of polymeric materials. The substances that make up PNF are ammonium phosphates, which react with the materials to form a layer. This layer protects the surface of the material from fire. PNF-based fire retardants are very effective because they have a mechanism to stop the fire and prevent the material from burning. PNF (phosphorus-nitrogen compounds) are added to polymeric materials. During the combustion process, they react with each other to form a layer on the surface of the material. The resulting layer protects against fire and prevents the material from burning. This layer reduces thermal conductivity and prevents the material from decomposing. The mechanism of stopping the combustion process occurs by stopping combustion reactions in the gas phase by forming non-combustible NH3 gas.

CONCLUSION

The experiments show that the flame resistance of cotton fabric treated with flame retardant was 2.5 cm and 5 cm after washing. This indicates an increase in fire resistance and meets the requirements of GOST. However, this process had a certain effect on the dyeing process. Scientific studies show that the active groups (–NH₂, –PO₄, –OH) in the dyestuff and flame retardant interacted with each other chemically. As a result: The color of the fabric becomes dull, but the FTIR analysis showed that the aromatic ring vibrations of the active dye in the range of 1600–1500 cm⁻¹ indicated the successful binding of the dye molecules to the fiber. Also, the fact that the aromatic dye peaks were preserved after treatment with flame retardant indicates that the flame retardant did not damage the dyed fiber. The active coloring agent can be used together with Setaflan PNF flame retardant. This can prevent problems such as paint failure, uneven coloration, and reduced adhesion.

When an ammonium phosphate-based flame retardant was applied to cotton fabric dyed with an active dye, the flame resistance of the samples increased and the color intensity decreased. However, the dye and the part of the antiperine attached to the fiber remained.

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