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Research and development of effective dye composition formulations for use in the dyeing process of cotton fabric

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Abstract: This article presents the results of studies of the process of modifying cotton fabric with fibroin oligomers, the influence of pH, temperature, concentration of polyvalent metal salts, sodium nitrite and resorcinol on the formation of metal complexes in the structure of cotton fiber, as well as the established optimal compositions of the developed powder composite dyes for the production of composite dyes and for dyeing textile cotton and viscose fabrics.

Keywords: dye composition, cotton fiber, metal complexes, textile materials, polyvalent metals, fibroin oligomers.

1. Introduction

Globally, the demand for textile industry products, especially for multi-colored materials made from cotton, viscose and their blends, is increasing from year to year. Expensive imported synthetic dyes are used to dye these textile materials [1-3]. However, existing synthetic dyes have certain disadvantages, in particular, they are very expensive, scarce, and have relatively low strength characteristics. In connection with this, the development of new compositions of relatively cheap compositions based on polyvalent metal salts and local raw materials with high strength characteristics for dyeing cotton textile materials is important [4-5].

The republic is conducting scientific research on the development of the textile industry, searching for ways to intensify the process of dyeing textile fabrics based on cotton, viscose and their mixtures, on the basis of which the competitiveness of finished products increases, and certain results are achieved [6].

Based on the analysis of modern literary sources, it should be noted that in the development of dyeing materials for dyeing textile materials, including cotton and viscose and their mixtures, insufficient research has been carried out on the development of optimal compositions of dyeing materials, including composite dyes based on local raw materials. It should also be noted that when developing and obtaining effective compositions of dyeing compositions based on salts of polyvalent metals and organomineral ingredients, the process of dyeing cotton and viscose fabrics and their mixtures in the presence of various types and concentrations of acid and salts of polyvalent metals is not sufficiently taken into account [8].

In this regard, the development of more effective powder compositions based on local raw materials, making it possible to obtain colors in a wide range of colors that are resistant to various physical and chemical influences such as light, washing, friction, and the action of organic solvents, is an urgent problem.

Object and research methods. The objects of research are cotton (chintz), viscose fabric and their mixtures, fibroin oligomers, salts of alkali and polyvalent metals, aromatic hydroxy compounds (resorcinol and pyrocatechin) and others.

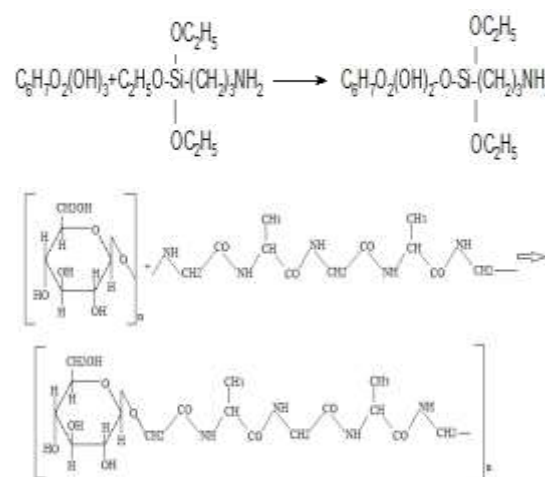
Research methods. The dissertation work used modern physico-chemical methods of analysis, including IR spectroscopy, thermal methods (DTA, TGA), photocalorimetry, complexometry, pH-metry and other

physico-chemical methods of analysis, as well as other standard methods of analysis permitted for CIS countries.

2. Research results and their analysis

In this regard, we conducted research on the formation of metal complexes in the structure of cotton fiber and developed optimal compositions for dyeing textile materials.

First of all, we studied, with the aim of introducing functional groups into fibers, the process of amination of cotton fabric with γ -aminopropyltriethoxysilane (a) and fibroin oligomers (b):



To substantiate the process of amination of cotton cellulose with γ -aminopropyltriethoxysilane and fibroin oligomers, IR spectra of the original and aminated cotton fabric were taken. The results of the IR spectra are shown in Fig. 1. It is known that the hydroxyl groups of cotton cellulose are characterized by absorption bands in the region of 3650-3100 cm^{-1} . Modification of cotton fabric with fibroin oligomers helps to increase the intensity of the absorption band at 3000-3600 cm^{-1} (Fig. 1), which is evidence of amination of cotton fabric, since the absorption of the primary amino group is located in the range of 3300-2500 cm^{-1} (stretching vibrations) and 1640 -1535 cm^{-1} (deformation vibrations).

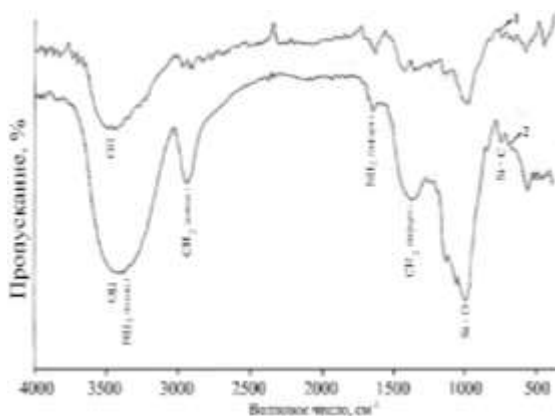


Fig.1. IR spectra of original (1) and aminated (2) cotton fabric

The modification of cotton fabric by fibroin oligomers is also evidenced by the absorption band at 2930 cm^{-1} , responsible for the deformation vibrations of $-\text{CH}_2$ groups, the absorption band at 1000 cm^{-1} , responsible for the Si-O silaxane groups (stretching vibrations) and the absorption bands at $820\text{--}850\text{ cm}^{-1}$ in the spectrum of aminated cotton fabric are responsible for Si-C (stretching vibrations).

It was found that the tensile strength of cotton fabric containing fibroin oligomer is slightly higher than that of the original fabric.

According to the basic law of color theory, almost any color can be represented as the sum of three linearly independent colors. Red (R), green (G) and blue (B) are used as primary colors, i.e. three monochromatic radiations with a wavelength of 700.0 ; 546.1 and 435.8 nm , respectively.

The influence of the pH value of the processing medium in the dye bath on the formation of metal complexes of iron (III) and cobalt (II) was studied. It has been established that when cotton fabric is treated in a dye bath with a complexing solution with a pH of 3.6 to 4.2 , the fabric acquires the most intense color, which indicates the formation of the maximum number of metal complexes in the structure of the cotton fiber.

Consequently, the least bright channel turned out to be blue (B) and the brightest red (R). Thus, the optimal pH range of a complexing solution with iron and cobalt when dyeing cotton fabric is $3.6\text{--}4.2$.

The influence of temperature on the process of formation of metal complexes in the structure of aminated cotton fiber was studied. The processing process was carried out at temperatures of 20 , 50 , 80 and 98°C . With increasing temperature, the rate of formation of metal complexes in the structure of cotton fiber is achieved at a temperature of 98°C for $5\text{--}7$ minutes.

In order to develop optimal compositions of complexing compositions and solutions based on them in a dye bath, we conducted studies of the influence of the concentration of polyvalent metal salts on the formation of the optimal amount of metal complexes in the structure of cotton fiber based on nickel chloride, cobalt chloride, iron and copper sulfite.

As an example in Fig. 2 (a, b) shows the dependence of the optimal formation of metal complexes in the structure of cotton fiber on the concentration of the polyvalent metal nickel chloride, which is observed at 0.5 g/l , and for cobalt chloride 0.25 g/l , which has an extreme character, passing through minimum.

Similar results are observed with complexing solutions containing iron and copper sulfate.

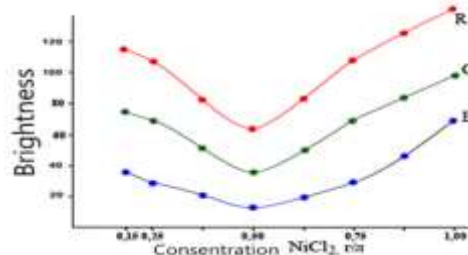
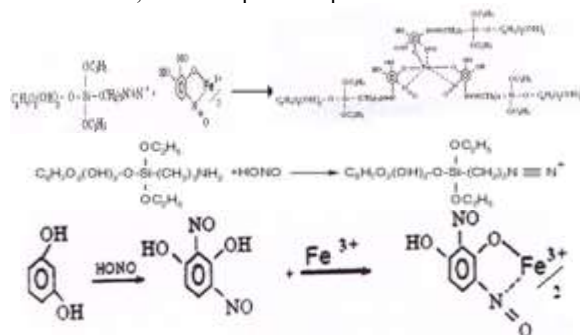


Fig. 2. Dependence of R-, G-, B- components of the color of cotton fabric containing metal complexes of nickel chloride (a) and cobalt chloride (b) in a complexing solution

From the data obtained it follows that under the influence of nitrous acid, the amino groups of the fibers are transferred to the azo group, which, when interacting with metal cations, form complex compounds.



In the IR spectra of dyed cotton textile materials, there is a shift of the absorption band at 1360 cm^{-1} , which belongs to the amino group, to 1600 cm^{-1} , indicating the formation of a Me-N coordination bond, and a peak appears in the region of $550\text{--}560\text{ cm}^{-1}$, responsible for the valence bonds vibrations of the Me-N bond (Fig. 3). The data obtained allow us to conclude that coordination occurs through nitrogen atoms in the diazo compound. The presence of an aromatic azo group in cotton fiber complexes is confirmed by absorption bands at 1550 cm^{-1} and $1046\text{--}1060\text{ cm}^{-1}$. From the data obtained it follows that under the influence of nitrous acid, the amino group of the fiber transforms into an azo group, which, when interacting with metal cations, form complex compounds.

It should be noted that with an increase in polyvalent metal cations in the processing dye bath to a certain stage for each type of cation, the number of metal complexes in the structure of cotton fiber increases, which is associated with the concentration factor.

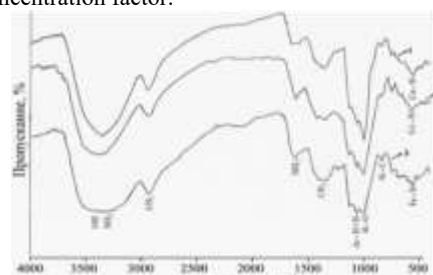
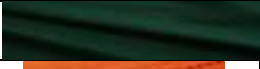

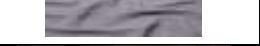



Fig. 3. IR spectra of cotton fabric containing metal complexes Cu^{2+} (1), Co^{2+} (2), Fe^{3+} (3)

Thus, the optimal concentration of polyvalent metal salts that promote the formation of the maximum amount of metal complexes in the structure of cotton fiber is: for iron sulfate and nickel chloride 0.5 g/l, for cobalt chloride - 0.25 g/l, for copper sulfate – 0.1 g/l. The choice of metal salt and dyeing conditions depends on the desired color and the desired effect. Table 1 shows samples of dyed cotton fabrics based on polyvalent metal salts.

Table 1
Samples of cotton fabrics dyed with dye compositions based on polyvalent metal salts

№	Dyed fabric samples	Formula of polyvalent metal salts used
1.		$\text{Fe}_2(\text{SO}_4)_3$
2.		CoCl_2
3.		CuSO_4
4.		NiCl_2

Next, we studied the effect of the concentration of sodium nitrite (NaNO_2) on the formation of the optimal amount of metal complexes in the structure of cotton fiber, which NaNO_2 in an acidic environment transforms into nitrous acid, which promotes the production of dinitrosoresorcinol and the transition of the amino groups of fibroin oligomers into a diazotized amine.

In Fig. 4 shows the dependence of R-, G-, B-, components of the coloring of cotton fabric containing metal complexes of nickel, cobalt, iron and copper, on the concentration of NaNO_2 in the complexing dye solution. The studies also showed the interaction of polyvalent metal cations with the formation of complex compounds of various compositions, corresponding strength with increasing concentration of sodium nitrite in the complexing dye bath. In this case, the color intensity increases and reaches its maximum value at a sodium nitrite concentration of 0.4-0.5 g/l.

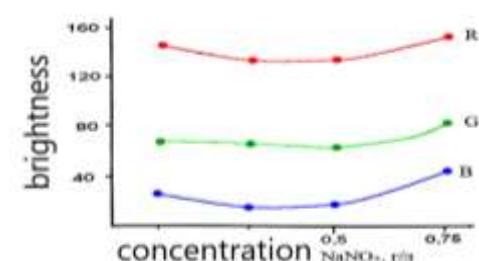


Fig. 4. Dependence of R-, G-, B- components of the color of cotton fabric containing metal complexes a) nickel chloride (NiCl_2), b) cobalt chloride (CoCl_2) on the concentration of NaNO_2 in the complexing solution

The influence of resorcinol concentration on the formation of the optimal amount of metal complexes in the structure of aminated cotton fiber was studied. Research results have shown that the most optimal concentration of resorcinol in a complexing bath is 0.5-0.6 g/l.

Based on the analysis of the results of the above studies, optimal compositions and ratios of organomineral ingredients have been developed that contribute to obtaining the maximum amount of metal complexes in the structure of cotton fiber, ensuring high-quality dyeing of cotton and viscose fabrics.

These are the following compositions: for the

composition of the composition, ferrous sulfate, sodium nitrite, resorcinol 1:1:1; for the composition nickel chloride, sodium nitrite, resorcinol 1:1:2; for the composition of cobalt chloride, sodium nitrite, resorcinol 1:1:2; for the composition copper sulfate, sodium nitrite, resorcinol 1:5:7.5.

Based on the research carried out, we can conclude that in order to obtain the most saturated colors, the dye bath should have the following composition: NiCl_2 -0.5 g/l; resorcinol-0.75 g/l; NaNO_2 -0.5 g/l. Composition: NiCl_2 -29%, NaNO_2 -29%, resorcinol -42%.

3. Conclusion

Based on the research results, rational compositions of composite dyeing materials for dyeing cotton, viscose fabrics and their mixtures, based on polyvalent metal salts and organomineral ingredients, were developed. It has been established that the introduction of fibroin oligomers into cotton fiber significantly affects the physicochemical, mechanical and technological parameters of cotton fabric, helping to improve its processing and the physical, mechanical and technological characteristics of the fabric; a mechanism for the process of dyeing aminated cotton fabrics with polyvalent metals (sulfate) has also been proposed iron, nickel chloride, cobalt chloride and copper sulfate), coloring compositions based on sodium nitrite and resorcinol salts. It has been established that the optimal amount of metal complexes in the structure of cotton fiber is obtained at a temperature of 980C at a pH of 3.6-4.2. It was revealed that the formation of metal complexes in the structure of cotton and viscose fiber helps to improve the physical and mechanical properties and polishing of textile materials based on cotton, viscose and their mixtures.

The possibility of replacing synthetic dyes with created coloring compositions is substantiated, where the dyeing speed increases by 2.5-3 times and thereby reduces their cost by 1.3-2.0 times.

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