STUDY OF THE INFLUENCE OF PREPARATION OF KNITTED FABRIC ON THE SURFACE AND SUPRAMOLECULAR STRUCTURE OF COTTON FIBER

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Abstract: *The main goal of this research is to study the effect of preparation methods (alkaline boiling, basic combined boiling and bleaching technology, developed preparation technology) on the surface structure of cotton fibers and changes in the supramolecular structure of cellulose. The surface of the cotton fibers was examined using a scanning electron microscope and the supramolecular changes were studied by radiography. The results of the study of surface and supramolecular structure of cotton fibers explain the increase in the sorption ability of the knitted fabric, which was prepared according to the developed technology.*

Keywords: *preparation, cotton knitted fabric, fiber surface, supramolecular structure.*

1 INTRODUCTION

Preparation is an important technological stage of cotton knitted fabric finishing. It is at this stage that the basic properties of the textile material are formed, which provide not only the quality of the subsequent dyeing and final treatment, but also the hygienic properties of the finished products. These are primarily sorption properties that cotton knitwear gets after the removal of natural and technological impurities in the conditions of the preparation process. Sustained whiteness is another of the basic properties that a knitted fabric gets after preparation and is an important quality indicator of this range of textile materials.

The classic preparation technology of the cotton knitted fabrics includes boiling and bleaching, which are carried out sequentially. The boiling is carried out in an alkaline or neutral medium in the presence of surfactants. Alkaline solutions of hydrogen peroxide are most often used for bleaching [1, 2].

Currently, technology of the cotton knitwear preparation aimed at reducing time, temperature and water consumption. For this purpose, intensification of the preparation processes using physical, biological and chemical methods is carried out.

Physical methods include the use of gamma radiation [3], supercritical carbon dioxide [4], ozone [5] and atmospheric pressure plasma treatment [6]. The use of physical methods of exposure provide an opportunity to carry out the process with a reduction in water consumption or even in an anhydrous environment with a reduced temperature and a reduction in the duration of the process. It should be noted that the limited use of these intensification methods is due to the need for additional high-tech equipment.

Biological intensification methods suggest the use of enzymes that have an eco-friendly character in cotton knitwear preparation technology [7-9]. Their application is complicated by the need for strict control of the preparation process conditions.

The simplest and most effective way to intensify the technology of boiling and bleaching of cotton knitwear is the use of chemicals that can speed up the process of removing associated impurities of cotton and give the cotton textile material capillarity and whiteness at low treatment temperature [10-13].

A promising direction of resource saving is combination of boiling and bleaching [14-17]. According to the results of previous studies, the combined preparation technology of cotton knitted fabric was developed [18, 19]. It has been established that the developed preparation principle for cotton knitted fabric promotes the maximum removal of wax-like and colored substances from textile material and, as a result, increases capillarity and whiteness. The strength of knitwear decreases slightly. This became possible due to the application of the previously developed highly effective surfactants composition, which allows to combine the operations of boiling and bleaching and to carry out the preparation process at a reduced temperature of 80°C.

As a result of the research [20], it was established that the developed preparation technology affects the sorption kinetics and the fixation degree of the reactive dyes by cotton knitted fabric. The developed preparation method has advantages in comparison with the base one, which consist in the combination of boiling and bleaching. The reduction of the processing temperature from 100°C to 80°C ensures efficiency of the developed technology. One of the reasons for the increase in dyes sorption by cotton fiber can be its damage due to excessive oxidation of cellulose of textile material with hydrogen peroxide during bleaching or during undesired oxidative destruction of the substrate by oxygen of the air during boiling [21]. As a result, the polymer chains of cellulose are broken and the degree of polymerization reduces. At the supramolecular level, the degree of crystallinity decreases and the volume of amorphous regions increases. This in turn causes an increase in the sorption capacity of the fiber, including in relation to dyes. It should be noted that the obtained colors on a cotton substrate that was damaged during the preparation process are characterized by reduced resistance to physical and chemical influences, despite the intense sorption of the dye. In addition, the textile material loses strength, which is exacerbated during the operation of finished products.

2 THE GOAL OF THE STUDY

The goal of the work is to study the changes in the surface and supramolecular structure of cotton fiber cellulose under the influence of the developed preparation technology of knitted fabric.

3 MATERIALS AND METHODS

Study was carried out on grey cotton rib knitted fabric 1×1 with surface weight 150 g/sm^2. Preparation of knitted fabric was carried out under the conditions given in Table 1.

The textile auxiliaries used are Ultravalon TC, Albafluid CD, Albaflow FFC-01, Clarite by Huntsman NMG and Oxipav A1214C.50 by LLC Research and Production Association NII PAV. The surfactant composition contains in a certain ratio Ultravon TC as a wetting agent, Albafluid CD as an anticrease agent, Albaflow FFC-01 as a defoamer and Oxipav A1214.50 as a detergent.

Micrographs of the cotton fibers surface were obtained using a JSM 6060 LA (Jeol, Japan) scanning electron microscope after preliminary deposition of a thin layer of gold by the cathode method using a JFC-1600 instrument.

The amorphous-crystalline structure of cotton knitted fabric samples was investigated by wide-angle X-ray diffraction using a DRON-4-07 diffractometer. The X-ray optical scheme of the specified diffractometer was made to pass the primary X-ray beam through the thickness of the sample under study according to the Debye-Scherrer method. The studies were carried out in monochromatic CuK$_\alpha$-X-rays (wavelength λ=0.154 nm) using a Ni-filter.

The size of the crystallites was estimated using the Scherrer method [22, 23] by determining the value of L in the direction of the angular position of the singlet maxima and then obtaining the average value of $\langle L \rangle$. First, the angular half-width (1/2β)×2 of the left and right parts of the diffraction maxima was determined at $2\theta_m$=14.8° and $2\theta_m$=22.6° respectively with the subsequent calculation of the average value.

$$L=K\cdot\lambda\cdot(\beta\cdot\cos\theta_m)^{-1} \tag{1}$$

where K is a constant that is associated with the form of crystallites (with an unknown form of crystallites K=0.89) and β is the angular half-width (width at half height) of the clearest singlet diffraction maxima.

Table 1 Cotton knitted fabric preparation conditions

Preparation method	Preparation conditions
Base technology	*Boiling:* TF-129B (washing agent) – 2 g/l; Albafluid CD (anticrease agent) – 0.8 g/l; Soda ash – 1.5 g/l. Treatment at 80°C for 20 min. Hot washing. *Bleaching:* Ultravalon TC (wetting agent) – 1.1 g/l; Albafluid CD (anticrease agent) – 0.8 g/l; Albaflow FFC-01 (defoamer) – 0.5 g/l; Clarite (hydrogen peroxide stabilizer) – 0.4 g/l; Hydrogen peroxide 60% w/w – 1.5 g/l; Sodium hydroxide – 1.5 g/l. Treatment at 98°C for 20 min. Hot washing, neutralization, hot washing, drying.
Developed technology	Surfactant composition – 1.5 g/l; Hydrogen peroxide 60% w/w – 1.5 g/l; Sodium hydroxide – 1.5 g/l. Treatment at 80°C for 20 min. Hot washing, neutralization, hot washing, drying.
Alkaline boiling	Ultravalon TC (wetting agent) – 1 g/l; Sodium hydroxide – 5 g/l. Treatment at 100°C for 20 min. Hot washing, neutralization, hot washing, drying.

The estimation of the relative crystallinity level of the structure (X_{cr}) of cotton knitted fabric samples was carried out according to the method given in [24]:

$$X_{cr}=Q_{cr}\cdot(Q_{cr}+Q_{am})^{-1}\cdot 100 \quad (2)$$

where Q_{cr} is the area of diffraction maxima, which characterize the crystal structure and ($Q_{cr}+Q_{am}$) is the area of the entire X-ray diffractogram in the 2θ interval, in which the amorphous-crystalline structure of the samples is observed.

4 RESULTS AND DISCUSSION

Earlier studies [18-20] established that as a result of preparation according to the developed principle, the knitted fabric gets high sorption properties with relation to reactive dyes and the resulting colors are characterized by high resistance to light.

The reason for the increase in the sorption ability of cotton fibers in relation to dyes can be a change in the molecular and supramolecular structure. As a result of the influence on the molecular structure of cotton fiber, cellulose is damaged and degree of polymerization decreases, which affects the strength of the textile material. A change in the supramolecular structure of cotton fiber cellulose does not affect fiber strength [25]. For example, a change in the supramolecular structure of cellulose during mercerization does not reduce the degree of polymerization of cellulose, but increases its sorption ability [26, 27].

It was found that the cotton knitwear after preparation according to the developed technology does not lose strength and the sorption of reactive dyes increases at the same time.

The presented micrographs of the cotton fibers surface (Figure 1), obtained using an electron scanning microscope, confirm the preservation of the knitted fabric structure.

The results of microscopic examination of the fibers surface structure showed that the surface of cotton fibers after preparation by the studied methods (Figures 1b-1d) does not undergo changes as compared with the surface of the original grey fiber (Figure 1a).

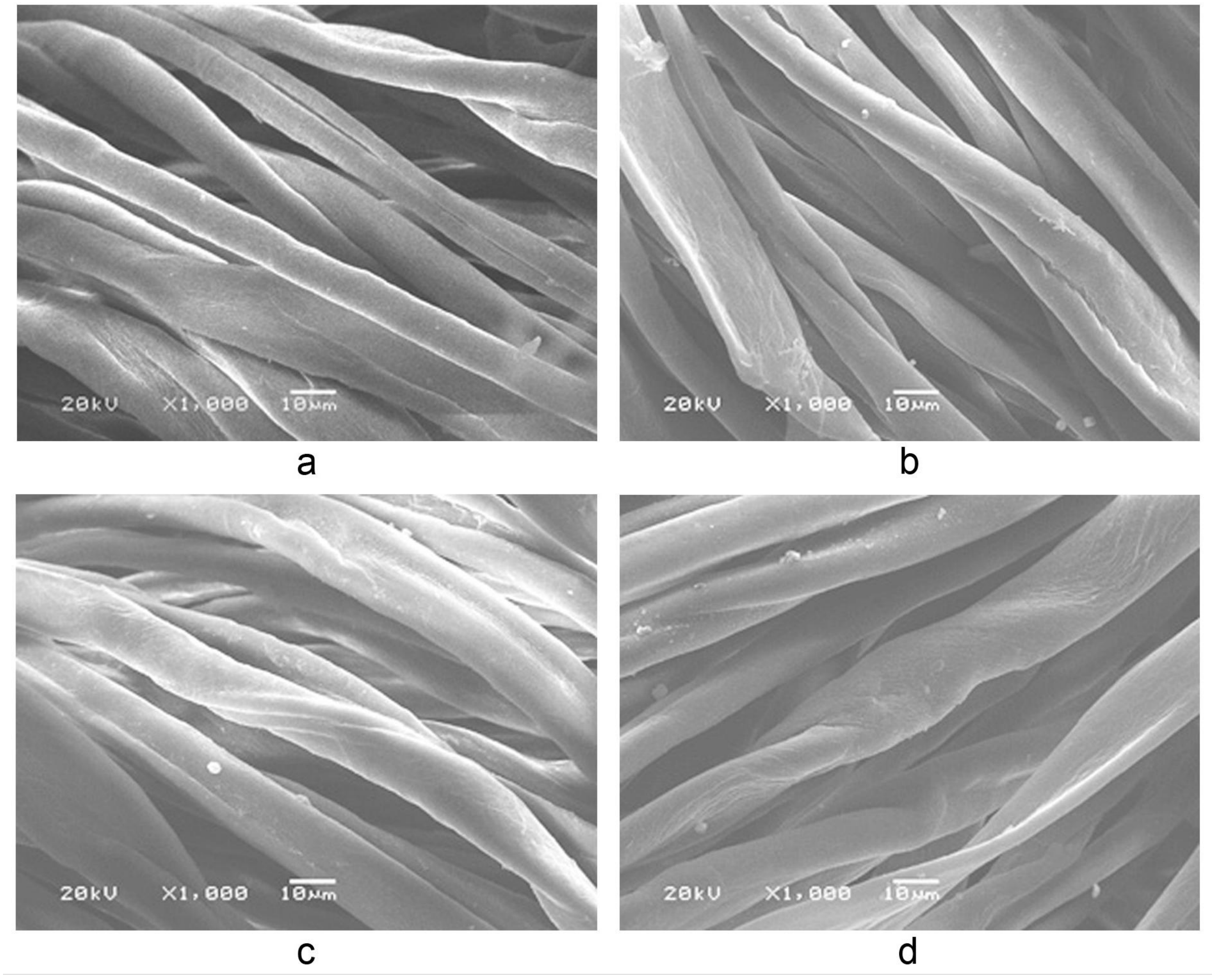

Figure 1 Micrographs of cotton fiber samples: a) gray, b) base technology, c) developed technology, d) alkaline boiling

Thus, it is necessary to investigate the supramolecular structure of cotton knitwear prepared by the developed technology. Radiographic study is one of the ways to study the supramolecular structure of textile materials [26]. The received roentgenograms of the cotton knitted fabric samples prepared in the different ways are presented in Figure 2.

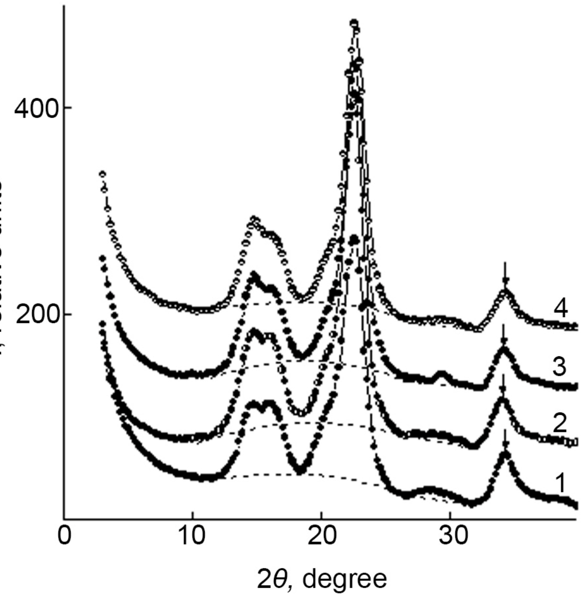

Figure 2 Wide-angle X-ray diffractograms of cotton knitted fabric samples: 1) gray, 2) base technology, 3) developed technology, 4) alkaline boiling

When analyzing X-ray diffractograms of knitted fabric (Figure 2), it was revealed that all samples have an amorphous-crystalline structure with a high level of crystallinity of more than 75%. This is evidenced by the presence of three main groups of diffraction maxima against a background of an imaginary diffraction maximum of diffuse type – amorphous halo – with the angular position of their peaks from $2\theta_m$=18.5° to $2\theta_m$=20°, which are shown by dotted lines. This is a doublet maximum with apexes at $2\theta_m$=14.8° and 16.0°, an asymmetric multiplet maximum with apex at $2\theta_m$=22.6° and a low-intensity singlet maximum, the angular position of the apex of which depends on the preparation method of sample and is in the range of angles X-ray scattering $2\theta_m$=34.2…34.4° (in Figure 2 it is indicated by an arrow).

It should be noted that a slight change in the angular position of the low-intensity singlet maximum, shown by an arrow at $2\theta_m$=34.2…34.4°, is observed regardless of the preparation method for the studied samples compared to the grey one.

The most noticeable changes on the diffractograms of cotton knitwear occur in samples prepared according to the developed technology (curve 3) and by the method of alkaline boiling (curve 4). At the same time, there is a change in the ratio of intensity of the components of doublet maximum at $2\theta_m$=14.8° and 16.0°, a change in the shape and intensity of the diffuse maximum, which is in the interval 2θ from 26.6° to 31.8° on the diffractograms. In addition, a diffraction maximum of a discrete type appears at the diffraction curve 3 of the knitted fabric sample prepared according to the developed technology at $2\theta_m$=29.4°. This leads to an increase in the area of the amorphous halo and indicates an increase in the amorphous fraction of cellulose in the specified sample. This fact explains the increase in the sorption ability of the cotton knitwear sample, which was prepared according to the developed technology.

Next, the sizes of crystallites $\langle L \rangle$ in the studied samples were calculated. To do this, we first determined the angular half-width $(1/2\beta)\times 2$ of the left and right sides of the diffraction maxima at $2\theta_m$=14.8° and $2\theta_m$=22.6° with the subsequent calculation of the average value. The results are presented in Table 2.

The results of determining crystallite sizes $\langle L \rangle$ in the volume of the cotton knitted fabric samples and the values of $L_{14.8°}$, $L_{26.6°}$ indicate that large and possibly defective crystallites have grey knitwear and knitwear prepared by the basic technology. For samples of cotton knitwear, prepared by the developed technology and by the method of alkaline boiling, the presence of smaller ordered crystallites is characteristic.

Table 2 The parameters of the amorphous-crystalline structure of knitted fabric samples depending on the preparation method

Samples	Crystallite size at $2\theta_m$=14.8°, $L_{14.8°}$ [nm]	Crystallite size at $2\theta_m$=22.6°, $L_{26.6°}$ [nm]	Average crystallite size, $\langle L \rangle$ [nm]
Gray knitted fabric	4.8	6.1	5.4
Base technology	4.8	5.8	5.3
Developed technology	4.6	5.7	5.1
Alkaline boiling	4.5	5.6	5.1

The results of calculating the relative crystallinity level X_{cr} of the cotton knitted fabric samples under study are presented in Figure 3.

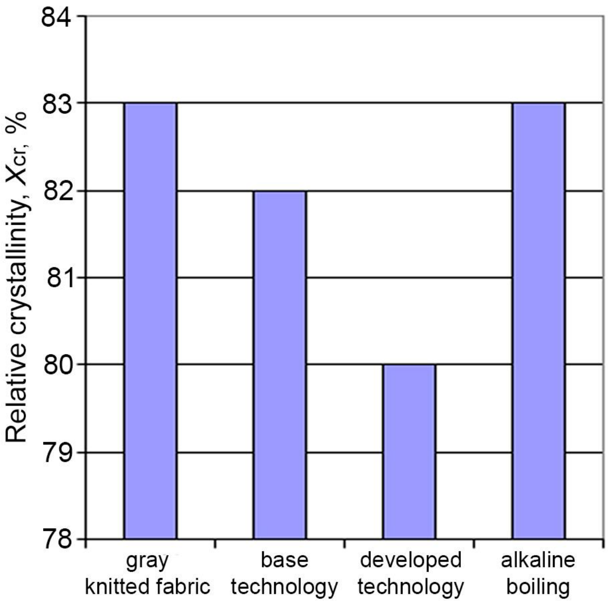

Figure 3 The relative level of crystallinity of cotton knitted fabric samples: 1) gray, 2) base technology, 3) developed technology, 4) alkaline boiling

The relative crystallinity of the structure of cotton knitted fabric samples was evaluated in the range of 2θ from 11.6° to 40.0°, in which the amorphous-crystalline structure of the samples under study are observed. It was established that the value of the relative crystallinity level X_{cr} of the studied samples is about 80% regardless of the preparation method (Table 2). The smallest crystallinity level X_{cr}=80% has a sample prepared according to the developed technology and the highest crystallinity level X_{cr}=83% has a grey knitted fabric.

Thus, X-ray studies of the amorphous-crystalline structure of cotton knitted fabric samples showed that they have a highly crystalline structure, which varies depending on the method of samples preparation.

Moreover, for curves 3 and 4 (the developed technology and alkaline boiling, respectively), the ratio of intensities of the components of doublet maximum and the angular position of low-intensity singlet maximum are observed, which is indicated by the arrow. In addition, there is a change in the shape and intensity of the diffuse maximum, which on the diffractograms is in the range of 2θ from 26.6° to 31.8° and also appears on its background a diffraction maximum of discrete type at $2\theta_m$=29.4° for sample 3, prepared by the developed technology.

5 CONCLUSIONS

With the help of SEM, it was determined that the preparation according to the methods under study does not damage the cotton fiber surface. On the diffractograms of knitted fabric samples after preparation, there is a slight increase in the intensity and integral value of scattering. The preparation of knitwear according to the developed technology causes a change in the nature of diffraction curve and an increase in the area of amorphous halo, which indicates an increase in the amorphous share of cellulose in the specified sample.

A quantitative analysis showed that the parameters of fibers diffraction of cotton knitwear that was prepared by the developed technology and by the method of alkaline boiling are characterized by the presence of smaller sized ordered crystallites. The relative level of crystallinity at the same time is the lowest among the studied preparation methods in comparison with the grey knitted fabric sample.

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