The influence of the capacitive coupled radio-frequency discharge on flax fiber

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Abstract. The article presents the results of a study of the effect of radio-frequency (RF) capacitive plasma discharge at low pressure on flax textile fiber. The study object was a standard bleached and boiled flax yarn. In the considered modes, plasma treatment does not significantly affect the cellulosic component of flax fiber. However, the results of scanning electron microscopy showed signs of etching on the samples of bleached yarn. The results of x-ray diffraction demonstrated slight increase of the proportion of the crystalline phase. This is due to selective etching amorphous sections of the fibers surface. The results of determining the lignin content showed that it is relatively resistant to the action of RF plasma discharge and plays a protective role in relation to cellulose. However, damage (etching) of the fibers surface facilitates the rapid penetration of water into the fiber; therefore, lignin passes from fiber to aqueous solutions faster. The increase of the capillarity by 2–3 times, and the increase of the colourability and the breaking load of flax yarn by 25% are the technological effects of plasma processing of flax fiber (in the considered modes).

1. Introduction

Low-temperature plasma is a universal tool which modifies the surface properties of various materials, including textiles [1]. Modification effects depend on the type of plasma-forming gas and processing parameters. The processing of materials by radio-frequency (RF) capacitive discharge plasma in dynamic vacuum is one of the best types of processing, the effectiveness of which has been theoretically and practically proved [2-4]. Natural cellulose fibers, namely flax fiber, have a complex structure and composition, therefore they are of a great interest as an object of plasma processing. Flax fiber consists of a complex of elementary fibers (10-40 each), bonded together by binders in bundles with a diameter of more than 300 microns. Flax fiber intercellular substance is an adhesive complex of pectin, hemicelluloses and lignin. Impurities are contained in the cell wall of elementary fibers; they complicate the finishing process of linen textile materials, but at the same time increase their flexibility and strength. For processing flax into textile raw materials, intensive primary processing is required, which consists in isolating fiber complexes from the bast and giving them softness and spinning ability. The authors examined the effect of capacitive coupled RF discharge plasma of low pressure on the basic properties of flax fiber and textile materials based on it in order to improve their finishing processes.
2. Research objects and methods

The objects under study were boiled and bleached flax yarn with a linear density of 46 tex [2]. Samples were processed in plasma of a RF capacitive discharge in a facility with the following parameters: \( P = 13,3 \) Pa, degree of ionization \( 10^{-7} - 10^{-5} \), concentration of charged particles \( n = 10^{15} - 10^{17} \text{ m}^{-3} \), gas temperature \( 60-90^\circ \text{C} \), ion current density on the sample surface \( j_{ip} = 0.3 - 1,0 \text{ A/m}^2 \), the energy of ions entering the sample surface \( W_i = 70 - 100 \text{ eV} \) [1]. The plasma-forming gas was air. The main characteristics of the initial and experimental (plasma-treated) samples of flax yarn (viscosity of copper-ammonia solutions of cellulose, the content of lignin (by the hydrolytic method), the content of fatty substances by extraction with carbon tetrachloride, capillarity and specific breaking load) were determined by standard and generally accepted methods [5]. X-ray diffraction (XRD) analysis of the samples was carried out using a «D2 Phaser» diffractometer (Bruker, Germany). From the obtained X-ray diffraction patterns, was calculated the degree of crystallinity (DC). The surface of flax fibers was studied by scanning electron microscopy (SEM) using the «AURIGA Cross Beam» workstation (Carl Zeiss NTS, Germany) in the secondary electron detection mode. Samples were stained in laboratory conditions using standard techniques [6].

3. Results and discussion

The results of experimental studies are shown in the table.

| Indicator                                           | Sample       | Samples                  |
|-----------------------------------------------------|--------------|--------------------------|
|                                                     | boiled yarn  | bleached yarn            |
|                                                     | control      | experimental            |
|                                                     | control      | experimental            |
| Viscosity of copper-ammonia solutions of cellulose, \( cP \) | 5,27         | 5,1                      |
| Degree of crystallinity, %                          | 67,97        | 67,36                    |
|                                                     | 68,78        | 69,51                    |
| Lignin content, %                                   | 5,94         | 2,98                     |
|                                                     | 6,86         | 3,91                     |
| Content of substances extracted with organic solvents, % | 1,31         | 0,58                     |

Plasma treatment in the considered modes does not significantly affect the cellulose part of flax fiber, since the viscosity of copper-ammonia solutions of cellulose is almost unchanged. However, this indicator tends to decrease due to changes (including etching) of the surface layer of the fiber. This fact is confirmed by the results of SEM fiber from bleached yarn (Figures 1. a, b). It demonstrates the presence of “etched” sections of the interfibrillar space with a width of about 10-20 nm (Figure 1. b). At the same time, there are no obvious signs of etching on the surface of boiled yarn fibers (Figures 1. c, d).
Comparisons of the degree of crystallinity of boiled and bleached yarn samples demonstrate the following: when boiling and bleaching, the effects on the cellulose fiber of liquid treatment and active agents increase the disorder of its structure. In addition, when bleaching, the material undergo more aggressive processing by bleaching preparations. This leads to a more noticeable decrease of the degree of crystallinity.

Lignin is an important non-cellulosic component of flax fiber, affecting its basic properties. After plasma treatment the relative content of acid lignin in flax fiber for boiled and bleached yarn increases by 15.5 and 35.3%, respectively (Table 1). It is due to a more intensive removal of other constituent components in the fiber and, consequently, the decrease in its relative content. The amount of lignin after processing bleached control yarn in hot water (at a temperature similar to dyeing) does not change, while in modified it decreases by 58.8%. After processing the control sample of boiled yarn in hot water, the lignin content decreases by 24.5%, for modified sample - by 27.1%. Thus, the degree of lignin removal during scouring process of the modified yarn is higher than of the control yarn. Damage (etching) of the fibers surface facilitates the rapid penetration of water into the fiber; therefore, lignin passes from fiber to aqueous solutions faster. The results obtained indicate the relative stability of lignin to the effects of RF plasma discharge and confirm its protective role in relation to cellulose. The amount of substances extracted with organic solvents in the flax fiber after plasma treatment is reduced by 2.2 times.
The changes occurring on the surface of flax fiber after RF plasma treatment lead to a change of the properties of textile materials - yarn and fabric. For example, the capillarity of boiled yarn increases by 3.2-3.3 times, bleached yarn – 2.1-2.6 times, in relation to the original. It should be noted that the processing effect is on the average 1.5 times higher for boiled yarn than for bleached. The effects obtained are affected by the presence of non-cellulose compounds in the fiber: the more they are contained on the surface of the fibers and the lower the initial values of wettability and capillarity, the higher the treatment effect. The results of testing the yarn for mechanical stress showed that after plasma processing, the specific breaking load of the yarn increased slightly. This fact can be explained by the increase in friction between the fibers due to the development of the microrelief.

At the next stage of the work, the influence of the RF discharge plasma to the color indicators of flax yarn after dyeing was determined. The pre-modified samples in terms of color intensity are better than the control samples by an average of 25%. The increase in color intensity is due to an increase in the amount of dye bound to the fiber. It is due to the partial removal and modification of non-cellulosic compound of flax fiber and an increase in the external surface because of the development of surface topography. It should be noted that a more pronounced effect of plasma processing is observed in terms of uniformity of color - an increase occurs by 27-40%.

The uneven coloring of flax fiber is associated with the presence in it of difficult to stain non-cellulose natural compounds and their uneven distribution over the surface. As a result of plasma exposure, the “surface” properties of the fiber are “aligned” due to changes in surface topography and the appearance of functional hydrophilic groups. As a result, conditions are created for uniform penetration and binding of the dye to the fiber.

4. Conclusions
The capacitive coupled RF discharge does not cause a significant change in the cellulosic component of flax fiber. Non-cellulosic impurities, in particular lignin, have a protective function with respect to cellulose. The increase of the capillarity by 2–3 times, and the increase of the colourability and the breaking load of flax yarn by 25% are the technological effects of plasma processing of flax fiber (in the considered modes).

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