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EXAMINING A CHANGE IN THE PROPERTIES OF COARSE WOOL FIBER UNDER THE INFLUENCE OF ELECTRICAL DISCHARGE TREATMENT

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Досліджено вплив електророзрядної обробки на зміну властивостей грубої вовни. Встановлено, що вплив електророзрядної нелінійної об'ємної кавітації на грубе вовняне волокно протягом 3 хв. призводить до покращення його поверхневих характеристик, збільшення кількості мікропор і зміни надмолекулярної структури кератину. В результаті модифікації відбувається підвищення сорбційної здатності грубої вовни при збереженні міцності волокна

Ключові слова: груба вовна, модифікація вовни, електророзрядна кавітація, морфологія поверхні, порова структура, хімічна структура кератину

Исследовано влияние электроразрядной обработки на изменение свойств грубой шерсти. Установлено, что воздействие электроразрядной нелинейной объемной кавитации на грубое шерстяное волокно в течение 3 мин. приводит к улучшению его поверхностных характеристик, увеличению количества микропор и изменению надмолекулярной структуры кератина. В результате модификации происходит повышение сорбционной способности грубой шерсти при сохранении прочности волокна

Ключевые слова: грубая шерсть, модификация, электроразрядная кавитация, морфология поверхности, поровая структура, химическая структура

1. Introduction

There has been an increase in the world consumption of wool fibre in recent years, which due to its unique range of natural properties is a valuable raw material for manufacturing a wide range of textile materials.

At present, however, woolen goods, produced in Ukraine, occupy a small part of the market. A decrease in the volume of production of woollen materials is related to a scarcity of Ukrainian raw materials, along with an increase in foreign currency cost for the purchase of wool from foreign manufacturers.

A basic condition for decreasing a dependence of the wool industry of Ukraine on import is a sustainable development of own raw material base. Today, Ukrainian wool, a significant part of which is coarse, is not capable of meeting the needs of textile enterprises in Ukraine. A decrease in wool production is associated with a decrease of total number of sheep and an increase in the share of low grade raw materials, coming from the private sector. Current conditions make the wool sector of light industry face the need to improve quality of the products under existing structure of the raw material base. This is possible to achieve through introduction of new technologies that will make it possible to produce competitive textile materials.

Changing the physical-mechanical and chemical properties of low-grade Ukrainian wool raw materials with the help of innovative technologies of finishing will lead to a decrease in peelability, enhance resistance to shrinkage and increase the optimized dyeing of wool fabrics. New methods for wool treatment will help solve the problem of import substitution and produce high-quality woolen fabrics for various purposes at reduced cost.

Along with the application of modern methods of wool treatment, economic efficiency and ecological safety of the technological process are becoming increasingly important. That is why the research, aimed at finding and implementing economically-sound and environmentally-safe ways to wool improvement, is a relevant task.

2. Literature review and problem statement

Wool has unique properties such as resilience, elasticity, low thermal conductivity, and is the raw material for manufacturing textile materials of high aesthetic qualities and comfort. Nevertheless, given the scaly surface structure, wool fiber is prone to felting and peel formation, which leads to the shrinking of woollen fabrics in the process of wet treatment and to the worsening physical appearance of finished products.

An effective way to improve quality of the manufactured woolen products may include wool modification. Modification is a directed change of the fiber surface, which allows, to some extent, improving its technological parameters, increasing consumer properties of manufactured textile materials.

At present, it is possible to distinguish three fundamental methods of wool fibre modification: chemical, biochemical and physical.

Chemical methods of modification are the oldest and mostly involve chlorination with gaseous chlorine and different aqueous solutions of chlorine-containing compounds, as well as other oxidation treatments in aqueous medium. However, contamination of wastewater by high concentrations of the employed chemical substances and the negative impact of clorine-containing compounds on the environment restricts the use of these methods.

An alternative to the use of environmentally hazardous ways of chemical modification is the enzymatic treatment. Proteolytic enzymes are most widely used as biocatalysts for giving low felting properties to woolen textile materials, as well as for enhancing softness of fabrics and increasing their absorbency. It should be noted that the diffusion of proteolytic enzymes inwards wool fibre, especially in alkaline medium, leads to the uncontrolled hydrolysis of wool keratin and, as a result, excessive weight loss of fiber, which causes its weakening. Thus, enzymatic modification of woolen textiles requires strict control over parameters of the process, such as the type and concentration of proteolytic enzymes, pH, temperature, time of treatment. In addition, it was found that white wool is more susceptible to enzymatic treatment than the black and the brown [1]. Therefore, enzymatic reaction is sensitive to the natural coloring of wool.

In order to decrease a degree of damage to wool fibres, in article [2], authors selected the concentration of proteolytic enzyme. It was found that 4 g/l concentration of alkaline protease is optimal.

For the controlled surface hydrolysis of wool, authors of papers [3, 4] proposed using bromelain, a proteolytic enzyme, active in acidic medium. It was proved that treatment with bromelain in the presence of sodium chloride at pH 6, provides a desirable antifelting degree with a decrease in weight and strength loss, compared to enzymatic treatment in alkaline medium.

Authors of [5] proposed a single-stage technology of the combined woollen fabrics bleaching and treatment with proteolytic enzymes. Mild conditions of reaction of alkaline protease with hydrogen peroxide provide the best physical-chemical properties of woollen textiles, which makes this method promising.

Thus, enzymatic methods for the modification of wool materials in general increase economical efficiency and ecological safety of the processes. The shortcomings of application of biological preparations include a disproportionate effect of enzymes on the fiber, elevated thermal sensitivity, high cost and non-versatility.

It is also possible to improve the properties of wool fabrics by application of natural polymers. In this case, the aquired low felting property and improvement of optimized dyeing of wool is achieved due to formation of a polymer film on the surface of the fiber. Paper [6] studied the use of chitosan, and it was established that application of chitosan in the form of nanoparticles is more effective than the traditional treatment with chitosan Authors in [7] proposed the use of protein, derived from chicken feathers by the method of enzymatic hydrolysis. The results showed that application of obtained protein on woolen fiber in the presence of joining agent (diglycydyl glycerin ether) leads to lasting consolidation of a protein layer on the surface of wool. However, these methods have not been widely used in textile industry.

One of the promising directions of modification of woolen textiles is the application of physical methods. Such methods include plasmochemical treatment, during which a scaly layer is partially destroyed to the depth of 30–50 nm, affecting *F*-layer of fatty acids and *A*-layer of the cuticle [8]. As a result of modification, wettability of woolen fiber increases, frictional properties change and adhesive characteristics and optimized dyeing improves [9, 10].

Combined treatment by low-temperature plasma and enzymes was developed [11]. The advantage of this technology lies in the joint application of protease and transglutaminase, which enhances functional-technological characteristics of keratin due to its ability to catalyze intra- and intermolecular cross-joining of protein molecules.

On the whole, advantages of plasmochemical technology can be characterized by the improvement of performance of processes and a decrease in the number of technological stages. Disadvantages of plasmochemical processes include high power consumption and instability of surface properties, acquired by fibre over time.

An analysis of literature allows us to make a conclusion that the existing methods of modification are material- and power-consuming and do not provide the required quality of fiber. In addition, explored technologies are focused mainly on the modification of thin and semi-fine wool. Therefore, at the present stage of development of wool textile industry, the search for and development of new effective ways of modifying coarse wool fibers are of serious practical interest.

In recent years, many industries are increasingly applying new electrophysical methods for treatment of materials, characterized by a high concentration of energy, high pressure and temperature. Such processes include high-voltage electrical pulse discharge in a liquid, accompanied by the emergence of electric discharge non-linear bulk cavitation (EDNBC). Papers [12–15] proved a fundamental possibility of its use in various technological processes. In addition, EDNBC has a number of advantages to improve the rate of technological process and equipment performance, reduce its power and material consumption compared with other types of cavitation.

In the course of the study [12–14], the process of electric discharge treatment of wool was examined and its basic technological parameters were determined. According to results of the experiments, it was found that due to characteristics of histological structure of wool fibre, EDNBC affects the wool of different fineness in various degrees.

In this regard, it is of interest to study effectiveness of application of EDNBC as a method of surface modification of coarse wool fiber in order to improve quality of the finished textile products and decrease their cost.

3. The goal and objectives of the study

The goal of present work was to study an impact of EDNBC on the formation of a new complex of properties of coarse wool fibers in the process of modification.

To accomplish the goal of the research, the following tasks have been set:

– to study the impact of EDNBC on the surface of coarse wool fiber;

– to explore structural changes of coarse wool fiber under the influence of EDNBC;

– to determine the impact of EDNBC on the change in chemical structure of coarse wool.

4. Materials and methods to study the influence of electric discharge non-linear bulk cavitation on the properties of coarse wool fiber

4. 1. Studied materials and equipment used in the experiment

The studies were performed with the use of washed coarse wool of the Tushinsky breed of sheep in the form of a tape. This wool is characterised by predominance in the composition of transitional hair and downy fibers, which are close to it by fineness at limited content of coarse hair. It is white, quality 44, length I.

Modification of the wool fiber was conducted on the laboratory set-up «Vega-6» [13]. Electric discharge treatment of wool on the set-up was carried out for 3 min. in tap water (bath module 1:150) at constant values of voltage and pulse frequency.

4. 2. Methods for studying wool fiber

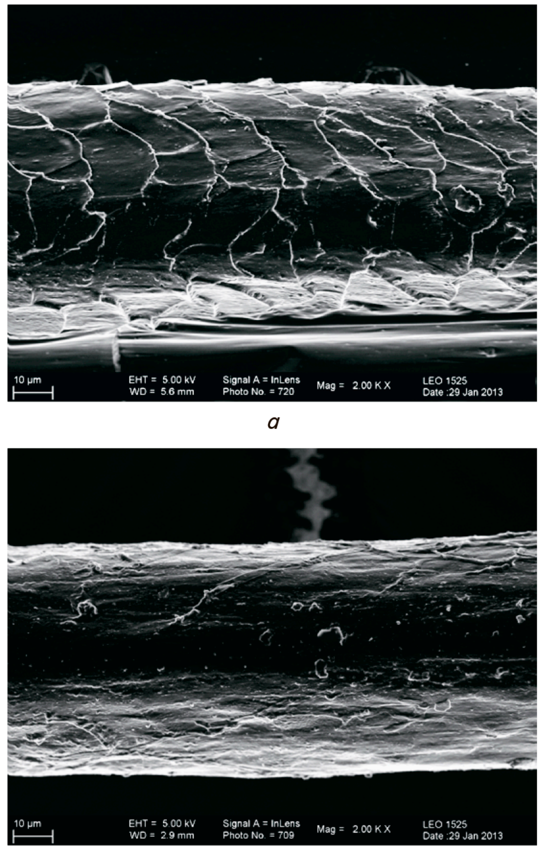
Morphological features of wool fibers were studied on the scanning electron microscope LEO 1525 Gemini SEM (LEO Electron Microscopy Inc., USA). To provide the flow of electrons from the surface, a thin film of gold with palladium was sprayed on the samples [16].

A change in the porous structure of wool after electric discharge treatment was studied, and the interlocation of macromolecules of wool fibre and their associates was determined using an X-ray structural analysis. Wide-angle diffractograms were obtained at the diffractometer DRON-2.0 in the radiation of copper anode with nickel filter in the primary beam by the Bragg-Brentano method. The explored finely dispersed powders were placed in 0.5-mm-thick gutters with windows from 17-μm thick Darcon. Optical circuit of the diffractometer was modified to make translucent filming. Details of conducting the experiment and parameters of the X-ray optical circuit are shown in [17]. To obtain diffractograms, we used copper $K\alpha$ radiation with an average wavelength of $\lambda=1.54E$. K_β radiation was filtered using a nickel filter. Accelerating voltage was 30 kW, tube current was 10 mA. Diffraction angles 2Θ were measured in the range of 4°–70°. Filming was carried out under continuous mode with a step of 0.0143° and at exposure time of 2 s [18].

The study of chemical structure of wool was conducted using the method of infrared (IR) spectroscopy. IR-spectra of the samples were filmed in the spectrophotometer with a Fourier-transform Tensor-37 (Bruker, USA) in the range of wavenumbers 400–4000 cm^{-1} under the transculent mode. The samples were preliminarily crushed and pressed with a KBr powder for the purpose of obtaining the substance in tableted form [19, 20].

5. Results of study of the impact of electric discharge non-linear bulk cavitation on the properties of coarse wool fibre

Results of the study into morphology of the surface of untreated and modified coarse wool fiber are shown in Fig. 1.



a

b

Fig. 1. Microphotographs of the surface of coarse wool fiber: *a* — untreated fiber; *b* — EDNBC-modified fiber

Fig. 1, *b* indicates that upon the electric discharge treatment, wool fibre has a smoother surface compared to the untreated sample, shown in Fig. 1, *a*. Smoothing and compaction of wool surface occurs under the influence of hydraulic shocks, induced by the pulse-occurring high pressure as a result of collapse of cavitation cavities.

It should be noted that in the process of electric discharge treatment, the effect of modification is predetermined by a imultaneous influence of electro-hydraulic shock and chemical processes in operation medium on the treated material. These chemical processes are the redox reactions, proceeding in water under the action of cavitation with the formation of water cleavage products.

Wool, having a complex chemical and physical structure, is very sensitive to the influence of various oxidizing and reducing agents that actively affect, first of all, disulfide bonds of keratin. In this connection, it was interesting to examine a comprehensive impact from the acting factors of EDNBC on a change in the supramolecular and porous structure of wool fibre.

Results of the X-ray structural analysis of the samples of untreated coarse wool fibre and those modified by the method of wide-angle scattering are shown in Fig. 2.

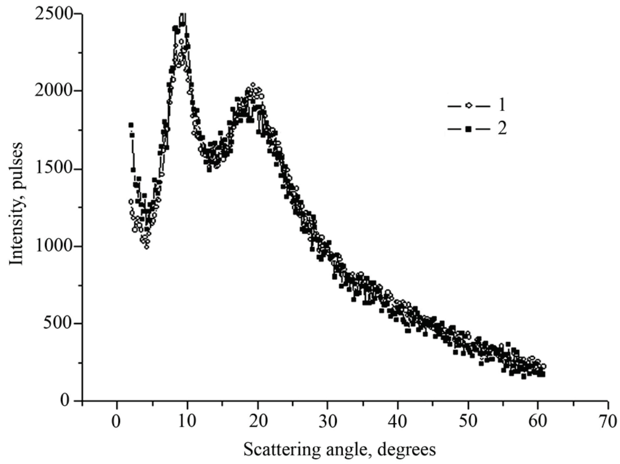


Fig. 2. Wide-angle scattering diffractogram of the coarse wool samples: 1 — untreated fiber; 2 — EDNBC-modified fiber

On the curves, shown in Fig. 2, we can observe two blurry maxima at scattering angles of 8.8Θ and 18.6Θ. This shape of a scattering curve indicates the amorphous character of close oderliness of the studied samples, that is, the absence of traces of a crystalline phase. Position of the first of the observed maxima is consistent with the Bragg periodicity of about 1 nm, and position of the second – 0.46 nm.

In the first approximation, the features of close orderliness of wool fibre can be compared with those in the linear polymer of polystyrene, whose diffraction curve also exhibits two blurry diffraction maxima– at scattering angles of 10Θ and 19.2Θ.

The first maximum on the polystyrene diffractogram [21] determines lateral periodicity in the distribution of molecular chains of polystyrene, that is, it corresponds to the averaged distance between their centers. The second maximum corresponds to the mean distances between atoms and atomic groups of amorphous material [22]. In this case, the first maximum of the scattering curve of wool corresponds to the mean distances between the axes of molecular chains, twisted in rod-like structures, and the second – to the mean close orderliness of the material.

Fig. 2 shows that the diffractogram of the treated material almost coincides with the diffractogram of the source material. Only minor differences in the intensities of diffraction maxima are observed. The most noticeable are the differences between the two presented curves in the initial region of scattering – on the curve of scattering by the modified material in the region of scattering angles from the minimal 2Θ to 5Θ, a noticeable increase in intensity is observed. This scattering region is known [22] to belong to the so-called small-angle scattering. The fundamental difference between this scattering and the wide-angle scattering is that the cause of its manifestation is the existence of nano-dimensional (1–200 nm) regions with a density that is different from the matrix material. These may include both the regions of elevated density (such as nanofillers) and nanoscale cavities, which, presumably, is observed in the given case.

Changing the porous properties of the modified wool fiber towards an increase in the micropores can indicate orderliness of the supramolecular keratin structure. To prove this hypothesis, we conducted a study of the chemical structure of coarse wool after the electric discharge treatment. Results of IR-spectroscopy of the samples of untreated and modified wool fibre are shown in Fig. 3.

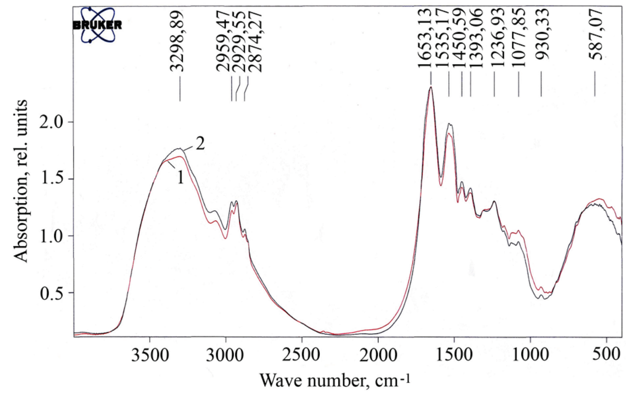


Fig. 3. IK-spectra of coarse wool fiber: 1 — untreated fiber; 2 — EDNBC-modified fiber

The obtained IR-spectra (Fig. 3) represent ф characteristic spectrum of protein, the main bands of which are:

– absorption bands of valence oscillations of NH-groups in the region of 3000–3600 cm^{-1};

– absorption of valence oscillations of the (C=O)-bond with a maximum of 1653 cm^{-1} (amide I);

– deformation ocsillations of NH-groups with a maximum of 1535 cm^{-1} (amide II).

Comparing IR-spectra of the samples of original and modified coarse wool in Fig. 3, we can see that the profiles of the main absorption bands are similar while small differences are mainly observed in the area of valence and deformation oscillations of NH-groups.

A broad band of valence oscillations of NH-groups of the original raw wool has two peaks with maxima of 3406 cm^{-1} and 3298 cm^{-1}. A peak of 3406 cm^{-1} is responsible for loosely-hydrogen-bound NH-groups, and the peak of 3298 cm^{-1} is responsible for the bound NH-groups. In the wool sample, subjected to the effects of EDNBC, intensity of the band with a maximum of 3298 cm^{-1} is higher, which proves the existence of a larger number of bound NH-groups compared with the sample of untreated wool.

Similar changes are observed in the region of deformation oscillations of NH-groups, thus, a complicated band of amide II has 3 peaks: 1555 cm^{-1}, 1535 cm^{-1} and 1516 cm^{-1}. The profile of this band, that is, a position of the intensity of peaks differs between the IR-spectra of untreated and modified wool, which is connected with the changes in the microstructure of the given samples. Absorption bands of amide I do not change, and the ratio of free and bound (C=O)-bonds remains constant. This pecularity is predetermined by the fact that the bond (C=O) is more stable and is less influenced by surrounding groups.

Based on the conducted comparative analysis of IR-spectra of coarse wool fibers, we can draw a conclusion on that the main differences are related to intermolecular structural changes of hydrogen-bound NH-groups. In the preliminarily modified samples, the number of bound NH-groups increases, which proves reallocation of transverse bonds in wool keratin, induced by operating factors of EDNBC.

6. Discussion of results of examining the influence of electric discharge non-linear bulk cavitation on the properties of coarse wool fibre

An analysis of the experimental data obtained makes it possible to argue that a change in the properties of coarse woolen fibers occurs as a consequence of modification not only at the surface level, but also at the internal level. This is also demonstrated by the accumulated experience of work [12, 13, 15] on the application of EDNBC for intensification of various technological processes of wool finishing.

The state of fiber surface is the most important factor in determining strength characteristics of wool, which is predetermined by high density of disulfide bonds in the cuticle. Earlier, EDNBC was found [23] to affect directly the hydrophobic lipid layer of wool fibre epicuticle, without influencing deeper layers of the substrate. Thus, safety of the main part of the cuticle of coarse fibre after electrical discharge treatment will provide high strength characteristics of wool, including subsequent bleaching process. In addition, wool, modified by EDNBC treatment, is characterised by low felting ability [23], which will make it possible to improve efficiency in the subsequent processes of its finishing.

The established fact of increasing the volume of micropores of coarse wool fibers after electrical discharge treatment is consistent with data, obtained in the study of capillary-porous properties of wool by a thermogravicalorimetric method [24]. Presented results indicate high sorption capacity of the modified fiber. Altered capillary-porous properties, aquired by wool fiber under the influence of EDNBC, make it possible to improve its absorption sensitivity and reactivity relative to active, acidic and basic dyes. This fact is proved by results of the study into dyeing kinetics [24].

Analysis of the obtained IR-spectra data revealed that in the samples of wool fibre after electric discharge treatment we can observe an increased number of hydrogen bonds, which indicates a change in the supramolecular structure of modified wool. It can be assumed that under the influence of atomic hydrogen, forming in water during electric discharge treatment of wool fibers, interpeptide disulfide bonds are restored and cystine is transformed into cysteine. In this case, polypeptide chains with restored cystine bonds can move freely relative to each other under the influence of electrohydrolic shock. Influenced by oxidants, present in the working medium, sulfhydryl groups of cystine can easily oxidize again, forming new cystine bonds. In addition, under conditions of cavitational treatment, reactive SH-groups can chemically interact with the formation of other, stronger transverse lanthionine bonds $-CH_2-S-CH_2-$.

Thus, we can conclude that in the process of electric discharge treatment under comprehensive influence of EDNBC the disulfide bonds of wool keratin are modified, which leads to a change in its natural properties. Thus, formation of new transverse bonds in keratine results in an increase in strength and resistance to the hydrolysis and effect of oxidants and reductants, as well as to a decrease in the tendency to fiber felting [25].

This circumstance allows us to assume that the application of EDNBC as a method of preliminary modification of coarse wool fiber will contribute to the improvement of its mechanical and technological properties. Therefore, the proposed method of treatment will make it possible to apply coarse wool for the production of high quality textile materials.

It should be noted that under modern conditions it is economically feasible to develop combined methods of wool fiber modification and basic technological processes of its finishing. In this regard, further research will focus on the development of technology for bleaching the EDNBC-modified coarse wool.

7. Conclusions

1. It was established that in the process of modification, under the influence of the shock wave of electrical discharge, there occurs the smoothing and compaction of the scaly layer of coarse wool with preserving the bulk of the fiber cuticle.

2. Structural changes in the coarse wool fiber exposed to EDNBC were studied using the method of an X-ray structural analysis. Electric discharge treatment was proved to contribute to the formation of nano-dimensional voids in the fiber structure, which differ by density from the original wool.

3. The EDNBC impact on chemical structure of coarse wool fibers was explored employing a method of IR-spectroscopy. It was established that the main differences between IR-spectra of the studied wool samples are associated with intermolecular structural changes of hydrogen-bound NH-groups. In the modified samples of wool fibre, the number of free and bound NH-groups changes in favor of the bound groups, which testifies to the orderliness of the structure of protein molecules.

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