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DEVELOPMENT OF A TWO-STEP TECHNOLOGY OF SCOURING WOOL BY THE METHOD OF HIGH-ENERGY DISCRETE TREATMENT

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Розроблено двостадійну технологію очищення вовни від природних забруднень на основі застосування високоенергетичної дискретної обробки. Визначено оптимальні параметри запропонованої технології. Встановлено, що попередня високоенергетична дискретна обробка вовни протягом 3 хв на стадії промивки дозволяє досягти необхідного ступеня очищення вовни, а також поліпшити фізико-хімічні характеристики та ступінь вилучення вовняного жиру

Ключові слова: цигайська вовна, Сульсід, залишковий вміст жиру, кислотне число

Разработана двухстадийная технология очистки шерсти от природных загрязнений на основе применения высокоэнергетической дискретной обработки. Определены оптимальные параметры предложенной технологии. Установлено, что предварительная высокоэнергетическая дискретная обработка шерсти в течение 3 мин на стадии промывки позволяет достичь необходимой степени очистки шерсти, а также улучшить физико-химические характеристики и степень извлечения шерстного жира

Ключевые слова: цигайская шерсть, Сульсид, остаточное жиросодержание, кислотное число

1. Introduction

The quality and outward appearance of woolen cloths and products, as well as their wear resistance in the process of operation in many respects depend on the pretreatment of wool. In Ukraine and in foreign countries, every year more attention is paid to the studies of improvement in the technological process of pretreatment of wool. The incorrect or imperfect technology of pretreatment of wool leads to enormous losses of woolen fiber both at the preprocessing plants and at wool processing enterprises. Special attention should be paid to the problem of extraction of wool grease from the used washing waters – at present, no wool preprocessing plant in Ukraine extracts wool grease from the used wool washing water.

Along with the application of contemporary methods of treatment, the cost-efficiency of technological process is of great significance. Therefore, the studies directed toward the search and implementation of the most economically advantageous technological modes of the wool pretreatment are relevant.

2. Literature review and problem statement

Traditional technology of the wool pretreatment consists of the following main operations [1, 2]:

– scutching unwashed wool with the purpose of separating it from plant (straw, leaves, parts of branches and stems of plants) and mineral (sand, clay) impurities as well as loosening wool for the best penetration of the washing solution;

– washing and quality control of the washed wool. The stage of washing of contaminated wool is performed at the equipment of continuous action with washing solutions of surface active substances (SAS), and what it is more frequent, with their compositions at the temperature of 40–60 °C. The washing action of SAS is predetermined by their comprehensive properties, such as moistening, dispersion and emulsification;

– drying and storage of the washed wool.

The main purpose of wool pretreatment is not only to scour the surface of the wool fiber, but also to maximally preserve its physical and mechanical (winding, hygroscopicity, humidity, strength, luster, thermal conductivity) and technological (spinning ability, suitability for felting) properties. The most common technology of the wool pretreatment, which is used at many factories of Ukraine, implies application of diverse washing SAS or their compositions [3]. The main advantage of this technology of treatment is a relative simplicity of realization. However, the method has the following disadvantages:

– significant power consumption;

– use of a large amount of textile auxiliary substances and volumes of water, which makes the process of wool processing considerably more expensive, and, therefore, increases its prime cost;

– formation of a large amount of wastewater, which negatively affects the state of the environment and water reservoirs, thus worsening ecological situation;

– difficulty of wastewater purification, since the majority of the utilized SAS are non-biodegradable;

– in many cases the state of fiber deteriorates – its hardness increases while the strength and elasticity of fiber decreases.

The method of scouring the wool fiber with the aid of organic solvents does not have such side effects as the method mentioned above [4]. At the same time, there are the problems of toxicity and explosiveness of technology, high financial cost of purchasing the appropriate equipment and the need for constant presence of highly qualified personnel. It is not possible to scour heavily soiled wool using this method; furthermore, the treatment by organic solvents frequently leads to significant yellowing of wool and a loss of strength of fiber. The consequence of the indicated disadvantages is an increase in the brittleness of separate fibers and formation of woolen dust in the production premises of factories.

The technology of intensification of the process of wool washing by the treatment of the material in the medium of inverse emulsions is also known [5, 6]. However, this technology has not become widely used.

The authors [7, 8] proposed the technology of scouring wool with hot water by adding SAS and subsequent centrifugation of washing waters for extraction of wool grease.

There are also the methods of fermentative wool fiber washing [9, 10]. However, the action of fermentative preparations on the wool fiber is insufficiently studied; therefore, this method of washing has not become widely spread in manufacturing.

The scientists [11] developed a technology of ultrasonic scouring of wool, which makes it possible to conduct treatment under the following technological parameters: concentration of SAS – 1 g/l, the temperature of treatment – 75 °C, duration of treatment – 25 s. As a result of washing, the necessary level of removal of the natural contamination from wool is achieved.

The researchers [12] developed technology of scouring wool by means of consecutive treatment by the ultrasound and the ultra-high frequency heating. It is also known that such treatment of wool raw material makes it possible to increase the strength of fibers due to formation of transverse disulfide and hydrogenous bonds, as well as new longitudinal peptide bonds. The drawback of the proposed technology is the complexity of operating the equipment.

Analyzing the aforesaid, it is possible to draw a conclusion that the development and introduction of new technology, which will ensure good quality of the produced washed wool and will not have side negative effects, is expedient and timely.

At the present stage of scientific development, the physical methods of intensification of processes of the decoration production of textile materials find their wide application, in particular the high-energy discrete processing (HDP), the essence of which is the phenomenon of electric discharge cavitation.

The electric discharge cavitation is the most powerful form of cavitation, which does not have any analogs in the increase in the power of influence on specific objects. At the generation of this process, the set of phenomena occurs – powerful pulse electrical and magnetic fields, acoustic emissions of low-frequency range, intensive hydro-flows, shock waves and chemical transformations of water. A certain level of influence on the process of removal of grease, mineral and organic impurities from wool is reached with the help of the electric discharge cavitation. Furthermore, the application of the electric discharge cavitation in the technology of washing wool has a number of important advantages regarding the energy and resources saving.

3. The aim and the tasks of the study

The purpose of the work was to study the influence of HDP on the process of washing wool from natural contaminants and further extraction of wool grease from the obtained washing waters.

To achieve the aim of the research, the following tasks were set:

– to study the influence of different types of cleaning agents on the quality indices of the washed wool;

– to explore the possibility of applying HDP for washing wool;

– to examine qualitative and quantitative characteristics of the water, obtained after washing wool fibers;

– to determine the influence of HDP of wool on the physical and chemical properties of wool grease with the acidic method of grease extraction.

4. Materials and methods of studying HDP, the degree of scouring wool, washing waters, the amount of wool grease and its physical and chemical properties

4. 1. The tested materials and the equipment used in the experiment

The studies were carried out with the use of the semi-thin Tsigeian wool of white color (Fig. 1).

HDP of wool fiber was conducted at the laboratory unit "Vega-6" [13], designed in cooperation with the scientists of the Institute of pulse processes and technologies of NAS of Ukraine, the city of Nikolayev. HDP of wool at the unit was carried out at the constant magnitudes of voltage and pulse frequency.

The following substances were used as cleaning agents in the work:

– the soap-soda solution (5 g/l of laundry soap (GOST 30266-95) and 3 g/l of calcinated soda (GOST 5100-85));

– Sulfoxide-61 – the composition of anionic SAS and non-ionogenic SAS with addition of foam extinguisher (TU 2484-143-05744685-95);

– Savenol NWP – the degreasing composition based on the mixture of hydrated SAS (TU 19401552-005-01);

– Sulside – the composition, which includes sulfanole, ricinox-80, Synthanol DS-10 and dimethylsulfoxide [14].

Fig. 1. Unwashed wool of Tsigeian sheep

4. 2. Methods of studying wool fiber

Determination of the moisture content of wool was carried out according to GOST 18080-95.

A residual quantity of greasy substances was determined by extracting the washed samples of wool by diethyl ether on the Soxhlet apparatus using the procedure, represented by GOST 21008-93.

The degree of destruction of keratin of wool was assessed according to the loss of wool mass following the action of the solution of 4n HCI by the procedure, given in [15].

The mass fraction of residual non-wool components (mineral and plant additions) were determined according to GOST 29239-91.

4. 3. Method of mathematical planning of the experiment

The main technological parameters of HDP and the washing in the SAS solution were determined by optimization of the process of scouring with the use of the method of mathematical planning – a full factor experiment of type 2^3, consisting of 8 experiments [16].

4. 4. Methods of studying the wool-washing water

The content of suspended substances was determined according to the procedure, indicated in [17].

The size of the particles of contaminants was determined by measuring the full-time deposition [18].

The content of grease in the washing waters was determined through extraction by petroleum ether [19].

The general hardness of water was determined by the complexometric titration of cations of hardness by the solution of trilon B [20].

The transparency of washing water was determined by the Snellen font [10].

The chemical consumption of oxygen (CCO) was determined by the dichromate arbitration method [21].

The biological consumption of oxygen (BCO) was determined by the method of dilution [21].

4. 5. Methods of extracting and studying the physical and chemical properties of wool grease

The wool grease, which is contained in the obtained washing waters, was extracted by the acidic method of grease extraction [2], for which wool-washing waters were acidified by sulfuric acid to pH=3–3,5 for decomposition of washing emulsion. The acidified liquid was heated up to 60–70 °C, in this case, wool grease appeared on the surface in the form of brown mass. The layer, located under grease, was disposed of and the greasy mass was processed in an autoclave, where contaminants and particles of water were separated from grease.

Physical and chemical characteristics of the extracted wool grease were determined according to the standard methods of analysis of oils, fats and their derivatives (IUPAC Commission on Oils, Fats and Derivatives) [22].

5. Results of studying the influence of cleaning agents on the quality indicators of the washed wool

The results of the study of influence of different types of cleaning agents on the quality indicators of the washed wool (humidity, residual grease content, a quantity of removed grease, loss of mass) are represented in Table 1.

Analyzing the obtained results, it should be noted that not all washing preparations allow obtaining the necessary degree of wool scouring. The greatest scouring ability is possessed by soap and soda solution and the Sulside preparation, using which eliminates from 91,0 % to 94,6 % of all grease and wax contaminants of wool. In this case, the humidity index of fibers reaches the values in accordance with GOST 18080-95 (12–17 %).

Analysis of the data that characterize the loss of mass of wool after the influence of solutions HCI (Table 1) proves direct dependency of the degree of damage of woolen fibers from the character of the used cleaning agent.

A number of authors noted [23] that anionic and cationic SAS most strongly interact with polypeptides and denature them. The denaturing effect of nonionogenic SAS is insignificant; however, they possess sufficiently high power to penetrate into protein structure and, therefore, in the mixture with cationic SAS in certain ratios they can strengthen unfavorable effect of the latter.

In this case it should be noted that the largest influence on the surface structure under washing conditions is produced by the soap and soda solution, obviously as a result of combination of the following factors unfavorable for wool: alkaline medium and the influence of temperature and anionic SAS. Furthermore, Sulfoxide-61 had a more destructing effect on the cuticle of wool fiber than Savenol NWP, which is explained by the content of anionic SAS in its composition.

The preparation Sulside is characterized by the lowest influence on the cuticle of fibers. This fact can be explained by the presence of dimethylsulfoxide in its composition, which has a softening effect on the polymeric materials and allows scouring wool under the milder conditions.

Such action of dimethylsulfoxide determines its application in the role of auxiliary component for the production of cosmetic-hygienic cleaning agents [23].

Organoleptic estimation of general state of the washed wool shows that the samples of wool fibers, washed in the soap and soda solution, are most entangled and display some tarnish, which is explained by deposition of calcium sediments on their surface. The other samples of wool are characterized by elasticity, softness and are less felted.

Table 1

Influence of cleaning agent on quality indicators of washed wool

Type of cleaning agent	pH of washing solutions	Qualitative indices of washed wool			
		Humidity W, %	Residual grease content X, %	Quantity of removed grease, %	Loss of mass L_a, %
Unwashed wool	–	9,45	20,7	–	8,68
Soap+soda	9–10	12,10	1,8	91,0	12,85
Sulfoxide-61	7,0–8,0	11,79	2,63	87,3	12,15
Savenol NWP	8,0	10,72	2,8	86,5	10,40
Sulside	7,0	12,58	1,12	94,6	9,19
By GOST	–	12,0–19,0	0,5–2,0	–	–

The washing of wool based on Sulside preparation made it possible to obtain the fluffiest and the least felted samples of wool fibers.

However, the use of the washing substances only does not make it possible to obtain the washed wool of high quality, because wool becomes felted in the process of repeated washing.

The solution to this problem is the implementation of washing in the solution of the washing composition in combination with HDP.

6. Results of studying a possibility of applying HDP during wool washing

The application of HDP for washing wool is possible in two directions:

1. One-stage washing of wool by introduction of a cleaning agent to the electric discharge reactor. The scheme of scouring wool is represented in Fig. 2.

In this case, the following mechanism of scouring is possible: intensive mechanical influence during HDP contributes to rapid destruction and dispersion of the particles of the greasy contaminants, which are separated from the surface of fiber and pass into the solution in the form of the smallest droplets, concluded in the adsorptive films from the molecules of the washing agent.

However, this scheme of cleaning will require frequent change of the washing solution for prevention of the resorption of contaminants as a result of rapid saturation of the solution with contaminated substances. The repeated deposition of contaminants can also be caused by the destruction of solvate membranes from molecules of the washing substance as a result of intensive mechanical influence of HDP. The use of two-stage technology of washing is more rational.

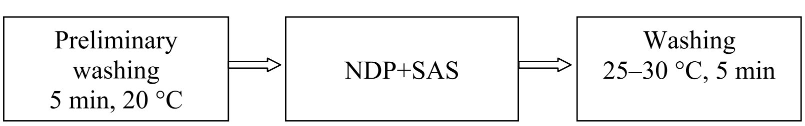

Fig. 2. Scheme of one-stage washing of wool

2. Two-stage technology of washing wool.

The scheme of scouring wool is represented in Fig. 3.

This scheme of scouring wool implies the following sequence of technological stages: preliminarily scoured wool enters the electric discharge reactor, where the removal of mineral and organic contaminants and destruction of the film of greasy contaminants on the surface of fiber take place. Then the wool enters the SAS solution for washing, where the final removal of mineral, organic and greasy impurities occurs.

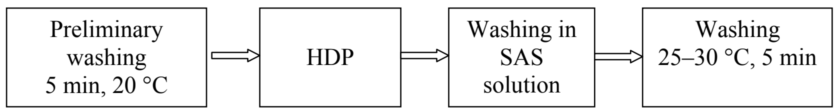

Fig. 3. Scheme of two-stage washing of wool

For determining the main technological parameters of HDP and washing in the SAS solution, optimization of the process was conducted.

The aim of optimization of the process of scouring wool was determining such technological parameters, which would ensure obtaining the necessary optimization criterion (Y), in this case – the final content of greasy contaminants.

It was assumed that the efficiency of scouring wool based on HDP depends on three basic parameters: concentration of SAS (C, g/l),temperature of processing in the SAS solution (T, °C), duration of HDP (τ, min).

The factors, levels of factors variability and the intervals of variation are shown in Table 2.

Table 2

Factors and levels of factors variability

Factors	Levels of variation			Interval of variation ε
	–1	0	+1	
Concentration of SAS C, g/l	0,5	1,5	2,5	1
Temperature of washing water T, °C	40	45	50	5
Duration of HDP τ, min	1	3	5	2

Processing the results of the experiment enabled us to calculate coefficients of the equation of regression, which characterize the dependency of the final content of grease on the concentration of SAS, the temperature of processing in the SAS solution and the duration of HDP.

After the analysis of the obtained equation of regression, the hypothesis of adequacy of the results of the study and of significance of the regression coefficients was verified. The reliable interval for regression coefficients was determined with the help of the Student criterion. After the exclusion of insignificant coefficients of the equation of regression and conversion of normalized values of the input variables to the natural, the expression takes the form:

$$\hat{Y} = 3{,}3 - 0{,}38 \cdot C - 0{,}36 \cdot \tau. \quad (1)$$

According to the obtained equation of regression (1), in the explored factor space, the dependency of the residual grease content of wool (Y) on the concentration of the washing agent (C) and the time of treatment by the electric discharge (τ) takes the form of a three-dimensional plane (surface of response), which is represented in Fig. 4.

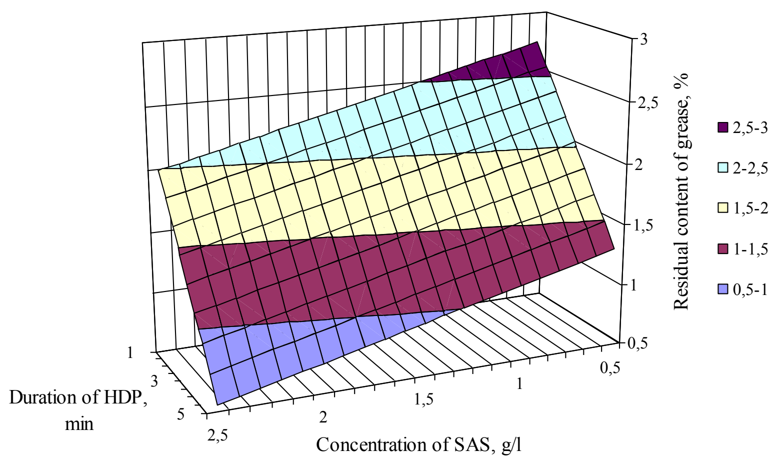

Fig. 4. Dependency of residual content of grease on the concentration of washing preparation and duration of HDP

Analysis of the equation of regression allows us to draw a conclusion that the criterion of optimization (residual grease content) in the selected factor space is influenced by two factors – the concentration of SAS and the duration of HDP. The obtained negative values of coefficients of the equation of regression attest to the fact that an increase in the value of any of the factors will lead to a decrease in the value of residual amount of wool grease.

The obtained mathematical model of the process of scouring wool attests to the fact that the desired value of the criterion of optimization – the content of grease and wax substances – is within the limits of the tested factor space. In this case, the optimum content of grease and wax substances (1,63 %) can be achieved with the values of factors corresponding to the zero level.

The obtained washed wool, scoured with the use of the developed technology, is characterized by the following indices of quality of washing:

– residual grease content – 1,63 %;
– humidity – 15,6 %;
– content of plant impurities – 0,8 %;
– content of mineral impurities – 1,54 %.

6. Results of the analysis of qualitative and quantitative characteristics of the water obtained after washing the wool fiber

Table 3 presents characteristics of the indices of obtained washing waters, from which wool grease was subsequently extracted.

Table 3

Indices of quality of obtained washing waters

Index	Value
Content of suspended substances, g/l	16,6
Size of contaminants particles, mkm	100
Content of wool grease, g/l	18,6
General hardness, mg-equ/l	11,6
Transparency, sm	2
Turbidity, mg/l	132
CCO, g/l	4,15
BCO, g O_2/l	12,5
pH of the medium	7,65

According to the data represented in Table 3, the obtained washing waters are a coarsely dispersed system, the content of wool grease is 18,6 %.

7. Results of studying the influence of HDP of wool on the physical and chemical properties of wool grease with the acidic method of grease extraction

The physical and chemical characteristics of the obtained wool grease are represented in Table 4.

According to the data, represented in Table 4, the grease extracted by the acidic method is dark brown, has high acidity, which is confirmed by the pH index and by the acid number, as well as by the increased saponification number. The obtained results agree with the data given in [2].

Table 4

Influence of HDP on physical and chemical characteristics of wool grease

Index	Wool	
	unprocessed wool	after HDP
Color	dark-brown	brown
Acid number, mg KOH per 1 g of grease	50,40	39,20
Saponification number, mg KOH per 1 g of grease	134,64	129,03
Ether number, mg KOH per 1 g of grease	95,44	78,63
Amount of glycerin, %	5,22	4,30
Iodine number, g I_2 per 1 g of grease	10,15	6,35
pH of water extract	3,50	3,00

When comparing physical and chemical properties of the grease, extracted from unprocessed and processed wool, it becomes obvious that HDP leads to an improvement in the qualitative characteristics of wool grease. Thus, with the use of HDP, the acid number decreases (from 50,40 to 39,20 mg KOH per 1 g of grease), the ether number decreases (from 95,44 to 78,63 mg KOH per 1 g of grease) and so does the iodine number (from 10,15 to 6,35 g of I_2 per 1 g of grease). Saponification number decreases by 4 % (from 134,64 to 129,03 mg KOH), which numerically is correlated with the indicator of amount of glycerin in grease.

It should be noted that the application of HDP of wool with the acidic method of extracting grease leads to an increase in the output of wool grease up to 96 % in comparison with the quantity of grease obtained from the unprocessed fiber.

8. Discussion of the results of studying the influence of HDP on scouring wool from natural contaminants and consequent extraction of wool grease by the acidic method

Taking into account our own accumulated experience and works by other scientists [24–33] on the application of HDP for intensification of different technological processes and the obtained data, we believe that HDP has a comprehensive influence during processing wool fiber in the course of washing and further extraction of wool grease.

Firstly, the influence of HDP manifests itself by destruction of the solid greasy film on the surface of fiber, which leads to the increase in the rate of extracting grease and the removal of natural contaminants.

Secondly, chemical transformations occur in water during HDP, as a result of which free radicals (4,2 mmole/liter under the influence of HDP for 2 minutes) and peroxide of hydrogen (0,1 mmole/liter under the influence of HDP for 2 minutes) are formed, which, in turn, influence both wool fiber and wool grease [33].

The obtained data attest to the fact that the influence of HDP on water and wool, put into it, is not possible to explain by cavitation mixing of water only.

Independent of the nature of dissolved substances, the electrical discharge affects one substance – water, which leads to a change in its physical and chemical properties: restructuring and activating water molecules, formation of free radicals and the products of their recombination and, as a result, to an increase in pH (from 7,12 to 7,49 under the influence of HDP for 2 minutes) and in electrical conductivity of water or aqueous solution (from 1612 mkCm/cm to 1922 mkCm/cm under the influence of HDP for 2 minutes) [32]. All acting factors of HDP enumerated above allow exposing the water and the objects put into it (wool and wool grease) to very diverse physical and chemical influences.

Thus, it was established earlier [27] that preliminary HDP of wool in the process of its pretreatment leads to modifying fiber. The structural, supramolecular and sorption properties acquired by woolen fiber contribute to an improvement of its sorption receptivity and reactivity with respect to the active, acidic and basic dyes, to the decrease in the average value of the fiber thinness and degree of felting, to an increase in the degree of crimp and to an increase in the relative breaking load.

The effect of HDP on the wool grease is similar to the action of alkaline refining, as a result of which the release of free aliphatic acids from the grease occurs, as well as other hydrated (protein) substances in the form of soaps. This assertion can explain a decrease in the acid number of wool grease as a result of HDP action and a change in its color from brown to cream, since soaps, when precipitating, absorb the coloring substances. As a result of the conducted studies, the authors of the work developed a unique technology of scouring wool fiber, which makes it possible not only to reach the necessary quality of the washed fiber, but also to intensify the process of extraction of wool grease from the obtained washing waters.

It should be noted that the obtained results of the study are limited by laboratory tests. For the solution of the problem, it is planned in future to create the industrial plant "Vega-6" for applying the HDP of wool and the use of the proposed technology of scouring wool and extraction of wool grease from the obtained washing waters at the Ukrainian wool pretreatment factories LTD "Kedr", Tatarbunary, Odessa Region; LTD "LANATEX", Sumy; LTD "SEBO PLUS", Sumy.

9. Conclusions

1. It was found that the most effective cleaning agent for removal of natural impurities from wool fiber is the preparation Sulside – the composition of which includes sulfanole, ricinox-80, Syntanol DS-10 and dimethylsulfoxide. However, the use of washing substances only does not make it possible to obtain the washed wool of high quality since in the process of repeated washings the felting of wool occurs.

2. The expediency of applying the two-stage technology of scouring wool by the method of HDP was defined, which includes the following sequence of technological operations: mechanical scouring of wool, HDP of wool (3 minutes), washing of wool in the SAS solution (Sulside 1,5 g/l, T=45 °C), washing wool in clean water, drying.

3. It was established that the obtained washing waters are a coarsely dispersed system, the content of wool grease is 18,6 %.

4. It was determined that the wool grease, extracted by the acidic method of grease extraction from the obtained washing waters, is characterized by a substantial improvement in the qualitative characteristics – by reduction in the acid, ether, iodine numbers as well as in the saponification number. Furthermore, it was found that the application of HDP of wool fiber for 3 minutes at the stage of washing contributes to the increase in the output of wool grease up to 96 % in comparison with the amount of grease, extracted from the unprocessed fiber.

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