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Studying the Possibility of Using Caseic Whey for Close-Cycle Technology of Pickling Fur Raw Materials.

Elena Gennadievna Leonova*, Dmitry Valeryevich Shalbuev, Vera Dashievna Radnaeva, and Nikolay Vasilyevich Sovetkin.

East Siberia State University of Technology and Management, 40V, Klyuchevskaya Street, Ulan-Ude, Russia, 670013.

ABSTRACT

The existing technologies of manufacturing fur raw materials use chemical materials that have a negative impact on the environment. To solve the environmental problems, fur companies develop and implement costly projects of water treatment facilities. An alternative solution to this problem is to use technologies that exclude generation of wastewater. The authors have proposed a new composition and a closed cycle method of pickling fur raw materials. The pickling composition containing caseic whey and products of collagen dissolution in the form of gel is applied to the leather of sheepskin by smearing. The study also identifies the technical requirements to the composition: the ratio of caseic whey and products of collagen dissolution at the rate of 3:1, the content of lactic acid in the caseic whey of 20 g/dm^3, pH of the pickling composition should be 3.4%; and dynamic viscosity of the pickling composition should be 117.12 cPs. The proposed pickling compositions, unlike the known ones, did not contain sodium chloride, which significantly reduced the cost of their preparation and disposal of salt in exhausted solutions. Technological parameters have been defined: the consumption of the pickling composition was 20 cm^3 per 1 dm^2 of fur raw material, and the duration of aging after applying the coating composition was 24 to 48 hours.
Keywords: Caseic whey, collagen dissolution products, sheepskin, closed cycle technology, pickling.

**Corresponding author*

INTRODUCTION

The process of pickling in the fur industry plays an important role, since this stage forms the consumer properties of fur semi-finished product like elasticity and plasticity. The effect of pickling is achieved by the action of the acid on the tight structure of the dermis and its separation into mobile thin elementary fibers. This state of the dermis structure becomes permanent after subsequent tanning and finishing.

Modern fur-producing companies use the immersing method of pickling, which is characterized by a considerable after consumption, use of organic acids, sodium chloride, proteolytic enzymes and auxiliary materials (combined effect synthetic surface-active substances). A wide range of chemicals for pickling fur raw material is offered by companies like [BASF] (Germany), [Lowenstein] (USA), [Clariant] (Switzerland), [Fanbo] (China), and the Research Institute of Fur Industry (Russia) [1, 2, 3, 4, 5]. Despite the diversity of chemical materials, world scientific practice is in constant search and development of new materials for dividing leather tissue into fibers.

The authors of this paper have proposed to use a pickling composition based on caseic whey and products of collagen dissolution. The choice of caseic whey for pickling fur raw materials is associated with the traditions of tanning leather and fur by nomadic peoples of the North with the help of dairy products [6]. To do so, dairy products (sour milk, etc.) were rubbed into the leather tissue, and the leather was pommeled either manually or using special devices, tumblers. It was a very long and labor-consuming process.

The caseic whey studied by the authors is a by-product of the dairy industry [7, 8], and using it for pickling fur raw material has several advantages:

- A promising solution to the problem of rational use of whey in dairy industry enterprises;
- Reducing the anthropogenic load on the environment from fur companies due to eliminating the formation of aggressive waste water after pickling, which contains acid and salt;
- Improving elastic-plastic properties of the obtained semi-finished fur product.

The aim was to study the possibility of using caseic in the process of pickling fur raw materials.

To achieve this goal, the authors have solved the following tasks:

1. The chemical composition and indicators of the organoleptic and physical-chemical properties of caseic whey and those of collagen dissolution products have been studied;
2. The temperature regime of caseic whey exposure has been studied;
3. Formulations based on caseic whey and collagen dissolution products have been obtained, and their physico-chemical properties have been studied;
4. The possibility of using formulations based on caseic whey and collagen dissolution products for closed cycle technology of pickling of fur raw materials has been studied.

Study subjects

The subjects of the scientific work were caseic whey, collagen dissolution products and sheepskin.

Caseic whey [9] is a by-product (liquid waste) of the dairy industry that is formed in curd production. In our work, we researched caseic whey with the titrated acidity value 70^{o}T and used it instead of water and organic acids for obtaining compositions for pickling sheepskin.

The collagen dissolution products (CDP) were obtained by treatment with salt and alkali and salt, followed by dissolution of solid waste of rawhide [10]. CDP were used to increase caseic whey viscosity and to prevent runoff of the pickling composition from the surface of the leather of the fur semi-finished product. In addition, CDP have a filling effect on leather of the fur semi-finished product, which fact positively influences its consumer properties.

Sheepskin [11] is the skin of a sheep after the processes of soaking, degreasing and fleshing, transferred to the steamed condition and characterized by evenly moistened leather and loose hairs. Samples of sheepskin after soaking, which were selected with the use of the method of asymmetric fringe [12], were treated with pickling composition, and their pickling state was assessed.

RESEARCH METHODS

The organoleptic methods were used to describe thickness, color and smell of caseic whey [13], collagen dissolution products and the pickling compositions, as well as to assess appearance of the leather tissue and hairs of the fur raw materials and the obtained semi-finished product.

The properties of caseic whey and the products of collagen dissolution were determined by chemical, physical and physico-chemical research methods.

The mass fractions of fat and dry matter in caseic whey were analyzed using the standard methods according to GOST [14, 15].

The content of organic acids in the caseic whey was determined with the use of the "Kapel-105" capillary electrophoresis system at the shared knowledge center “Progress” (The Federal State-Funded Educational Institution of HVE ESSUTM). The method is based on separating charged components of a complex mixture in a quartz capillary exposed to high voltage [16]. Experimental electrophoregram had peaks with different time of substance release. The obtained results were compared with the calibration curve, and the presence of certain organic acids in the whey was assessed.

The IR analysis was performed using the IR-Fourier spectrometer [Nicolet-380] with the use of an attachment [Smart Multi-Bounce HATR] in the window [ZnSe], with the number of scans 32 units at the resolution of 4 units. Interpretation of the spectra used empirical data about the relation between the infrared absorption bands and the structural elements of the molecules [17].

The value of titrated acidity is the main indicator of quality for all dairy products; it was determined titrimetrically and expressed in Turner degrees (°T). 0.1 n solution of sodium hydroxide was used as the titrant, and spirit solution of phenolphthalein was used as the indicator [18].

Activity of hydrogen ions was measured by potentiometric titration with the use of a [pH] meter [19].

The surface properties of whey were studied with the use of a Traube stalagmometer. The surface tension (σ, j/m^2) of the liquid was calculated by formula 1:

$$\sigma = \sigma_{h2o} \times \frac{d_i \times n_{h2o}}{d_{h2o} \times n_i}$$

where [σ_{H2O}] is water surface tension, j/m^2; [d_i] is density of the solution studied, g/cm^3; [d_{h2o}] is water density, g/cm^3; [n] is the number of droplets of the studied solution; and [n_{h2o}] is the number of droplets of water [20].

Density of solutions was determined by the scale of a hydrometer, and the calculated density was expressed in kg/m^3 [21].

The dynamic viscosity of solutions was determined at the rotational Brookfield viscometer, the operation principle of which is based on converting the torque generated during uniform spindle rotation in the medium [22].

The molecular weight of collagen dissolution products was determined with the use of the viscometric method, using the Mark-Howink equation (Formula 3):

$$[\eta] = KM^{\alpha}$$

where [η] is the characteristic viscosity of the studied system; [M] is the molecular weight of the polymer; [K], [α] are constants, whose values depend on the nature of the polymer and the solvent, and on the temperature.

In order to assess the pickling state of the fur raw material, the cure temperature and the duration of the leather tissue dissolution in the caustic and elastic-plastic properties were determined.

The curing temperature of the leather tissue was determined by heating samples of leather in water, and expressed in degrees Celsius as the arithmetical mean of the results of at least five measurements [23].

To study the duration of the dissolution of the leather tissue, 2×2 cm pickled tissue samples after pre-incubation at 60°C in distilled water were placed in a solution of sodium hydroxide, where further dissolution occurred [24]. The time of leather tissue dissolution in alkali was recorded at the moment when the sample became a homogeneous colloidal solution.

The elastic-plastic properties of fur semi-finished product after pickling and drying was determined by the change in the area of the sample under load (tension) and after removing the load, and was calculated by formula 2:

$$P = \frac{S_2 - S_1}{S_1} \times 100$$

where [P] is the elongation (stretch) of the leather tissue, %; [S_1] is the area of the sample of leather tissue of sheepskin before stretching, cm^2; and [S_2] is the area of the sample of leather tissue of sheepskin after stretching, cm^2 [12].

RESULTS AND DISCUSSION

At the first stage of the research, we studied the chemical composition, organoleptic and physico-chemical properties of caseic whey and collagen dissolution products intended for obtaining pickling compositions. The results are shown in Table 1.

Table 1: Caseic whey indicators

Name	Value	
	Caseic whey	Collagen dissolution products
Organoleptic properties (texture, color, smell)	Homogeneous liquid with white sediment; pale green color; odor that id characteristic of milk whey	Homogeneous gel-like material; transparent; odor characteristic of collagen
Mass fraction, % Minerals Fatty substances Dry substances	 0.50±0.01 0.20±0.01 6.80±0.32	 0.12±0.01 2.56±0.03 3.60±0.10
pH index, units	3.40±0.05	3.20±0.05
Density, kg/m^3	1,028±8	1,124±1
Dynamic viscosity, cPs	1.12±0.01 (the "cone-plate" system)	31,176.50±310 (SSA-spindle)
Surface tension, ×10-3 J/m^2	45.8±2.3	-
The value of titrated acidity, °T	70±2	-
Molecular weight, units.	-	275,000±13,750

The studied collagen dissolution products had viscous gel-like consistency (the value of the dynamic viscosity was 31,176.50 cPs), which is characteristic of the products with molecular weight of 275,000 units. The CDP pH value was 3.2 units and was slightly different from the pH of whey, which is important to consider in obtaining pickling compositions.

Analysis of caseic whey showed that the content of lactic acid in it amounted to 6.3 g/dm^3 (at pH of 3.4 units), which corresponded to the value of titrated acidity 70°T (Table 1). In the known pickling compositions that are proposed for immerse fur processing, the content of acids depends on the chemical nature of the acid. Mikhailov A. N. established that for the maximum swelling of the collagen, the flow rate of the acid had to be the following, g/dm^3: formic acid – 4.60, lactic acid - 18, and acetic acid – 60 [25]. Earlier studies of the authors showed that the content of lactic acid in the whey in case of immersion fur pickling should be in the range between 15 and 25 g/dm^3 [26]. Therefore, to achieve the desired degree of separation of fibers of the leather tissue in fur raw materials, the acidity of caseic whey is to be increased from 70 to 200°T and more.

Bearing in mind that the acidity of the whey depends on the temperature and duration of exposure, the influence of the temperature of caseic whey exposure on the value of its titrated acidity was studied for 19 days (Figure 1).

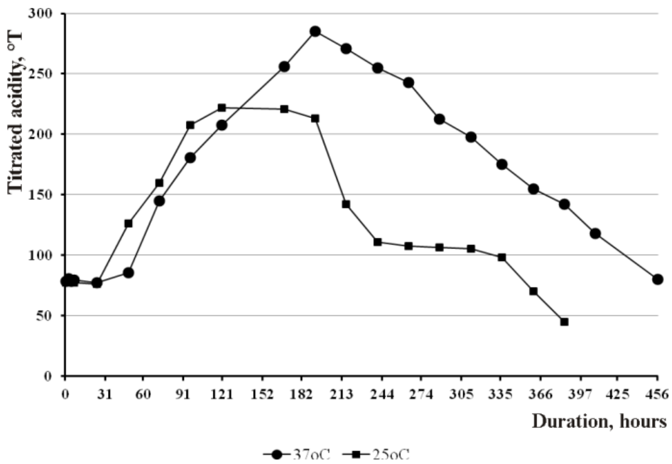

Figure 1: Changes in titrated acidity of caseic whey at different temperatures of exposure

Increasing the value of titrated acidity of caseic whey occurs in vivo due to the homo - and heterofermentative digestion of lactose and other sugars, which results mainly in forming lactic acid. To create a favorable environment for growth and development of lactic acid organisms in caseic whey, the temperatures of exposure of 25 and 37±2°C were tested. Figure 1 shows that at the exposure temperature of 37±2°C, the highest value of acidity was achieved on day 7, and was 285°T (25.65 g/dm^3 of lactic acid), at a temperature of 25±2°C - on day 5 at 225°T (20.25 g/dm^3 of lactic acid). The observed increase in acidity of the whey can be explained by the development, in the first case, of lactic acid rod cells ([Lactobacterium bulgaricum], [Lactobacterium acidophilum], [Lactobacterium casei]), and in the second case, of lactic acid rod cells and streptococci ([Streptococcus lactis], [Streptococcus cremoris], [Streptococcus citrovorus]), and yeast ([Saccharomyces], [Torulopsis], [Candida]) [27].

In pickling fur raw materials, the microorganisms of caseic whey may use protein-carbohydrate complexes that bond the structure of the dermis as power sources, thereby splitting the leather tissue even further. In addition, the additional effect of pickling is also provided by proteolytic enzymes found in whey.

Thus, exposing caseic whey to the temperature of 25±2°C and 37±2°C resulted in an increased content of lactic acid from 6.3 to 20.0 g/dm^3 and more, which is an indicator of suitability of using the whey as a pickling composition. The optimum temperature of whey exposure was 25±2°C, which is economically reasonable.

The increase in the value of the titrated acidity of whey from 70 to 225°T in the first case, and to 285°T in the second case of exposing was caused by the presence of organic acids resulting from microorganisms' activity, which fact is evidenced by the results of IR-spectroscopic analysis.

Figure 2 shows that the peaks in the IR spectra of caseic whey with the titrated acidity of 70, 225 and 285 °T are located in the same area of absorption, and correspond to the same set of functional groups in the wavelength range between 500 and 4,000 nm. The peaks are characteristic of OH groups that are, in turn, characteristic of chelates; aromatic compounds of all types of substitution; variations of saturated ketones and acids, cyclic ketones of 6-membered α, β-unsaturated acids, γ-lactams, β-diketones, ionized acids; fluctuations of groups of $CH_3C≡$, $(CH_3)_2C=$, $(CH_3)_3C$-, $-CH_2$-; groups inherent to cyclohexane, cyclobutane, cyclopropane; variations of RCHO, C_6H_5CHO, C_6H_5COR, CH_2OH, and groups of anhydrides, benzoates, groups of ≡C-OH, -$CONH_2$. Variations of $CONH_2$ are likely to be explained by the presence of whey proteins. So, Gavrilov G. B. [28] asserts that chemical composition of whey protein includes immune globulin (high molecular protein), α-lactalbumin (20-25 %), β-lactoglobulin (50-54 %), and minor proteins (components of the proteose-peptone fraction, enzymes, lactoferrin (a glycoprotein containing iron)). It is likely that peaks in the area of 1,400 - 1,500 cm^{-1} are caused by the presence of dairy (CH_3-CHOH-COOH) and succinic (HOOC-CH_2 -CH_2-COOH) acids confirmed by the method of capillary electrophoresis. Electrophoregrams of determining the content of organic acids in the whey showed the peaks of lactic and succinic acids manifestation in the quantities necessary for efficient pickling fur raw materials – over 20 g/dm^3 (Figure 3).

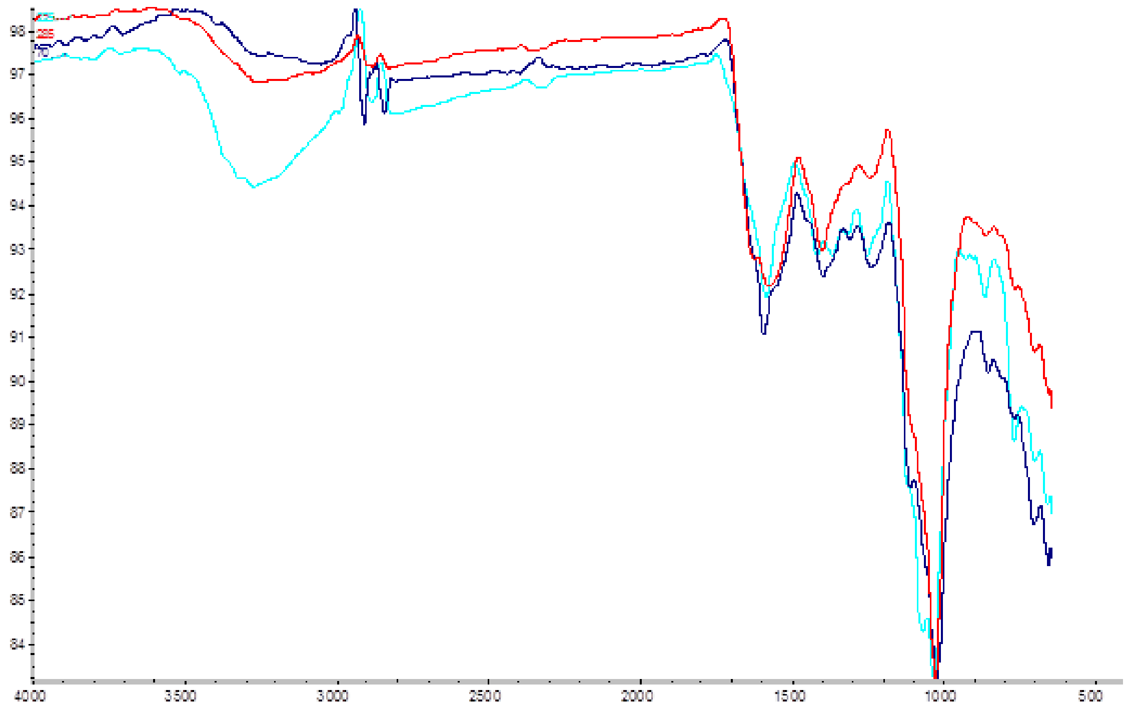

Figure 2: IR spectra of whey

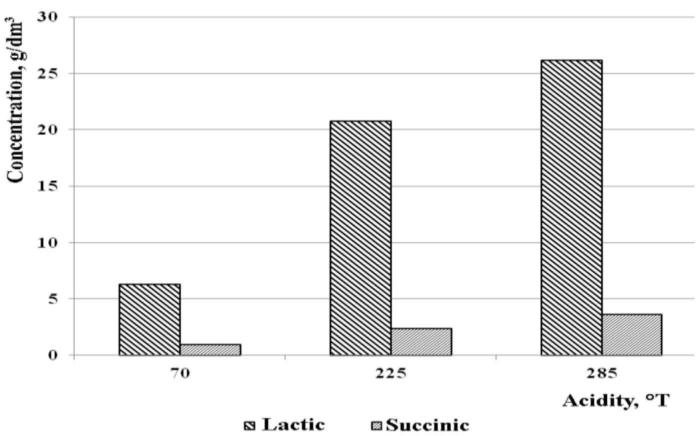

Figure 3: The content of organic acids in whey

For the development of the closed cycle technology of pickling at the third stage, formulations based on whey (CW) and collagen dissolution products (CDP) have been obtained. The value of titrated acidity of whey was 225°T, which corresponded to pH of 3.4%, and the contents of lactic and succinic acids were 20.73 and 2.34 g/dm^3, respectively (Figure 2). Collagen dissolution products were used as a thickener for whey with the molecular weight of 275,000 units and the pH value of 3.2 units. The CW to CDP ratios in formulations No. 1-3 are 1:3, 1:1, 3:1. The consistence of the experimental compositions met the basic requirement, i.e., the compositions held to the surface of leather tissue in course of aging and subsequently diffused into the dermis. The minimum value of dynamic viscosity was characterized by composition No. 3 - 117.12 cPs ([ULA]-spindle), whereas the reference composition No. 4 for immerse pickling had the value of dynamic viscosity of 1.2 cPs (the "cone-plate" system).

It is interesting that classical pickling composition (composition No. 4), besides formic acid (5 g/dm^3) and auxiliary materials (enzyme [Elbro-SR] 2.0 cm^3/dm^3, detergents [Wetter] 0.5 cm^3/dm^3), also included sodium chloride (50 g/dm^3) to prevent "plumping" of the leather tissue, whereas the experimental pickling compositions did not contain salt. This significantly reduced the cost of their preparation and disposal of salt in exhausted solutions.

The organoleptic properties (color, texture, and odor), pH, and the dynamic viscosity of the pickling compositions were determined (Table 2).

Table 2: Indicators of organoleptic and physical-chemical properties of pickling compositions

Pickling composition	Organoleptic assessment	Hydrogen index, units	Dynamic viscosity, cPs
No. 1	Gel-like consistence; pale green color, specific odor of milk whey	3.60±0.05	13,400.00±134 ([ULA]-spindle)
No. 2		3.56±0.05	3,174.40±31.74 ([ULA]-spindle)
No. 3		3.43±0.05	117.12±1.172 ([ULA]-spindle)
No. 4	Transparent colorless solution with a characteristic odor of formic acid	2.67±0.05	1.2±0.010 (the "cone-plate" system)

The experimental pickling compositions No. 1, No. 2, and No. 3 had the pH value about 3.4 to 3.6%, which is well above the pH of the reference composition (2.67%). It is assumed that by reducing the aggressiveness of the pickling bath, more equal division of the leather tissue structure of the fur raw material into elementary fibers may be achieved.

In the fourth stage, the possibility of using experimental pickling compositions for pickling fur raw materials was studied. For this purpose, formulations No. 1-3 were applied with a brush to the surface of the leather tissue of sheepskin at the rate of 20 cm^3 per 1 square dm^2 of raw fur materials. The consumption of the composition was chosen so that it completely covered the surface of the sheepskin sample. After 0, 24, 48, 72 and 96 hours of aging, the indirect indicators of raw materials pickling were analyzed - the scalding point, elongation (stretching), and the duration of leather tissue dissolution in alkali. Pickling in the composition No. 4 served as the reference variant, which, in accordance with the technology [Lowenstein], was performed by immersing with the duration of the process of 16 hours [2].

The results showed (Figure 4) that in 24 hours after aging, the scalding point of leather tissue in the fur samples treated with compositions No. 3 (experimental) and No. 4 (reference) was approximately 48-49°C, which is comparable with the data in literature [29]. Whereas the samples of sheepskin that have been processed with experimental compositions No. 1 and 2 reached similar values of scalding point of leather tissue only after 48 h of aging. Probably, composition No. 3 had the optimal viscosity (117.12 cPs) and, accordingly, the maximum rate of pickling composition diffusion in the thickness of the leather tissue of the sheepskin, which caused the decreasing of the scalding point of the samples after 24 hours aging. Thus, the duration of sheepskin aging after pickling is affected by the viscosity of the experimental compositions.

This work has established that an increase in the duration of aging up to 48 h results in additional division of dermis structure, which has been confirmed by the results of studying the duration of dissolving the samples of leather tissue of sheepskin in alkali. This indicator depends on the number and type of destroyed intermolecular bonds in course of pickling. The samples were dissolved after 71.6-74.2 hours - after aging for 24 hours, and after 49.7-51.6 hours after aging for 48 hours, i.e., the degree of the dermis structure separation into individual fibers was higher after 48 hours. Therefore, the duration of aging after applying the experimental pickling compositions should be more than 24 hours.

It is known [25] that mineral acids feature a greater affinity to collagen, and are mainly absorbed by the top layers of the leather, which does not ensure thin separation of the fiber bundles during pickling. The proposed composition contains only organic acids, which, having lower affinity to collagen, are gradually distributed in the layers of the dermis and optimally disorder its structure. This situation explains the long aging of fur raw materials after applying experimental compositions, which is 24 to 48 hours.

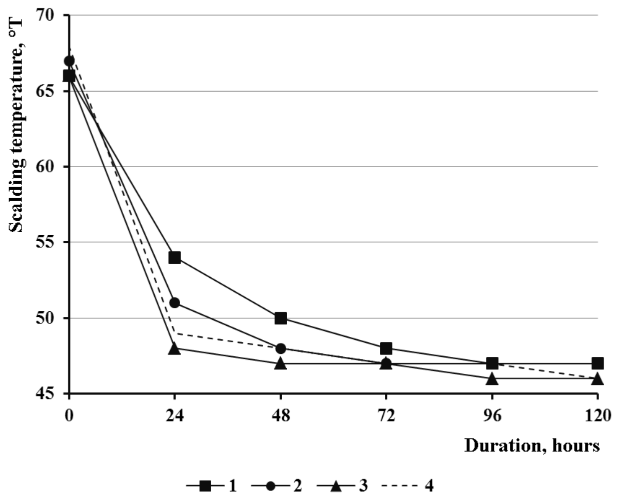

Figure 4: Change of the scalding point of the leather tissue of sheepskin during pickling

Table 3: Indicators of pickling the leather tissue in samples of sheepskin

Duration of aging, hours	Elongation (%) / Duration of leather tissue dissolution in alkali (hours)							
	Composition No. 1		Composition No. 2		Composition No. 3		Composition No. 4	
0	9.8	96.0	10.4	96.0	11.8	96.0	10.3	96.0
24	16.2	74.2	18.1	74.0	24.5	71.6	24.2	72.5
48	20.7	51.6	21.3	50.2	24.8	49.7	24.5	50.1
72	20.8	50.0	21.9	50.0	24.9	49.6	24.5	49.6
96	20.8	49.8	22.0	50.0	24.9	49.3	24.6	49.6

Elongation of the leather tissue in samples of sheepskin treated with compositions No. 1 to 4 after 24 h of aging amounted to 16.2, 18.1, of 24.5 and 24.2%, respectively. After 48, 72 and 96 hours of aging, a slight increase in the elongation was observed in the leather tissue of the samples. This can be explained by the fact that within the first 24 hours, diffusion, distribution and binding of components in the pickling composition with the collagen of the leather tissue occurred, and after the 24 h, further distribution of the pickling effect into the layers of dermis occurred.

It should be noted that the higher the values of elongation (stretching) are, the more efficient the process of pickling is, and the obtained semi-finished product will have high plastic-elastic properties. The maximum values of elongation had the samples processed by experimental composition No. 3 and the reference composition No. 4, which can be explained by a more intensive separation of the collagen bundles into elementary fibers. Thus, samples of sheepskin treated with composition No. 3 featured the best stretching.

CONCLUSION

The authors have shown the possibility of using caseic whey and collagen dissolution products for closed-cycle processing of fur hides. Based on the results of the study, a pickling composition has been developed, which contains caseic whey and collagen dissolution products instead of the traditional organic acids, sodium chloride and water. Due to lower affinity of the lactic acid contained in the whey to the collagen of the dermis in the fur raw materials, gradual diffusion of the pickling compositions into the fibrous structures occurs, which contributes to finer separation of fibers in the leather tissue. The collagen dissolution products contained in the pickling composition can have a filling effect on the leather tissue. Such processing ensures high elastic-plastic properties of the semi-finished product, which is very important in using a fur garment. Besides, the method of smearing pickling makes it possible to retain the structure and the shine of the hair cover of semi-finished fur product. The advantage of the proposed technology is in reducing the cost of chemical materials and water, in excluding formation of aggressive wastewaters in the process of pickling, and in using biologically degradable materials.

Summarizing the results of the research, the technical requirements for the composition have been established: the titrated acidity of the whey should be 225°T, the whey to collagen dissolution products rate should be 3:1, the dynamic viscosity of the pickling composition should be 117.12 cPs, and the pH value should be 3.4 units. The technological parameters have been defined: the consumption of the pickling composition was 20 cm^3 per 1 dm^2 of fur raw material, and the duration of aging after applying the coating composition was 24 to 48 hours.

Further study of the subject will be associated with searching for and studying new materials for thickening the whey.

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