

Досліджено комплекс споживних властивостей гідрофобізованого хутрового велюру нутрії з використанням алкенмалеїно-акрилсинтанової композиції. Склад цієї композиції включає алкенмалеїновий полімер, поліакриловий полімер і синтанний дубитель. Процес наповнювання-гідрофобізації хутрового велюру нутрії проводиться за рН робочого розчину 6,8–7,0, температури 40–43 °С та тривалості три години і закінчується за рН 3,8–4,0.

Гідрофобізований хутровий велюр нутрії отримували з неякісної за волоссяним покривом сировини, неоднорідної структури в різних топографічних ділянках та зниженої щільності, але із задовільними фізико-механічними властивостями шкірної тканини.

Встановлено вплив складу наповнювально-гідрофобізуючої композиції на фізико-хімічні властивості хутрового велюру. Зокрема досліджено характеристики: сумарний тепловий опір, динамічне водопоглощення, всмоктування краплі води, крайовий кут змочування, намокання, паропроникність, повітропроникність та ін.

Встановлено також оптимальний склад алкенмалеїно-акрилсинтанової композиції, який забезпечує формування модифікованого хутрового велюру зі шкурки нутрії з комплексом підвищених споживних властивостей порівняно з велюром овчини. Зокрема при гідрообробленні зниження показників сумарного теплового опору та паропроникності гідрофобізованого хутрового велюру нутрії проявляється у меншій мірі відповідно в 9,7 і у 1,7 рази порівняно з велюром овчини. При цьому естетичні показники велюру нутрії – колористичне оформлення та якість оздоблення шкірної тканини – також переважають велюру овчини на 39–41 %.

Одержані результати гідрофобізації хутрового велюру з комплексом необхідних споживних властивостей свідчать про можливість розширення асортименту матеріалів з хутрової сировини з неякісним волоссяним покривом, але задовільними фізико-механічними показниками шкірної тканини. При цьому виготовлені хутрові вироби з гідрофобізованого велюру нутрії будуть придатні для експлуатації в умовах підвищеної вологості

Ключові слова: хутровий велюр нутрії, наповнювання-гідрофобізація, алкенмалеїно-акрилсинтанова композиція, споживні властивості

A COMPREHENSIVE ANALYSIS OF CONSUMER PROPERTIES OF NUTRIA VELOUR HYDROPHOBICIZED WITH ALKENMALEIN-ACRYLSYNTANE COMPOSITION

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1. Introduction

Present-day fur production involves fabrication of a wide range of furs and fur coat materials including velour. Sheepskin is mainly used in production of fur velour with taking into consideration the features of skin tissue and hair. At the same time, considering conditions for wearing the fur velour products, physical and mechanical properties of skin tissue are essential. Fur velour is obtained from fur coat sheepskins after their wet polishing and rejecting those unsuitable for velour production [1]. Taking into consideration the shortage of such raw materials in Ukraine [2], there is an objective necessity and urgency in a search for other types of fur raw materials with a corresponding set of consumer properties. In this regard, male nutria skins with low-grade overhair unsuitable for the manufacture of fur material are of interest. They can be effectively used in production of fur velour after overhair epilation and improvement of physical and mechani-

cal properties of the skin tissue by applying new chemical filling materials followed by hydrophobicizing.

2. Literature review and problem statement

Structure of fur skin materials is hydrophobicized using monomeric and polymeric reagents in a wide variety of chemical compositions, in particular, silanes, fluorocarbon resins, polydimethylsiloxane rubbers, complexes of aluminum and wax or paraffin compounds [3], etc. Improvement of water resistance of the skin is also achieved by coating painting which results in formation of a multilayer protective film coating during multiple application of a coating composition followed by drying, pressing and use of special equipment [4, 5]. It should be noted that such technology is highly complex, time-consuming and labor intensive.

Semi-finished fur skin materials are volumetrically modified with nitrogen-containing compounds, derivatives of

hydroxycarboxylic acids, fatty acid esters, etc. Among the most common hydrophobicizing agents, organosilicon compounds including silanes and silicones as well as paraffins are used. A copolymer of acrylic acid and hydrophobic acrylate monomers in a number of chemical compositions were used in [6] at the stage of retannage and staffing of chrome tanned semi-products. At the same time, the degree of filling increases and water resistance, plasticity and mechanical strength improve. Considerable influence of copolymers with direct hydrocarbon chains on these properties has been established. The highest hydrophobic effect was achieved at a side chain length above C_{16} .

Emulsion of fluorine-containing copolymer [7] based on maleic anhydride, rapeseed oil or fish oil with addition of dodecafluorheptanol and octadecyl alcohol make it possible to obtain chrome-tanned leather of increased water resistance. In this case, the maximum effect was achieved at a 5 % content of fluorine in the copolymer molecules. The resulting skin was characterized by wetting angle of 155° , a static water absorption coefficient of 9 wt. % and dynamic water permeability of 55 min. Use of hybrid polyfunctional polyurethanes of organo-inorganic amphiphilic polymers with hydrophobic, oleophobic and hydrophilic radicals to improve water resistance and dirt repulsion is known [8]. However, skin stiffness increases and appearance worsens after such modification.

The studies that give results of raising water resistance of skin materials due to the use of combined treatment with low-temperature plasma and chemical reagents are of significant scientific and technical interest. For example, when applying hexamethyldisiloxane and tetraethylorthosilicate [9], water resistance of the skin grows. Authors of [10, 11] investigated influence of A-187 organic-silicone polymer and conditions of plasma treatment on physical, mechanical and hygienic properties of leather obtained from cattle and sheep skin raw materials. The materials produced according to the proposed technology were characterized by a 23 % higher strength after integrated plasma treatment. In this case, duration of absorption of water drops increased by 86 % and hygroscopicity decreased by 87 and 76 % [10] for sheep and cattle skin, respectively.

It should be noted that the overwhelming number of these studies relate to the skin hydrophobicity. At the same time, due to the considerable difference between tissues of fir skin and leather consisting in its increased plasticity and structural features, this fact determines high demands to the technological process. In particular, this refers to the process of filling and hydrophobicizing fur velour with preservation of elastic properties of the skin tissue.

Thus, combination of hydrophobicizing fur skin materials with other technological processes is considered an effective method. At the same time, quality of the resulting natural material featuring water resistance essentially depends on chemical structure of reagents, development of new compositions and technologies of their application. In determining water resistance of the skin tissue and hence thermal protection properties of the fur velour with polished frieze surface, significant difficulties arise in objective determination of these indicators. Therefore, it is necessary to use a system of structural and colloid-chemical properties of the investigated materials. Therefore, solution of these issues becomes especially important in development of technologies for manufacturing fur velour from raw materials with a

specific structure of skin tissue which can effectively serve in wear products under conditions of high humidity.

3. The aim and objectives of the study

The study objective was obtaining of fur velour from nutria skins with low-grade overhair by means of hydrophobicizing the skin tissue with alkenmalein-acrylsyntan composition.

To achieve the objective, the following tasks were set:

- analyze technological features of production of hydrophobicized velour from nutria skins;
- establish influence of component ratio of the filling and hydrophobicizing composition on physical and chemical properties of nutria fur velour;
- determine essential consumer properties of fur velour obtained from nutria skins at an optimal component ratio of the filling and hydrophobicizing composition.

4. The study materials and methods

Male nutria 24–25 dm² area skins with rough overhair were taken for obtaining hydrophobicized nutria fur velour (NFV) after chrome tanning, resting for 22–24 hours, centrifugation and beating [1].

The process of filling and hydrophobicizing of the semi-finished nutria skins was carried out in a paddle with a solution of alkenmalein (AM) polymer at a temperature of 40–43 °C and stirring for 20 minutes. After 1 hour, a filling mixture of Melio Resin A-821 polyacrylic emulsion from Clariant (Germany) and BNS synthetic tannin (TU 17-06-165-89) was added to the working solution (Table 1). After 40 minutes of stirring the system contents, the rest of the hydrophobicizing AM polymer was added to it. The polyacrylic emulsion had dry residue of 24 % and density of $1.03 \cdot 10^3$ kg/m³, the BNS syntane contained 62 % of tannins. A composition based on C_{20-24} α -alkenes and maleic anhydride which is a 50–52 % aqueous-organic polymer solution with a mean-value molecular weight of $38 \cdot 10^3$ was used for hydrophobicizing of NFV.

The process liquor had pH of 6.8–7.0. On the expiry of 30 min stirring of the system, pH of the solution was adjusted with formic acid to 3.8–4.0. Subsequent pressing and free drying of the semi-finished product was carried out at a temperature of 20–23 °C. Overhair was removed by epilation. The obtained hydrophobicized semi-finished nutria skins were subjected to polishing by No. 4 abrasive cloth.

The filled and hydrophobicized NFV was characterized by a set of physical and chemical properties after pre-conditioning of samples by a desiccator method in normal conditions.

Thermal protection ability of the fur velour was determined on whole skins by total thermal resistance [12] using PTS-225 device, Russian Federation. Samples of fur velour were tested with and without blowing at a speed of 5 m/s and direction of air flow at an angle of 45° to the sample surface. Total thermal resistance R , m²·°C/W, was calculated from formula:

$$R = \frac{3C_1}{FK \cdot 3C_1z - a + C_2},$$

where C_1 is total heat capacity of the instrument plate, J/°C; F is the plate constant, J·°C/m²; K is coefficient of dissipation of heat flow over the fur sample; z is the rate of cooling of the instrument plate, s; a is correction for dissipation of the heat flux in the device, s⁻¹; C_2 is the total heat capacity of the plate and the sample, J/°C with

$$C_2 = 1.675 \cdot 10^3 qS,$$

where $1.675 \cdot 10^3$ is specific heat of material of organic origin, J/kg·°C; q is weight of 1 m² sample, kg; S is the device plate area, m².

Water impregnation in dynamic conditions was determined with the help of PVD-2 instrument, Russian Federation. Air permeability, vapor permeability and other indicators were determined by appropriate methods [13]. Water resistance of NFV was determined after its hydro-testing according to ISO 4920:2012 standard for 10 minutes in the same way as textile materials by spraying water at a temperature of 24–26 °C. Tensile strength and elongation of velour under load of 4.9 MPa and uniaxial tension were determined on RT-250M-2, belt A, tearing machine, Russian Federation, at deformation rate of 80 mm/min.

Thus, a set of physicochemical indicators including thermal protecting and hygienic properties was used to assess consumer properties of hydrophobicized nutria fur velour.

5. Influence of component ratio of alkenmalein-acrylsyntane composition on properties of nutria fur velour

Results of the study of influence of chemical composition of filling and hydrophobicizing agents (Table 1) on physical and chemical properties of NFV are given in Table 2. Variants of filling and hydrophobicizing of nutria skins are characterized by different consumption of ingredients of the process liquor at a sevenfold flow rate of water relative to the skin weight. Moreover, the third variant of treatment differs from the first one by 1.7 and 3 times higher values of consumption of alkenmalein polymer and acrylsyntane component, respectively. Filling and hydrophobicizing ingredients in the structure of nutria skin tissue were fixed by reducing pH to 3.8–4.0 with formic acid. Sheepskin fur velour (SFV) whose semi-finished product was retanned using chrome tannin with basicity of 35–40 % and BNS syntane according to the current technology was used as a control sample [1]. At the same time, spending of the process liquor in all studied technologies exceeded working one by 5–15 %.

Table 1

Consumption of alkenmalein and acrylsyntane complex ingredients in filling of semi-finished nutria skins

Treatment variant	Material consumption, g/l					Solution spending, %
	AM polymer	polyacrylic emulsion	BNS syntane	AM polymer	formic acid	
1	2	4	3	5	0.5	86
2	5	9	6	7	0.5	80
3	5	12	9	7	0.5	76

As can be seen from Table 2, with an increase in consumption of hydrophobicizing and filling ingredient, physicochemical properties of NFV varied dramatically. In particular, with consumption of reagents by the second variant, duration of

dynamic water wetting and water drop absorption increased 2.8 and 2.65 times, respectively, compared with the first variant. Relative to the control variant, 17.7 and 106 times increase in these indicators was observed. It should be noted that at the same time, air permeability of the NFV reached the maximum value in variant 3 (1.8 times increase) at a slight decrease in vapor permeability compared to variant 1.

Table 2

Physicochemical properties of nutria fur velour

Indicator	Variant			
	1	2	3	control
Welding temperature, °C	63	64	64	61
Water wetting in dynamic conditions, s	360	1,020	720	60
Water drop absorption, s	1,200	3,180	2,940	30
Wetting angle, deg.,				
– in 1 min	110	130	130	–
– in 30 min	–	120	120	–
Soaking, %,				
– in 2 hrs	142	123	131	197
– in 24 hrs	184	160	172	213
Vapor permeability:				
– absolute, 10 ⁻⁶ kg/(m ² ·s)	5.28	4.72	4.44	5.83
– relative, %	54	48	45	59
Air permeability:				
– absolute, s	11	13	16	9
– relative, 10 ⁻⁶ m ³ /(m ² ·s)	0.16	0.13	0.11	0.19
Increase in area, %	3.2	5.8	6.3	0

The obtained data show a considerable influence of hydrophobicizing and filling ingredients on structure and physicochemical properties of the fur velour due to their interaction with fibrous collagen of the NFV skin tissue and corresponding reduction of fibrillar interactions. This effect was accompanied by a 1.8 and 1.97 times increase in area, respectively, as compared with the minimum consumption of the filling component.

Thus, the results of physicochemical studies have made it possible to determine composition of ingredients of the filling and hydrophobicizing complex for production of fur velour from nutria skins possessing a set of improved properties.

An a priori ranking of the consumer indicators of the material determining its quality was conducted in further studies to establish the most important wearing and aesthetic properties of the obtained hydrophobicized fur velour of nutria skins using the developed technology [14]. As a result of this ranking, 10 significant indicators were established with coefficient of concordance of 0.894. Statistical significance of this coefficient was confirmed by the χ^2 criterion equal to 136.8 at its critical value of 27.6 at the level of significance 0.05.

Results of the experimental studies of influence of ingredients of the filling and hydrophobicizing complex on essential consumer properties of the NFV are given in Table 3 and in Fig. 1, 2. Treatment of NFV with alkenmalein polymer is accompanied by a sharp increase (24 times) of duration of dynamic water wetting of NFV (Table 3) compared with non-hydrophobicized SFV which indicates its significant hydrophobicizing effect on hydrophilic structure of the material. Accordingly, if the hydrophobicized NFV revealed somewhat lower values of TTP in its initial state (Fig. 1) in comparison with SFV, this indicator increased 2.1 times after

hydro-processing. In this case, deformation properties after NFV hydro-processing were characterized by significantly greater stability compared to SFV which, accordingly, had almost twice the value of residual elongation. The obtained results indicate that the products made from hydrophobicized NFV will ensure high stability of their shape, even after 10 minutes of water action.

Effect of hydrophobicizing of NFV is well revealed in analysis of its hygienic properties (Fig. 2).

Thus, hydrophobicized NFV is characterized by 53 and 60 % higher values of vapor permeability and air permeability parameters, respectively, compared with SFV. Difference between these indicators significantly increases to 4.7 and 2.5 times after hydro-processing. It should be noted that hydrophobicized NFV also has a clear advantage in aesthetic indicators after hydro-processing (Table 3). This is manifested in a smaller loss of quality of leather dressing and coloristic design of the skin tissue.

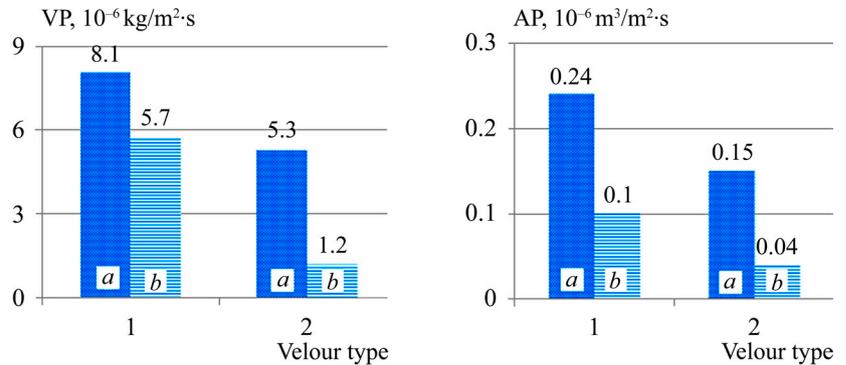


Fig. 2. Dependence of vapor permeability (VP) and air permeability (AP) of skin tissue on the type and condition of fur velour: 1 – NFV, 2 – SFV; a – before hydro-treatment, b – after hydro-treatment

Thus, processing of fur velour from nutria skins with the developed filling and hydrophobicizing composition provides formation of a modified material with a complex of improved consumer properties in comparison with the fur velour produced from fur-coat sheepskins. This effect significantly increases after hydro-processing.

Table 3

Consumer properties of fur velour obtained from nutria and sheep skins before and after hydroprocessing

Indicator	Fur velour			
	nutria		sheepskin	
	before and after hydro-processing			
	before	after	before	after
Tensile strength of the skin tissue, MPa	0.69	0.63	0.94	0.82
Water wetting in dynamic conditions, s	1,380	870	57	0
Relative complete elongation of the skin tissue at a stress of 4.9 MPa, %	21	23	22	29
Relative elastic elongation of the skin tissue at a stress of 4.9 MPa, %	12	12	10	8
Relative residual elongation of the skin tissue at a stress of 4.9 MPa, %	9	11	12	21
Coloristic design of the skin tissue, units	1.00	0.85	0.95	0.61
Dressing quality of the skin tissue, units	1.00	0.85	0.90	0.60

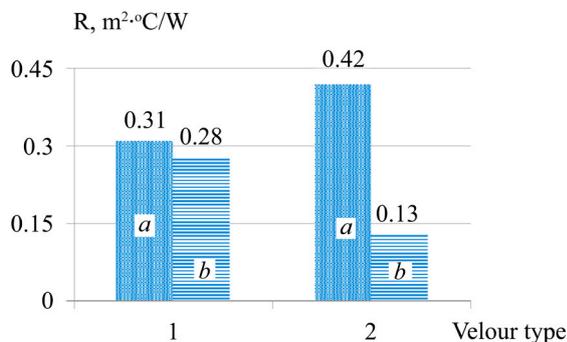


Fig. 1. Dependence of the total thermal resistance (R) on the type and condition of fur velour: 1 – NFV, 2 – SFV; a – before hydro-processing, b – after hydro-processing

6. Discussion of the results obtained in the study of the consumer properties of nutria fur velour

To improve water resistance and other consumer properties of natural leather and fur materials, processing of surface and volumetric types was applied using a wide range of chemical reagents. Volumetric hydrophobicizing modification of the semi-finished products is used in the processes of filling, retanning, stuffing and coating painting. Given the heterogeneous and reduced density of nutria skin tissue in both volumetric and topographic areas, a need for filling of its structure appears first of all. This measure makes it possible to significantly raise density of the skin tissue and improve its physical and mechanical properties. In order to improve heat-protective and hygienic properties and preservation of product shape during wear in conditions of high humidity, it is necessary to hydrophobicize semi-finished products. Significant advantages of bulk processing of semi-finished products in a forward trace [1] during implementation of the NFV manufacturing cycle should be noted.

In order to effectively carry out the NFV filling and hydrophobicizing process, introduction of AM polymer in the process liquor in two steps (before and after filling) was used in the study. Primary treatment of NFV with AM polymer ensures its deep diffusion in the intermicrofibrillar structure of the semi-finished product and promotes dispersion of polyacrylic polymer and the BNS syntane followed by their sorption. After filling of the interfibrillary gaps in the structure of the semi-finished product, it is necessary to reintroduce AM polymer for its sorption in the modified surface of elements of the fibrillar structure of the semi-finished product and achieve the hydrophobic effect. When using an optimal amount of alkenmalein-acrylsyntane composition, maximum value of water resistance of NFV is achieved. However, the hydrophobic effect decreases when amount of hydrophilic filler is increased. At the same time, maximum hydrophobic effect causes increased mobility of elements of the fibrillar structure in the NFV skin tissue and its plasticization and, accordingly, an increase in the material area output.

An adequate change in porosity of the hydrophobicized NFV skin tissue takes place which is expressed in the nature of change in air and vapor permeability. With an increase in the filling component content at an optimum content of AM composition, the effect of dispersion of structure of the semi-finished product and hence air permeability grows. Difference of the nature of vapor permeability is due to the mechanism of diffusion of water vapor through the porous structure of NFV skin tissue, in particular, due to different contribution of the pore size and shape, the character of the pore size distribution and hydrophobic-hydrophilic nature of the wall surface in the process of sorption-desorption of water molecules during their transportation through the material structure.

The effect of using the alkenmalein-acrylsyntane composition with an optimal content of elements in the NFV manufacture was well observed in comparative study of the set of essential consumer properties after hydro-processing. The hydrophobic-hydrophilic character of structure of the modified nutria fur velour is expressed in the practically stable elastic elongation of the skin tissue at a stress of 4.9 MPa or a slight increase in elongation at the practically unchanged strength. At the same time, considerably larger changes in deformation indicators of SFV skin tissue and especially residual elongation are explained by its greater water sensitivity connected with increased hydrophilicity of its structure. This is also reflected in significantly higher dynamic water resistance of the hydrophobicized NFV in comparison with the sheepskin velour. Significantly higher value of the SFV wetting causes low value of its total thermal resistance. At the same time, this important consumer indicator gets smaller in a less degree in the hydrophobicized NFV indicating its high thermal protective properties.

In future, it is necessary to perform computer multi-criteria optimization of the alkenmalein-acrylsyntane composition and conditions of its effective use in the technology for industrial testing of the developed process of filling and hydrophobicizing of nutria fur velour. To do this, it is necessary first to work out a corresponding plan of an experiment to determine element content of the composition and properties of the nutria fur velour and then determine type of a plan of establishing filling and hydrophobicizing technology and elucidating properties of the obtained velour. After realization of corresponding experiments, mathematical models shall be constructed on the basis of which parameters of optimal element content of the composition and its use in the technological process can be determined.

As a result of the studies to be conducted, permissible limits of element contents in the alkenmalein-acrylsyntane composition and the process factors shall be established. The obtained results of optimization will be used for testing the developed technology in an experimental shop of an industrial enterprise. After development of technological regulations, the process of filling and hydrophobicizing nutria fur velour can be implemented under industrial conditions.

Consequently, proceeding from the discussed set of essential consumer properties, the hydrophobicized nutria fur velour obtained by the developed technology can be used as an effective material for replacing sheepskin fur velour in the manufacture of water-resistant wear products.

7. Conclusions

1. To manufacture nutria fur velour with a set of consumer properties suitable for use in conditions of high humidity, it is necessary to carry out filling and hydrophobicizing of the semi-finished product in a single technological cycle of material production. The process of filling and hydrophobicizing of nutria fur velour is carried out in two stages. About 40 % of the alkenmalein polymer is introduced in the process liquor at the first stage and the rest after filling of the skin tissue with the acrylsyntane component. This process gives a significant compaction of the skin tissue structure and increases its water resistance and shape stability of the wear products made from nutria fur velour.

2. It was established that duration of dynamic water wetting and water drop absorption of the skin tissue of the hydrophobicized nutria fur velour increased with increasing content of alkenmalein polymer in the filling and hydrophobicizing composition. These indicators reached maximum value at the polymer flow rate of 12 g/l. At the same time, increase in the content of acrylsyntane tanner from 15 to 21 g/l and the wetting angle reached 130 and 120 degrees in 1 and 30 minutes, respectively. Air permeability of nutria velour increased by 23 % at practically the same vapor permeability with a 5.8 and 6.3 % increase in area growth compared with the control variant.

3. Ten essential consumer properties of nutria fur velour were determined by the method of *a priori* ranking. They most fully and objectively characterize wearing and aesthetic properties of the material. Application of the optimal component ratio in the alkenmalein-acrylsyntane composition in the manufacture of modified nutria fur velour contributed to formation of a material with increased total thermal resistance and vapor permeability after hydro-processing of the material. These indicators were 2.1 and 4.7 times higher than those of the sheepskin fur velour. At the same time, aesthetic indicators of nutria fur velour (coloristic design and quality of leather dressing) also exceed those of sheepskin fur velour by 39–41 %.

The results from hydrophobicizing fur velour with a set of necessary consumer properties indicate the possibility of expanding the range of materials produced from fur skins with low hair quality but satisfactory physical and mechanical indicators of skin tissue. The fur wear products made from hydrophobicized nutria velour will be suitable for use under conditions of high humidity.

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