

Diffusion of Fillers in the Structure of Chrome Leather and the Impact on Their Air and Water Performance



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Abstract: The main results of the study of the diffusion of fillers in the structure of chrome leather are presented. The effect of fillers on the air - and water permeability of leather is shown. The products of protein hydrolyzate of chrome-plated leather wastes and modified protein hydrolysates with acrylic acid and plasticized with glycerin were prepared. Leather prototypes are filled with protein hydrolyzate products. The control leather is filled with barium chloride, molasses and potassium alum. The diffusion of the fillers of experimental and control samples of leather with both the front and the bahtar side was investigated. The water resistance of the leather treated with the studied fillers confirms that repeated cycles of watering and drying of the leathers practically do not change the indicators of wettability. Uniform diffusion is observed when using barium chloride and alumino-potassium alum. In the leather samples of used molasses, its uneven distribution over the thicker leather and salinity is observed. The porosity of the prototypes of the leather and, consequently, their air permeability after filling is reduced in comparison with the control samples.

Key words: filler, hydrolyzate, collagen, modified, porosity, penetration, distribution, diffusion, air, water permeability, water lacqueriness, wetness.

I. INTRODUCTION

Recently, much attention has been paid to the development of new effective ways of filling the leather. On the basis of the research carried out with the use of new synthesized chemical materials, some regularities have been identified for filling leather and fur for various purposes [1-3]. The main results of the study of the laws of synthesis and the properties of graft copolymer of collagen with acrylic acid are given. The optimal synthesis parameters were found: the initial ratio of the components, the effect on the concentration of the catalyst, time and temperature. It was determined that with an increase in the amount of acrylic acid in the copolymerization process,

the reaction rate, the viscosity of the solutions and the yield of the copolymers formed increase due to the participation of the acrylate double bond in chain transfer reactions. These results allow directional synthesis to produce copolymers with a uniform distribution of functionally active groups, which in the future may determine their high hydrophilic properties.

It is shown that acrylic acid does not inhibit graft copolymerization, but actively reacts with copolymerization with collagen over wide ranges of initial comonomer concentrations [4-6]. In this work, the diffusion of fillers in the structure of chrome leather and their effect on air and water permeability are investigated.

II. EXPERIMENTAL PART

When performing the work, various salts, acids, solvents, monomers, polymers and other chemical materials were used.

According to the well-known technique [7], various concentrations of analytical solutions, tanning agents, compositions were prepared and their chemical analysis was performed.

Objects of study

Acrylic acid brand "h" was distilled at atmospheric pressure over copper shavings. The fraction boiling at 140-141°C, $n_D^{20} = 4224$ was collected [8].

Acetone was absolutized by holding over NaOH for several hours, periodically adding a new batch, then distilled over a fresh batch of P₂O₅. The selected fraction was distilled three times. Boiling point 56,0 °C. $n_D^{20} - 1,3590$ [9].

Ethyl alcohol (C₂H₅OH) was absolutized using calcined calcium oxide (CaO), followed by distillation, with a boiling point of 78.4 °C [10].

Aluminum-potassium alum - white hygroscopic crystals, well soluble in water. It is isolated from aqueous solutions in the form of KAl(SO₄)₂·12H₂O crystalline hydrate. Melting point 92 °C (in its own water of crystallization). Molar mass 258.20; Density is 2.75 g/cm³. Solubility in water 3⁰, 5,9²⁰, 71.0⁸⁰ g/100 ml. Reg. CAS number 10043-67-1. PubChem 62667. ChemSpider 56418. Used as a mordant in dyeing fabrics, coagulant in wastewater treatment, crystalline deodorants, artificial food, etc. [9]

Treacle (dextrin maltose, maltodextrin) GOST 32902-2014, C₁₈H₃₂O₁₆ - a product of incomplete acid (diluted acids) or enzymatic hydrolysis of starch. It is similar in consistency to a thick syrup. Composition, in%: - dextrin up to 70, glucose up to 50, maltose up to 85. Depending on the carbohydrate composition, it can be used in various sectors of the food industry and in non-food production.

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Reg. CAS 9050-36-6. PubChem 62698. Reg. EINECS number 232-940-4. ChEBI 25139. ChemSpider 56445. Data for standard conditions (25 °C, 100 kPa) [10].

Barium chloride BaCl₂, GOST 4108-72 - binary inorganic substance belonging to the salt class, solid. Molar mass: 208.23 g/mol [9]. Barium chloride under standard conditions is a colorless rhombic crystals. It is slightly soluble in alcohol, insoluble in diethyl ether. Barium chloride dihydrate, colorless crystals with a monoclinic lattice, crystallizes from aqueous solutions. Toxic, MPC 0.5 mg/m³. Barium chloride is used in the production of pigments (for example, barite yellow, casserole greens and other compounds), as an additive to electrolytes in the production of magnesium, for hardening high-speed steel (as melts of a mixture of barium chloride with alkali and alkaline-earth metal chlorides) as a component of ceramics, reagent on (sulfate ion), for weighting and lightening of the leather in the leather industry, as a zoocide [11].

Glycerin C₃H₈O₃ - the simplest representative of trihydric alcohols with the formula C₃H₅(OH)₃. It is a viscous clear liquid. Density: 1.26 g/cm³. Chemical formula: HOCH₂-CH(OH)-CH₂OH. Refractive index: 1.4729. A thick transparent liquid, extracted by chemical processing of fats, used for medical and technical purposes. Glycerin is a colorless, viscous, very hygroscopic liquid that mixes with water in any proportions. Sweet to the taste. In conjunction with propylene glycol, it becomes more fluid. Scope: food industry, tobacco production, electronic cigarettes, medical industry, production of detergents and cosmetics, agriculture, textile, paper and leather industries, plastics, paint and varnish industry, electrical engineering and radio engineering. Registered as a food additive E422, and used as an emulsifier, by means of which various immiscible mixtures are mixed [12].

Preparation of collagen solution. The main raw material for the production of collagen was an aspiration split of bovine leathers. A split of at least 3.0-4.0 mm thick, qualitatively fleshy, white, without lime spots and alkalis was selected at the doubling stage. It was cut into pieces no more than 10x10 mm and washed in running water for 8 hours to remove lime and mechanical impurities. After washing, the solution was hydrolyzed with an alkaline-saline solution of 10% sodium hydroxide and saturated sodium sulfate. The hydrolysis was carried out with stirring for 48 hours at a temperature of 19 ± 2.0 °C. After hydrolysis, the spent solution was decanted, the split was left to drain moisture for 30 minutes. After hydrolysis, the split was washed with distilled water. Then, alkali was neutralized to compact the structural elements and reduce the degree of swelling of the collagen mass. Neutralization was carried out with 3% boric acid to pH 5-6, at a temperature of 18-21 °C. Processing time was 24 hours with stirring. Next, treated with buffer solution to remove globular proteins. The buffer solution was prepared as follows: to 21.008 g of citric acid was added 20 ml of 1.0 N sodium hydroxide and diluted with distilled water to 1 liter. Then a 0.1 N solution of hydrochloric acid was taken with a volume equal to the previously prepared solution and added to the previously prepared solution to a pH of 3.6-3.8. Processing time 24 hours with stirring at a temperature of 18-21 °C. At the end of the treatment, the buffer solution was decanted and the split was washed with running distilled water for 6 hours until neutral. Then the collagen mass was placed in a refrigerator for 24 hours at a

temperature of 6-8 °C. To obtain a collagen solution, the collagen mass was dissolved in a mixture of a 6% solution of acetic acid and ethyl alcohol in a ratio of 9:1. The dissolution was carried out for 24 hours at 20 °C. During the first two hours, it was stirred vigorously, and then for 1 hour with an interval of 3 hours. The resulting collagen solution was kept at 20 °C for 24 hours. The solution was filtered twice through a nylon filter material. Then the solution matured in a refrigerator at 7-8 °C for 24 hours and was filtered again. In this case, the solution underwent additional homogenization [13].

Properties of collagen. Collagen is insoluble in cold water, dilute solutions of acids, alkalis, and salts, but it splits and swells. In hot water, cooks, and then goes into the glue.

The resulting collagen is a white protein substance in a highly elastic state. When dehydrated, it loses its elasticity and becomes brittle. The density is 1.35 g/cm³, the pH of the isoelectric point is 6.8. Sorption capacity in the range of 200-300% by weight of absolutely dry matter. It dissolves in weak solutions of acids and alkalis, with increasing temperature its solubility increases markedly. Not soluble in alcohols such as isopropanol, methanol, in ethers, in dioxane as well as in aliphatic hydrocarbons. It is precipitated in acetone and in a saturated solution of sodium chloride [14].

Modification of collagen with acrylic acid. To this end, a sample of dry powdered collagen was weighed out in 20 g each; it was poured into a 200 ml volumetric flask and poured with distilled water to the flask mark. The contents of the flask were left for 5-6 hours for swelling and complete dissolution at room temperature. Thus, a collagen solution of 10% concentration was prepared.

Next, in the flask was added acrylic acid in the amount of 3.0% by weight of dry collagen. Then the contents of the flask were stirred with a mechanical stirrer for 20 minutes. and then 0.05% hydrogen peroxide (60%) was added and stirring was continued for another 3.5 hours at a temperature not higher than 40 °C. After the process of modifying collagen with acrylic acid, water was added to a density of 1.08 g/cm². After the time, solutions of the product of modification of collagen with acrylic acid to form films were cast from the flask into Petri dishes and over them were left to dry completely at room temperature. Next, the films were removed from the cups and dried in an oven at a temperature of 102-104 °C to an absolute state. The output was 16.8 [15].

Thus, in this study, chrome tanned leathers for shoe tops, treated with filling agents (protein hydrolyzed chrome-plated leather waste, modified collagen with acrylic acid and modified collagen with acrylic acid, plasticized with glycerin — experimental) were investigated.

III. METHODS EXPERIMENTS

Filling. Semifinished chrome-tanned yalovki medium bychina easy. Thickness of planing is 1,6-1,7 mm. Consumption of the filling mixture in percentages was calculated depending on the weight of the planed leather. Washing 1st L.C (liquid coefficient). = 2.5, temperature 30 °C, 15 min. Washing 2nd L.C. = 2.5 temperature -30 °C, 10 min. Filling: L.C. = 0.6-0.8 30 °C, when the drum rotates through the hollow axis, a naponitel was introduced in the amount of 3.0% (calculated on the dry matter),

the filling time was 30-40 minutes, then, while the drum was rotating, it was added in the form of 10% a solution of sodium formate or sodium sulfite in the amount of 0.5% and the rotation is continued for 15-20 minutes, the pH of the bath at the end of treatment is 3.5. Further processes: washing, neutralization, dyeing, greasing was performed by the current method. Poddublivanje organic tanning agents did not produce [16].

Determination of air permeability. Breathability is determined on the basis of GOST 938.18 - 70. This standard applies to the leather of all kinds and establishes a method for determining the air permeability. To determine the permeability of the device used.

The air permeability of the leather and the leather of the fur fabric characterizes their ability to pass air at a pressure difference on both sides of the test specimen.

The air permeability indicator, $\text{cm}^3 / (\text{cm}^2 \cdot \text{h})$, is the volume of air in cubic centimeters passing through 1 cm^2 of the area of the test sample for 1 h with a pressure difference on both sides of the sample equal to 9.81 MPa. The results of the test of absolute air permeability B_a , s, was calculated by the formula:

$$B_a = t - t_0 \quad (1)$$

Where, t – time, 100 cm^3 of air spent for transmission, s; t_0 - time of expiration of 100 cm^3 of water from the device without a sample, c.

The final result was calculated as the arithmetic average of the two determinations. Relative breathability found by the formula:

$$B_0 = \frac{V \cdot 3600}{S B_a} \quad (2)$$

Where, V - the volume of air passed through the sample (according to the test conditions is 100 cm^3); S - working area of the sample, equal to 10 cm^2 ; B_a - absolute breathability, with, s [7].

Tests of permeability under static conditions. Water resistance and water permeability under static conditions are determined on the basis of GOST 938.21 - 71. This standard applies to all types of leather and establishes a method for determining water resistance and water permeability when testing leather samples in static conditions. Permeability is characterized by the amount of water that has passed through a wet sample. Water resistance and water permeability of leather samples are determined on a PVA - 2 device.

To test the sample, 2 ml of water was passed through it (provided that it took no more than 2 hours), after which the water level on the graduated tube of the instrument was noted with an absolute error of 0.05 ml and the time was recorded on the counter. Then, 5 ml of water was passed through the sample and its level in the tube and time were again recorded. From the water level and time stamps, the amount of water that passed through the sample in the relevant time was calculated. When testing more dense samples, at first they were left under the pressure of water for 2 hours (during this time no more than 2 ml of water should pass through the sample), after which the water level in the tube and time were noted. Then the level was noted after 2 hours. If an insignificant amount of water passed through the sample for 2 hours, the tests continued for another 2 hours. With each sample, at least two determinations were carried out, the difference between which did not exceed 10% of the average value. The test shall be carried out at a temperature of 20 ± 2.0 °C.

Permeability B , $\text{ml} / (\text{cm}^2 \cdot \text{h})$, was calculated by the formula:

$$B = \frac{V \cdot 60}{tS} \quad (3)$$

Where, V - amount of water, passed through the sample during the test, ml; t - test time, s; S - sample area, cm^2 [17].

Optical microscopy. Microscope Trinocular model KXL-2001 (with 5 mR digital camera) / Trinocular microscope model KXL-2001. The binocular head is rotated 360 ° C and tilted 45 ° C with antifungal coating for lenses. The system was focused by a coaxial system with a planetary gears having a lens of 100 × and a pair of eyepiece 10 ×. Leather samples for microscopic studies were previously fixed with a 10% formaldehyde solution. For fixation, the samples were cut into pieces of size 20 × 20 mm, the amount of fixing liquid was taken from the calculation of $LC = 100$, the processing time was 24 hours. In order to clearly distinguish the individual elements of the structure of the objects under study, they were contrasted, using the ability of these elements to be colored selectively. Orsein is chosen because it paints elastin fibers in brown, and collagen fibers in pinkish brown. When stained, the washed sections were kept for 3-5 minutes in the solution of orsein. Then the slice was placed on a glass slide, filled with 2-3 drops of a mixture of glycerin and gelatin and covered with a cover glass and inspected and filmed

IV. RESULTS AND ITS DISCUSSION

Control samples of leather were also treated by the standard method in the presence of known used fillers such as barium chloride, syrup and potassium alum. For all treatment options used 50% solution of fillers.

In fig. 1 shows the products of pure and modified protein hydrolyzate of chrome-plated leather waste.



A



B



Fig. 1. Products of protein hydrolyzate of chrome-plated leather waste:

a - unmodified protein hydrolyzate; *b* - modified protein hydrolyzate with acrylic acid; модифицированный; *v* - protein hydrolyzate with acrylic acid plasticized with glycerin.

In tab. 1 shows the results of the study of the diffusion of the fillers of experimental and control samples of leather with the front and bakhtarmy side.

Table 1 Diffusion of the fillers of experimental and control samples of leather with the front and bakhtarmy side

Filler	Depth of penetration,%	
	from the front	from the bakhtarma surface
Water	97	100
Barium chloride	63	70
Aluminum-potassium alum	90	95
Syrup	68	77

Protein hydrolyzed chrome leather waste	70	76
Modified protein hydrolyzate with acrylic acid	72	78
Modified protein hydrolyzate with acrylic acid plasticized with glycerin	76	80

The depth of penetration of the filling preparations was determined on the basis of microscopic examination of leather sections, and was measured with an KXL-2001 microscope microscope eyepiece. The results of the observations are presented in Fig. 2

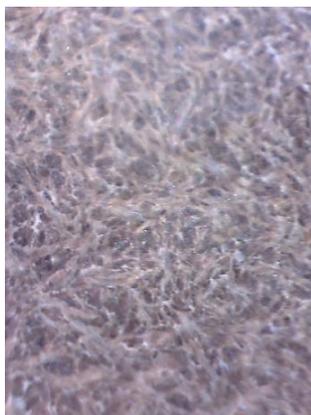
As established from visual observations, intense diffusion of filling drugs throughout the entire leather section is observed in the case of using barium chloride and potassium alum. This is due to the fact that these reagents have a low molecular weight and moreover are true solutions.

Since the main part of the molasses is crushed in the face layer due to its uneven absorption by the leather, the front surface of the leather was sticky and spots appeared on it.

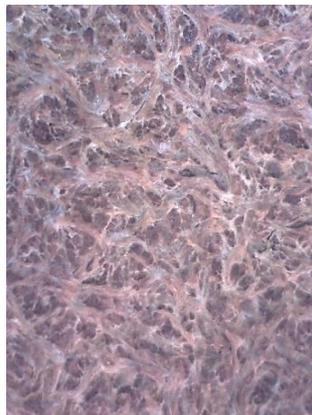
When leathers are treated with protein hydrolysates and products of its modification, possessing a high reactivity, diffusion of filling preparations occurs moderately, and its binding to the substrate occurs slowly, which contributes to their heavy migration.

Crucial importance of preserving the air and water permeability of natural leather is the nature of the distribution of filling preparations in the structure of the dermis.

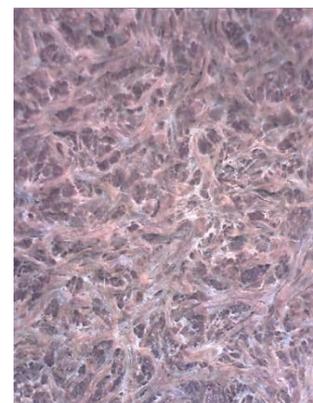
As follows from the micrographs, filling the leather with the studied drugs leads either to a discrete distribution of filler particles on the fibers of the leather, or to enveloping the surface of structural elements with a preparation film. Apparently, stereotypical non-repeating, dense and vigorous chaotic films in the form of agglomerates are formed on the surface of the fibers of the leather.



a



b



v

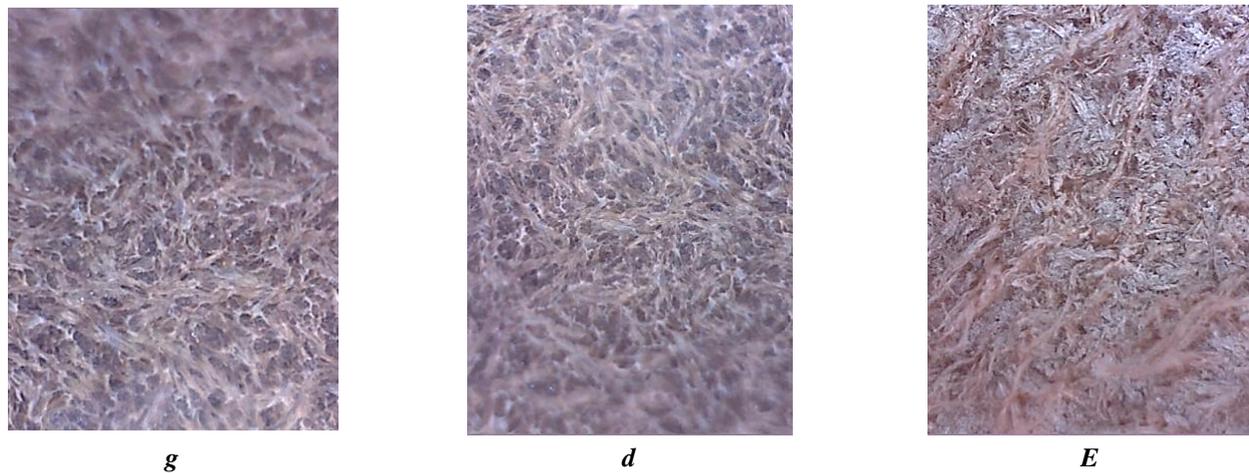


Fig. 2. Micrographs of the structure of chrome tanned leather (magnification × 2000): barium chloride filled (a), molasses (b), aluminum-potassium alum (v), protein hydrolyzate chrome waste leather (g), modified protein hydrolyzate with acrylic acid (d) and modified with protein hydrolyzate with acrylic acid plasticized with glycerin (e).

In contrast to the above treatments, leather filling with drugs such as barium chloride, molasses, potassium alum, protein hydrolyzed chrome-plated leather waste, modified protein hydrolyzate with acrylic acid, modified protein hydrolyzate with acrylic acid, plasticized with glycerol, leads to a discrete distribution of filling, and in the case of a modified protein hydrolyzate with acrylic acid, the plasticized glycerin on individual elements of the structure can be observed its kind of "caps" of the filling drug. It should be borne in mind that when processing modified protein hydrolyzate with acrylic acid, plasticized glycerin results in a significant decrease in water impermeability and leather soak, which may result in a change in the supramolecular structural elements of the leather.

As is known [18,19], the nature of the distribution of pores by effective diameters in combination with the total porosity and chemical nature of the filler have a significant impact on the hygienic properties of the leather, in particular on steam and air permeability, which is confirmed by the data given in Table . 2

Table . 2 Hygienic properties of the leather filled with various functionally active substances

The name of the fillers		Hygienic indicators		
		Porosity, % *	Air permeability, cm ³ / (cm ² · h)	Vapor permeability%
controlling	Barium chloride	42	459	68,9
	Aluminum-potassium alum	37	515	63,4
	molasses	48	382	69,3
Experienced	Protein hydrolyzed chrome leather waste	54	364	71,6
	Modified protein hydrolyzate with acrylic acid	57	242	78,4
	Modified protein hydrolyzate with acrylic acid plasticized with glycerin	56	235	84,2

*Note. * The porosity of the samples was determined by the weight pycnometric method using kerosene.*

From the data table. 2 it follows that the porosity of the prototypes of the leather and, consequently, their air permeability after filling decreases in comparison with the control samples.

So, when leather is treated with modified protein hydrolyzate with acrylic acid plasticized with glycerin, deposited on the surface of structural elements in the form of a film, air permeability decreases about 2 times, and these samples are also characterized by greater porosity. With a discrete distribution of fillers in the leather structure, the vapor permeability indices change accordingly, depending on the type and nature of the materials used.

Table . 3 The wetness of the leather samples filled various functionally active substances

2-hour wetness, % *		Leather samples					
		controlling			Experienced		
		Unfilled	Aluminum-potassium	molasses	Barium chloride	Protein hydrolyzed chrome leather waste	Modified protein hydrolyzate with acrylic acid
Original	74,5	62,5	63,4	51,8	39,3	38,7	38,2
Watering and drying cycle	I	76,8	62,7	64,0	52,1	39,8	39,3
	II	76,1	63,1	60,3	52,6	40,0	39,8
	III	78,3	63,5	60,7	52,9	40,3	40,1
	IV	79,2	63,9	61,4	60,3	40,7	40,9

The vapor permeability of prototypes of leather in individual cases exceeds the control ones, which is probably explained by the alternation in micro, meso and macropores [20-21] of hydrophilic and hydrophobic areas, which is one of the conditions for vapor permeability of finely dispersed structures.

The water resistance of the leather treated with the investigated fillers also confirms the fact that repeated cycles of watering (soap and soda treatment) and drying of the leathers practically do not change the characteristics of wetting, and in some cases reduce the leather's ability to absorb water (Table 3).

This fact can be explained by the fact that after several soap-soda treatments simultaneously with the washing out of the injected substances, fillers are fixed in the leather.

V. CONCLUSIONS

The reduction of leather namostiemosti, treated with modified protein hydrolyzate with acrylic acid plasticized with glycerin, occurs, obviously, as a result of additional polymerization of low molecular weight fractions of unsaturated monomer present in the technical product, catalysable bases in the process of soap and soda treatments.

A moderately uniform distribution is observed when leather is treated with protein hydrolysates and products of its modification.

It has been established that filled leather with protein hydrolysates and products of its modification to the surface of microfibrils fibers of the leather's fabric are covered with a film of filling preparations. It has been shown that stereotypical non-repeating, dense and vigorous chaotic films in the form of agglomerates are formed on the surface of the fibers of the leather.

It was revealed that for a more complete preservation of the hygienic properties of the leather during the filling process, it is necessary to achieve uniform distribution of the fillers in the leather structure. The hydrophobic preparations from the studied ones should be referred to such filling reagents as collagen hydrolysates and products of its modification

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