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Influence of Sorption and Vapor Permeability in Providing Normal Conditions when Operating Hydrophobized Footwear

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ABSTRACT

The analysis showed that natural experimental hydrophobized skin in all treatment options, unlike the control one, is able to remove a significant amount of moisture from the shoe area due to its high sorption ability. However, a comparison of the human skin's moisture loss with the sorption capacity of natural skin showed that sorption capacity alone is not enough to completely remove the released moisture. Since the possibilities of increasing the sorption capacity of genuine leather are extremely limited, the only way to improve the hygienic properties of shoes made from genuine leather is to less disturb the high permeability of this natural material as little as possible.

KEYWORDS: *vapor permeability, hydrophobized, polyvinyl ethinyl-dihydroxychlorosilane, moisture exchange, sorption properties.*

Introduction

In the face of increased competition in the global shoe markets, the domestic footwear industry is faced with the task of developing and producing competitive leather waterproof shoes with improved consumer and performance properties. The desire to create comfortable, lightweight shoes that would enjoy walking and improve people's quality of life is the "philosophy" of the modern shoe industry, and genuine leather that most fully meets the operational, hygienic and aesthetic requirements for shoes will remain the main one for a long time material in its production. [1-3].

Numerous studies are known in this area, but so far the question of the role and influence of various factors in ensuring comfort in shoes remains debatable. Most researchers believe that the decisive role in ensuring comfortable conditions belongs to moisture exchange processes - the ability of materials to absorb and pass water vapor - sweat [4-5].

Other authors believe that researchers pay little attention to the root causes of increased sweating due to the influence of external factors on the sweat glands and nerve endings of the human body [6]. Therefore, in the study of the hygienic properties of materials, the primary task, in our opinion, is to study the process of moisture transfer in the system "human foot - shoes - environment" and the identification of factors affecting this process.

At present, it can be considered with certainty that the degree of hydrophilicity of the polymer and the nature of its porous structure are the most important factors contributing to the removal of moisture from inside the shoe space. In this regard, the establishment of a relationship between the sorption and vapor permeability of materials plays a significant role in understanding the processes that ensure the hygienic properties of the material.

Experimental part. In the present work, the possibilities of obtaining new water repellents and their application in the skin finishing technology were investigated using various chemical materials and systems. For this purpose, various natural film-forming agents, film-forming polymers, plasticizers and other auxiliary materials were used in this work.

Objects of study. Polymethylsiloxane liquids (PMS-100, PMS-200) are transparent, chemically inert liquids. They are a mixture of polymers of linear structure $(CH_3)_3Si[Osi—(CH_3)_2]_n$,, where n = 3-700. Density at a temperature of 20 ° C was in the range of 0.98-0.99 g / cm^3, boiling point 300 ° C, pour point 62-64 ° C [7].

A surfactant preparation (OP-10) (a mixture of ethoxylated dialkylphenols) $C_nH_{2n+1}C_6H_4O(C_2H_4O)_mH$, where n = 9-10, m = 10-12 is a light yellow oily paste, the pH of the aqueous solution is 7-8. The clarification of the aqueous solution occurred at a temperature of 90 ° C [7].

Acrylic emulsion No. 1 is an aqueous dispersion of polymethyl acrylate, plasticized during the polymerization with a small amount of dibutyl phthalate.

Methacryl emulsion, 20% – an aqueous dispersion of polymethyl acrylate, plasticized during the polymerization process with a small amount of dibutyl phthalate [8].

Polyacrylamide is a white, odorless polymer; soluble in water, formamide, glacial acetic and lactic acids, glycerin; swells in propionic acid, propylene glycol, diethyl sulfoxide; insoluble in methanol, ethanol, acetone, hexane. Tglass ≈ 200 °C, molar mass reaches ≈1·106.

Hydrocarbons are organic compounds consisting of carbon and hydrogen. Hydrocarbons serve as the fundamental basis of organic chemistry: molecules of any other organic compounds are considered as their derivatives. The ratio between carbon and hydrogen in hydrocarbons varies widely (10-90%).

Hydrocarbon compounds differ from each other:

Polyvinylethinyldihydroxychlorosilane [8]. In a four-necked flask with a volume of 500 ml, equipped with a mechanical stirrer with a shutter, a thermometer, a dropping funnel and a reflux condenser, 103.78 ml (74 g. 1.0 mol) of ethyl ether (or 88.73 ml. (78 g. 1.0 mole) of benzene). Then, 53.29 ml were poured with stirring. (85 g. 0.5 mol.) Silicon tetrachloride. After that, the temperature was raised to 60 ° C and 68.42 ml was added portionwise. (77.5 g. 0.5 mol.) Vinylethynyl magnesium bromide for 45-50 minutes. Then, with vigorous stirring, the temperature was raised to 70 ° C, the reaction continued for another 4.0-5.0 hours. After time, the stirrer was stopped, allowed to cool for 2.0 hours and cooled to 10.0 ± 2.0 ° C.

Then, in small amounts, the reaction mixture was transferred to a separatory funnel, washed several times with double-distilled water (until chlorine ions appeared in the washing water, with a breakdown with silver nitrate) to remove magnesium chloride.

Ethyl ether solvent was distilled off by vacuum distillation at a temperature of 35.6 °C (or benzene at 80.1 ° C), using a vacuum pump, with a pressure for ethyl ether of 1.8-2.0, and in the case of benzene, 5.0-5.2 mm.

The resulting vinyl ethynyl trichlorosilane was introduced into the sump and 81.63 ml was released from there. intermediate in the mixer. 182.57 ml was added to the mixer with stirring of vinylethynyl trichlorosilane. water,

within 2.0 hours at a temperature of 146 ± 5.0 ° C for the purpose of hydrolysis of the intermediate.

The hydrolysis of vinylethynyldihydroxychlorosilane was 182.565 ml. water for 2.0 hours in a temperature range of 146 ± 5.0 ° C.

Then, the target product of vinyl ethynyl dihydroxychlorosilane was subjected to distillation, and a product of polyvinyl ethinyl dihydroxylorsilane with ethyl ether of 75.46 ml (58%) was obtained in the case of benzene 70.60 ml (58%). The final product, polyvinyl ethinyl trichlorosilane, had a boiling point of 96 ° C / 6 mm T. Art., With characteristics n20D 1.4560 and d204 1.0183.

For treating with water repellents, the “yalovka” chrome-tanned leather was selected

Experimental technique

Methodology for the preparation and use of water repellents

Production technology. Water repellents were prepared by sequential mixing of the starting materials at various ratios (wt.%) As an experimental option: acrylic emulsion-20% 40-70, PVEDGOHS-80% 2-12, industrial oil, IA-20 12-53, penetrator 4-6 and control polyethylhydrosiloxane (GKZh-94) 100 at a temperature of 20–22 ° C for 3-4 hours.

Technology application. Application by plush brush: not diluted, or diluted in a 1: 3 ratio. Spraying: diluted in an aqueous medium in a ratio of 1: 2 is sprayed in 1-2 passes from a distance of 20-30 cm. It should be noted that the action of the hydrophobic composition occurs immediately after the complete evaporation of the solvent. Liquid formulations do not change the tone of dyed skin.

Methodology for studying the properties of water repellents

Viscosity. To determine the viscosity of sample solutions, methods were used based on measuring the flow rate of a certain volume of liquid through a capillary, as well as measuring the resistance provided by a solution of a certain concentration.

Determination of conditional viscosity of water repellents. To determine the viscosity of water repellents, aqueous solutions of standard concentration were prepared. Standard solution of water repellent (GOST 3252-80) in terms of anhydrous and ashless product [8]. It has been established that the determination of the conditional viscosity of the studied water repellents is conveniently determined on a VZU viscometer, because changing the diameter of the fluid passage on the indicated viscometer a wide range of definitions.

The nominal viscosity of the tested water repellent ($^{o}E^{40}_{20}$**)** was calculated by the formula:

By = t_1 / t_2,

where t_1 is the expiration time of water repellents at a temperature of 40 ° C; t_2 - time of water outflow at a temperature of 20 ° C.

The viscosity was calculated with an error of not more than 0.1 about. The discrepancy between parallel determinations did not exceed 0.1% [8].

Determination of the density of water repellents by the pycnometric method. The density P^{20}_{4} was calculated by the formula

$P^{20}_{4} = (m_1 – m) / V$,

where, m is the mass of the empty pycnometer, g; m1 is the mass of the pycnometer with the analyte, g; V is the volume of the pycnometer determined by calibration, cm3 [86].

Sorption properties of the samples — the study was carried out on a McBan vacuum balance with a quartz spring, in the range from 0-100% relative humidity, at 25 °C [6].

Determination of density and porosity. Density in kg / m3 is one of the main physical properties of the material, which is the ratio of the mass m of a substance to its volume V:

$\rho = m / V$

For porous materials (skin), apparent and true density are distinguished. "Apparent" density is the ratio of the mass of the sample of the skin to its total volume, including the pore volume. Such skin density depends on the pore volume in them, i.e. from the type of raw material from which they are produced, as well as from technological processes of production. Since the apparent density is associated with porosity, it can also be judged indirectly by the value of the skin’s ability to pass air, water, and heat. Determining the apparent density of the skin comes down to determining the mass of the samples and their volume.

Determination of breathability. Breathability is determined on the basis of GOST 938.18-70. This standard applies to all types of skin and establishes a method for determining breathability. A device was used to determine breathability.

The breathability of the skin and skin tissue of the fur characterizes their ability to pass air at a pressure difference on both sides of the test sample.

The air permeability index, $cm^3 / (cm^2 \bullet h)$, is the volume of air in cubic centimeters passing through 1 cm^2 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 absolute breathability test B_a, s, were calculated by the formula:

$B_a = t - t_0$

where t is the time taken to pass 100 cm^3 of air, s; t_0 is the outflow time of 100 cm^3 of water from the device without a sample, s.

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

$B_0 = V \cdot 3600 / S\ B_a$

where, V is the volume of air passing through the sample (according to the test conditions it is equal to 100 cm^3); S is the working area of the sample, equal to 10 cm^2; B_a - absolute breathability, s. [9].

Water permeability tests in static conditions.

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

To test the sample, 2 ml of water was passed through it (provided that it would take no more than 2 hours), after which the water level was noted on the graduated tube of the device 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 noted a second time. The water level and time were used to calculate the amount of water that passed through the sample for the corresponding time. When testing denser samples, at first they were left under water pressure 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 in 2 hours, the tests continued for another 2 hours. At least two determinations were made with each sample, the difference between them did not exceed 10% of the average value. The test shall be carried out at a temperature of 20 ± 2.0 ° C.

Water permeability B, $ml / (cm^2 \bullet h)$, calculated by the formula:

$B = V \cdot 60 / t S$

where, V- is the amount of water passing through the sample during the test, ml; t- is the test time, s; S is the sample area, cm^2 [9].

Determination of wetting under static conditions [9] . The weight wetting of Hb was determined in the same way as the moisture capacity, but the calculation is made not on the mass of absolutely dry skin, but on the one containing 18% moisture.

For this, after determining the moisture content in the sample, its mass was calculated at a moisture content of 18% q_1:

$q_1 = q (100 - B) / 18$

where, the B-moisture content in the sample before testing,%; q -is the mass of the sample with a moisture content of, g

Weight wetness Hb% (2- or 24-hour), was determined by the formula:

$H_в = q_{2, 24} - q_1 / q_1 \cdot 100$

where q_2, 24 is the mass of the sample after getting wet in water, g; q_1 is the mass of the sample at a moisture content of 18% (before getting wet), g. To recalculate the weight wettability of the skin to the moisture capacity, we used the formula:

$B_{2, 24} = H_в + 18 / 100 - 18 \cdot 100$

The moisture capacity was recalculated to the wet weight by the formula:

$H_{2, 24} = 0{,}82 B_{2, 24} – 18$

Determination of vapor permeability. Vapor permeability is determined on the basis of GOST 938.17 - 70. This standard applies to all types of skin and establishes a method for determining vapor permeability. The following equipment and reagents are used to determine vapor permeability: technical scales in accordance with GOST 24104 - 2001; desiccators according to GOST 25336 - 82 with an inner diameter of 250mm; metal glasses 45 mm high and 55 mm in diameter.

Vapor permeability is the ability of the skin to pass water vapor. Vapor permeability is one of the most important indicators used to assess the hygienic properties of the skin. Vapor permeability is expressed in milligrams of moisture passing through 1 cm 2 of sample area in 1 h, or as a percentage of the mass of water vapor passing through the test sample to the mass of water evaporated from an open surface under the same conditions.

Vapor permeability was determined in special metal cups on round samples with a diameter of 55 mm; the diameter of the working part of the sample is 36 mm.

The relative vapor permeability P_0,%, was calculated by the formula:

$P_0 = q \cdot 100 / q_1$

where, q is the decrease in mass of the glass with the contents after 6 hours of testing, g; q_1 is the arithmetic mean of the decrease in the mass of control glasses with water, g.

In the case of vapor permeability of the samples, P must be expressed in milligrams of moisture passing through the unit area of the sample per unit time, then the calculation was performed according to the formula:

$P = q / t\pi r^2$

where, q is the decrease in mass of the glass with the contents, mg; t is the duration of the experiment, h; πr^2 is the working area of the sample, cm^2. The arithmetic average of the test results of two samples taken from one sample was taken as the test results [9].

Results and discussion

At present, it can be considered with certainty that the degree of hydrophilicity of the polymer and the nature of its porous structure are the most important factors contributing to the removal of moisture from inside the shoe space. In this regard, the establishment of a relationship between the sorption and vapor permeability of materials plays a significant role in understanding the processes that ensure the hygienic properties of the material.

The object of the study are polymer-composite materials based on polyvinyl ethinyldihydroxychlorosilane, acrylic emulsion, penetrator, polyhydrosiloxane and others. The interaction of water repellents was studied on samples of genuine leather of chrome tanned tanning from cattle skins (GOST 939–88) made in Uzbekistan.

It seems to us that in order to determine the role of sorption and permeability of materials in the implementation of moisture exchange processes, it is necessary to compare the amount of human skin's moisture loss while wearing shoes with the ability of the material to absorb moisture and remove it through vapor permeability. Such a comparative analysis was carried out on the basis of a study of the sorption properties of natural hydrophobized skin using data from [10]

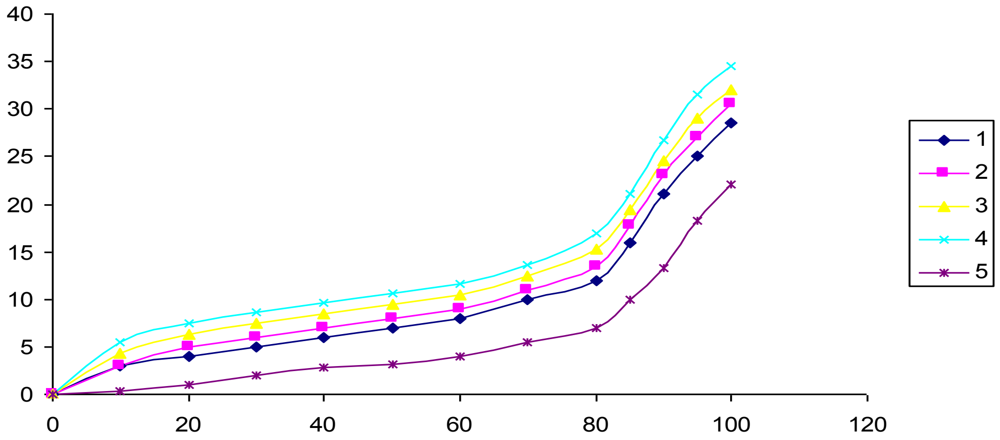

Fig. 1. Sorption isotherms (G) of natural samples 1, II, II, IV-Treated with a hydrophobized composition based on polyvinyl ethinyl dihydroxychlorosilane,V-control polyethylhydrosiloxane) skins at a temperature, ° C: - 20;

Sorption studies were carried out under conditions corresponding to the operating conditions of the shoe: at a temperature of 20 and 30 ° C for 7 hours.

The vapor permeability and moisture absorption of the samples were determined under nonisothermal conditions (temperature T_1 = 20 ° C and T_2 = 30 ° C; relative humidity φ_1= 60% and φ_2= 100%) for 7 hours (GOST 22900–78).

When calculating the moisture balance in the system, it is also necessary to take into account the fact that even before wear the material contained an equilibrium amount of moisture under the given initial conditions (φ = 40-50%, T = 20-210C in the environment). For genuine leather, this value is 5.5-6%.

If the surface area of the shoe upper is 5-6 dm2, and the weight is on average 50 g, then the equilibrium amount of absorbed moisture under the initial conditions for genuine leather will be about 3 g [11]

If we take the maximum sorption value (at φ_1= 100% and T = 20 ° C), which is equal to about 25% for natural skin (Fig. 1.), Then after 7 hours of wear the sample is able to absorb 12.5 g of moisture (3 of them g was already contained in the material before socks). Therefore, as a result of sorption, up to 9.5 g of moisture can be removed from the shoe space. At lower values of relative humidity and a higher temperature, which are usually found in shoes (see table 3.6), the amount of absorbed moisture will be even less. During the same time, from a steam source operating at a constant capacity, from 17.5 g (in the sitting position) to 40 g (when walking) of moisture is released into the intra-shoe volume. A comparison of these figures clearly shows (see Fig. 1.) that the sorption ability of genuine leather alone is clearly not enough to completely remove the released moisture from the shoes. From the analysis of the kinetics of sorption, it also follows that the active removal of moisture from shoes through sorption (in the absence of vapor permeability) can be carried out only up to a certain point.

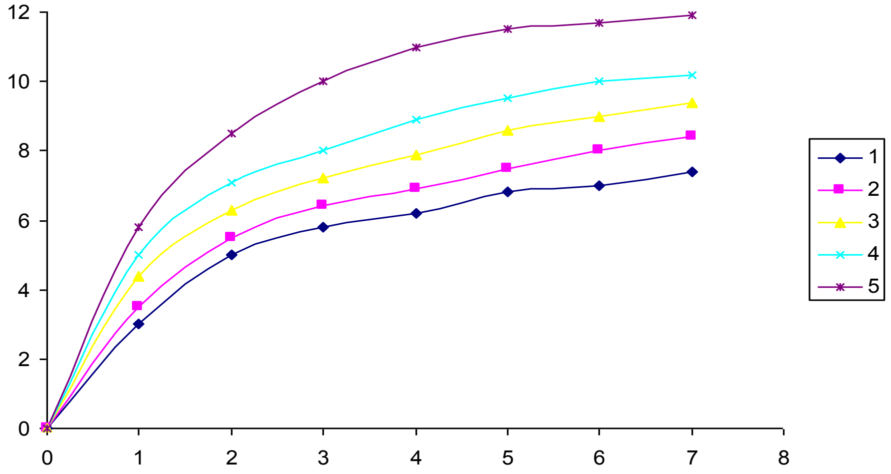

Fig. 2. Kinetic sorption curves I-IV - experimental and V - control components of water repellents. at relative humidity,%: 70 – 90

Fig. 2. kinetic curves of sorption are shown. According to the experimental data presented in this figure, the moisture content of natural leather samples was calculated, by weight equal to the workpiece for the upper shoe, and the result is shown in Fig. 3 .

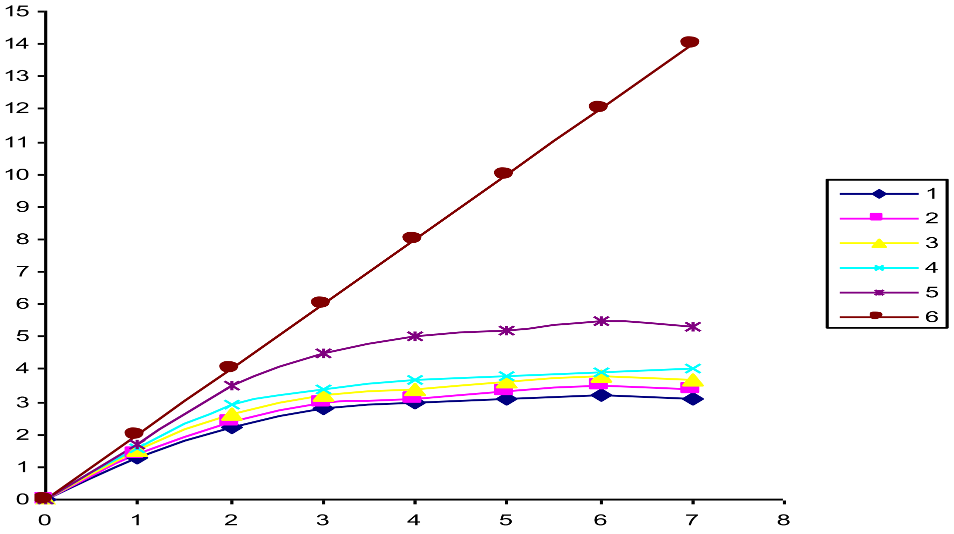

Fig. 3. Kinetic curves of skin moisture content I-IV - experimental and V - control components of water repellents.at relative humidity,%, temperature - 70 - 90C0;

The kinetic curves show two characteristic regions: fast and slow sorption. A steep rise in kinetic curves at the initial time may be associated with moisture diffusion into the porous structure and adsorption on the surface of the pore walls. For genuine leather - in 2-3 hours. The site of the slow rise of the kinetic sorption curves is associated with the absorption of water vapor by the polymer.

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The microclimate parameters in shoes during experimental wear according to the methods of Dibshlag [8] and Langmeier [2]

Table 1.

Test characteristics	Experienced				Control sample	Dibshl-agom	Langma yerom
	I	II	III	IV			
Relative humidity,%	40	40	40	40	40	40	50
Relative humidity inside the shoes,%	39	42	43	41	45	43-45	86
Ambient temperature, ^{0}C	20	20	20	20	20	20	21
The temperature inside the shoe, ^{0}C	28	29	27	28	30	30-31	27

It can be assumed that the insufficient sorption capacity of the upper materials of the shoes can be compensated by using lining materials from genuine leather in the manufacture of shoes. But a simple calculation by summing the values characterizing the hygroscopicity of the top and lining shows that although such a multilayer system has a higher sorption capacity with respect to water, it is not able to completely remove evaporating moisture from the shoe during the operating cycle (see Fig. 1.) Consider the case where the materials used to make shoes have some vapor permeability. From the work of Dibshlag [10], it follows that during 4 hours of operation (in the position of the toe sitting) in shoes made of genuine leather, a constant relative humidity of ~ 45% is maintained, which approximately corresponds to the humidity of the environment (see table 1). The closeness of the relative humidity values of the footwear space and the environment indicates that 2.5 g of steam arriving hourly into the footwear volume is almost completely eliminated. In our opinion, steam is removed from shoes in this case, mainly through vapor permeability: the material is not capable of significantly absorbing moisture, since before the socks it already contained its equilibrium amount at a humidity of 40%.

The conclusion obtained, however, does not mean that in this case, sorption does not affect the removal of steam from the shoes. Note that in permeable materials, sorption promotes the removal of moisture from the material in the presence of moisture and temperature gradients as a result of transfer in the adsorption layer: at low humidity, corresponding to a small filling of the surface of the skin structural elements with the adsorbate, surface diffusion takes place, and at a sufficiently high humidity, film flow.

In porous systems with a developed specific surface and hydrophilic pore walls, the quantitative effect of the sorption flow on the vapor removal can be significant.

Thus, the results obtained suggest that the role of vapor permeability of materials along with their ability to absorb moisture in the implementation of moisture exchange processes and in providing comfortable conditions when wearing materials is important and obvious.

Studies have shown that a decrease in vapor permeability of samples of natural hydrophobized skin, characterized by close values of total porosity.

A comparison of the values of vapor permeability, moisture absorption and hygroscopicity shows that the samples of natural hydrophobized skin moisture absorption does not exceed their hygroscopicity. The differences in the vapor permeability of such samples are mainly associated with the number of through pores having an exit to the surface of the material. Obviously, in these samples, steam is removed both by the diffusion mechanism in the gas phase through the pores and by transfer in the adsorption layer, the influence of which can be significant with a developed specific surface. [12-13]

It should be noted that in the process of moisture transfer (in laboratory conditions and when worn), steam condensation can occur both in small (with a radius of less than 0.1-0.2 μm), and in larger pores as a result of the temperature difference on the sample. However, in well-permeable materials, condensate does not form in macropores and moisture absorption does not exceed the hygroscopicity of the samples.

With a substantial overlap of the through pores during the application of finishing coatings, the vapor permeability of the samples decreases, and excess moisture accumulates in the shoe area and inside the material in the form of a liquid phase, creating a feeling of discomfort. [12-14] As observations showed, on samples of natural hydrophobized leathers characterized by vapor permeability below 3.5 mg / (cm^2 h), there were drops of condensed moisture after the test, and organoleptic such samples were characterized by increased humidity.

Conclusions

The analysis showed that natural experimental hydrophobized skin in all treatment options, unlike the control one, is able to remove a significant amount of moisture from the shoe area due to its high sorption ability. However, a comparison of the human skin's moisture loss with the sorption capacity of natural skin showed that sorption ability alone is not enough to completely remove the released moisture. Since the possibilities of increasing the sorption capacity of natural leather are extremely limited, the only way to improve the hygienic properties of shoes made of genuine leather is to less disturb the high permeability of this natural material as little as possible.

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