Modern American Journal of Engineering, Technology, and Innovation
ISSN(E): 3067-7939
Volume 01, Issue 05, August, 2025
Website: usajournals.org

INVESTIGATION OF SORPTION BEHAVIOR OF LEATHER PROCESSED WITH FILLERS OBTAINED FROM MODIFIED SILK HYDROLYSATE

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Abstract
This study investigates the sorption properties of leather treated with fillers obtained on the basis of modified silk hydrolysate. The research highlights the role of protein-based fillers in improving the structural and functional characteristics of chrome-tanned leather. Modified silk hydrolysate provides active functional groups that enhance the binding capacity of the filler with collagen fibers, thereby increasing water vapor permeability, moisture absorption, and hygienic performance.

Keywords: Leather, modified silk hydrolysate, fillers, sorption properties, collagen, eco-friendly technology

Introduction

During the process of filling leather materials with fillers based on modified silk hydrolysate, the optimal conditions are largely governed by the sorption-hygienic properties of the leather. These properties, in turn, are influenced by the development of the internal surface, the volume, number, and configuration of pores, as well as the degree of blocking of collagen active groups capable of interacting with vapors. Numerous studies on the sorption behavior of collagen

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and gelatin [1–2] have been conducted by various researchers for diverse applications.

Recognizing the usefulness of the data obtained from these studies, which contributed to expanding the understanding of structural changes in the collagen that forms the basis of leather and fur tissues, it should be noted that the available instruments and methods are not capable of detecting a number of subtle alterations in collagen [3]. The aim of this work is determined by the growing global focus on green production processes, in which sericin has become the object of research as a bioactive compound with potential applications in the leather and fur industry.

II. METHODOLOGY AND DISCUSSION

The main part of the research was conducted using the desiccator method. Studies on the formation of the porous structure of leather materials during the filling process with fillers based on modified silk hydrolysate, and its subsequent characterization, have shown that the structural framework is primarily formed during the filling stage. In this context, the most important parameters are the physical indicators of leather structure associated with porosity, which help to expand our understanding of the filling processes.

To investigate porosity within modern approaches, adsorption methods were applied to study the pore characteristics of imitation-enriched leather filled with modified silk hydrolysate-based fillers [4]. For the analysis of adsorption processes in leather, a Mac Ben-Bakra spring balance device was employed. The sorption of water vapor was monitored through independent weighing, which significantly enhanced the accuracy of the experiments [5].

Determination of density and porosity. Density is one of the fundamental physical properties of materials, representing the ratio of the mass of a substance to its volume. [6]. Density – p, kg/m^3

$$p = \frac{m}{V}$$

where, m – mass of the substance, kg;

V – volume of the substance, m³.

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The density of leather tissue reflects its porosity and the degree of filling. During technological processing, knowledge of the porosity level, pore size, and their distribution within the leather makes it possible to properly control process parameters and obtain leather and fur tissues with the desired properties.

Pores may have different structures: closed, blind, perforated, or loop-shaped. They can account for up to 50 m^2/g of the internal surface of leather and fur tissue. Based on the cross-sectional area, pores are classified into macropores, micropores, and ultramicropores. The number, size, and distribution of pores significantly influence such properties of leather and leather tissue as air permeability and water permeability.

For porous materials, including leather and fur tissue, there are two types of density: true density and apparent density. Apparent density is defined as the ratio of the weight of a leather or fur tissue sample to its total volume, taking into account the pores.

True density is defined as the ratio of the weight of a leather or fur tissue sample to the volume of its dense substance (without pores).

A 5–10 g leather or fur tissue sample is weighed and cut into strips measuring 20 cm in length and 2–3 mm in width. The crushed sample is placed into a 50 ml pycnometer (or a volumetric flask) and filled with kerosene up to the mark using a burette. The volume of kerosene poured, V_1, is measured, and the pycnometer is sealed with a stopper or filter paper and left for 24 hours. During this time, the pores of the leather or fur tissue are completely filled with kerosene, leaving a certain amount of kerosene in the pycnometer whose volume does not change. The pycnometer is then refilled with kerosene up to the mark, and the volume V_2 is measured.

The difference between the pycnometer volume V_0 and the sum of the first and second kerosene volumes poured equals the true volume of the dense substance of the leather or fur tissue:

$$V_{true} = V_0 - (V_1 + V_2)$$

The kerosene in the pycnometer is poured out, and excess kerosene on the surface of the crushed sample is gently blotted with filter paper (without pressing).

Then, the blotted pieces are placed back into the same pycnometer, and it is refilled with kerosene up to the mark.

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The difference between the pycnometer volume V0 and the third kerosene volume poured V_3 corresponds to the volume of the dense substance together with the pores of the leather or fur tissue, i.e., the apparent volume Vapp.

Knowing the true and apparent volumes, the density of the leather and fur tissue, the pore volume Vpore, and the porosity P can be determined:

$V_{pore} = V_{app} - V_{true}$

Porosity can also be calculated based on the apparent and true densities. **Sericin** is a natural hydrophilic protein derived from the silk cocoon (Bombyx mori). It consists of a polypeptide chain mainly composed of serine (30–40%), threonine, asparagine, glycine, and tyrosine. The molecular weight of sericin varies widely, typically ranging from 20,000 to 400,000 g/mol.

Structurally, sericin is a polypeptide in which amino acids are interconnected through peptide bonds (–CO–NH–). Although its exact molecular structure is undefined due to its polymeric nature, it can be generally represented as $–(NH–CH(R)–CO)_n–$, where R denotes amino acid side chains.

The presence of hydroxyl (–OH), amide ($–CONH_2$), and carboxyl (–COOH) functional groups imparts hydrophilicity and high water solubility, making sericin valuable for biomedical, cosmetic, and textile applications.

Glyoxal (ethanedial, diformyl, oxaldehyde) [6] is the simplest dialdehyde, with the chemical formula OHC–CHO. It appears as yellowish crystals or a liquid with a formalin-like odor.

Carboxymethylcellulose (cellulosoglycolic acid, simple ethers of cellulose and glycolic acid, known commercially as tylosa, valocel, blanose, edifas) [7] has the general formula $[C_6H_7O_2(OH)_{3-x}(OCH_2COOH)_x]_n$, where x = 0.08–1.5. It is an amorphous, colorless substance with weak acidity ($K = 5.25 \cdot 10^{-7} – 5.0 \cdot 10^{-5}$ at x = 0.1–0.8).

Plasticizers are substances used in polymer structures, particularly in leather collagen, to increase elasticity and softness. They penetrate between polymer fibers through physical and chemical interactions, thereby reducing the strength of intermolecular bonding. Chemically, plasticizers are hydrophilic (well miscible with water) or hydrophobic (poorly miscible with water) organic molecules, most commonly alcohols, ethers, ether-alcohols, polymers, or natural oils.

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III. EXPERIMENTAL RESULTS

Taking the above into account, our objective was set to develop a new method of leather tissue filling that would improve its physicomechanical and chemical properties, ensuring long-term durability of the product.

Table 1 presents various approaches for obtaining modified silk hydrolysate.

Table 1 Different variants of obtaining fillers based on modified silk hydrolysate (in % relative to the mass of leather tissue)

№	Components	Control	Experiment		
			I	II	III
1	Eurosyntan MC	2	-	-	-
2	Mimtan AT-1	5	-	-	-
3	Mimosa RG	3	-	-	-
4	Acid Black Jet 135%	5	5	5	5
5	Silk hydrolysate (sericin)	-	1	4	7
6	Carboxymethylcellulose	-	2	2	2
7	Glyoxal	-	3	3	3
8	Plasticizer (castor oil)	-	1	1	1
9	Water	65	68	65	62

Note: The liquid coefficient is 0.8.

In this study, our main focus was on the physical parameters of leather structure related to porosity during the filling processes, with the aim of broadening our understanding of these characteristics. In modern approaches, one of the methods for studying porosity is the adsorption technique, which was applied here to investigate the porosity of leather filled with fillers based on modified silk hydrolysate, simulating enriched leather structures.

To examine the adsorption processes in leather, a Mak Ben-Bakra spring balance device was employed. The sorption of water vapor was monitored through independent weighing, which significantly increased the accuracy of the experiment.

For this research, in order to study the porous structure of leather treated with fillers, we selected samples of bovine hides, both as control and those treated with fillers based on modified silk hydrolysate.

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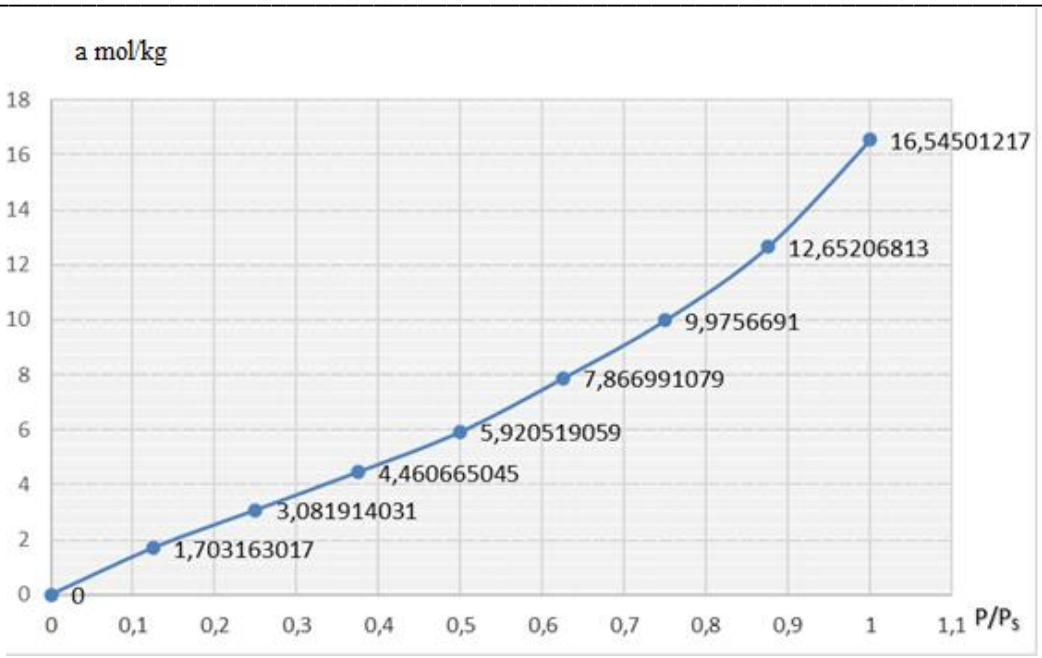

Figure 1. Dynamics of water vapor sorption processes in leather tissue treated under Variant I compared with the control.

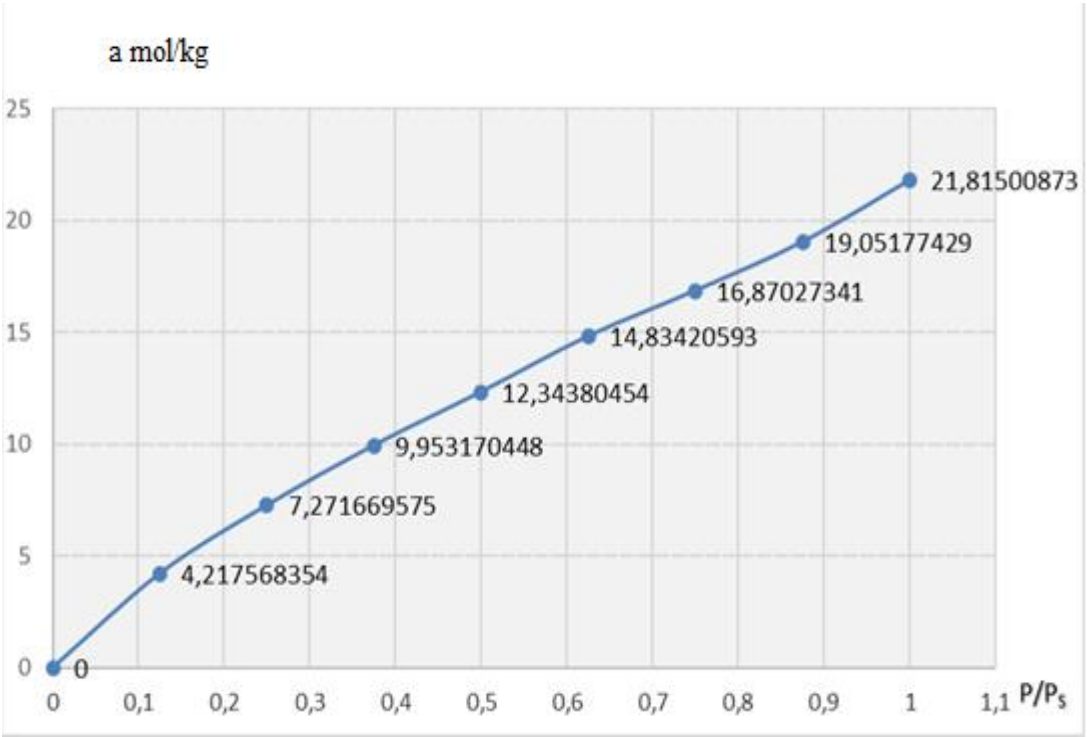

Figure 2. Dynamics of water vapor sorption processes in leather tissue treated with fillers based on modified silk hydrolysate (Variant II).

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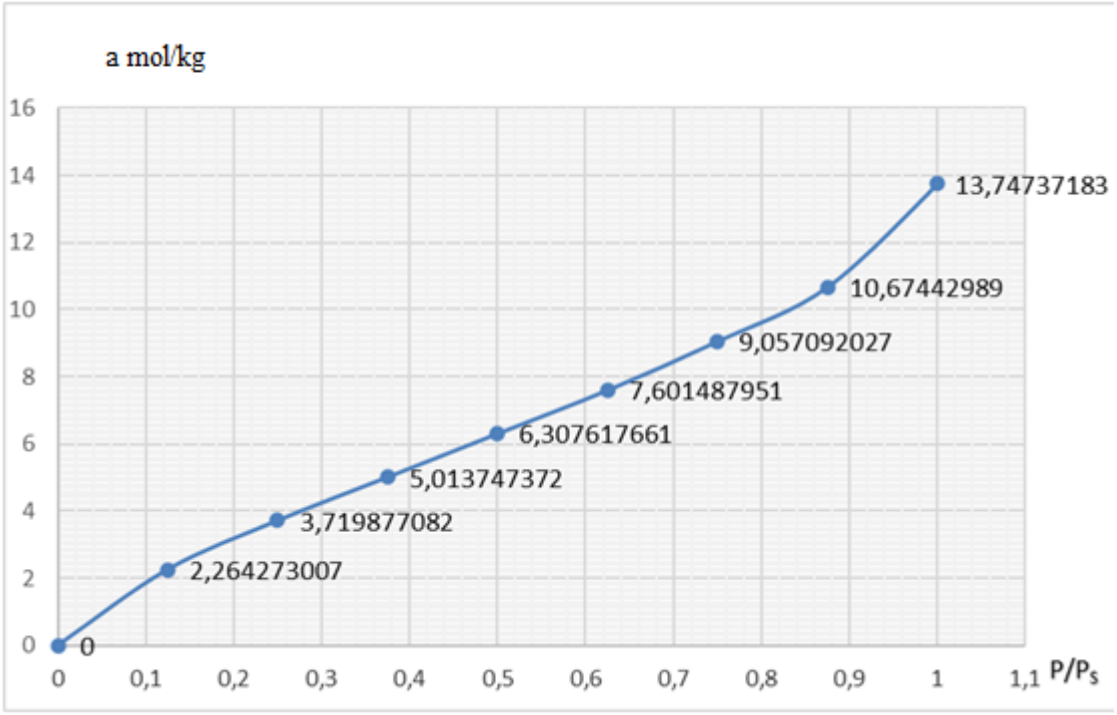

Figure 3. Dynamics of water vapor sorption in leather tissue treated with fillers based on modified silk hydrolysate (Variant III).

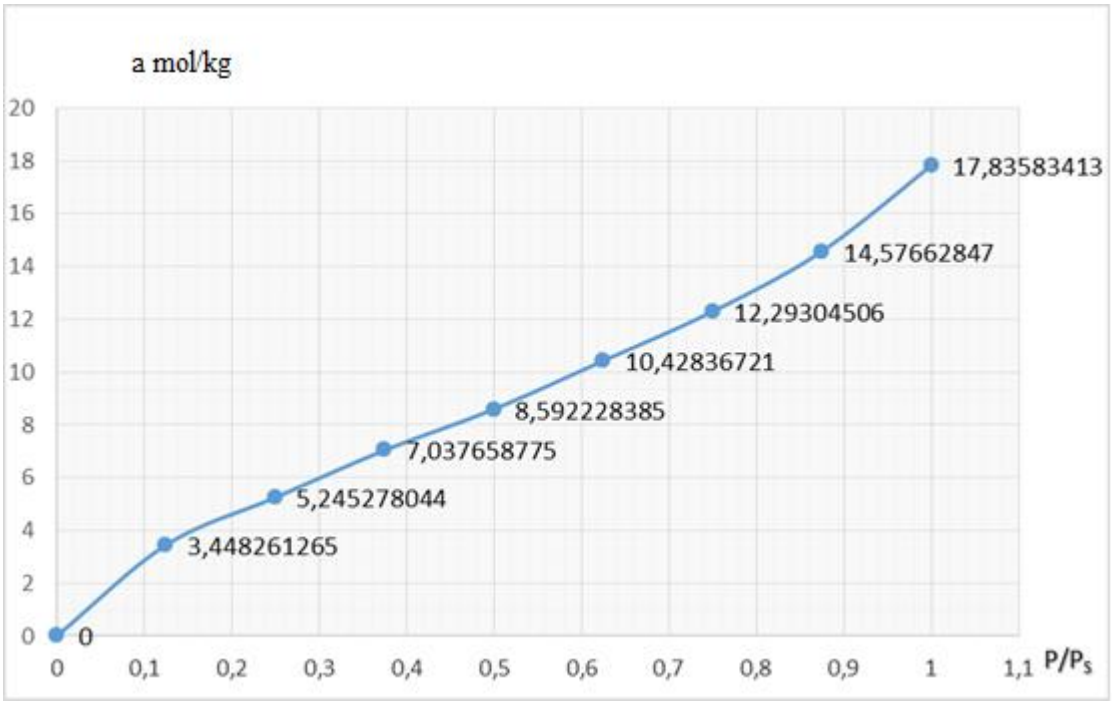

Figure 4. Dynamics of water vapor sorption in leather tissue treated with fillers based on modified silk hydrolysate (Variant IV).

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The specific surface area (S) among the porosity parameters of the adsorbent structure was determined using the Brunauer–Emmett–Teller (BET) theory equation. In this case, when the ordinate is plotted as P/P_s / $a(1–P/P_s)$ and the abscissa as P/P_s, a straight-line coordinate is obtained. The specific surface area of the adsorbents was calculated using the following formula [8]:

$S=am \cdot NA \cdot \omega 0$

S – specific surface area (m^2/g);

am- monomolecular layer (mol/kg);

NA- Avogadro's number;

ω0- surface area occupied by a single molecule (mm^2).

The results obtained from adsorption experiments and calculated according to the BET equations are presented in Table 2.

Table 2 Sorption characteristics of the porous structure of leathers

Indicators		**Leather treated with fillers obtained on the basis of modified silk hydrolysate**			
		Control	**Experiment**		
		1	2	3	4
Monolayer capacity A_m, mol/kg		2,490	2,888	5,737	4,088
Specific surface area S, m^2/g		161,91	187,77	373,01	265,81
Micropores W_o, cm^3/g		0,178	0,164	0,303	0,221
Saturation volume Vc, cm^3/g		0,297	0,246	0,392	0,320
Mesopores Wme, cm^3/g		0,12	0,08	0,09	0,10
Pore radius rk, Å	Å	36,7	26,3	21,0	24,1
	nm	3,67	2,63	2,10	2,41

The sorption characteristics and the modifications in the porous structure of leather fabrics treated with fillers derived from modified silk hydrolysate reveal a marked enhancement of their active surface properties, adsorption potential, and the formation of capillary networks [9]. Among the fundamental parameters that reflect mass transfer capacity is the monolayer capacity. Under the influence of silk hydrolysate, this parameter increased more than twofold compared with the control, indicating a significant rise in the number of active adsorption centers within the leather matrix. In particular, the maximum monolayer capacity of 5.737

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mol/kg confirms the development of highly active surfaces and the formation of thin, functional structures within both the external and internal architecture of the leather [10–11].

A similar trend was observed in the increase of the specific surface area. Notably, in the second experimental sample treated with the filler based on modified silk hydrolysate, the surface area expanded up to 373.01 m^2/g. This considerable growth indicates the intensification of adsorption processes across the leather's overall surface as well as an increase in the density of microporous structures. The observed increase in specific surface area demonstrates that the enhancement of mass transfer processes occurred not only on the outer surface but also throughout the micropores within the leather tissue [11–12].

The variations in micropore volume reflect fundamental transformations within the internal structure of the leather tissue. In the control sample, the micropore volume was 0.178 cm^3/g, while in the second experimental sample of leather treated with a filler based on modified silk hydrolysate, this value increased to 0.303 cm^3/g due to the formation of additional chemical bonds. This indicates the development of ultrafine (nanoscale) capillary channels and thin porous structures within the leather matrix [13]. Such structures not only enhance the adsorption capacity but also improve the rate and extent of substance penetration into the tissue, thereby facilitating the effective incorporation of various functional molecules into the leather.

Similar trends were observed in the saturation volume. While the control sample exhibited a value of 0.297 cm^3/g, treatment with the modified silk hydrolysate-based filler raised this indicator to 0.392 cm^3/g. This increase demonstrates the enhanced overall sorption capacity of the leather, meaning its ability to absorb and retain larger amounts of substances or liquids. Such a property is of practical importance in terms of moisture retention, liquid uptake, and absorption of active agents.

By contrast, mesopore volume showed relatively minor changes, which highlights the greater contribution of micropores and indicates that microporous structures play the dominant role in the sorption process. In the fourth experimental sample, the mesopore volume stabilized at 0.10 cm^3/g, confirming the persistence of this structural balance.

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The reduction in pore radius represents another indicator of the effectiveness of chemical bonding. Its decrease from 3.67 nm to 2.10 nm reflects the formation of highly dense, thin, channel-like structures within the leather matrix. These structures amplify capillary forces, thereby significantly accelerating the penetration of substances into the tissue [14]. As a result, both air and liquid permeability of the leather are enhanced, imparting a "breathable" property to its surface.

IV. CONCLUSION AND FUTURE WORK

In summary, fillers obtained on the basis of modified silk hydrolysate considerably intensify the microstructural activity of leather tissue, creating sorption centers both on the surface and within the bulk volume, thereby substantially enhancing its adsorption performance. This improvement is not limited to the increase in sorption capacity but also extends to the activation of mass transfer processes, which allow liquids, gases, and functional molecules to penetrate more effectively into the leather matrix.

The modified structures demonstrate clear advantages in terms of mechanical stability, maintaining the strength and flexibility of the material while simultaneously improving sanitary-hygienic and technological characteristics. Such properties are of great significance in the production of functional leather goods, including footwear, clothing, accessories, and protective materials, where both durability and comfort are equally important.

From a structural perspective, the presence of active amino acid groups in silk hydrolysate enables strong interactions with the collagen fibers of leather, resulting in the development of a thin, uniform, and well-balanced porous architecture. This not only increases the number of active adsorption centers but also enhances the diffusion of moisture and active agents throughout the leather. As a consequence, the material acquires a "breathable" quality, which is especially valuable for hygienic and biomedical applications.

Furthermore, the method has significant technological implications. The ability to form finely tuned porous structures within the leather matrix demonstrates the scientific validity of the approach and its practical potential for industrial application. Such leathers can be used in the design of high-performance materials

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with improved moisture-retention capacity, resistance to microbial growth, and enhanced comfort in wear. These findings confirm that the use of modified silk hydrolysate-based fillers represents a scientifically sound, environmentally friendly, and practically effective strategy for the development of advanced leather materials.

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