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Investigation of the Effect of a Natural Extract From Oak Bark on the Properties of the Leather

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Abstract
Researchers are constantly looking for the use of new and effective types of natural dyes to reduce the impact of harmful factors from the use of synthetic dyes. In this study, the effects of a natural plant extract based on oak bark, used as a dye during the finishing process, on the physico-mechanical and hygroscopic properties of the leather were studied. Extraction was carried out by the traditional method without chemical additives using only distilled water and oak bark. The application of finishing solutions based on a standard chemical pigment and oak bark extract was carried out using an automatic rotary spray machine in combination with a drying tunnel. The physico-mechanical properties of colored leathers based on oak bark extract were determined in comparison with a standard chemical pigment and with a sample without finishing. The hygroscopicity of the leather samples were also evaluated, such as: water absorption, water penetration and color fastness to water spotting.
The results of the conducted studies have shown that the samples stained with a natural dye have good physico-mechanical and water resistance properties.

Keywords
leather, plant extract, oak bark, physico-mechanical properties, hygroscopicity of the leather, water resistance.

1. Introduction

Leather is an important intermediate industrial product in the sectors of the consumer goods industry [1]. Common leather applications include shoes, clothing, car covers, and furniture [2, 3].

The physical properties of leather form the most important quality parameters that determine the performance characteristics in the fields of application. The physical properties of leather form vital quality parameters that determine the performance characteristics in their fields of application. However, the transformational processing of skin into leather involves a number of both chemical and mechanical changes that affect these mechanical properties [4].

In this case, physical properties play an important role in determining a specific application area, since they determine the performance characteristics required [5,6].

Skin treatment can be divided into several operations [7-9]. Leather production is a complex sequence of chemical reactions and mechanical processes, among which leather finishing is one of the most important stages responsible for improving the appearance of the leather and determining the final characteristics of products required by consumers [9]. Thus, finishing operations have taken an important place in the production of leather due to the tradition of aesthetic qualities, and also contribute to the improvement of the physical and mechanical properties of leather.

The finishing process of leather is defined as a set of operations performed to improve the appearance and properties of the leather surface [10]. The coating procedure is performed mainly for decorative purposes, at the same time it affects the physical properties of the leather.

The finishing process gives a tendency to increase the rigidity of leather, while the air and water resistance permeability of the leather are decreased [9]. These changes depend on the type of finish and finishing technology applied to leathers. The type of polymeric binder, the penetration of the finishing solution into the leather, and the thickness of the overall coating are extremely important, defining the final properties expected from the leather [11].

The finishing compositions used may vary depending on the type of leather and the desired properties of the final product [12]. Among the many chemicals used in finishing formulations, polymer binders are necessary for the formation of a layer on the surface and between the fibers of the skin, as well as for the adhesion of pigments and other chemicals. Although they form a coating film on the surface, they often penetrate into the leather in base coats and adhere to the fibres to facilitate the adhesion of latter coating layers. Therefore, the layers penetrating into the leather and remaining on the surface are expected to affect the mechanical properties of the finished leather [13].

There are many different types of finishes in the leather industry: two-tone, transparent, semi-aniline and opaque.

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The composition of each type of finish is different, therefore each of them affects not only the performance characteristics, but also the hygienic properties of the finished leather [14].

Natural products are incorporated into various leather production processes. For example, plant extracts are used for antimicrobial action, elimination of formaldehyde and for natural colouring in skin treatment processes. In this regard, there is a need to use environmentally friendly plant extracts in the treatment of skin, which will solve the problems of environmental protection [15-20].

Flavones are organic compounds. The dye quercetin, belonging to the class of flavonols, was first obtained from oak bark [21]. In addition to flavones, tannin compounds such as gallic acid and organic acids were also detected in the measurements [22].

It is well known that the physical and mechanical characteristics of leathers are mainly determined by the type and quality of untreated leather, as well as by chemical and mechanical operations in wet processes. It was also revealed in [4, 13, 23], that the appearance and composition of leather finishing materials have a great influence on the mechanical and hygienic properties of leather.

The use of natural extract from oak bark during finishing is a new approach to the ecological and economical production of leather, as well as improving the strength of leather materials. The main goal of this work was to improve the properties of leather by replacing the chemical pigment with a natural extract from oak bark. For comparative analysis, leather finished with chemical pigment and leather without finishing were used and compared with a sample finished with oak bark extract.

№	Dye solution	pH
1	Oak bark (*Quercus cortex*)	3,5
2	Chemical pigment	6,5

Table 1. pH value of extract and chemical pigment

CHEMICALS	APPLICATION Coat (gramme)	DESCRIPTION
Stage 1		
CPT 2350	150	Acrylic Binder (Alpa Chemistry)
CPT 2345	150	Binder (Alpa Chemistry) Acrylic Polymer
CPU 1641	150	Polyurethane Binder (Stahl)
CRE 1036	200	Acrylic Binder (Alpa Chemistry)
CST 6760	200	StukoWax (Alpa Chemistry)
CW 171	50	Synthetic Wax (Alpa Chemistry)
CW 159	50	StukoWax (Stahl)
CST HD	50	Polyurethane Binder (Stahl)
Dyestuff	2000	Plant Extract (oak bark) or chemical pigment
1) 3×spray – RotoPress (80°C, 150 Bar) - 3×spray – RotoPress (80°C,70 Bar) - 3×spray (80°C,70 Bar)		
Stage 2		
CK 1622	150	Polyurethane lacs (Stahl)
Dyestuff	300	Plant Extract (oak bark) or chemical pigment
1) 2×spray – RotoPress (90°C, 70Bar)		

Table 2. Finishing recipe used in application

2. Materials and Methods

2.1. Materials

For the study, undyed chrome-tanned bovine leather was used, processed by the «Turan-Skin» factory, located in Kazakhstan in the city of Shymkent.

2.2. Process of finishing leather samples

In the finishing process of the leather, natural extract from oak bark was used. The process of preparation of the extract is given in another study [24-25]. For the control sample, a basic finishing formulation using a chemical pigment and samples without finishing were used. Characteristics of the dye solution are shown in Table 1.

The finishing was carried out by spraying, the process of which consisted of two stages. The first stage was painting the coating, and the second was fixing the coating with lacs and dyes. The finishing recipe is shown in Table 2.

The finishing formulation used in this research belongs to the factory itself. The proportions of the materials used in this research may vary according to the desired properties. For this reason, it may be appropriate to try plant extracts in different formulations.

Unfinished, and finished leather samples using chemical pigment and oak bark are shown in Figure 1. The leather sample with the oak bark extract turned a pale pink colour, while that treated with the chemical turned a beige colour.

2.3. Sample conditioning procedure

Prior to the tests all finished and unfinished leathers were conditioned according to Standard EN ISO 2419:2012 Leather - Physical and mechanical tests -Sample preparation and conditioning.

The alternative specific standard atmosphere has a temperature of 20.0±2°C and a relative humidity of 65.0± 2%.

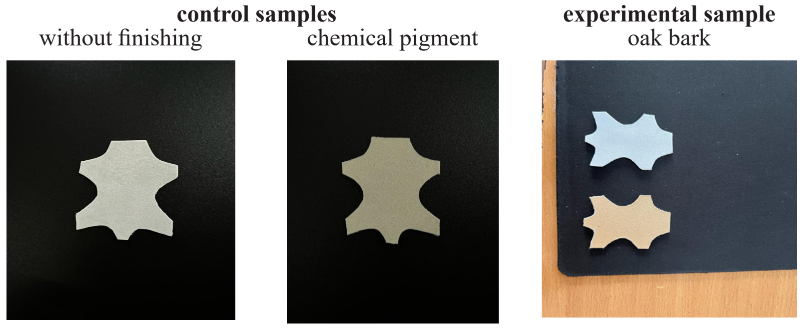


Fig. 1. Leather samples

2.4. Determination of mechanical parameters

A Shimadzu AG-IS tensile testing device was used for all tests. The measurement of sample thickness was performed in accordance with EN ISO 2589:2002. Leather - Physical and mechanical tests - Determination of thickness.

The tensile strength and percentage extension was determined with EN ISO3376:2011. Leather - Physical and mechanical tests - Determination of tensile strength and percentage extension, single edge tear load with EN ISO 3377-1:2011. Leather - Physical and mechanical tests - Determination of tear load - Part 1: Single edge tear, double edge tear load with EN ISO 3377-2.:2002. Leather - Physical and mechanical tests - Determination of tear load - Part 2: Double edge tear.

During the sampling, half of test pieces were cut parallel to the backbone and the other half perpendicular to the backbone.

2.5. Determination of water absorption

The percentage water absorption is determined according to ISO 5403-1:2011. Leather - Determination of water resistance of flexible leather - Part 1: Repeated linear compression.

The percentage water absorption, W_0, is calculated using the formula:

$$W_0 = \frac{(m_1 - m_0).100}{m_0} \qquad (1)$$

Where, m_1-the mass of the test piece after any time period, in grams; m_0 - the initial conditioned mass of the test piece, in grams.

2.6. Determination of water penetration

The water transmission is determined according to ISO 5403-1:2011. Leather - Determination of water resistance of flexible leather - Part 1: Repeated linear compression.

The water transmission, m_{wt}, in grams, is calculated using the formula:

$$m_{wt} = m_{am1} - m_{am0} \qquad (2)$$

Where, m_{am1} - the mass of the absorbent material after the test, in grams; m_{am0} - the initial conditioned mass of the absorbent material, in grams.

2.7. Determination of colour fastness to water spotting

To control the colour stability of the dye extract contained in the finished leather, studies were conducted on the colour fastness to water spotting in accordance with the standard TS EN ISO ISO 15700:1998 - Leather - Tests for colour fastness.

In this study, the absorption of water by the leather was controlled by instilling drops of distilled water over two time periods, i.e. 30 minutes and 16 hours. The experiment was repeated 3 times.

Two methods were used to monitor the results of the study. The first was evaluation by visual inspection using an organoleptic method in accordance with the ISO 105-A02 standard, and the second method was instrumental evaluation according to the ISO 105-A05 standard on a gray scale.

3. Results and Discussion

3.1. Single and double edge tear loads

In a single tear test a specimen which is partially slit from one edge is pulled so that a tear is propagated from the end of the slit. In the double edge tear load test, the specimen is pulled from the both edges of a specifically shaped hole in the sample. In both tests the highest force exerted during tearing is measured. Both tests gave information about the mechanical strength of the leathers in the case of an applied force on a created tear on the leather.

The single edge tear load in the samples without finishing is 90.1 N, with a chemical pigment - 88.9N, and in the samples finished with oak bark it is on average 101.3 N. The average double edge tear load in the samples without finishing is 157.1 N , with a chemical pigment - 138.8 N, and in the samples finished with oak bark it is 164.7 N. These results clearly show that the tearing strength of the materials can be affected by process variation, as similarly reported by Mukhopadhyay *et al.* for tearing behavior [26-27].

3.2. Mechanical tests

For the experiment, samples without finishing and samples finished with the use of chemical pigment and oak bark extract in the parallel and perpendicular directions were prepared. A graph of the effect of the plant extract on mechanical properties of the leather is shown in Figure 2.

Samples	Edges	Thickness, mm	Tear load, N
Control			
Without finishing	Single edge (Parallel)	1.73	83.6
	Single edge (Perpendicular)	1.72	96.7
	Double edge (Parallel)	1.72	158.2
	Double edge (Perpendicular)	1.78	155.9
Chemical pigment	Single edge (Parallel)	1.75	87.5
	Single edge (Perpendicular)	1.77	90.4
	Double edge (Parallel)	1.76	140.1
	Double edge (Perpendicular)	1.74	137.5
Experimental			
Oak bark	Single edge (Parallel)	1.78	94.1
	Single edge (Perpendicular)	1.78	108.5
	Double edge (Parallel)	1.75	150.2
	Double edge (Perpendicular)	1.76	179.5

Table 3. Values of the single and double edge tear load of the finished and control groups

According to the results of the study, the tensile strength of the sample in the parallel direction using oak bark is 4.8 N/mm^2 and in the perpendicular direction - 9.2 N/mm^2 more than in samples without finishing, and 1.3 N/mm^2, more than in the sample with a parallel section using a chemical pigment (Figure 2a).

The percentage extension of the sample using oak bark is also higher than that of control samples. For example, the elongation of the sample along the parallel section using oak bark is 94.8%, without finishing 62.9%, and with the use of chemical pigment, the percentage extension of the sample is 87.3% (Figure 2b).

The results of the study showed that leather samples finished by natural extract from oak bark have the best mechanical properties.The partial penetration of extract from oak bark through the pores of the leather and its interaction with the surface layers leads to an improvement in mechanical and hygienic properties of the leather.

The difference in mechanical properties of the leather samples regarding the parallel and perpendicular directions of the sections is related to the structure and fibers of the leather [28].

For the samples cut along the backline, the majority of their fibres are already aligned in the same direction as the strain applied, hence they have little scope to orientate towards the strain axis. This therefore means that the fibres themselves are directly strained at low levels of nominal strain and that the process of fibre orientation is limited.

For samples cut perpendicularly, many fibres are aligned normal to the direction of the strain applied; therefore, the fibres orientate towards the strain axis. This orientation minimizes levels of nominal strain and deformation occurs by straining themselves. Further deformation is associated with straining of the fibres themselves. Since tensile strength is proportional to the number of fibres aligned in that direction, its value is higher in the parallel orientation because the strain is applied to a more orientated network of fibres than in the of perpendicular sampling. Fibre orientation will cause frictional damage to the fibres. Since tensile strength is proportional to the number of fibres aligned in that direction, its value is higher in the parallel orientation because the strain is applied to a more orientated network of fibres than in the case of perpendicular sampling.

This shows a significant difference between samples cut perpendicularly and those cut along the backline regarding physical and mechanical properties of the leather.

3.3. Water absorption of leather

Determination of water absorption and water penetration are very important indicators for leather used in the shoe industry. Increased moisture capacity leads to a weakening of the fastening of the upper and lower parts together, faster wear due to abrasion, and a decrease in the comfort of shoes. The results of determining water absorption are shown in Table 4, displaying that the water absorption of leathers finished with oak bark extract is less than in control samples.

According to the results of the study, the water absorption of the leather sample with a parallel section using oak bark is 8.7%, and with perpendicular - 17.8%, and in samples without treatment with a parallel section it is 40.1%, and with a perpendicular - 32.3%.

With the use of a standard chemical pigment, water absorption decreases by an average of 21%.

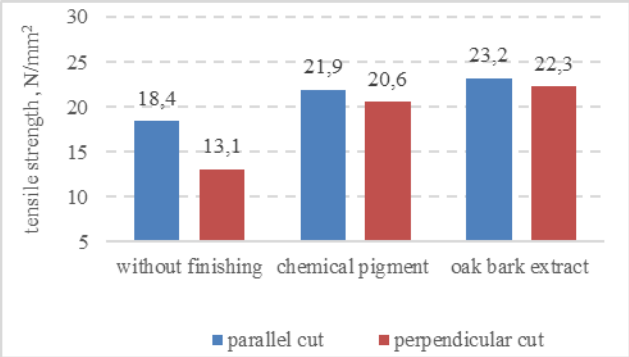
It is known that the main amount of moisture is absorbed by the leather as a result of molecular and capillary wetting by filling the interstructural voids of the skin tissue with water. Water absorption occurs in two stages [2].

At the beginning − rapid absorption of water, lasting 10-15 minutes. At this time, the largest pores are filled. During the second stage, water absorption slows down, water-soluble substances are washed out and some swelling of the skin fibers occurs.

A decrease in water absorption, apparently, can occur due to a decrease in porosity andhydrophobization of the surface of the leather.

3.4. Water penetration of leather

The results of determining water penetration are shown in Table 5, displaying that as a result of finishing using oak bark extract, water penetration in the leather samples is significantly reduced. For example, for leather samples without finishing, water penetration averages 1.93 g, and when finished with a standard chemical pigment, it averages 1.29 g. In leather samples using oak bark extract, water penetration averages



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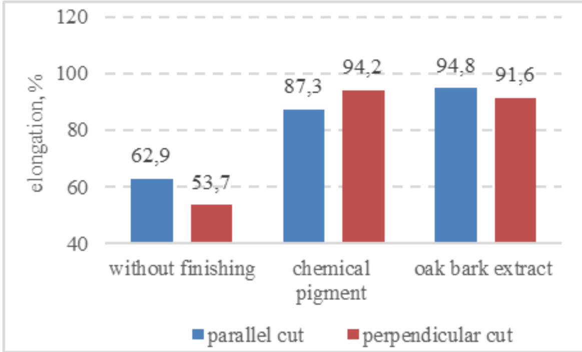

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Fig. 2. Physical and mechanical properties of leather samples

Type of leather	m_1, g	m_0, g	W_0, %
Control			
Without finishing (Parallel)	7.78	5.55	40.1
Without finishing (Perpendicular)	7.46	5.64	32.3
Chemical pigment (Parallel)	6.13	4.93	24.3
Chemical pigment (Perpendicular)	6.79	5.35	26.9
Experimental			
Oak bark (Parallel)	5.25	4.83	8.7
Oak bark (Perpendicular)	5.15	4.37	17.8

Table 4. Results of water absorption

Type of leather	m_{am1}, g	m_{am0}, g	m_{wt}, g
control			
Without finishing (Parallel)	7.71	5.56	2.15
Without finishing (Perpendicular)	7.34	5.62	1.72
Chemical pigment (Parallel)	6.05	4.91	1.14
Chemical pigment(Perpendicular)	6.77	5.32	1.45
Experimental			
Oak bark (Parallel)	5.01	4.82	0.19
Oak bark (Perpendicular)	5.08	4.34	0.74

Table 5. Results of water penetration

Type of leather	Time of test	
	30 min	16 hours
Control group		
Without finishing 1	3.5	3
Without finishing 2	3	2.5
Without finishing 3	3	2.5
Chemical pigment 1	4	4
Chemical pigment 2	4	4
Chemical pigment 3	3.5	3.5
Experimental group		
Oak bark 1	4	4
Oak bark 2	4/5	4/5
Oak bark 3	4	4

Table 6. Colour fastness to water spotting

0.46 g, which is on average 1.15 g less than in control samples.

The reduction of water penetration in leather samples using oak bark extract is their positive difference.

Reduction of water absorption by reducing porosity, hydrophobization of the surface of leather elements (fiber and bundle), as well as limited swelling of leather fibers were achieved.

3.5. Colour fastness to water spotting

The results of the study of leather samples for colour fastness to water spotting are shown in Table 6.

From the above data, it can be seen that leather samples finished using oak bark extract show better results of colour fastness to water spotting compared with other samples. For example, the indicators of colour fastness to water spotting in leather samples without finishing after 30 minutes of testing averaged 3, after 16 hours 2.5, in leather samples with oak bark extract finishing after 30 minutes averaged 4, and showed the same results after 16 hours of testing. And leather samples finished with chemical pigment have an average colour fastness of 4.

During the finishing process, the appearance and functionality of the skin improved, and a protective film was created on its surface. This made this a delicate and important stage when we decided to invest in the development of increasingly environmentally sustainable production. The use of a plant extract of oak bark for leather finishing also solves a very important environmental problem. When finishing, binders, waxes, pigments and dyes were applied to the leather texture to impart colour, as well as improve organoleptic and mechanical characteristics.

4. Conclusion

The present study revealed the influence of various coating compositions on the tensile strength, percentage extension, single and double edge tear loads, water absorption , water penetration, and colour fastness to water spotting properties of leather products. The best results were obtained when processing leathers with oak bark extract.

Thus, the tensile strength of the leather finished by oak bark extract when cut in the parallel direction is 23 N/mm^2, without finishing - 18.4 N/mm^2, and with a chemical pigment - 21.9 N/mm^2, which is on average 1.1 N/mm^2 higher compared with samples finished with chemical pigment and 4.6 N/mm^2 higher than for the samples without finishing.

The same positive results are shown in studies of the water resistance and colour fastness to water spotting of leather samples finished with natural extract from oak bark.

The use of such an extract in the leather industry is able to create favourable environmental protection.

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