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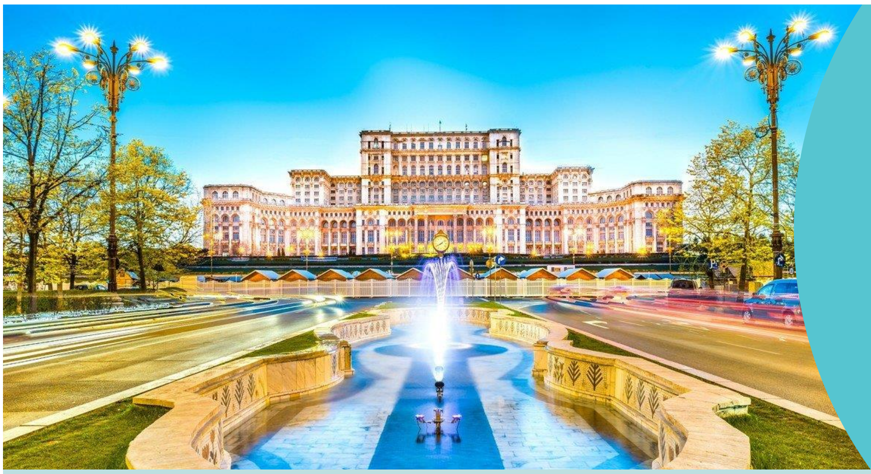

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RESEARCH ON DEVELOPING A SIZE SYSTEM AND DESIGNING SHOE LASTS FOR MEN WITH DIABETES IN VIETNAM

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RESEARCH ON DEVELOPING A SIZE SYSTEM AND DESIGNING SHOE LASTS FOR MEN WITH DIABETES IN VIETNAM

ABSTRACT. This paper presents the method and results of developing a shoe last sizing system on the basis of the foot parameter system, designing shoe lasts for men with diabetes in Vietnam. Diabetic patients often have foot complications and need to use extra depth shoes or custom shoes to protect their feet and prevent foot ulcers. The parameters of 5 shoe last sizes by length and 3 sizes by width according to the French sizing system have been developed. The results show that the shoe last parameters for men with diabetes are much larger than those for healthy men, especially the ball girth and width are larger by about 20 mm and 5 mm, respectively. Afterward, shoe lasts were designed and modified using 3D CAD tools for style, fit and comfort specifically suited for men with diabetes. Using shoe last design software, 3D printing technology to design and fabricate a shoe last, the proposed approach proved the viability of designing and manufacturing extra depth shoes for men with diabetes in Vietnam.

KEY WORDS: shoe lasts, sizing system, extra depth diabetic shoes

CERCETĂRI PRIVIND DEZVOLTAREA UNUI SISTEM DE MĂRIMI ȘI PROIECTAREA CALAPOADELOR DESTINATE ÎNCĂLȚĂMINTEI PENTRU BĂRBAȚII CU DIABET DIN VIETNAM

REZUMAT. Această lucrare prezintă metoda și rezultatele dezvoltării unui sistem de dimensionare a calapoadelor pe baza sistemului de parametri ai piciorului, proiectând calapoade de încălțăminte pentru bărbații cu diabet din Vietnam. Pacienții diabetici au adesea complicații ale piciorului și trebuie să folosească pantofi cu adâncime suplimentară sau pantofi personalizați pentru a-și proteja picioarele și pentru a preveni ulcerul piciorului. S-au stabilit parametrii a 5 mărimi de calapod pe lungime si 3 mărimi de calapod pe lățime, conform sistemului francez de dimensionare. Rezultatele arată că parametrii calapodului de încălțăminte pentru bărbații cu diabet sunt mult mai mari decât cei pentru bărbații sănătoși, în special circumferința în zona metatarsienelor și lățimea sunt mai mari cu aproximativ 20 mm, respectiv 5 mm. Ulterior, s-au proiectat și modificat calapoade folosind instrumente CAD 3D pentru stil, potrivire și confort, corespunzătoare bărbaților cu diabet. Folosind un software de proiectare a calapoadelor și tehnologia de imprimare 3D pentru a proiecta și fabrica un calapod, abordarea propusă a dovedit viabilitatea proiectării și fabricării pantofilor cu adâncime suplimentară pentru bărbații cu diabet din Vietnam.

CUVINTE CHEIE: calapoade, sistem de mărime, pantofi pentru diabetici cu adâncime suplimentară

RECHERCHE SUR LE DÉVELOPPEMENT D'UN SYSTÈME DE TAILLE ET LA CONCEPTION DE FORMES DE CHAUSSURES POUR LES HOMMES DIABÉTIQUES AU VIETNAM

RÉSUMÉ. Cet article présente la méthode et les résultats du développement d'un système de dimensionnement des formes de chaussures sur la base du système de paramètres du pied, en concevant des formes de chaussures pour les hommes atteints de diabète au Vietnam. Les patients diabétiques ont souvent des complications aux pieds et doivent utiliser des chaussures de profondeur supplémentaire ou des chaussures sur mesure pour protéger leurs pieds et prévenir les ulcères du pied. On a développé les paramètres de 5 tailles de formes de chaussures en longueur et 3 tailles en largeur selon le système de pointure français. Les résultats montrent que les paramètres de la forme de chaussures pour les hommes diabétiques sont beaucoup plus grands que ceux pour les hommes en bonne santé, en particulier la circonférence dans la zone des métatarsiens et la largeur et sont plus grandes d'environ 20 mm et 5 mm, respectivement. Par la suite, des formes de chaussures ont été conçues et modifiées à l'aide d'outils de CAO 3D pour un style, un ajustement et un confort spécifiquement adaptés aux hommes atteints de diabète. En utilisant un logiciel de conception de chaussures et une technologie d'impression 3D pour concevoir et fabriquer une forme de chaussure, l'approche proposée a prouvé la viabilité de la conception et de la fabrication de chaussures à profondeur supplémentaire pour les hommes diabétiques au Vietnam.

MOTS CLÉS : formes de chaussures, système de dimensionnement, chaussures à profondeur supplémentaire pour les diabétiques

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INTRODUCTION

Shoe lasts, 3D molds utilized to make footwear, play an important role in shoe production, affecting the shape, fitting, and size of shoes. Wooden or metal shoe lasts are tools for designing and manufacturing shoes, satisfying the biomechanical prerequisites of the inside, style, fit, and comfort. To achieve these requirements, shoe lasts are designed based on foot shapes, sizing systems, and parameters. The complication of the shoe last making process is to determine the necessary data for building a standardized last from various foot dimensions (Figure 1).

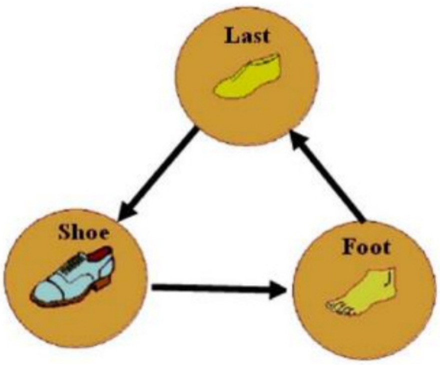

Figure 1. Correlation between foot, last, and shoe [1]

In the footwear industry, shoe last design is an arduous job. This is related to the requirements of shoe comfort, production technology and aesthetics [2]. Varied countries have developed a size system or a shoe last parameter system for numerous types of shoes (dress, casual, sport, hiking, work shoe, sandal, moccasin) [3]. The dimensions of a shoe last are not exactly equivalent to the homologous dimensions of the foot, though the foot is the model for it [2, 4]. Setting up the shoe last size system is the conversion of foot parameters into homologous last parameters. This represents the relationship between the foot and the last. And it depends on the age, gender of the shoe user, the type of shoes, the height heel of the last, and the purpose of the shoe or the requirements for the shoes.

The length of the last is longer than the foot length. The last bottom length is its curved length and is equal to the bottom pattern length. This length is equal to the length of the foot minus the room around heel and plus the toe allowance [2-6]. The toe allowance includes the minimal addition to toe movement, the addition for child's foot growth for about 1 year, the addition according to aesthetics [2, 3].

The last width is closely related to the foot print and girth, which may influence the design of the last, the fit and the aesthetics of the footwear. According to AKA64-WMS system, the ball width of the last bottom, depending on the type of shoe, is equal to 37 to 40% of the ball girth of foot [4]. The width of the last bottom also depends on its heel lift. When the last heel lift is increased, its bottom width is narrowed [2, 4, 6]. The last width should be neither greater than the foot width, nor smaller than the footprint width, or the shoe will be too flat or too narrow in the metatarsophalangeal joint (MPJ) area [2-4, 6]. Therefore, the last width should be an intermediate value between the two widths, according to different situations. For example, the last bottom width with high heel lift will be closer to the foot print. Whereas the last with low heel lift will have a bottom width closer to the foot width [2, 3, 6].

Several foot girths may be utilised in shoe last design, such as ball girth, waist girth, instep girth, heel girth and ankle girth. The ball girth, which is the circumference length of the MPJ, is one of the most important dimensions in shoe last design. The ball girth of a last is also slightly longer or shorter than the ball girth of the foot. This depends on the type of shoe and its use conditions [2-6]. For example, a last for leather shoes for men or women may have girths shorter than the homologous girths of the foot. This means allowing the shoe to slightly compress the foot without causing discomfort [2, 3]. Meanwhile, the lasts of children's shoes, sports shoes, and safety shoes have girths longer than the homologous girths of the foot. The waist girth also changes according to different situations and is smaller in lasts for high-heeled shoes [2, 4]. To ensure comfortable wear, the instep girth of most lasts should be greater than that of the foot [4, 6]. The insole is inserted into the shoe after the shoe last is removed from it.

Therefore, it is necessary to take into account the thickness of the insole into the parameters of the shoe last girths, especially the closed girths [7].

Research shows that there is an angle of about 15 degrees between the toes and the bottom of the foot, when the foot hangs naturally [2, 4]. This is called the toe spring. Shoes with appropriate toe spring are able to support the toes and reduce the bending of the MPJ during walking or running. They will also deter excessive wrinkling of the upper, and wear and tear on the outsole, so extending serviceable life. The toe spring is also reduced with an increase in heel height [2-6].

The optimal last heel height reported in many studies is 25–45mm [2-4, 8]. However, high heels are designed mainly for fashion and aesthetics, and may be from 50mm to 80mm, and in extreme cases, over 100mm. Heel elevation also affects the design of shoe lasts, as the room around the heel of the foot or heel shift will become smaller [4, 6], or bigger [2, 3] thus altering the length of the last.

Currently, there are no standards or regulations on the shoe lasts and footwear for diabetic patients in Vietnam, thus general recommendations are given on designing extra depth diabetic shoes. Suggestions and recommendations from published reports could be used in shoe last design such as the heel height should be up to 20 mm [9], between 15 to 25 mm [10], from 20 to 30 mm [11, 12], not more than 50 mm [13]; an addition to the bottom length at the toe at least 10 mm [9]; shoe insole thickness from 5 to 10 mm [9]; shoe weight less than 700g/pair. Other issues such as a change in foot parameters, the presence of edema that leads to increased foot parameters, and the recommendation of choosing shoes in the afternoon, also need to be taken into consideration before designing the last and manufacturing extra depth shoes for the foot characteristics of diabetic patients.

The design of shoe lasts for diabetic patients is an urgent issue, particularly, for patients with foot problems or a high risk of ulcers. Footwear-related factors aggravate foot ulceration, leading to infection and in the worst case partial or full amputation. Modified shoe last has been studied to meet foot shape and biomechanical functions among individuals, therefore, reducing the risk of foot ulcers. However, a systematic procedure has not been established to customize shoes for these patients. Shoe lasts made by skilled craftspeople could be time-consuming, unreliable, heavily relied on techniques and experience. So far, there have been a number of studies on custom shoe last design based on 3D foot shape and parameters [1, 14-16]. These CAD systems can identify diabetic footwear to adjust sizing geometry for individuals thus minimizing pressure infliction while preserving the fashion style.

Novel CAD tools have been employed to design shoe last for diabetic patients with the technologies of risk reduction for foot ulceration and preserving shoe styles. The footwear and original aesthetic parameters necessary to measure geometrical models and design features were identified based on the foot model and geometric algorithms for shoe last processing. The last design for diabetic patients was based on biomechanical variables regarding diabetic feet having high pressure at the joint of the metatarsal and hallux. Ball, instep, and heel circumference are demonstrated to cope with the space required for the foot and the custom insole. Limited changes in the foot shape were guaranteed during walking to avoid friction with the upper and shear loading. The ball girth was clarified to have enough room to fit the foot comfortably; otherwise, a shoe too tight leads to ulcers in the medial area of the first metatarsal head and in the lateral area of the fifth metatarsal head. A shoe, having a high and rounded toe box, provides the best fit for the toes allowing toes to move comfortably inside the shoe [17]. Computer-aided reverse engineering system (CARESystem) are used to develop the design process and fabrication of ankle foot orthotics (AFO) for patients with diabetes [18]. The study results showed that the shape of the shoe fit the standard AFO and the first patient experienced comfort for the 4-week long testing period. The use of this technology reduced the time for both the design and fabrication of the AFOs by 64%

[18]. The reverse engineering (RE) combined with an artificial neural network, a termed self-organizing map (SOM) was used to select a shoe last for footwear design to help relieve the pain associated with diabetic neuropathy and foot ulcers [19]. With this technology, the most suitable shoe last for each patient with a mild diabetic foot can be determined by calculating the relative fitness function for each patient [19].

In Vietnam, Bui *et al*. conducted a study on anthropometric characteristics of 412 female feet with diabetes and investigated the patient's requirements for shoes. The authors identified the types of foot lesions and built a system of patient foot parameters [20, 21]. This data, combined with the results of a patient survey about the requirements for extra depth shoes have been the basis for building a shoe last size system, favorable for designing shoe lasts for women with diabetes in Vietnam [7, 22]. The shoe last quality is assessed by wearing tests and pressure measurements on patients' feet using a pressure sensor system [23]. The results show that the shoe last met the requirements for up-scale manufacturing [24]. The results of the study on foot shape and parameters of men with diabetes in Vietnam show that there is a difference in foot width and circumference of men with diabetes compared with the feet of healthy men [25]. In addition, extra depth shoes for diabetic patients need to meet specific requirements such as using insoles with a minimum thickness of 5 mm, shoes with a high and wide toe, heel height not exceeding 3 cm, and so on [9-12]. Therefore, developing a sizing system and designing lasts to fabricate extra depth diabetic shoes for men with diabetes in Vietnam are necessary and novel. Therefore, in this study, we used the established foot parameter system [25], and based on the shoe requirements, to build a shoe last size system, design the last of extra depth shoes for men with diabetes in Vietnam.

EXPERIMENTAL

Methods

The process of designing a shoe last of extra depth shoes for men with diabetes is alike compared to that for women with diabetes [7], as shown in the flowchart in Figure 2.

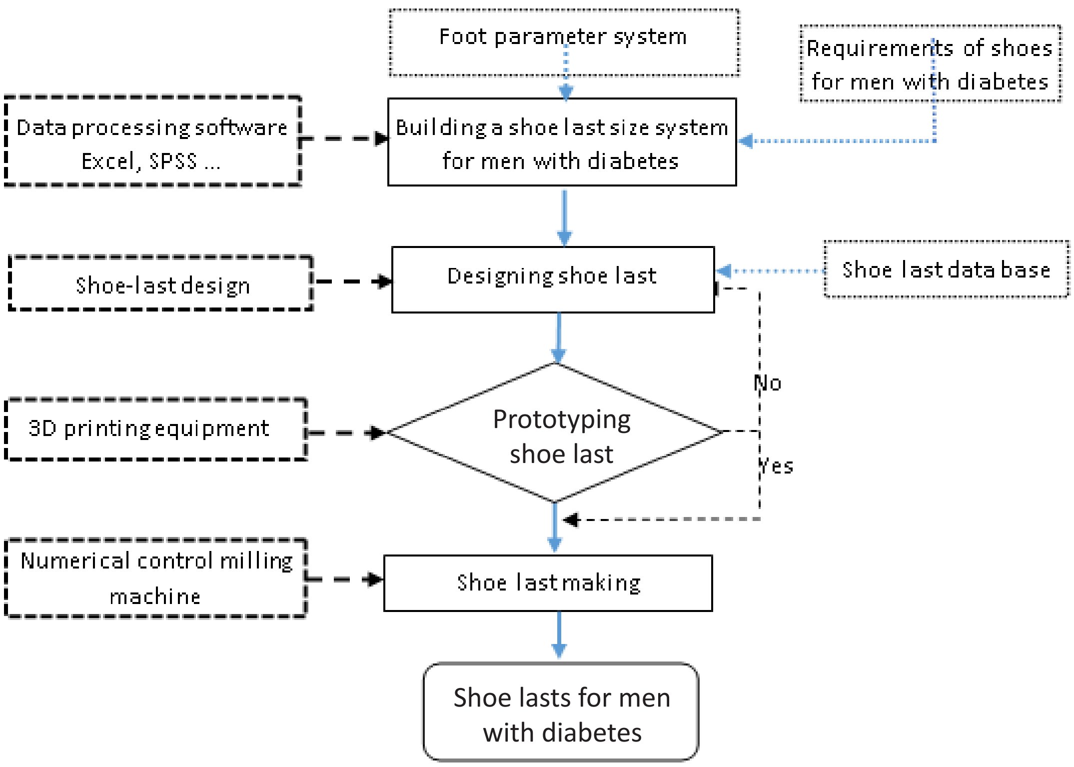

Figure 2. Flowchart for developing a sizing system and designing a shoe lasts of extra depth shoes for men with diabetes

To design the lasts, it is necessary to have data on the shape and parameters of the foot. From here, a system of foot parameters is built including foot parameters and their increments of adjacent sizes. From the main data, the foot parameters, combined with the requirements of shoes for men with diabetes such as high and wide toe shoes, 15 mm toe allowance, shoes that do not constrict the foot, insole thickness at least 5 mm, heel height not more than 25 mm and so on will be calculated or converted into the corresponding foot parameters of the shoe last. Building a shoe last size system is fundamental before further steps utilizing a 3D shoe last design software, 3D printing equipment, a CNC milling machine to design and manufacture the lasts.

Method for Determining Shoe Last Parameters in the Size System

The shoe last parameters for men with diabetes were determined on the basis of foot measurements in the established system of foot parameters [25] and shoe requirements for diabetics. In the study [25], we built a system of foot parameters according to the French size system including 5 sizes by length (231.5, 238, 244.5, 251, 257.5 mm) with increments between sizes is 6.5 mm. Each size by length has 3 sizes by width with 10 mm increments. The foot length determines shoe last sizes according to the length of its bottom surface. The ball girth determines shoe last sizes by width. Parameters of average foot size 244.5 mm by length with 3 sizes by width are shown in Table 1.

Table 1: Parameters of average foot size 244.5 mm by length with 3 sizes by width of Vietnamese men with diabetes [25]

№	Foot parameters	Values by width, mm		
		A	B	C
1	Foot length **Lf**	244.5	244.5	244.5
2	Length to medial ball **Lmb**	180.0	180.0	180.0
3	Length to lateral ball **Llb**	159.4	159.4	159.4
4	Length to the end of 5th toe **L5toe**	202.4	202.4	202.4
5	Length to instep point **Lins**	78.5	78.5	78.5
6	Length to center of lateral ankle **Lla**	55.7	55.7	55.7
7	Width of medial ball **Rmb**	95.4	99.5	103.7
8	Width of lateral ball **Rlb**	92.2	96.2	100.1
9	Width of ball **Rb**	98.6	102.9	107.2
10	Width of heel **Rh**	62.6	65.3	68.0
11	Height at 1st toe **C1toe**	19.2	20.0	20.8
12	Medial ball height **Cmb**	31.4	32.8	34.1
13	Instep height **Cins**	51.1	53.3	55.5
14	Height at lateral ankle center **Cla**	57.1	59.5	62.0
15	Medial ball girth **Vmb**	221.1	230.6	240.2
16	Lateral ball girth **Vlb**	225.2	235.0	244.7
17	Ball girth **Vb**	231.0	241.0	251.0
18	Waist girth **Vw**	225.5	235.2	245.0
19	Instep girth **Vins**	235.0	245.1	255.2
20	Heel (cross) girth **Vh**	301.7	314.7	327.8
21	Ankle girth **Va**	196.4	204.9	213.4

Determining the Lengths of the Shoe Last Bottom

The length of the bottom surface of the shoe last (bottom length Lsl) is determined by the formula [2]:

$$Lsl = Lf - Sh + P1 + P2 + P3 \qquad (1)$$

where Lf is the foot length; P1 is the minimal addition to toe movement. Because shoes for diabetic patients have high and wide toes, so P1 = 15 mm [2, 4]; P2 is the addition for foot growth for about 1 year. In the study [7], 30 female diabetic feet were re-measured after 1 year. The results showed that the patient's foot length was almost unchanged. Therefore, in this study, inherit the research results [7] and take P2 = 0 mm for the shoe-last for men with diabetes; P3 is the addition according to aesthetics. Shoes for diabetics have wide and high toes, so P3 = 0 mm; Sh is the room around heel or heel shift [2, 3]:

$$Sh = 0.02Lf + 0.05Hh \qquad (2)$$

where Hh is the heel lift, mm. The maximum Hh for shoes for diabetics is 30 mm [9-12]. According to the results of the patient survey, the majority of patients required this height to be 10 mm, similar to shoe last for sport shoes. This height ensures both comfort and aesthetics for the shoe. Therefore, in this study, Hh = 10 mm was used.

The remaining parameters according to the length of shoe last bottom surface Lsln are determined according to the corresponding parameters of the foot Lfn by the formula [2, 4]:

$$Lsln = Lfn - Sh \quad (3)$$

Determining the Girths of the Shoe Last

The value of the ball girth and the girths in the closed vertical cross sections of the shoe last Vslg are determined according to the respective parameters of the foot Vfg using the formula [7]:

$$Vslg = Vfg.К + 2Tis \quad (4)$$

where Tis is the insole thickness; К is the coefficient, determined by the formula [2]:

$$К = 1 + (\Delta O1 + \Delta O2 - q)/Vb \quad (5)$$

where Vb is the ball girth of the foot, mm; ΔO1 is the average change in ball girth of the foot at the end of the day and with walking, ΔO1 is about 5 mm [2, 3]; ΔO2 is the mean change of patient's ball girth after 1 year. The results of the study for the feet of diabetic women showed that after 1 year, the foot size increased by 2.8 mm, or ΔO2 = 3 mm. Therefore, in this study, inheriting the research results [7] and using ΔO2 = 3 mm to design a shoe last for men with diabetes; q is the allowable reduction in foot circumference when compressing. Studies on the feet of diabetic patients [26-28] have concluded that there is poor blood circulation in the patient's feet, the skin of the feet is vulnerable to impact, compression, etc. Therefore, shoe uppers should not be pressed against the skin surface of diabetic patients' feet. This is similar to shoes for children's feet. That is, q = 0 mm should be used.

Thus, the value К = 1 + 8/Vb. With an average foot length of 244.5 mm, Vb = 241.0 mm, К = 1 + 8/241 = 1.033.

The heel cross girth of the shoe last or back length V4 may not take into account the thickness of the insole, calculated by the formula [2]:

$$V4 = Vh.К \quad (6)$$

where Vh is the heel cross girth of the foot.

Determining the Bottom Widths of the Shoe Last

Diabetic men's shoes are designed with a low heel, high and wide toe to ensure no compression on the foot similar to children's shoes. Therefore, the calculation of the bottom width parameters of the child's shoe last (for sizes 18-35) according to the AKA64-WMS system can be used for the shoe last of men with diabetes [4]. The ball width of last bottom R1 is 38% of the ball girth Vb. In which, the width of the medial side is 15% and the width of the lateral side is 23% from the longitudinal axis of the shoe last bottom surface. The heel width of last bottom R2 = 2/3*R1 + 2mm (7) and its waist width R3 = 0.80*R1 (8).

Determining the Thickness of the Shoe Last

The thickness of the shoe last is usually greater than the height or thickness of the foot. Minimum forepart height Csln of the last are determined by forefoot height Cfn and insole thickness. That is, Csln ≥ Cfn + Tis (9).

Toe Spring

For diabetic shoes use the last with a higher toe spring than that of the last for casual shoes. This creates favorable conditions for the foot when walking. The shoe has a big toe spring, increasing the rolling effect of the foot, reducing the bend in the ball joint area. The high toe spring will also help reduce pressure on the ball joint [29, 30]. So, in this study, the toe spring of the last for diabetic men was 20 mm, which is 5 mm higher than the typical shoe for healthy men [31].

Determining the Increments of the Parameters in the Shoe Last Size System

The increments of adjacent size parameters in the shoe last size system are similar to those for the foot parameter system (see Table 2). The increments of the shoe last parameters are rounded to 0.5 mm.

Table 2: Increments of diabetic men's foot parameters of adjacent sizes by length and by width rounded to 0.5 mm

Nº	Foot parameters	Regression [25]	Increments, mm by length*	Increments, mm by width**
1	Lmb	Lmb = 0.736Lf	5	0
2	Llb	Llb = 0.652Lf	4	0
3	L5toe	L5toe = 0.828Lf	5.5	0
4	Rb	Rb = 0.427Vb	2	4
5	Rh	Rh = 0.271Vb	1.5	3
6	C1toe	C1toe = 0.083Vb	0.5	1
7	Cmb	Cmb = 0.136Vb	1	1.5
8	Cw	Cw = 0.976Vb	5	10
9	Vins	Vins = 1.018Vb	5	10
10	Vh	Vh = 1.306Vb	6.5	13

** The increment between adjacent sizes by length according to the French shoe sizing system is 6.67 mm (rounded to 6.5 mm)*
*** The increment between adjacent sizes by width (ball girth) in this study is 10 mm.*

Method of Designing Shoe Last for Men with Diabetes

The design data is a shoe last size system for men with diabetes, using the parameters of the average size by length and width. After designing the medium-sized last, the full range of sizes by length and width will be graded.

The shoe last for men with diabetes was designed by modifying the basic standard shoe last of casual shoes for Vietnamese men, that had heel height 10 mm, addition P1 = 15 mm, P2 = 0, and P3 = 0 [31]. The parameters to be controlled in the shoe last size system are shown in Figure 3 and Table 3. A shoe last design software [3] was used to design and grade the lasts.

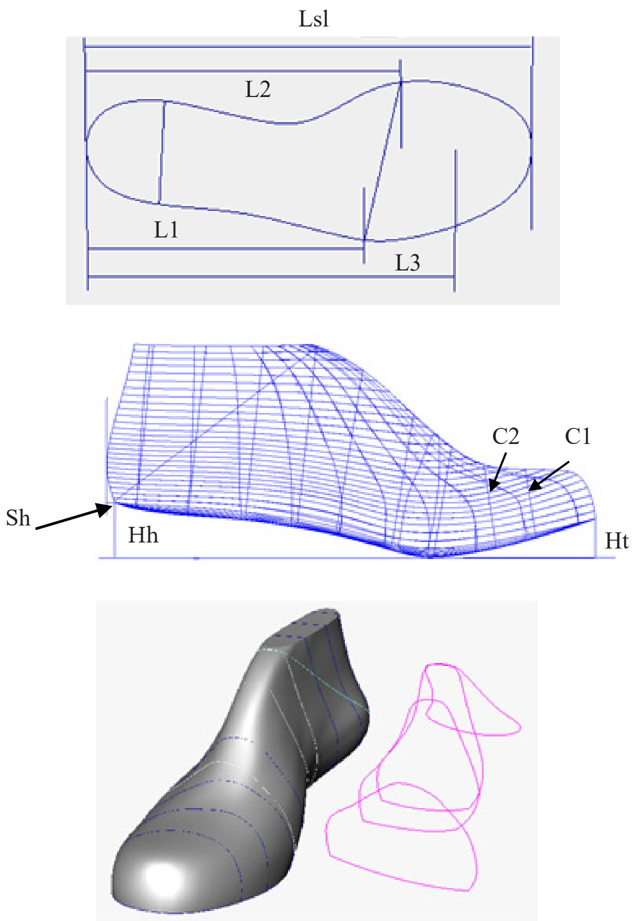

Figure 3. Shoe last parameters are controlled when designing the lasts of extra depth shoes for diabetic men using shoe last design software

The last 3D surface designed with shoe last design software [3] is transferred to the 3D multifunction printer ProJet MJP3600 Series for prototyping. The 3D printed last is the basis for correcting and perfecting its design.

RESULTS AND DISCUSSIONS

The Result of Building the Shoe Last Size System

Using formulas to convert foot data to shoe last parameters, taking into account the shoe requirements for diabetic patients, shoe last parameters for men with diabetes were determined. The results of the comparison of the last parameters for men with diabetes with that of the last for healthy men [31] are shown in Table 3.

Table 3: Comparison of shoe last parameters for men with diabetes with that of shoe last for healthy men (size 38)

№	Shoe-last parameters	Value		
		Men with diabetes, mm	Healthy men, mm	Difference, mm
1	Bottom length **Lsl***	254	≥ 253.5	-
2	Bottom length to medial ball $\mathbf{L_1}$	174.5	171	3.5
3	Bottom length to lateral ball $\mathbf{L_2}$	154	152	2
4	Bottom length to the end of 5th toe $\mathbf{L_3}$	197	190	7
5	Bottom ball width $\mathbf{R_1}$	92	87	5
6	Bottom heel width $\mathbf{R_2}$	63	60	3
7	Bottom waist width $\mathbf{R_3}$	74	70	4
8	Ball girth $\mathbf{V_1}$	259	239	20
9	Waist girth $\mathbf{V_2}$	253	237	16
10	Instep girth $\mathbf{V_3}$	263	246	17
11	Back length $\mathbf{V_4}$	337	310	27
12	Toe thick $\mathbf{C_1}$	≥ 25	24	≥ 1
13	Medial ball thick $\mathbf{C_2}$	≥ 38	38	-
14	Toe spring **Ht**	20	15	5
15	Heel height **Hh**	10	20	-10
16	Heel shift **Sh**	5.5	6	-0.5
17	Sock thick **Tis**	≥ 5	3	≥ 2

* The foot length of a diabetic man is 244.5 mm, and that of a healthy man's foot is 245 mm.

The results in Table 3 show that, except for the bottom length, the remaining parameters of shoe last for men with diabetes are larger than those of the shoe last for healthy men. Especially the ball girth and ball width differ by up to 20 mm and 5 mm, respectively. This confirms the need to develop a sizing system and design the lasts of extra depth shoes for men with diabetes. The causes of the difference in parameters are: 1) the foot circumference and width of diabetic men are larger than that of healthy men's feet; 2) shoes for diabetics using insoles of greater thickness; and 3) shoes should not compress the patient's foot.

Using the increments of the parameters for adjacent sizes by length and width (see Table 2), a shoe last size system with 5 sizes has been developed according to the bottom surface length from 241 mm to 267 mm, respectively sizes from 36-40 by the French system. Each size by length has 3 sizes by width (A1-A3), with 10 mm increments. With the same sizes by length, the length parameters of the shoe last (Lsl, L1, L2, L3) and Ht, Hh, Sh, Tis of the 3 sizes by width are unchanged, only its girth and width parameters are changed (Table 4).

Table 4: Values of the last parameters of extra depth shoes for men with diabetes in 3 sizes by width

Nº	Shoe-last parameters	Shoe-last size														
		36			37			38			39			40		
		A1	**A2**	A3	A1	**A2**	A3	A1	**A2**	A3	A1	**A2**	A3	A1	**A2**	A3
1	Bottom length **Lsl**	241	241	241	247.5	247.5	247.5	254	254	254	260.5	260.5	260.5	267	267	267
2	Bottom length to medial ball $\mathbf{L_1}$	164.5	164.5	164.5	169.5	169.5	169.5	174.5	174.5	174.5	179.5	179.5	179.5	184.5	184.5	184.5
3	Bottom length to lateral ball $\mathbf{L_2}$	146	146	146	150	150	150	154	154	154	158	158	158	162	162	162
4	Bottom length to the end of 5th toe $\mathbf{L_3}$	186	186	186	191.5	191.5	191.5	197	197	197	202.5	202.5	202.5	208	208	208
5	Bottom ball width $\mathbf{R_1}$	84	88	92	86	90	94	88	92	96	90	94	98	92	96	100
6	Bottom heel width $\mathbf{R_2}$	57	60	63	58.5	61.5	64.5	60	63	66	61.5	64.5	67.5	63	66	69
7	Bottom waist width $\mathbf{R_3}$	67.5	71	74.5	69	72.5	76	70.5	74	77.5	72	75.5	79	73.5	77	80.5
8	Ball girth $\mathbf{V_1}$	239	249	259	244	254	264	249	259	269	254	264	274	259	269	279
9	Waist girth $\mathbf{V_2}$	233	243	253	238	248	258	243	253	263	248	258	268	253	263	273
10	Instep girth $\mathbf{V_3}$	243	253	263	248	258	268	253	263	273	258	268	278	263	273	283
11	Back length $\mathbf{V_4}$	311	324	337	317.5	330.5	343.5	324	337	350	330.5	343.5	356.5	337	350	363
12	Toe thick $\mathbf{C_1}$	23	24	25	23.5	24.5	25.5	24	25	26	24.5	25.5	26.5	25	26	27
13	Medial ball thick $\mathbf{C_2}$	34.5	36	37.5	35.5	37	38.5	36.5	38	39.5	37.5	39	40.5	38.5	40	41.5
14	Toe spring **Ht**	18	18	18	19	19	19	20	20	20	21	21	21	22	22	22
15	Heel height **Hh**	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
16	Heel shift **Sh**	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5
17	Sock thick **Tis**	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5

Results of Shoe Design for Men with Diabetes

The 3D shoe last surface of the medium size (size 38) designed on shoe last design software [3] (Figure 4) has the dimensions that meet the last size system built in Table 4, and ensures its aesthetic.

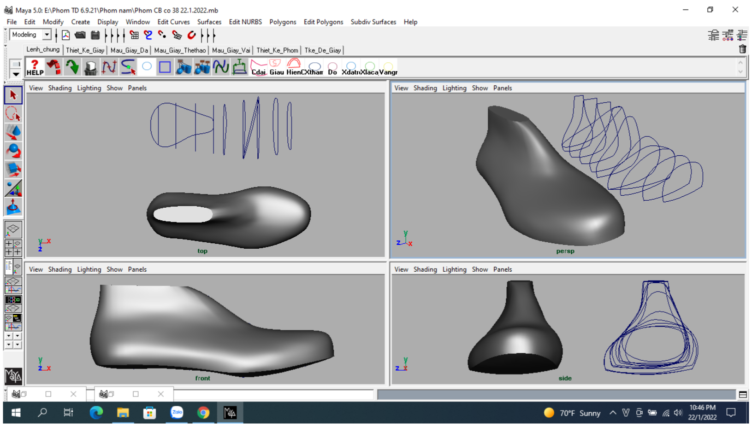

Figure 4. The designed 3D last surface of extra depth shoes for men with diabetes

The results of manufacturing the shoe last prototype on the 3D printer (Figure 5), using thermoplastic ABS, show that the designed last meets the requirements for shape and parameters as well as aesthetics. Its surface is smooth, transitions evenly between its regions.

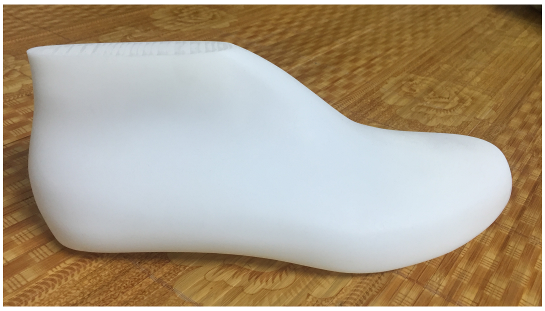

Figure 5. 3D printed shoe last prototype

Shoe last grading is performed with increments of 6.5 mm by length, 1.5 mm by ball width and 5 mm by ball girth. As a result, the 3D last surface data are obtained for the size range by length (Figure 6). It is possible to grade the shoe last by width sizes to get a full range of sizes for the design and production of extra depth shoes for men with diabetes.

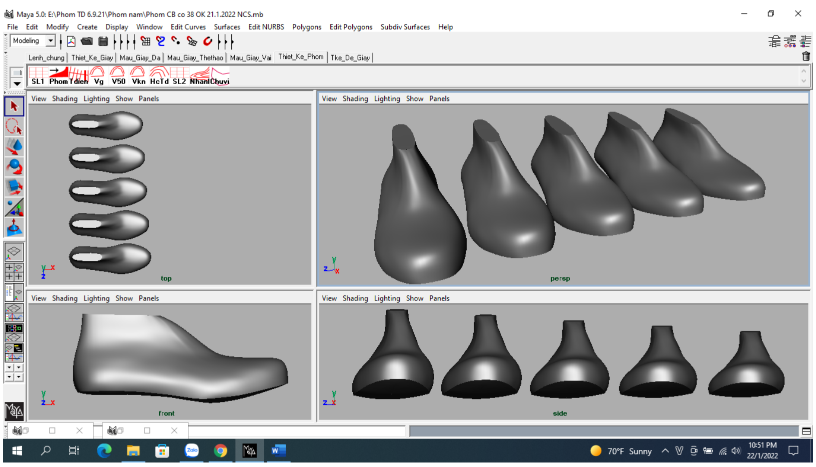

Figure 6. The size range by length of shoe last

CONCLUSIONS

This study has contributed to the establishment of procedures and methods for developing a sizing system and designing the lasts of extra depth shoes for diabetic patients. On the basis of the foot parameter system and the requirements of extra depth shoes for men with diabetes, the last size system has been built with 5 sizes by length (from size 36 to size 40 according to the French size system). Each size by length has 3 sizes by width with 10 mm increments. The shoe last parameters for men with diabetes are much larger than those for healthy men, especially its girth and width. The shoe last designed and fabricated in this study meets the requirements for shape and size, as well as aesthetics. This is the base for manufacturing shoe lasts and extra depth shoes that are suitable for the feet and requirements of men with diabetes in Vietnam.

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3D MODELLING OF CUSTOMIZED LASTS BASED ON ANTHROPOMETRIC DATA ACQUIRED FROM 3D FOOT SCANNING – ONE CASE STUDY

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3D MODELLING OF CUSTOMIZED LASTS BASED ON ANTHROPOMETRIC DATA ACQUIRED FROM 3D FOOT SCANNING – ONE CASE STUDY

ABSTRACT. Designing and manufacturing personalized lasts are the first steps in obtaining the right fitted footwear for various users, especially for sports or/and medical purposes. The accurate dimensional relationship between foot and last represents the key element for this activity. The critical shape of the last should always be determined by the shape of the foot and the cumulative relationship between lengths, widths, heights and girths, whatever method is used. Some corrections and constraints must always be considered because the shoe-last is not identical to the foot. The foot anthropometric measurements are modified based on biomechanical constraints and technological limitations and they are interactively transformed into last's dimensions by using 3D modelling. The present study brings together the modern scanning technique with the new methodology for modifying a reference last, and it is aimed to explore the philosophy of re-designing functional lasts. It also tests and highlights the limits of the actual methodology for shoe-last virtual prototyping based on anthropometric data acquired from a commercially available 3D foot scanning system.
KEY WORDS: shoe last; footwear; 3D modelling; manufacturing customized lasts

MODELAREA 3D A CALAPOADELOR PERSONALIZATE ÎN BAZA DATELOR ANTROPOMETRICE OBȚINUTE PRIN SCANAREA 3D A PICIORULUI – STUDIU DE CAZ

REZUMAT. Proiectarea și fabricarea calapoadelor personalizate sunt primii pași în obținerea încălțămintei potrivite pentru diverși utilizatori, în special în scopuri sportive și/sau medicale. Relația dimensională exactă dintre picior și calapod reprezintă elementul cheie pentru această activitate. Forma critică a calapodului ar trebui să fie întotdeauna determinată de forma piciorului și de relația cumulativă dintre lungimi, lățimi, înălțimi și circumferințe, indiferent de metoda utilizată. Unele corecții și constrângeri trebuie întotdeauna luate în considerare deoarece calapodul nu este identic cu piciorul. Măsurătorile antropometrice ale piciorului sunt modificate pe baza constrângerilor biomecanice și a limitărilor tehnologice și sunt transformate interactiv în dimensiunile calapoadelor prin utilizarea modelării 3D. Prezentul studiu reunește tehnica modernă de scanare cu noua metodologie de modificare a formei de referință și își propune să exploreze filosofia reproiectării calapoadelor funcționale. De asemenea, testează și evidențiază limitele metodologiei actuale pentru prototiparea virtuală a calapodului de încălțăminte pe baza datelor antropometrice preluate de la un sistem de scanare 3D a piciorului disponibil comercial.
CUVINTE CHEIE: calapod, încălțăminte, modelare 3D, fabricarea calapoadelor personalizate

LA MODÉLISATION 3D DES FORMES CHAUSSURE PERSONNALISÉES À PARTIR DE DONNÉES ANTHROPOMÉTRIQUES OBTENUES PAR LA NUMÉRISATION 3D DU PIED – ÉTUDE DE CAS

RÉSUMÉ. La conception et la fabrication de formes chaussures sur mesure sont les premières étapes pour obtenir des chaussures adaptées aux différents utilisateurs, notamment à des fins sportives et/ou médicales. La relation dimensionnelle exacte entre le pied et la forme chaussure est la clé de cette activité. Le contour critique de la forme chaussure doit toujours être déterminé par la forme du pied et par la relation cumulative entre les longueurs, les largeurs, les hauteurs et les circonférences, quelle que soit la méthode utilisée. Certaines corrections et contraintes doivent toujours être envisagées car la forme chaussure n'est pas identique au pied. Les mesures anthropométriques du pied sont modifiées en fonction des contraintes biomécaniques et des limites technologiques et sont transformées de manière interactive en pointures de formes chaussures à l'aide de la modélisation 3D. Cette étude combine des techniques de numérisation modernes avec la nouvelle méthodologie de modification de la forme de référence et vise à explorer la philosophie de la re-conception des formes chaussure fonctionnelles. Également on teste et met en évidence les limites de la méthodologie actuelle de prototypage virtuel de la forme chaussure à partir des données anthropométriques tirées d'un système de numérisation 3D du pied disponible dans le commerce.
MOTS CLÉS : forme, chaussure, modélisation 3D, fabrication de formes chaussures sur mesure

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INTRODUCTION

When purchasing footwear, consumers are looking for two main features: style and comfort. Footwear products that do not correspond dimensionally to the foot shape, and also do not take over the foot modifications while standing or walking, represent the main cause for prevalence and evolution of structural and functional foot anomalies [1, 2]. Moreover, the health of the entire body, as well as the human performance, could be affected. Customized footwear for different uses, including the medical and sports ones, is an important market niche [3, 4]. Personalized footwear based on customized lasts could solve some of the foot problems related to sizing, poor fitting or perceived a lack of comfort [5-7].

The footwear shape and its inner space are both influenced by the shape and dimensions of the technological lasts [8]. Often, several physical prototypes that go through a series of adaptations and adjustments are required. The virtual prototype can be created, analysed and modified long before producing a physical prototype. This technology diminishes the time for testing the physical prototype; also, other designing problems may be resolved in the virtual prototyping stage. The main advantages are given by reducing time and costs for trials of the new products [9].

There is an increasing demand for industrial applications of systems digitizing the human body, and there are markedly available innovative solutions regarding affordable imaging techniques, such as laser scanners, multiple video cameras, motion capture systems or projector-camera sets. The 3D images are transformed into digital forms, and the process of designing new products includes new stages, such as modelling and simulation for the virtual prototype. Virtual prototyping suggests new opportunities both for researchers and for customized footwear [10]. While the manual measuring of the foot introduces errors caused by the skills of the person who takes the measurements, the automatic measurement from a scanning device could offer error-free data [11].

Nowadays, the lasts can be rapidly designed due to the recent developments in computer-aided design (CAD) technologies [12]. The computerized method consists in the 3D modelling of the last; this method has the advantage of simulating the new shape before a physical last prototype is manufactured. When a last is interactively or/and automatically modified, the new changes are carried out while the designers visualize their models at every step; thus, the entire shoe-last designing process is less time consuming. Because of some limits in using commercially available CAD systems, most of the shoe-last designers still prefer manual methods [13]. The mixed techniques for designing new lasts use combinations between computer-aided designing based on modelling software and manual methods based on lasts construction type grouping and/or 2D templates [14]. Regardless of the method, important design features determine how the required fitting conditions are achieved; also, the dimensional design restrictions should refer to the technological constraints of lasts manufacturing [13].

Designing new lasts and re-designing existing ones are based on foot anthropometric. There are certain restrictive factors affecting the last's shape and its dimensions: acceptable limits of foot tightening by footwear, modification of foot dimensions while walking (biomechanics), footwear constructive type, physical and mechanical properties of materials, and footwear manufacturing technology. Also, one has to consider the general design requirements of the footwear. The footwear is comfortable when, throughout its inner volume and dimensions, it allows the foot to achieve its protective, biomechanical and orthopaedic functions [15-17].

EXPERIMENTAL

Materials and Methods

The paper presents one case study, but the hereby described method, as well as the developed methodology for analyzing the final results of the modelling process, can be replicated and applied for any new case study. The high degree of interaction between practitioner/designer and its computer, as well as the high level of customization are the main advantages of this re-designing process. Five working stages are considered in order to obtain the modified last: 1) scanning of the foot; 2) positioning of the anatomical points on foot; 3) measurement/calculation of the main anthropometric measurements; 4) comparison of the foot against the reference last; 5) modification of the last according to the foot shape.

All experimental protocols were approved by a named institutional review board. The subject has been informed and consented for study participation. All methods were carried out in accordance with relevant guidelines and regulations.

Scanning of the Foot

The studied case refers to a subject having visibly identified foot problems that ask for a careful interpretation of the design features based on anthropometric data, biomechanics and orthopaedic requirements. The subject (47-year-old, female) agreed to be studied on a voluntary basis. She has been previously diagnosed with arthritis and the results of clinical analysis of her feet allowed establishing the correct premises for designing a customized last according to the identified risk of developing a more severe arthritic foot. Several initial stages of structural modification related to arthritic feet have been identified, in this case, especially in the forefoot area. Thus, the subject presents incipient stages of modification on the first toe (Hallux-Valgus) on both feet and, visible differences in the height of the first toe of the right foot and the left foot.

The subject's foot is scanned by using a 3D foot scanning system; respectively the INFOOT USB Standard Model IFU-S-01, provided with eight progressive ¼' CCD cameras and four laser instruments, class 1M. INFOOT scans a foot and positions the anatomical landmarks, which are used to measure automatically/calculate up to 20 measuring items. It scans the 3D foot form and the anatomical points in about 10 seconds per foot, and the dimensions and angles are automatically calculated and viewed in a few seconds. The subject stands with one barefoot inside the scanner and one foot outside the scanner, and the entire mass of the subject is equally distributed on both feet.

The scanned foot data can be used for foot morphological analysis, footwear/last selection, and also for designing new lasts or re-designing existing ones. The scanned data have the points cloud format, wireframe or solid format, and they are saved as FBD binary data that gives both the 3D foot shape and the position of the anatomical points. The binary file can also be exported by a specific INFOOT software module (for example File Converter) as *.csv, *.dxf, *.vrml or *.stl formats. These exported formats could be imported into different modelling or designing software. For this study, the OrthoLast modelling software from Delcam Crispin has been used.

Positioning of the Anatomical Points on Foot

Accurate positioning of the anatomical points influences the value of anthropometric parameters. For the hereby-presented study case, the anatomical points mapping (Figure 1) suggested by the scanner's producer – INFOOT used (http://www.iwl.jp). The landmarks are automatically given by the software in a few seconds. Because several problems and structural modifications against the normal foot have been identified for this case, each anatomical point is checked, and it is moved (if necessary) in its right position. Also, each transversal section is checked and corrected in case shape distortions occurred during scanning. Even if the scanning process takes several seconds, the correction process can take a long time. The commercially available scanning systems recognize the anatomical points for normal feet; in the case of feet having anomalies, this standard facility is less useful. Therefore, the accurateness in measurements taken for customized footwear can be affected by introducing huge errors regarding positioning the anatomical points.

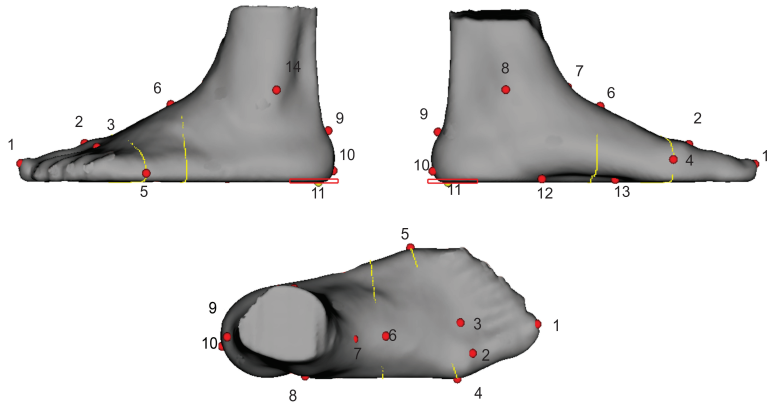


Figure 1. There are 26-foot landmarks suggested by INFOOT user manual

1 - Tip of 1st toe
2 - Head of the 1st metatarsal bone
3 - Head of the 2nd metatarsal bone
4 - Metatarsal tibiale – 1st metatarsal head
5 - Metatarsal fibulare – 5th metatarsal head
6 - Top of instep point
7 - Junction point
8 - Medial malleolus
9 - Upper heel point
10 - Extreme heel point
11 - Landing point
12 - Arch point 1
13 - Arch point 2
14 - Lateral malleolus

Measurement/Calculus of the Main Anthropometric Measurements

Anthropometry applied in the footwear industry aims to measure the foot. The foot measurements are assessed through precisely defined points that are called anatomical points. The anatomical points are some protuberances of the foot skeleton or its joints, and they are becoming well-shaped limits of the soft tissues. Several basic measurements are mentioned [18] for characterizing the foot dimensions and, therefore, its anthropometric measurements. The longitudinal measurements (lengths of the foot) represent the distances from the heel extreme point (landing point or nearby it) to a series of precise anatomical points (for example, 1st or 2nd toe, instep point, 1st metatarsal head and 5th metatarsal head, etc.). These distances are measured up along the longitudinal axis of the foot. There are different opinions among specialists regarding the right position for this axis [19-21]. To keep the same reference as for the longitudinal axis of the last, the longitudinal axis of the foot is given in this study by the line that joins the heel centre with the head of 2nd metatarsal bone. The transversal measurements are represented by widths and girths. The widths are measured on the outline of the foot or the footprint, perpendicularly on the foot's longitudinal axis or in line with ball direction. The girths are circumferences of foot measured up according to with previous defined sectional planes on metatarsal heads, instep, heel, ankle, etc. The heights represent the vertical distance measured up from the footing surface. These dimensions are measured through precisely defined points that are called anatomical points. The anatomical points are some protuberances of the skeleton or the joints, and they are becoming well-shaped limits of the soft tissues.

Fifteen anthropometric measurements (Figure 2) are significant dimensions from footwear designer point of view: FL-Foot Length, FL5-Foot Length to 5th metatarsal head, FL1-Foot Length to 1st metatarsal head, FBW-Foot Ball Width, FLW-Foot Lateral Width, FAW-Foot Arch Width, FHW-Foot Heel Width, FBG-Foot Ball Girth, FIG-Foot Instep Girth, FHC-Foot Heel Circumference, FHB-Foot Height to top of Ball girth, FHI-Foot Height to Instep point, FHL-Foot Height to Lateral malleolus, FHM-Foot Height to Medial malleolus, FHT-Foot Height to 1st Toe joint.

a) b) c)

Figure 2. Anthropometric measurements: a) upper view, b) lateral view, c) medial view

Due to lack of generalized, universal accepted rules for taking measurements on foot/last, as well due to the need of standardized models for transforming the anthropometric data into dimensional parameters of last, the foot anthropometry applied to designing well-fitted footwear has been found quite difficult [22].

Comparing the Foot against the Reference Last

The reference last, which is imported from an existing database, is subject to an interactively comparing process against the scanned foot. Following simplified hypotheses were considered for this study: the selected reference last has appropriate size towards subject's foot length, it has low heel height, and it has rounded toe. On these lines, by using the Compare module of OrthoLast-Delcam Crispin software, the two 3D shapes (foot and last) were brought together on the same screen. The reference last and the foot are successively moved and rotated to align them in the same plane (Figure 3).

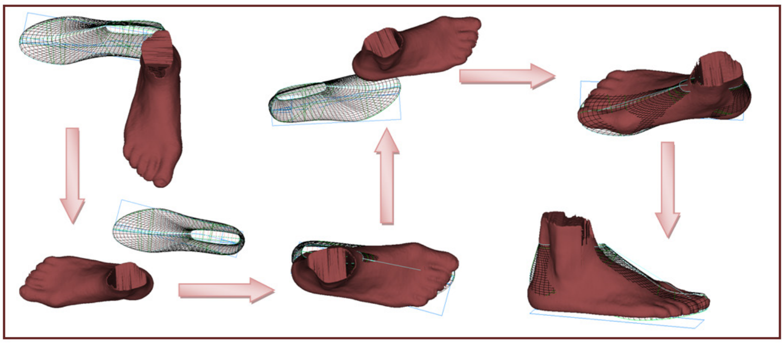


Figure 3. Interactively aligning the foot to the reference last

When the two forms (last and foot) are correctly positioned, the face centre line of the foot should match with the face centre line of the last and the centre back line of the foot should match with the back centre line of the last. This process is somehow time-consuming, and it requires from designer to have strong visual abilities for correct perspectives on 3D forms moving into 2D space available on flat computer screens. At this point, any further technological developments on similar software could be very useful regarding making this process in an automatic manner, just with several corrections at the end.

Modifying the Reference Last toward the Foot Shape

A shoe-last designer is using various foot anthropometrical data that are transformed into constructive parameters of the last. Foot length, ball width and ball girth, instep and heel region girths, toe height, toe spring and heel height have been identified as most important factors affecting designs of shoe-lasts [23]. Our study intends to analyze several more aspects and to extend the number of constructive parameters to highlight their importance and influence. Thus, using Delcam OrthoLast software the following parameters were modified and measured: Stick Length (SL), Lateral Width (LW), Bottom Length (BL), Ball Girth (BG), Ball Upper Girth (BUG), Ball Width Curved (BWC), Ball Width Linear (BWL), Instep Upper Girth (IUG), Instep Width Curved (IWC), Instep Width Linear (IWL), Short Heel Curved (SHC), Heel Width (HW), Heel Height (HH), Heel Counter (HC), Entrance Width (EW), Toe Spring (TS), Toe Length (TL), Toe Thickness (TT), Arch Curve (AC), Arch Width (AW). Figure 4 illustrates the methodology for measuring the hereby-mentioned parameters.

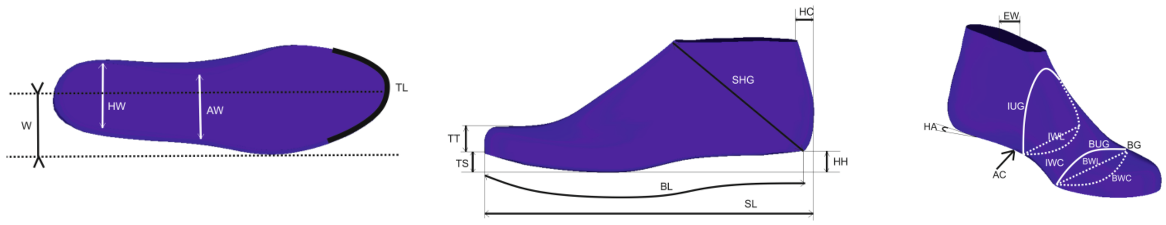


Figure 4. Illustrated methodology for measuring dimensional parameters of shoe-last

The technique of transforming an initial 3D structure into a new one, namely Free-From Deformation of Solid Geometric Models, represents one of the graphic procedures that allow for modifying a 3D structure by moving the basic points/nodes of its grid [14]. Mochimaru, M. *et al*. (2000) used this method for building new deformed grids suitable for grading the lasts. In our study, one structure (the last) represents the grid that will be interactively modified by moving precise points, and the other structure (the foot) represents the comparing form [24]. The last and the foot are being compared until they are overlapping in as many points as possible. The two 3D shapes have different appearances: draft solid for foot and gridded frame for last. By overlapping, the differences between the foot and the last can be seen. Therefore, the last will be modified in precisely selected areas (Figure 5).

Figure 5. The nine-step methodology for modelling the last

The last is modified acting on nine typical dimensional parameters, namely interactively modified parameters: SL, LW, BG, IUG, HW, HH, HC, TS, and TT. These parameters have been selected based on an initial analysis of the need for modification according to with the subject's foot. The other dimensional parameters that also describe the modified shape of the last represent the outcome-modified parameters, and they are BL, BUG, BWC, BWL, IWC, IWL, SHC, EW, TL, AC, AW. On each step, one single parameter from the first category is interactively modified. The modification upon one parameter is affecting all studied parameters that allow for collecting series of data to be statistically analyzed.

RESULTS AND DISCUSSIONS

When wearing shoes, the foot is constrained to modify its shape and dimensions among certain admissible limits of tightening. The constructive dimensional parameters of last provide limits of tightening the foot by footwear. As a result, a lower level of tightening the foot by footwear that will reduce the risk of high pressures on concrete foot surfaces is one functional requirement in this case study [25].

Table 1 shows the obtained data from 3D interactive modifications on dimensional parameters by following up nine successive steps. Each step is based on the results obtained in the previous step. When one parameter is modified, the entire range of the studied parameters is collected. While the modelling process advances, there can be determined paired relationships among sets of parameters that characterize the shape of last at each step of modification. Also, the set of dimensional parameters for the resulting last could be compared against the initial one.

Table 1: Values for interactively modified parameters and for outcome modified parameters

Parameters of the last	Reference Last (mm)	1st step of modification- on SL (mm)	2nd step of modification- on LW (mm)	3rd step of modification- on BG (mm)	4th step of modification- on IUG (mm)	5th step of modification- on HH (mm)	6th step of modification- on HW (mm)	7th step of modification- on HC (mm)	8th step of modification- on TS (mm)	9th step of modification- on TT (mm)
Interactively modified parameters										
SL	260	267,0	267,8	267,8	267,8	267,8	267,8	266,0	267,0	267,0
LW	49,8	49,8	58,0	57,5	57,2	57,2	57,2	57,2	57,2	57,2
BG	216,4	216,4	251,9	250,0	246,5	246,9	247,2	247,2	247,2	247,2
IUG	194,6	191,2	217,7	217,5	198,2	197,4	196,7	196,6	196,6	196,6
HH	15,0	14,7	17,1	17,1	17,1	10,0	10,0	10,0	10,0	10,0
HW	49,4	49,4	57,4	57,3	57,3	57,3	60,0	60,0	60,0	60,0
HC	5,3	4,9	5,0	5,0	5,0	4,9	4,9	9,0	9,0	9,0
TS	13,9	13,9	16,2	16,2	16,2	16,2	16,2	16,2	10,0	10,0
TT	21,7	21,9	25,5	25,5	24,9	25,1	25,1	25,1	29,0	25,0
Outcome modified parameters										
BL	258	259,7	260,6	260,6	260,6	260,9	260,9	260,9	262,0	262,0
BUG	133,0	133,0	154,7	153,4	150,0	150,5	150,8	150,8	150,9	150,9
BWC	83,5	83,5	97,3	96,6	96,5	96,4	96,4	96,4	96,3	96,3
BWL	82,4	82,4	96,0	95,4	95,2	95,2	95,2	95,2	95,2	95,2
IWC	49,0	49,0	57,0	57,0	57,2	57,4	57,5	57,5	57,5	57,5
IWL	48,3	48,3	56,2	56,2	56,4	56,6	56,7	56,7	56,7	56,7
SHC	134,3	137,0	145,1	145,1	140,1	140,7	140,4	140,4	140,4	140,4
EW	25,1	25,1	29,3	29,2	29,3	29,3	30,6	30,6	30,6	30,6
TL	74,5	77,2	77,2	77,2	73,9	74,5	74,8	74,8	75,8	75,8
AC	11,3	10,7	12,5	12,5	12,2	6,8	6,7	6,7	6,7	6,7
AW	32,3	33,1	38,6	38,5	39,3	39,6	39,7	39,7	39,7	39,7

The dimensional parameters of the last, both the interactively modified parameters and the outcome parameters have different values on successive steps of modification. For comparing on a common basis the range of variations (increase or decrease) for each parameter, the following calculus has been considered:

$$Di = (Pi/Pi\text{-}1 - 1) * 100 \quad (\%), \qquad (1)$$

Where:

i – step of modification, i=1, 2, 3, 4, 5, 6, 7, 8, 9;

Di – increase or decrease of modified parameter on step i, in %;

Pi – value of modified parameter on step i, in mm;

Pi-1 – value of modified parameter on step i-1 (previous step), in mm.

Table 2: Increase or decrease for interactively modified parameters and outcome parameters (empirical method)

Parameters of the last	1st step of modification-on SL (%)	2nd step of modification-on LW (%)	3rd step of modification-on BG (%)	4th step of modification-on IUG (%)	5th step of modification-on HH (%)	6th step of modification-on HW (%)	7th step of modification-on HC (%)	8th step of modification-on TS (%)	9th step of modification-on TT (%)	Variation between final modified last and reference last (%)
Interactively modified parameters										
SL	3,65	0,30	0,00	0,00	0,00	0,00	-0,67	0,38	0,00	3,65
LW	0,00	16,47	-0,86	-0,52	0,00	0,00	0,00	0,00	0,00	14,86
BG	0,00	16,40	-0,75	-1,40	0,16	0,12	0,00	0,00	0,00	14,23
IUG	-1,75	13,86	-0,09	-8,87	-0,40	-0,35	-0,05	0,00	0,00	1,03
HH	-2,00	16,33	0,00	0,00	-41,52	0,00	0,00	0,00	0,00	-33,33
HW	0,00	16,19	-0,17	0,00	0,00	4,71	0,00	0,00	0,00	21,46
HC	-7,55	2,04	0,00	0,00	-2,00	0,00	83,67	0,00	0,00	69,81
TS	0,00	16,55	0,00	0,00	0,00	0,00	0,00	-38,27	0,00	-28,06
TT	0,92	16,44	0,00	-2,35	0,80	0,00	0,00	15,54	-13,79	15,21
Outcome parameters										
BL	3,55	0,35	0,00	0,00	0,12	0,00	0,00	0,42	0,00	3,75
BUG	0,00	16,32	-0,84	-2,22	0,33	0,20	0,00	0,07	0,00	13,46
BWC	0,00	16,53	-0,72	-0,10	-0,10	0,00	0,00	-0,10	0,00	15,33
BWL	0,00	16,50	-0,63	-0,21	0,00	0,00	0,00	0,00	0,00	15,53
IWC	0,00	16,33	0,00	0,35	0,35	0,17	0,00	0,00	0,00	17,35
IWL	0,00	16,36	0,00	0,36	0,35	0,18	0,00	0,00	0,00	17,39
SHC	2,01	5,91	0,00	-3,45	0,43	-0,21	0,00	0,00	0,00	4,54
EW	0,00	16,73	-0,34	0,34	0,00	4,44	0,00	0,00	0,00	21,91
TL	3,62	0,00	0,00	-4,27	0,81	0,40	0,00	1,34	0,00	1,74
AC	-5,31	16,82	0,00	-2,40	-44,26	-1,47	0,00	0,00	0,00	-40,71
AW	2,48	16,62	-0,26	2,08	0,76	0,25	0,00	0,00	0,00	22,91

Di variations are calculated against the previously obtained values; thus, the dependencies between two successive modified lasts can be progressively noticed and quantified. Additionally, after the final iteration is performed (corresponding to the 9th step), the variation is calculated with the same relation (eq.1), by comparing final obtained parameters with parameters of the reference last (Table 2).

Because the order of modifying the last was empirically established, a mathematical method is proposed by the authors using a DSM matrix. The right method will be the one in which the last's parameters are closer to the scanned foot.

DSM matrix design was firstly described by Yassine in 1999, being developed and applied later in many fields [17]. In this type of analysis, three steps are followed:

1. The product is decomposed in elements and the relationships and connections between them are established;

2. The identified elements are written in the same order in a matrix. The connection between elements is marked inside the matrix;

3. The matrix is transformed using special algorithms in a low triangular shape by arranging rows such that the marked points are located close to the diagonal of the matrix. A specialized software facilitates and simplifies the procedure (Figures 6, 7). In our case, the algorithm used is the one A. Kusiak *et al*. developed in 1994 and available at: http://css.engineering.uiowa.edu/~ankusiak/process-model.html.

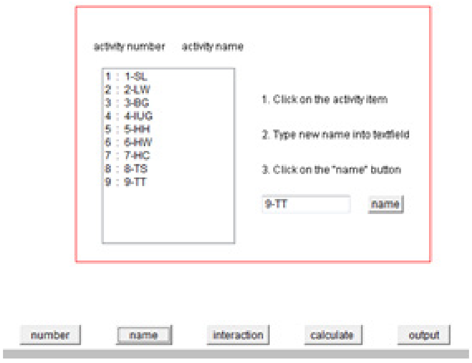


Figure 6. Establishing the matrix elements

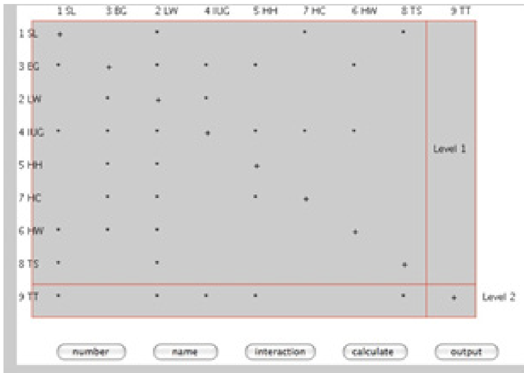


Figure 7. Generating the matrix

Based on the obtained results, the initial last will be modified respecting the order given by the DSM matrix. The results can be seen in the table below (Table 3).

Table 3: Increase or decrease for interactively modified parameters and outcome parameters (DSM method)

Parameters of the last	1st step of modification-on SL (%)	2nd step of modification-on LW (%)	3rd step of modification-on BG (%)	4th step of modification-on IUG (%)	5th step of modification-on HH (%)	6th step of modification-on HW (%)	7th step of modification-on HC (%)	8th step of modification-on TS (%)	9th step of modification-on TT (%)	Variation between final modified last and reference last (%)
Interactively modified parameters										
SL	3,65	0,00	0,00	0,00	0,00	-0,67	0,00	0,30	0,00	3,26
BG	0,00	15,53	-9,20	10,57	0,08	0,12	-0,04	-0,08	0,00	16,08
LW	0,00	16,87	-0,34	0,00	0,00	0,00	0,00	0,00	0,00	16,47
IUG	-1,75	-0,05	1,78	1,90	-0,55	-0,10	-0,20	0,46	0,00	1,44
HH	-2,00	0,00	-0,68	0,00	-31,51	0,00	0,00	0,00	0,00	-33,33
HC	-7,55	0,00	0,00	0,00	0,00	83,67	0,00	0,00	0,00	69,81

Parameters of the last	1st step of modification-on SL (%)	2nd step of modification-on LW (%)	3rd step of modification-on BG (%)	4th step of modification-on IUG (%)	5th step of modification-on HH (%)	6th step of modification-on HW (%)	7th step of modification-on HC (%)	8th step of modification-on TS (%)	9th step of modification-on TT (%)	Variation between final modified last and reference last (%)
HW	0,00	0,00	-0,20	0,00	0,00	0,00	21,70	0,00	0,00	21,46
TS	0,00	0,00	0,00	0,00	0,00	0,00	0,00	-28,06	0,00	-28,06
TT	0,92	0,46	4,09	0,00	0,87	0,00	0,00	10,39	-1,96	15,21
Outcome parameters										
BL	3,55	0,04	0,00	0,00	0,04	0,00	0,00	-0,15	0,00	3,47
BUG	0,00	16,84	-7,34	9,10	0,06	0,19	0,00	-0,13	0,00	18,27
BWC	0,00	13,29	-12,16	13,12	-0,11	0,00	0,00	0,00	0,00	12,46
BWL	0,00	13,59	-12,29	13,28	0,00	0,00	0,00	0,00	0,00	12,86
IWC	0,00	0,00	-0,20	0,00	0,00	0,00	0,00	2,25	0,00	2,04
IWL	0,00	0,00	-0,21	0,00	0,00	0,00	1,87	0,41	0,00	2,07
SHC	2,01	0,00	1,17	-0,65	0,15	0,00	0,00	0,00	0,00	2,68
EW	0,00	0,00	-0,40	0,00	0,00	0,00	20,40	0,00	0,00	19,92
TL	3,62	0,00	0,00	4,66	0,50	0,12	0,00	0,74	0,00	9,80
AC	-5,31	0,00	1,87	0,92	-33,64	0,00	0,00	0,00	0,00	-35,40
AW	2,48	0,00	-2,11	-0,62	0,62	0,00	1,24	-0,31	0,00	0,93

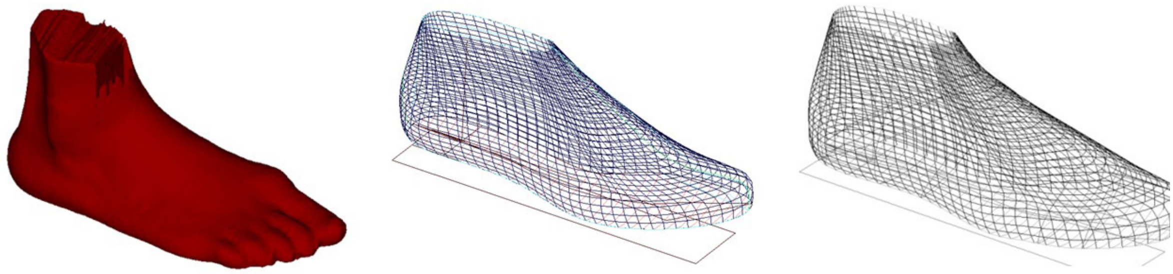
The results of the two methods, the empirical one and DSM method are presented in the table below (Table 4).

Table 4: Centralized results of two studied cases

1st case		2nd case	
The order of empirically modified parameters	Variation between final modified last and reference last (%)	The order of DSM modified parameters	Variation between final modified last and reference last (%)
SL	3,65	SL	3,26
LW	14,86	BG	16,08
BG	14,23	LW	16,47
IUG	1,03	IUG	1,44
HH	-33,33	HH	-33,33
HW	21,46	HC	69,81
HC	69,81	HW	21,46
TS	-28,06	TS	-28,06
TT	15,21	TT	15,21

Comparing the results obtained in both cases, the empirical method and the DSM method, lower deviation from the initial last are obtained in the user defined order: SL>LW>BG>IUG>HH>HW>HC>TS>TT. Research is recommended to continue, for testing another order for the parameters.

All successive modifications on the empirical method are made considering the foot's anthropometric measurements and the dimensional parameters of the reference last (Table 5). The result is an adapted last to the subject's foot (Figure 8, Table 6).



a) b) c)

Figure 8. Foot (a), initial last (b) and modified last (c)

Table 5: Correspondences among the foot, the initial last and the modified last

Anthropometric parameter on foot	Measurements Left foot (mm)	Right foot (mm)	Constructive parameter on last	Measurements Reference Last (mm)	Modified Last (mm)
Foot Length, FL	257.6	259.2	Stick Length, SL	260.0	267.0
			Bottom Length, BL	258	262
Foot Ball Width, FBW	105.3	104.2	Ball Width Linear, BWL	83.5	96.3
Foot Lateral Width, FLW	58.0	57.4	Lateral Width, LW	49.8	57.2
Foot Arch Width, FAW	33.0	32.0	Arch Width, AW	32.3	39.7
Foot Heel Width, FHW	68.5	67.6	Heel Width, HW	49.4	60
Foot Ball Girth, FBG	247.3	245.5	Ball Girth, BG= BUG+BWC	216.4	247.2
			Ball Upper Girth, BUG	133.0	150.9
			Ball Width Curved, BWC	83.4	96.3
Foot Instep Girth, FIG	240.2	239.6	Instep Circumferences, IC= IUG+IWC	243.6	254.1
			Instep Upper Girth, IUG	194.6	196.6
			Instep Width Curved, IWC	49	57.5
Foot Height to 1st Toe, FHT	20.7	24.4	Toe Thickness, TT	21.7	25.0

Table 6: Technological dimensional parameters of last

Parameters	Measurements Reference Last (mm)	Modified Last (mm)
Heel Height, HH	15	10
Instep Width Linear, IWL	48,3	56,7
Short Heel Curved, SHC	134,3	140,4
Toe Spring, TS	13.9	10
Toe Length, TL	74.5	75.8
Entrance Width, EW	25.1	30.6
Heel Counter, HC	5.3	9
Arch Curve, AC	1.13	0.67

The relations between human foot and last are critical for designing new footwear, lasts or footwear bottom components such as insoles, soles, orthoses, etc., as well as their manufacturing [8]. Lasts give the fitting volume and the inner dimensions of footwear. A last designed on empirical trials, without a scientific basis towards the consumer's foot specific conformation, will lead to a less comfortable product. Furthermore, inappropriate footwear could cause irreversible changes on foot and/or on gait patterns. The concept of shoe-last tailored to support foot's modification during human body locomotion is not precisely and finally defined yet. Complete customization of lasts and footwear is still under development, but some promising results have demonstrated clear good practices of how CAD/CAM systems for simulations and modelling can be employed to bring innovative solutions in the footwear industry [26]. Further research is needed to assess entirely customized lasts and customized footwear. The lack of information in this area still determines the designers to select the last just based on empirical approaches gained from previous experiences [27, 28].

The described method, as well as the developed methodology for analyzing the final results of the modelling process, could be replicated and applied for any new study case. In order to obtain the modified last, five working stages have been followed up: 1) scanning the foot; 2) positioning the anatomical points on foot; 3) measuring/calculating the main anthropometric measurements; 4) comparing the foot against the reference last; 5) modifying the last towards the foot shape.

The studied case refers to a subject (47-year-old, female, diagnosed with arthritis and incipient stages of Hallux-Valgus) having visibly identified foot problems that ask for a careful interpretation of the design features based on anthropometric data, biomechanics and orthopaedic requirements. Several initial stages of structural modification related with arthritic feet have been identified on this subject, especially in the forefoot area.

The obtained data from 3D interactive modifications on dimensional parameters follows up nine successive steps. Each step is based on the results obtained in the previous step. When one parameter is modified, the entire range of the studied parameters is collected. While the modelling process advances, paired relationships among sets of parameters are determined, that characterize the shape of last at each step of modification.

The dimensional parameters of the last, both the interactively modified parameters and the outcome parameters, have different values on successive steps of modification.

Each modification on a parameter brings changes to the other parameters. The designer has to follow each change, so that at the end, the last will be proper to the subject's foot [29, 30]. This research has indicated that modifying the last's parameters is not enough to design a customized last, the relationship between parameters has to be taken into account. The limitation of this paper is given by the fact that some foot areas could be tightened while wearing a footwear product, and to know if this is acceptable, the subject has to wear a shoe designed on these specific lasts.

CONCLUSIONS

This research follows up an extended and integrated application on combining CAD techniques for 3D shoe-last modelling with 3D foot scanning and measuring procedures. Both 3D scanning and 3D virtual modelling in the practice of designing fully customized lasts for special purposes are analyzed and presented as grounded related results for further innovation and development on software applications in the footwear industry. The present study brings together the modern scanning technique with the new methodology for modifying a reference last through interactive 3D modelling. Thus, it aims to go deeply into re-designing process of functional lasts based on anthropometric data obtained from 3D foot scanning. This research emphasizes the necessity of reuniting various modelling methods and scanning techniques into a new common standardized approach that should make the data transforming process more easily and precisely.

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EVALUATION OF PHYSICAL PROPERTIES OF LEATHER ON THE BATING PROCESS BY COMBINATION OF PAPAIN ENZYME WITH SURFACTANT

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EVALUATION OF PHYSICAL PROPERTIES OF LEATHER ON THE BATING PROCESS BY COMBINATION OF PAPAIN ENZYME WITH SURFACTANT

ABSTRACT. Bating is one of the important stages in the tanning process that has a role as a determinant of leather properties. It is commonly carried out with the assistance of protease enzymes such as papain. By using surfactants, sorbitol and Sodium Dodecyl Sulfate (SDS), it can help the penetration of enzymes into the skin or hides. Thereby, the study aimed to evaluate the use of papain on the bating process of *kacang* goat skin by assisted surfactants. Accordingly, three different concentrations (1, 1.5, & 2%) of enzymes were added with two different types of surfactants (SDS and Sorbitol) for the bating process. For the result, the leather was evaluated based on physical properties and histology studies. The presence of surfactant in the bating process improves the absorption of papain towards the hydrolysis of non-collagenous proteins. Based on the physical properties and histology studies, they perform better than leather without surfactant agents. Cleary, the surfactant agents can be used as bating auxiliary.

KEY WORDS: bating, papain, surfactant, *kacang* goat skin, physical properties

EVALUAREA PROPRIETĂȚILOR FIZICE ALE PIELII ÎN URMA PROCESULUI DE SĂMĂLUIRE PRIN COMBINAREA ENZIMEI PAPAINĂ CU UN SURFACTANT

REZUMAT. Sămăluirea este una dintre etapele importante ale procesului de tăbăcire, cu un rol determinant în obținerea anumitor proprietăți ale pielii. Se efectuează în mod obișnuit cu ajutorul enzimelor proteolitice, cum ar fi papaina. Folosirea unor surfactanți, sorbitol și dodecil sulfat de sodiu (SDS) poate facilita pătrunderea enzimelor în piele. Prin urmare, studiul și-a propus să evalueze utilizarea papainei în procesul de sămăluire a pielii de capră din specia *kacang* utilizând surfactanți. În consecință, s-au utilizat trei concentrații diferite (1, 1,5 și 2%) de enzime și două tipuri diferite de agenți tensioactivi (SDS și sorbitol) în procesul de sămăluire. S-au efectuat caracterizarea proprietăților fizice și studii histologice pentru a evalua pielea obținută. Prezența surfactantului în procesul de sămăluire îmbunătățește absorbția papainei până la hidroliza proteinelor non-colagenice. Proprietățile fizice și studiile histologice arată că pielea obținută are o performanță mai bună decât pielea fără agenți tensioactivi. În mod evident, agenții tensioactivi pot fi utilizați ca auxiliari pentru sămăluire.

CUVINTE CHEIE: sămăluire, papaină, surfactant, piele de capră *kacang*, proprietăți fizice

L'ÉVALUATION DES PROPRIÉTÉS PHYSIQUES DE LA PEAU APRÈS LE PROCESSUS DE CONFITAGE EN COMBINANT L'ENZYME PAPAÏNE AVEC UN SURFACTANT

RÉSUMÉ. Le confitage est l'une des étapes importantes du processus de tannage, avec un rôle décisif dans l'obtention de certaines propriétés de la peau. Il est généralement réalisé à l'aide d'enzymes protéolytiques, telles que la papaïne. L'utilisation de tensioactifs, de sorbitol et de dodécylsulfate de sodium (SDS) peut faciliter la pénétration des enzymes dans la peau. Par conséquent, l'étude a eu le but d'évaluer l'utilisation de la papaïne dans le processus de confitage de la peau de chèvre de l'espèce *kacang* à l'aide de tensioactifs. Par conséquent, trois concentrations différentes (1, 1,5 et 2%) d'enzymes et deux types différents de tensioactifs (SDS et sorbitol) ont été utilisés dans le processus de confitage. On a réalisé la caractérisation des propriétés physiques et des études histologiques pour évaluer la peau obtenue. La présence de l'agent tensioactif dans le processus de confitage améliore l'absorption de la papaïne jusqu'à l'hydrolyse des protéines non-collagéniques. Les propriétés physiques et les études histologiques montrent que la peau obtenue a de meilleures performances que la peau sans tensioactifs. De toute évidence, les tensioactifs peuvent être utilisés comme auxiliaires pour le confitage.

MOTS CLÉS : confitage, papaïne, tensioactif, peau de chèvre *kacang*, propriétés physiques

INTRODUCTION

Hides and skins are an end product of animal production, which are turned into a value-added product through a series of chemical, enzymatic, and mechanical procedures [1, 2]. To obtain the high quality of leather products is needed adequate preparatory treatment of hides or skins during the wet end phase [3]. The main features of desired customers, i.e., softness, grain smoothness and fullness, can be improved in the beam house operation [4]. Bating is one of the important stages of the beam house operation to produce the soft leather products such as gloves, upholstery, and garments [3]. Therefore, it is of high interest to promote novel biocatalysts in the bating process.

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Bating is one stage of the beam house operation, which employs protease enzyme as biocatalyst at neutral pH [5, 6]. This process is essentially performed to remove non-collagenous proteins from pelt, open up collagen fiber network and make the pelt soft and smooth for the purpose of tanning [6]. Non-structural proteins can be degraded by general proteases such as nagarse, trypsin [6, 7], chymotrypsin [8], papain [5], and pronse [9]. Previous studies revealed that trypsin enzyme is regarded as the best bating enzyme due to its moderate proteolytic activity and safety [8], and a mixture of trypsin and ammonium salts is commonly used in bating to provide better performance [6].

Currently, novel approaches are being carried out to identify other enzymes and examine the enzymes that have been used for bating of leather processing. For example, papain is very heat-stable [10], easy to produce, and has very broad specificities to break off specific peptide bond in pelt protein [5, 7]. However, the activation of enzyme is affected by other factors such as surfactants, cofactor, chelator, and reducing agents [5]. Surfactants have suitable properties to facilitate the penetration and adsorption of the enzyme in addition to fiber swelling [5, 11]. Accordingly, there are few studies on surfactans and other agents for enzyme processing were examined in the application of tanning process (soaking and fatliquoring) [5, 6, 11]. Here, this study evaluates the application of surfactants [Sodium Dodecyl Sulfate (SDS) and Sorbitol] in papain treatment of bating process of *kacang* goat skin. The existence of surfactants is expected to improve the papain solubility, and is predicted to help the penetration and adsorption of enzyme and fiber structure of pelt.

EXPERIMENTAL

Materials and Methods

Twenty-one *kacang* goat skins were used as the leather material. Chemicals used for leather processing were of commercial/ industrial grade. Papain was purchased from local company, Yogyakarta, Indonesia. The chemical analysis and physical testing of leathers were conducted at the Center for Leather, Rubber and Plastics-Yogyakarta, Indonesia. Microscopic studies were performed at Department of Anatomy, Faculty of Veterinary Medicine, University of Gadjah Mada, Yogyakarta, Indonesia.

Tanning process was carried out according to recipe in Table 1. For control, chromed leather was processed without surfactant of bating step, while experimental leather was processed by combination level of enzyme (1-2%) with surfactant (SDS and Sorbitol).

Table 1: The recipe for leather processing

Process	Product	(%)	Duration (min)	Remarks
Soaking	Water	300		
	Water	0.15	30	
	Calcium hypochlorite	0.5		
Unhairing	100% Water, 1% Sodium sulphide, 0.3% Degreasing agent		30	
	Sodium sulphide	1	30	
	1% Sodium sulphide, 0.5% Lime		20	Stop 40 min
Liming	Water	300	30	
	Sodium sulphide	2	60	18 h (run 5 min/h)
	Calcium hydroxide	6	30	
Fleshing, Trimming, Weighing				
Deliming	Water	100		
	Ammonium chloride	1	10	
	Deliming agent	1	25	

Process	Product	(%)	Duration (min)	Remarks
Bating	Water	100		Temp. 37°C
	Surfactant	X		Control = no surfactant X = SDS (0.2%) or Sorbitol (0.15%)
	Enzyme	Y	45	For control was used 1% of Basozym T 1000-BASF Y = 1, 1.5, and 2% of papain
Pickling	Water	100		
	Sodium chloride	7	10	
	Formic acid	0.8	30	pH = 2.9 – 3.0
	Sulfuric acid	0.7	120	
Tanning	33% Basic chromium sulfate	6	60	
	Sodium formate	1	60	pH = 3.8 – 4.2
	Sodium bicarbonate	1	70	

Determination of Tensile Strength and Percent Elongation at Break

Samples for various physical tests from experimental and control crust leathers were obtained as per IUP methods [13]. Specimens were conditioned at 20±2°C and 65±2% RH over a period of 48 hrs. Tensile strength and elongation at break was measured as per standard procedures. Each value reported is an average of three samples.

Determination of Shrinkage Temperature

The shrinkage temperature of both control and experimental tanned leathers were determined using the Theis shrinkage tester [14]. A 2 cm sample, cut out from the leather was clamped between the haws of the clamp, which in turn was immersed in a solution of glycerol: water mixture (3:1). The solution was stirred using a mechanical stirrer attached with the shrinkage tester. The temperature of the solution was gradually increased and the temperature at which the sample shrinks was noted. Each value reported is an average of three experiments.

Histological Studies

Histological examination was carried out on the bated *kacang* goat skins. Samples were taken and preserved in 10% formalin for 48 hours. The fixed samples were dehydrated in an aqueous alcohol series (50 to 100%) and then cleared in xylene. Samples were finally embedded in paraffin wax and 10 µm sections were cut on a microtome, mounted and stained with Van Gieson staining method [15].

Statistical Analysis

All the physical parameters tests performed in triplicate were compared for the different types group of surfactants with papain concentration level using variance of ANOVA. The Turkey test for post hoc analysis was chosen because of its robustness. Statistical Package for Graphpad prism 8 program was used for all statistical analyses. All tests were considered statistically significant when they had a *p*-alpha value of less than 0.05.

RESULTS AND DISCUSSIONS

Bating is very important process in leather making, which facilitates achieving the softness feature of leather. This process connects to the proteolytic enzyme system and cannot be substituted by any mechanical and chemical procedure. However, the acceleration of enzymes needs to be improved by using surfactants. Surfactants have been widely used in many industrial products such as detergents, medicines, and anti-corrosive treatments due to their unique structures consisted of two different molecular parts and the broad range of selection [16]. Applying a surfactant can help the enzymatic process of bating stages. Hence, this work evaluated the use of surfactants in papain treatment of bating process of *kacang* goat skin.

Table 2: Physical properties of tanned leather (control) and combination of papain concentration (1%) with surfactant (SDS or Sorbitol)

Physical properties	Control	Type of surfactant		BIS standards [17]
		SDS	Sorbitol	
Tensile strength (Kg/cm^2)	261.76±0.34	352.11±0.53	346.60±0.76	200
Elongation at break (%)	75.17±0.53	64.02±0.96	66.37±0.67	40-65
Shrinkage temperature (°C)	90.67±0.58	94.67±0.58	94.33±0.58	-

Note: The values are means ± S.D. of three values

Physical properties are essential to examine the addition of surfactant on bating process. The physical strength measurements and shrinkage temperature of experimental and control leathers are given in Table 2. The tensile strength measurement was found to be better for experimental leathers, while the elongation at break was slightly lower than control. However, all the physical properties for both control and experimental leathers are found to meet the requirement of BIS standards for upper leathers. While the shrinkage temperature data show that the addition of surfactants in the bating process gives a good shrinkage temperature around 94°C compared to control 90°C. Despite the meet requirements, the tensile strength value of the control is relatively close to the BIS standards [17]. Clearly, the collaboration of enzyme and surfactant resulted in an improvement in tensile strength and shrinkage temperature of leather.

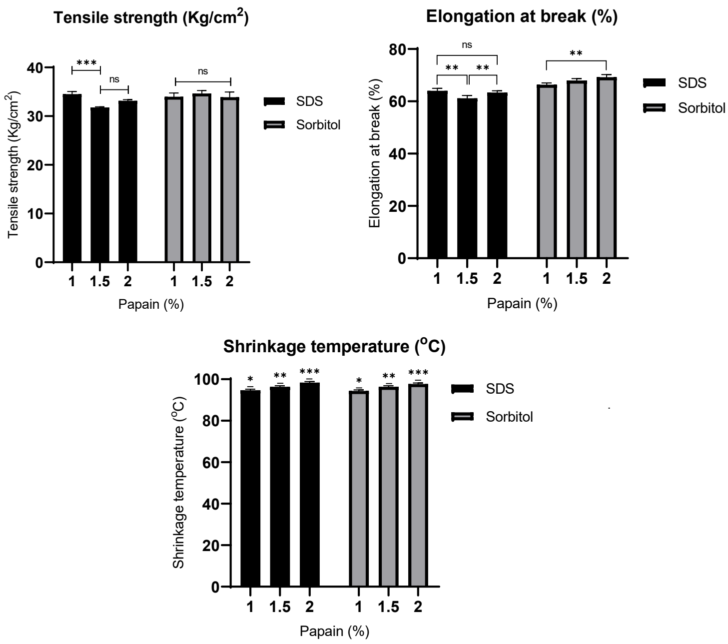
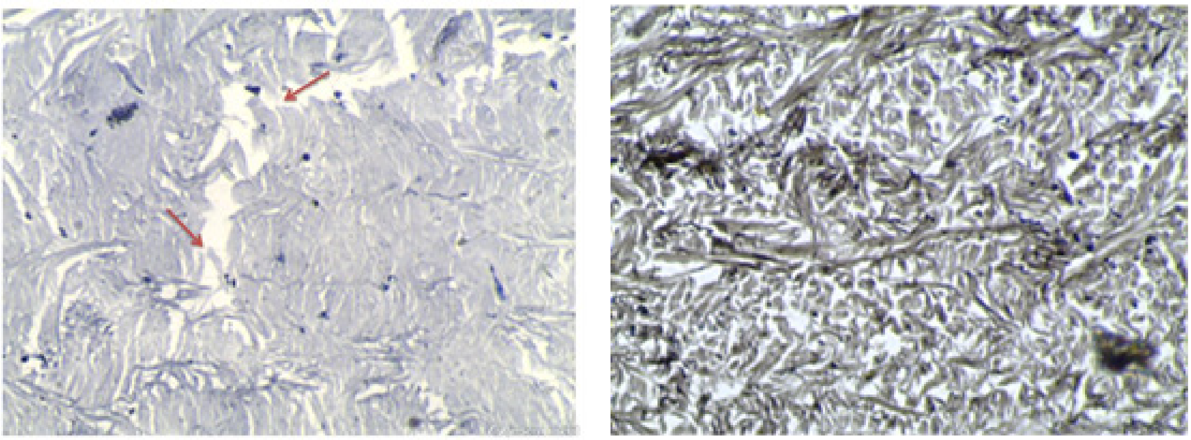


Figure 1. Physical properties of tanned leather from combination of papain concentration level with surfactant (SDS or Sorbitol)

Previous findings show that the use of enzymes and surfactants gives excellent physical properties of leather. This prompted us to investigate a papain concentration level (1%, 1.5%, and 2%) with two kinds of surfactant (anionic and nonionic) to gain information about the utilization of surfactant in bating process. Generally, the result of the examination gives an effect on the elongation at break and shrinkage temperatures of leather except for the tensile strength (Figure 1). For Sorbitol, elongation at break and shrinkage temperature of the samples are obviously improved linearly with the increase of papain in the range 1-2% whereas SDS is only on elongation at break measurement. Both tensile strength values have no effect by the increase of papain due to tanning enhancing the structural stability and the mechanical strength of the leather. While, the various types of surfactants in bating have an impact to the leather tensile strength, the elongation at break, and the shrinkage temperature [5]. The addition of surfactants can improve the removal of non-collagenous protein, and hence useful for the opening up of the fiber network [4, 18]. Concurrently, this causes the increase of the collagen reactivity group, thus the bond point between the chrome tanning agents and the skin was stronger [18].



A. Experimental leather **B. Control leather**

Figure 2. Residual of elastin fiber in grain layer (100 x hor.sec.)

The optical microphotographs taken for the bated skins are set out in Figures 2. The microphotographs show that the surfactant boost enzymes to open up more collagen fibers than the control, making the cavities among collagen as indicated by the red arrows. These results demonstrate that surfactants promote the acceleration of enzymatic reaction towards to the degradation of non-protein fibrous.

CONCLUSION

In conclusion, we here describe the application of surfactants in the bating process. The addition of surfactant improves the penetration of papain towards to proteolysis non-collagenous in satisfactory bating effect. Surfactant agents, such as SDS and sorbitol, perform better than control of leather non-surfactant according to the physical properties and histology studies. As a result, the surfactant agents can be considered as supplemental molecules for bating process.

Acknowledgements

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THE USE OF THE FUZZY INFERENCE SYSTEM IN DETERMINING THE QUALITY OF GOATSKIN AS RAW MATERIAL FOR THE LEATHER INDUSTRY

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THE USE OF THE FUZZY INFERENCE SYSTEM IN DETERMINING THE QUALITY OF GOATSKIN AS RAW MATERIAL FOR THE LEATHER INDUSTRY

ABSTRACT. Leather products are affected by the quality of the raw hide, the pre-treatment, and the tanning process. Furthermore, some leather products use quality goatskins with the right determination as raw materials. This study aims to determine the quality of goatskin through the use of Fuzzy Logic science. It was conducted using the Sugeno Fuzzy Inference System (FIS), while data analysis was carried out through the Matlab R2018a program. The criteria for using this method were the factors that affected the quality of raw goatskin such as thickness, length of the backline, skin area, number of scratches on the skin, number of holes, presence of boils, lice bumps, and hair loss. The results of the analysis showed that Sugeno FIS could be used to help determine the quality of goatskin with a good accuracy level with a MAPE value of 18%. Furthermore, the FIS method could be used as an appropriate modeling system for determining the quality of goatskin as a raw material for the leather tanning industry.
KEY WORDS: expert decision, quality of goatskin, tanning industry

UTILIZAREA SISTEMULUI DE INFERENȚĂ FUZZY PENTRU A DETERMINA CALITATEA PIEILOR DE CAPRĂ CA MATERIE PRIMĂ PENTRU INDUSTRIA DE PIELĂRIE

REZUMAT. Produsele din piele sunt afectate de calitatea pielii brute, de procesul de pre-tratare și de procesul de tăbăcire. În plus, unele produse din piele folosesc ca materie primă piei de capră a căror calitate înaltă a fost confirmată. Acest studiu își propune să determine calitatea pielii de capră prin utilizarea sistemului cu logică Fuzzy. Acesta a fost realizat folosind sistemul de inferență fuzzy Sugeno (FIS), în timp ce analiza datelor a fost efectuată cu ajutorul programului Matlab R2018a. Criteriile de utilizare a acestei metode au fost factorii care au afectat calitatea pielii brute de capră, cum ar fi grosimea, lungimea șirei spinării, segmentul pielii, numărul de zgârieturi pe piele, numărul de găuri, prezența furunculelor, a mușcăturilor de păduchi și pierderea părului. Rezultatele analizei au arătat că sistemul de inferență fuzzy Sugeno ar putea fi utilizat pentru a determina calitatea pielii de capră cu un nivel de acuratețe bun, cu valoarea MAPE de 18%. Mai mult, metoda FIS ar putea fi utilizată ca un sistem de modelare adecvat pentru determinarea calității pielii de capră ca materie primă pentru industria de pielărie.
CUVINTE CHEIE: decizie pe baza analizei experților, calitatea pielii de capră, industria de pielărie

L'UTILISATION DU SYSTÈME D'INFÉRENCE FLOUE POUR DÉTERMINER LA QUALITÉ DE LA PEAU DE CHÈVRE EN TANT QUE MATIÈRE PREMIÈRE POUR L'INDUSTRIE DU CUIR

RÉSUMÉ. Les produits en cuir sont affectés par la qualité du cuir brut, le prétraitement et le processus de tannage. De plus, certains produits en cuir utilisent des peaux de chèvre comme matières premières avec la confirmation de leur haute qualité. Cette étude vise à déterminer la qualité de la peau de chèvre grâce à l'utilisation du système de la logique floue. Elle a été réalisée à l'aide du système d'inférence floue Sugeno (FIS), tandis que l'analyse des données a été effectuée via le logiciel Matlab R2018a. Les critères d'utilisation de cette méthode ont été les facteurs qui affectent la qualité de la peau de chèvre brute, tels que l'épaisseur, la longueur de la moelle épinière, la zone de la peau, le nombre d'égratignures sur la peau, le nombre de trous, la présence de furoncles, les piqures de poux et la perte de cheveux. Les résultats de l'analyse ont montré que Sugeno FIS peut être utilisé pour aider à déterminer la qualité de la peau de chèvre avec un niveau de précision bon, avec une valeur MAPE de 18 %. En outre, la méthode FIS pourrait être utilisée comme système de modélisation approprié pour déterminer la qualité de la peau de chèvre en tant que matière première pour l'industrie du tannage du cuir.
MOTS-CLÉS : décision d'expert, qualité des peaux de chèvre, industrie du tannage

INTRODUCTION

The business processing system in the livestock industry has entered the digital era with the aim of increasing the use of technology. One of the technological improvements is on the part of the quality grouping system. This process must be carried out by the industry on a regular basis and needs to be carried out by industry players so that productivity can be achieved. However, sometimes the manual process cannot be carried out quickly due to the limitations of experts who select the quality of raw materials. This can have an impact on the quality and time in the process in the industry. On the other hand, technological developments and methods that continue to develop are very helpful for tanning industry players in carrying out the grading work to determine the quality of goatskin before the tanning process is carried out.

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Cattle hides have high economic value compared to other livestock by-products. Thus, this is the only livestock by-product still being exported by Indonesia. The number of exports to countries such as China, Hong-Kong, Italy, and Germany in 2015-2019 reached 5,500 tons. Meanwhile, these hides are exported in the form of non-food products, namely as raw materials for the leather tanning industry. Tanning treatment improves skin properties and increases its strength and flexibility.

The quality of the rawhides for tanning raw materials reduces when the skin is removed from the livestock's body. Moreover, this quality is influenced by the treatment before and during tanning, and at the time of processing [1chrome hydroxide was converted to chrome sulfate as tanning agent by addition of concentred sulfuric acid. Cr2O3 content of chrome sulfate was determined before being used for tanning. The result showed that Cr2O3 content was 3958.6 mg/l. Variation of recovered chrome sulfate concentrations for tanning jacket leather were 25, 50, 75 and 100% respectively. Controls were made with the use of 100% of industrial grade chrome sulfate. The best result were goat jacket leather tanned with recovered chrome of 75 and 100 % for its physical properties and 25% for its chemical properties. Test results of SEM showed that chrome was morphologically presence in tanned goat leather mass.","container-title":"Majalah Kulit, Karet, dan Plastik","DOI":"10.20543/mkkp.v31i2.176","ISSN":"18296971, 24604461","issue":"2","journalAbbreviation":"MKKP","language":"id","page":"107-114","source":"DOI.org (Crossref]. Factors that affect the quality of goatskin include thickness, backline length, skin area, scratches on skin, holes on skin, scars, tick and lice lumps, flea spots, and hair loss. Leather tanning is a method used to prevent skin degradation due to putrefactive bacteria, by mixing raw leather with tanning material [2].

A method used to determine the quality of rawhides is the Fuzzy Inference System (FIS) [3]. Moreover, Starkey *et al*. [4] stated that this system could be used to solve the problem of data and modeling uncertainty and optimize various objectives. FIS is a computational framework used to analyze systems that have a level of uncertainty in determining the numbers and limits of each factor for decision making [3]; Sitio [5] also stated that Fuzzy logic is capable of generating inputs and outputs without neglecting existing factors. Fuzzy logic could be applied as a problem-solving control system methodology applied to a system [6]. While according to Mukaromah [7], FIS is based on three components, namely basic concepts, databases, and reasoning mechanisms. Research related to the application of FIS for various needs including [8, 9], etc.

Previously, determination of skin quality was carried out manually, hence the process was deemed less efficient. Moreover, measuring the quality of goatskin is an important part of the raw material or leather tanning process, however, the this was still carried out manually, and was considered slow and imprecise. Therefore, this research aims to determine the quality of the skin using the Sugeno Fuzzy method because Sugeno FIS improves the weaknesses of a pure Fuzzy system by adding a simple mathematical calculation such as THEN. In this change, the fuzzy system has a Weighted Average Value in the IF-THEN fuzzy rules section [5]. The purpose of this study is to obtain the appropriate method for the precise and quick determination of quality of rawhides goatskin compared to the manual method.

EXPERIMENTAL

This study used salt-preserved goatskin from the Regional Technical Implementation Unit (UPTD) for leather processing in West Sumatra, Indonesia. The skin used is 100 samples which will be assessed by the expert and after being assessed by the expert, it is followed by the FIS method. It also implemented the FIS method developed by Sugeno, which is now referred to as the Sugeno method and was cited by Setiawan *et al*. [10]. The software used was the Matlab R2018a Tool Box. Fuzzy logic is the most suitable way to map an input space into an output space [11] and can be expressed in binary terms [12].

According to experts, the quality of goat skin is determined manually according to the custom in determining the quality of the skin. Experts are people who work in the leather tanning industry with more than 20 years of experience. Skin measurements were made using a roller measuring 80 cm. This size is used as a reference that the skin with a size of ≥80 cm is Quality 1. If goat skin has a size of less than 80 cm, then it is classified as Quality 2, Quality 3 and Reject. The quality assessment is also combined with the level of damage to the skin such as the surface of the goat skin damaged by ticks, hair loss and the size of the holes in the skin.

Sugeno FIS Logic

Fuzzy Sugeno is represented in the form of IF-THEN, and the system output is a constant or linear equation [11]. Sitio [5] states that the typical fuzzy rules in the Sugeno fuzzy model are formed: when x is A and y is B then z = f (x, y), where A and B are fuzzy sets in antecedents and z = f (x, y) function decisively in consequence. When f (x, y) is a first-order polynomial, the resulting FIS is called the first-order Sugeno fuzzy model. Furthermore, when f is constant, a zero-order Sugeno fuzzy model is generated. This fuzzy inference system has characteristics, which consequently are not part of a fuzzy set, but form a linear equation with data on the quality characteristics of goatskin according to the input criteria. This study used several variables that determine the quality of goatskin such as thickness, backline length, skin area, scratches on skin, holes on skin, scars, tick and lice lumps, flea spots, hair loss The following is the Figure 1 flow diagram of the fuzzification process.

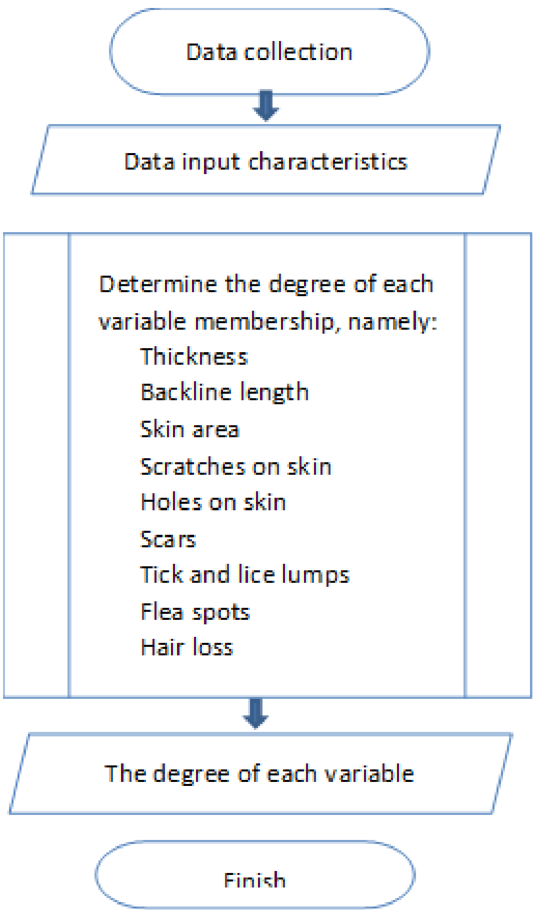

Figure 1. Flowchart of the fuzzification process

Fuzzification Process

After determining the variable membership function, a fuzzy logic rule is formed based on the existing data. In Table 1, the parameters, fuzzy and domain sets are shown for analyzing data on quality determination of goatskin. Fuzzy rules for the determination of goatskin quality were obtained from the opinion of experts at the Regional Technical Implementation Unit of as a leather processing facility Figure 2.

Table 1: Input parameter data, fuzzy set, and domain

Parameter	Fuzzy set	Domain (x)
	Thick	x >2
Thickness	Thin	0,5 <x≤2
	Very thin	x≤0,5
	Long	x >60
Backline length	Short	45 <x≤60
	Very short	x≤45
	Wide	x>3500
Skin area	Wide enough	3500 <x≤4500
	Narrow	x<5500
	Low	x≤3%
Scratches on skin	Medium	3%<x≤9%
	High	x>9%
	Low	x≤3%
Holes on skin	Medium	3%<x≤9%
	High	x>9%
	Low	x≤3%
Scars	Medium	3%<x≤9%
	High	x>9%
	Low	x≤3%
Tick and lice lumps	Medium	3%<x≤9%
	High	x>9%
	Low	x≤3%
Hair loss	Medium	3%<x≤9%
	High	x>9%

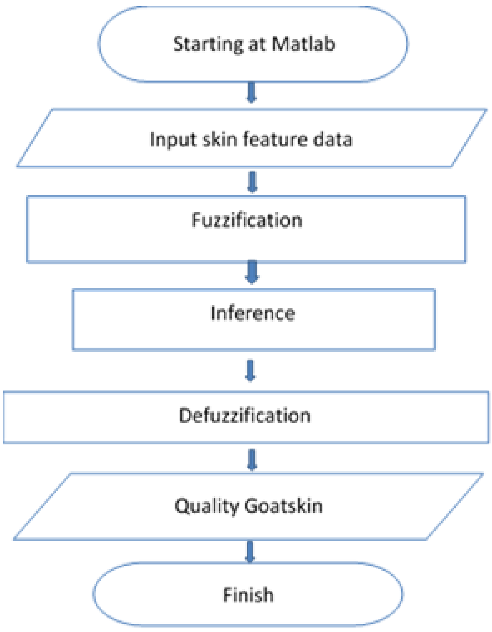

Figure 2. Flowchart of Sugeno's FIS method

Inference

The inference is a calculation framework based on IF-THEN fuzzy rules and thinking. The result output of each rule is z, which is in the form of a crisp set based on the predicate. This is also known as the Fuzzy Rule-Based System.

Defuzzification

Defuzzification was carried out using the Sugeno method, and is the final stage after fuzzification and inference are carried out [13]. Defuzzification is used to calculate the average output value of the alpha predicate and weighted calculation:

$$Defuzzification = \frac{\sum_{i=1}^{n} \propto i * zi}{\sum_{i=1}^{n} \propto i} \quad (1)$$

Description:

$\propto i$: *Degree of membership in rule to* $- i$

zi

The output parameter value of rule to $- i$

The quality classification and category of rawhides goatskin was obtained from experts in the leather tanning industry was obtained by interview. Therefore, they are grouped into: Quality 1, Quality 2, Quality 3 and Reject. Mean absolute percentage error (MAPE) is used to determine the percentage of accuracy from the use of the FIS method [14]. The MAPE value is used to analyze the predictions where the MAPE value is listed in Table 2 and the following calculation method:

$$\text{MAPE} = \frac{1}{n}\sum_{t-1}^{n}\left|\frac{At-Ft}{At}\right| \quad (2)$$

Description:
At = Value actual
Ft = Prediction value
n = data total

Table 2: Assessment based on quality category of goat skin

Category	FIS (Fuzzy Inference System)	Expert Opinion
Quality 1	66	61
Quality 2	17	26
Quality 3	10	6
Reject	7	7
Total	100	100

RESULTS AND DISCUSSION

Range and Membership Functions

The use of fuzzy application to determine the quality of goatskin as raw material for tanning is carried out by changing inputs such as thickness, backline length, skin area, scratches on skin, holes on skin, scars, tick and lice lumps, flea spots, hair loss. Therefore, the output is the quality of the goatskin. The basic fuzzy rules describe the relationship between the membership function and the membership function form by using the Sugeno method, it also shows that the system's output is in the form of constants or linear equations. The set starts when the domain with a zero degree of membership [0] moves to the right of the domain that has a higher degree of membership.

$$\mu[\text{x}] = \begin{cases} 0\,; x \geq a \\ \frac{1-a}{b-a} \quad ; a < x \leq b \\ 1\,; x \leq b \end{cases} \quad (3)$$

Description:

a^0 = The value of a domain that has zero membership degrees;

b^0 = The value of a domain that has a membership degree of one;

x^0 = The input values will be converted into fuzzy numbers.

The following is the fuzzification process of the determined variables used to measure the quality of goat skin:

Skin Thickness

Thickness variables are divided into three categories, namely thickness with very thin, thin, and thick levels and they are based on the thickness of the goatskin. The skin thickness variable is depicted using the membership function as shown in Figure 3. Among them are membership functions which include the following:

$$\mu[x]\text{Very thin} = \begin{cases} 0\,; x \leq 0{,}5 \\ \frac{1-x}{1-0{,}5} & ;0{,}5 < x \leq 1 \\ 1\,; x \geq 1 \end{cases} \quad (4)$$

$$\mu[x]\text{Thin} \quad = \begin{cases} 0\,; x < 1 \\ \frac{x-1}{1-0{,}5} & ;0{,}5 < x \leq 1 \\ \frac{2-x}{2-1}; 1 < x \leq 2 \end{cases} \quad (5)$$

$$\mu[x]\text{Thick} \quad = \begin{cases} 0\,;\ x \leq 1 \\ \frac{x-1}{2-1} & ;1 < x \leq 2 \\ 1\,; x > 2 \end{cases} \quad (6)$$

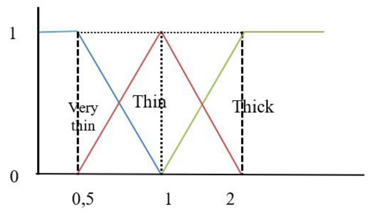

Figure 3. The fuzzy variable set for goatskin thickness

Back Line Length

The variable back line length is also divided into three types, namely the very short, short, and long levels. Where these levels are based on the back line length on the goatskin. The back line length variable is depicted using the membership function as shown in Figure 4. Among them are membership functions:

$$\mu[x]\text{Very short} = \begin{cases} 0\,; x \leq 45 \\ \frac{60-x}{60-45} & ;45 < x \leq 60 \\ 1\,; x \geq 60 \end{cases} \quad (7)$$

$$\mu[x]\text{Short} \quad = \begin{cases} 0\,; x \leq 55 \\ \frac{x-55}{55-45} & ;55 < x \leq 60 \\ \frac{65-x}{65-60}; 60 < x \leq 65 \end{cases} \quad (8)$$

$$\mu[x]\text{long} \quad = \begin{cases} 0\,;\ x \leq 60 \\ \frac{x-70}{70-60} & ;60 \leq x \leq 70 \\ 1\,; x > 70 \end{cases} \quad (9)$$

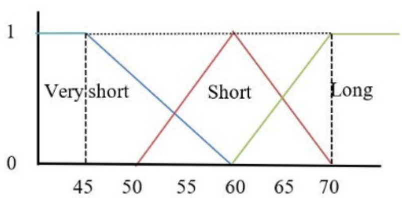

Figure 4. The set of fuzzy variables for the backline length of the goatskin

Skin Area

The goatskin area variable is divided into three, namely with a small wide, wide enough, wide. The skin area variable is depicted using the membership function as shown in Figure 5. The following membership functions include:

$$\mu[x]\text{Small wide} = \begin{cases} 0\,; x \leq 4500 \\ \frac{4500-x}{4500-3000} & ;3000 < x \leq 4500 \\ 1\,; x > 3000 \end{cases} \quad (10)$$

$$\mu[x]\text{Wide enough} = \begin{cases} 0\,; x \leq 3500, \\ \frac{x-2500}{4500-2500} & ;3500 < x \leq 4500 \\ \frac{5500-x}{4500-3500}; 4500 < x \leq 5500 \end{cases} \quad (11)$$

$$\mu[x]\text{Wide} = \begin{cases} 0 \,;\, x \le 4500 \\ \frac{x-4500}{5000-4500} \;;\, 4500 \le x \le 6000 \\ 1 \,;\, x > 6000 \end{cases} \tag{12}$$

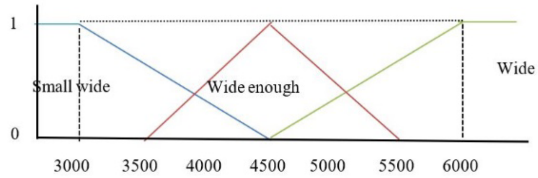

Figure 5. The set of fuzzy variables for goatskin area

Scratches on Skin

Variable scratches on the skin are also divided into three types, namely low, medium and high. The scratches on skin variable is depicted using the membership function as shown in Figure 6. Furthermore, these levels are based on scratches on goatskin with a membership function:

$$\mu[x]\text{Low} = \begin{cases} 0 \,;\, x \le 6 \\ \frac{6-x}{6-3} \;;\, 3 < x \le 6 \\ 1 \,;\, x \ge 3 \end{cases} \tag{13}$$

$$\mu[x]\text{Medium} = \begin{cases} 0 \,;\, x \le 6, \\ \frac{x-3}{6-3} \;;\, 3 < x \le 6 \\ \frac{9-x}{9-6} ;\, 6 < x \le 9 \end{cases} \tag{14}$$

$$\mu[x]\text{High} = \begin{cases} 0 \,;\, x \le 6 \\ \frac{x-6}{9-6} \;;\, 6 \le x \le 9 \\ 1 \,;\, x > 9 \end{cases} \tag{15}$$

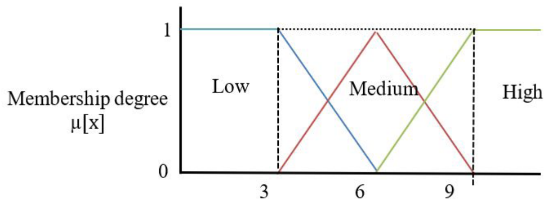

Figure 6. The set of Fuzzy variables for scratches on goatskin

Hole in Skin

The hole in skin variable is depicted using the membership function as shown in Figure 7. This hole in the skin variable is divided into three types, namely low, medium and high levels, and are based on holes in goatskin with a membership function:

$$\text{Low} = \begin{cases} 0 \,;\, x \le 6 \\ \frac{6-x}{6-3} \;;\, 3 < x \le 6 \\ 1 \,;\, x \ge 3 \end{cases} \tag{16}$$

$$\mu[x]\text{Medium} = \begin{cases} 0 \,;\, x \le 6, \\ \frac{x-3}{6-3} \;;\, 3 < x \le 6 \\ \frac{9-x}{9-6} ;\, 6 < x \le 9 \end{cases} \tag{17}$$

$$\mu[x]\text{High} = \begin{cases} 0 \,;\, x \le 6 \\ \frac{x-6}{9-6} \;;\, 6 \le x \le 9 \\ 1 \,;\, x > 9 \end{cases} \tag{18}$$

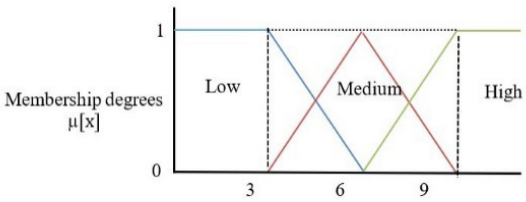

Figure 7. The set of fuzzy variables for holes in goatskin

Scars

The variable known as the presence of scars on the skin is divided into three levels, namely low, medium and high levels. The skin scars variable is depicted using the membership function as shown in Figure 8. These levels are based on the presence of scars in goatskin and they possess membership functions:

$$\text{Low} = \begin{cases} 0\,; x \leq 6 \\ \frac{6-x}{6-3} \quad ; 3 < x \leq 6 \\ 1\,; x \geq 3 \end{cases} \tag{19}$$

$$\mu[\text{x}]\text{Medium} \quad = \begin{cases} 0\,; x \ \leq 6, \\ \frac{x-3}{6-3} \quad ; 3 < x \leq 6 \\ \frac{9-x}{9-6}; 6 < x \leq 9 \end{cases} \tag{20}$$

$$\mu[\text{x}]\text{High} \quad = \begin{cases} 0\,;\ x \ \leq 6 \\ \frac{x-6}{9-6} \quad ; 6 \leq x \leq 9 \\ 1\,; x > 9 \end{cases} \tag{21}$$

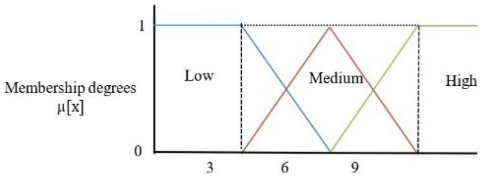

Figure 8. The set of fuzzy variables for the presence of scars on goat skin

Tick and Lice Lumps

The presence of ticks and lice on the skin is a variable that is divided into three levels, namely low, medium and high levels. The tick and lice lumps thickness variable is depicted using the membership function as shown in Figure 9. This ranking is based on the presence of ticks and lice lumps on the goatskin with a membership function:

$$\text{Low} = \begin{cases} 0\,; x \leq 6 \\ \frac{6-x}{6-3} \quad ; 3 < x \leq 6 \\ 1\,; x \geq 3 \end{cases} \tag{22}$$

$$\mu[\text{x}]\text{Medium} \quad = \begin{cases} 0\,; x \ \leq 6, \\ \frac{x-3}{6-3} \quad ; 3 < x \leq 6 \\ \frac{9-x}{9-6}; 6 < x \leq 9 \end{cases} \tag{23}$$

$$\mu[\text{x}]\text{High} \quad = \begin{cases} 0\,;\ x \ \leq 6 \\ \frac{x-6}{9-6} \quad ; 6 \leq x \leq 9 \\ 1\,; x > 9 \end{cases} \tag{24}$$

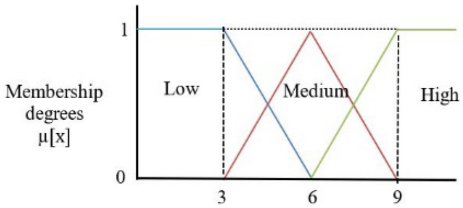

Figure 9. The set of fuzzy variables for the presence of tick and lice lumps goatskin

Hair Loss

The variable hair loss on the skin is divided into three levels, consisting of low, medium and high levels. The hair loss variable is depicted using the membership function as shown in Figure 10. These levels are based on the presence of tick and lice lumps on the skin of goats with membership functions:

$$\text{Low} = \begin{cases} 0\,;x \leq 6 \\ \frac{6-x}{6-3} \quad ;3 < x \leq 6 \\ 1\,;x \geq 3 \end{cases} \tag{25}$$

$$\mu[\mathrm{x}]\text{Medium} = \begin{cases} 0\,;x \leq 6, \\ \frac{x-3}{6-3} \quad ;3 < x \leq 6 \\ \frac{9-x}{9-6};6 < x \leq 9 \end{cases} \tag{26}$$

$$\mu[\mathrm{x}]\text{High} = \begin{cases} 0\,;\,x \leq 6 \\ \frac{x-6}{9-6} \quad ;6 \leq x \leq 9 \\ 1\,;x > 9 \end{cases} \tag{27}$$

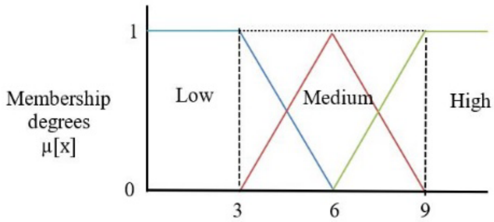

Figure 10. The set of fuzzy variables for the hair loss of goat skin

Inference

Inference is a fuzzy implication rule obtained by combining each input variable. Consequently, the 20 fuzzy rules were obtained from the opinion of experts from the Regional Technical Implementation Unit of Padang Panjang City in 2019. The fuzzy inference method used was the first-order Fuzzy Sugeno, while the antecedents and consequences were represented by propositions in a fuzzy set, and a linear equation respectively. The consequence in this research, refers to data obtained in the field and information from informants. Rules and implications were formed to state the relationship between input and output. Furthermore, the operator used to connect two inputs is known as the AND operator, and IF-THEN is used to draw maps between the input-outputs. Propositions that follow IF are called antecedents, while propositions that follow THEN are called consequent [10]. The results on the classification analysis of the quality of goat skin used MatLab with FIS Sugeno. The display of the rule composition and defuzzification can be seen in Figure 11 below:

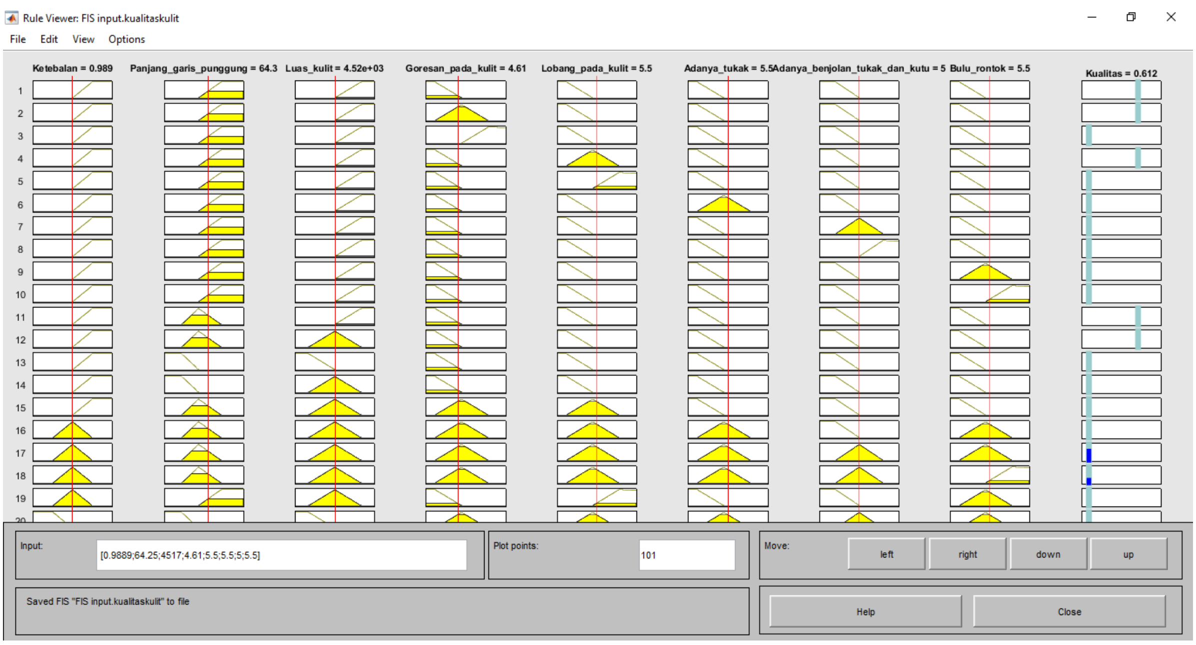

Figure 11. Simulation results of the quality of goat skin using MatLab

Testing Data

Table 3: Recapitulation of Sugeno FIS assessment

Variable	Fuzzy set	Domain	Number of skins with values based on domain
Thickness	Thick	x >2	
	Thin	0,5 <x≤2	4
	Very thin	x≤0,5	96
Back line length	Long	x >60	
	Short	45 <x≤60	2
	Very short	x≤45	98
Skin area	Wide	x>3500	
	Wide enough	3500 <x≤4500	17
	Narrow	x<5500	83
Scratches on skin	Low	x≤3%	
	Medium	3%<x≤9%	2
	High	x>9%	98
Holes in skin	Low	x≤3%	
	Medium	3%<x≤9%	4
	High	x>9%	96
Scars	Low	x≤3%	2
	Medium	3%<x≤9%	4
	High	x>9%	94
Tick and lice lumps	Low	x≤3%	5
	Medium	3%<x≤9%	7
	High	x>9%	87
Hair loss	Low	x≤3%	2
	Medium	3%<x≤9%	3
	High	x>9%	95

The quality classification of goat skin was obtained from 100 test data. Meanwhile, the results of the test with Fuzzy logic compared to expert opinion can be seen in Table 3. Based on the quality assessment of goat skin, it was found that determining the quality of rejected leather through FIS and expert opinion had similar results. This is because the criteria for rejected skin depends on the level of damage to the skin such as small skin, and defects with a high degree of damage. However, there are differences in the number of skin types consisting of Quality 1, 2 and 3. The value of Quality 1 using the FIS method shows a higher number than expert opinion. This is presumably because the FIS observation uses measurable variables and has clear boundaries, resulting in an overall value for determining the quality of goat skin. Furthermore, the inference system can read explicit values, which lead to desired results in the output variables. Meanwhile, the expert assessment system is carried out manually, by observing parameters such as the color of the fur which looks alive and not gloomy, hair loss due to bacteria, holes, scars and defects in the raw skin caused during peeling work, and defects because of various skin diseases. Therefore, the FIS method is a more appropriate method to determine the quality of raw goatskin.

Table 4: MAPE values to measure the accuracy level of the FIS method

MAPE value	Prediction accuracy
MAPE ≤ 10%	Hight
10% < MAPE ≤ 20%	Good
20% < MAPE ≤ 50%	Reasonable
MAPE > 50%	Low

Table 4 shows that the defuzzification process used can produce an overall value for determining the quality of goat skin from a fuzzy consequent area. This is because the inference system can read firm values, and produces the desired results in the output variable. After comparing 100 goat skin samples using Sugeno's logical assessment it was found that 7 goat skins had different quality determination results from experts.

From the research that has been done, the results of the comparison of the Sugeno FIS assessment using the average percentage or Mean Absolute Percentage Error (MAPE) are shown in Table 4. The MAPE value obtained from each quality indicates that the accuracy of this FIS method is high. Based on the total MAPE obtained is 18%. Thus, these results indicate that the accuracy of forecasting results using FIS has a good level of accuracy. Based on this, Sugeno FIS can be used as a determinant of the quality of goat skin based on thickness, length of back line, skin area, number of scratches on the skin, number of holes, presence of ulcers, tick bumps, and hair loss. Mean Absolute Percentage Error is a method that can be used to calculate error from predicted least square method of data [15], this way in calculating the predicted error gives the organization's choice to consider the utilization of a method of prediction [16].

CONCLUSIONS

Sugeno FIS can be used to determine the quality of goat skin with a good accuracy level. This system can be used as an appropriate model in determining the quality of goat skin as a raw material in the leather tanning industry.

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FUR SKIN – A VALUABLE MATERIAL, CONSIDERATIONS ON QUALITY ASSESSMENT

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FUR SKIN – A VALUABLE MATERIAL, CONSIDERATIONS ON QUALITY ASSESSMENT

ABSTRACT. This work is intended to reveal the most important physico-chemical and physico-mechanical characteristics of the fur skins that determine their quality. Values of these characteristics are discussed as depending on the semifinished product related to technological significance and consequence for the user. The main characteristics and values for fine furs are proposed, thus providing some guidelines in evaluating their quality. Investigations in this field can lead to the determination of some new quality characteristics of woolen sheepskins and fine furs. A case study on fine and common furskins is presented and reveals the uniqueness and versatility of the combination of two protein layers, represented by natural furs, composed of collagen and keratin, unmatched by any synthetic material.

KEY WORDS: woolen sheepskin, fine furs, quality evaluation

BLĂNURILE – MATERIAL VALOROS, CONSIDERAȚII ASUPRA EVALUĂRII CALITĂȚII

REZUMAT. Această lucrare are ca scop să prezinte cele mai importante caracteristici ale blănurilor care definesc calitatea acestora. Valorile acestor caracteristici sunt discutate în funcție de modul lor de prelucrare și domeniul de utilizare al produselor semifinite. Principalele caracteristici și valorile acestora pentru blănurile nobile sunt propuse, oferind astfel câteva repere în evaluarea calității acestora. Cercetările în această direcție pot conduce la identificarea unor noi caracteristici cu potențial de utilizare crescută a produselor finite. Un studiu de caz asupra blănurilor nobile și comune este prezentat și dezvăluie unicitatea și versatilitatea combinației dintre două proteine, colagenul și cheratina, neegalată de niciun material sintetic.

CUVINTE CHEIE: blănuri de ovine, blănuri nobile, evaluarea calității

LA FOURRURE – MATÉRIAU DE VALEUR, CONSIDÉRATIONS SUR L'ÉVALUATION DE LA QUALITÉ

RÉSUMÉ. Cet article présente les caractéristiques les plus importantes des fourrures qui définissent leur qualité. Les valeurs de ces caractéristiques sont discutées en fonction de leur transformation et du domaine d'utilisation des produits semi-finis. Les principales caractéristiques et valeurs des fourrures nobles sont proposées, donnant ainsi quelques repères pour évaluer leur qualité. La recherche dans ce sens peut conduire à l'identification de nouvelles fonctionnalités avec un potentiel d'utilisation accrue des produits finis. Une étude de cas sur les fourrures nobles et communes est présentée et révèle l'unicité et la polyvalence de la combinaison de deux protéines, le collagène et la kératine, inégalée par toute matière synthétique.

MOTS CLÉS : peau de mouton, fourrures nobles, évaluation de la qualité

INTRODUCTION

Fur skin production is one of the oldest human activities, remaining largely traditional in many regions worldwide. Recent archeological discoveries and analyses [1] showed that leather and fur skins clothing has been processed for 120,000-90,000 years. Apart from the climate hostility, the symbolistic and ornamental clothes were the main functions of animal skin clothes. The bone tools for animal skinning were found and identified next to sand fox, golden jackal, and wildcat remains. It is known that 120,000 years ago, 67 vertebrate species of animals were hunted for meat and hide in North Africa. It can be concluded that food procurement was always connected to the processing of hides or skins in imputrescible materials for clothes, contributing to the survival of the human species.

Fur wearing knew different social significance in historical human development eras, from efficient material for cold protection to decorative accessory for social status expression [2].

Nowadays wild furs (beaver, raccoon, muskrat, sable, coyote, red fox, lynx, cat lynx, marten, otter, wolf, wolverine and black bear) represent 15-20% of the traded fine fur skins and are hunted under programs set by governmental authorities in agreement with environmental biologists [3]. The most important fine fur skin production is generated by intensive breeding of minks, foxes, chinchilla, swakara and rabbit, especially for luxury goods [4]. The fine fur skins

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durability depends on the animal species [5], the most durable being water animal fur skins (the otter has the coefficient 100%), followed by mink (70%), marten (65%), karakul and sable (65%), muskrat, red fox (50%), blue fox (40%), chinchilla and farm rabbit (20%) and hare (5%). The main important farming producers are Denmark, Finland, the Netherlands, Poland, and China.

Woolen sheepskins represent more accessible and popular furs, traditionally produced in countries with developed animal husbandry sectors, like UK, Spain, Romania, Greece, France, Italy, etc. [6]. Fur skins are recognized as irreplaceable materials due to their unique resistance to water, thermal insulation, hygienic transfer of water vapors from inside to outside, and natural aspect [7]. The use of wild Canadian bear skins in royal army caps from UK, Belgium, Canada; karakul or merinos fur skins for collars or caps of the ceremonial or regular army are a few examples of fur skins uniqueness. The ethical aspects of fur skins wearing transcend the animal protection issues (hunting regulations, protected species, life quality of intensively grown animals) and rich global issues such as regenerative agriculture [8]. Fur skin production could be divided into the area of the woolen sheepskins – the most widespread and popular, and fine fur skins – very diverse and valuable. The present problems of competition, consumer protection, and quality assurance, as well as ecological ones, also call for this area of activity to find some specific methods for quality quantification and attestation. In this context the paper's aim is to point out some aspects of fur skin quality, which can be useful for scientists, state regulators, producers and commercial agents.

Quality Control of Fur Skins

The most important factors influencing the fur skin quality could be classified in factors during animal life (*in vivo*) and factors during fur skin processing (*in vitro*), all being summarized in Figure 1. Figure 1 shows that when estimating the quality of the processed fur skins, a lot of factors are influencing its level. The control of shown processing stages could ensure the manufacture of high-quality products. With woolen sheepskins, the relationship between hair fineness and leather strength is often in an inverse ratio ordering the end destination of the woolen sheepskin to the assortments processed on the leather or on the wool. Suede shearlings are usually made from coarse woolen skins, but have a strong leather able to undergo the buffing.

Final quality control for these woolen skins consists of organoleptically assessing the leather (elongation, softness, feel, fullness), the wool (fineness, defaulting level, hair length, individualization level of hairs, etc.), physical-mechanical, chemical tests, and by assessing the usable area. Physical-mechanical and chemical tests revealing the quality of suede shearlings and standardized values to the semifinished products are shown in Table 1 [8].

Table 1 presents tests mostly applied to leather, which must show good levels for elongation at break and load at the tensile break, stitch-tear strength, or tear strength so that it would be suitable for manufacturing clothes. The values for shrinkage tests, as well as the amount of chrome oxide, show the tannage level for these assortments and provide for the dimensional steadiness during processing. Wool dye manufacturers [9] suggest even higher values for these characteristics for tone-in-tone assortments requiring wool dyeing at 60-65°C.

The final appearance of the wool cover depends highly on the defaulting level and finishing method. Tests for to-and-fro rubbing fastness of leather dyeing, and colour fastness to water spotting are also particularly important for clothes durability. Fat and total ash levels in leather could give information about the final weight of fur clothes, which is an important characteristic for wearers' comfort. The pH of the aqueous extract ensures the clothes manufacturer and wearer that no undesirable acid hydrolysis phenomena will take place, which could affect the stitch strength. The test for wetting fastness requires slightly hydrophilic leather, allowing the fur clothes to be worn in rain without straining. Water repellency of nappalan fur skins (dermal layer covered with film forming polymers or with transfer foils) clothes as well as their soil repellency are very much appreciated as more durable and comfortable versions for dynamic persons. For woolen sheepskins processed on wool, the quality control is made organoleptically by assessing the look of leather (softness, bending resistance, fullness, elongation, smoothness, thickness, etc.), wool (hair length, felting level, gloss, individualization of hair, etc.), usable area, by physical-mechanical and chemical tests (Table 2) [9].

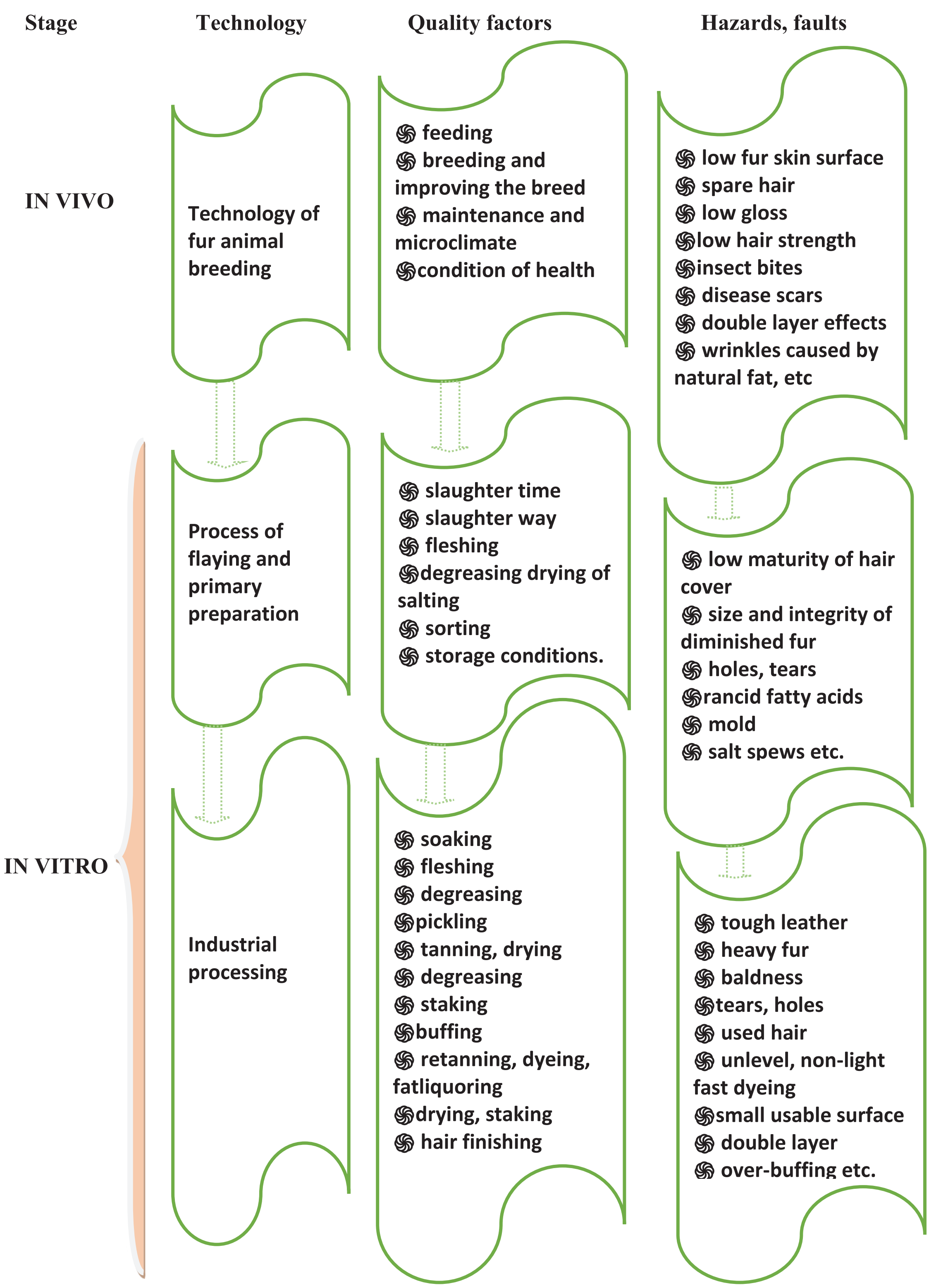


Figure 1. Diagram of *in vivo* and *in vitro* quality factors for fur skins

Table 1: Physical-chemical and physical-mechanical characteristics for shearling suede sheepskins

Characteristic	Admissible value	Method
Load at tensile break, longitudinal (N), min	100	ISO 3376
Load at tensile break, transversal (N), min	70	ISO 3376
Load at tensile crack of surface (N), min	40	ISO 3376
Elongation at break, longitudinal (%), min	40	ISO 3376
Elongation at break, transversal (%), min	45	ISO 3376
Elongation at 30N stress (%), min	20	ISO 3376
Stitch-tear resistance (N), min	30	ISO 23910
Tear load (N), min	15	ISO 3377-2
Pull strength (N), min	Good	Manual from 4 points
Colour fastness to to-and-fro rubbing (colour change and staining), 20 dry white felt cycles: - colour change of the leather, min - the staining of the felt, min	 4-5 3	ISO 11640
Colour fastness to water spotting	No halo after 14 h	ISO 15700
Shrinkage temperature (°C), min	80	ISO 3380
Boiling test at 80°C (%), min	5	IUP 24
Volatile matter (%), max - in leather - in wool	 14 10	ISO 4684
Total ash (%), max	5	ISO 4047
Cr_2O_3, (%), min	1	ISO 5398-1
Aqueous extract pH, min	4	ISO 4045
Dichloromethane extractable substances (%), max: - in leather - in wool	 7-16 2	ISO 4048

Table 2: Physical-chemical and physical-mechanical characteristics for woolen sheepskins processed on wool

Characteristic	Admissible value	Method
Load at tensile break, longitudinal (N), min	110	ISO 3376
Load at tensile break, transversal (N), min	80	ISO 3376
Load at tensile crack of surface (N), min	40	ISO 3376
Elongation at break, longitudinal (%), min	45	ISO 3376
Elongation at break, transversal (%), min	50	ISO 3376
Elongation at 30N stress (%), min	21	ISO 3376
Stitch-tear resistance (N), min	25	ISO 23910
Tear load (N), min	15	ISO 3377-2
Pull strength (N), min	Good	Manual from 4 points
Colour fastness to to-and-fro rubbing (colour change and staining), 20 dry white felt cycles: - colour change of the leather, min - the staining of the felt, min	 4-5 3-4	ISO 11640
Wool colour fastness to light: - colour change of the wool, min - colour change of the textile, min	 6 3	ISO 105-B02

Shrinkage temperature (°C), min	80	ISO 3380
Volatile matter (%), max - in leather - in wool	 14 10	ISO 4684
Total ash (%), max	4	ISO 4047
Cr_2O_3, (%), min	1	ISO 5398-1
Aqueous extract pH, min	4	ISO 4045
Dichloromethane extractable substances (%), max: - in leather - in wool	 12 2	ISO 4048

Table 2 shows that the admissibility levels are similar to those for shearling suede, some slightly higher levels are noted for break tests, thus revealing a derma structure closer to native structure, able to orientate itself in the stress direction because of the simpler technology and less interfibrillarily deposited chemical auxiliary materials.

More attention is paid to the dyeing fastness tests for wool where the rating 6 (for dark shades) assumes a severe dyestuff selection. Oxidizing dyestuffs, which could be a possible hazard for wearers, are not allowed.

Quality control for karakul fur skins is made by assessing the quality of wool cover (curling characteristics, shape, uniformity, density, strength, colour, gloss, elasticity, and fineness), leather (extensibility, suppleness, smoothness, defaulting level, size), the type and size of faults and physical-mechanical and chemical tests. Physical-mechanical and chemical characteristics and admissible values for karakul fur skins are shown in Table 3 [10].

Table 3: Physical-chemical and physical-mechanical characteristics for karakul and half-breed karakul fur skins

Characteristic	Admissible value	Method
Load at tensile break, longitudinal (N), min	40	ISO 3376
Load at tensile break, transversal (N), min	30	ISO 3376
Load at tensile crack of surface (N), min	20	ISO 3376
Elongation at break, longitudinal (%), min	40	ISO 3376
Elongation at break, transversal (%), min	40	ISO 3376
Elongation at 30N stress (%), min	18	ISO 3376
Stitch-tear resistance (N), min	30	ISO 23910
Tear load (N), min	6	ISO 3377-2
Colour fastness to to-and-fro rubbing (colour change and staining), 20 dry white felt cycles: - colour change of hair, min - the staining of the felt, min	 5 3	ISO 11640
Wool colour fastness to light: - colour change of the wool, min Wool colour fastness to light and weather: - colour change of the wool, min	 4 4	ISO 105-B02
Shrinkage temperature (°C), min	80	ISO 3380
Volatile matter (%), max - in leather - in wool	 14 10	ISO 4684
Total ash (%), max	5	ISO 4047
Cr_2O_3, (%), min	1.5	ISO 5398-1

Aqueous extract pH, min	4	ISO 4045
Dichloromethane extractable substances (%), max: - in leather	10-16	ISO 4048

The immature collagen structure of karakul skins brings about low break, tear and surface crack strengths. The constant light and weather fastness assure the wearer of a good behavior of the products made from these skins.

Regarding the fine fur skins, their small surface area, the high importance of the fur cover which has to be processed so as to preserve the native colour and integrity as much as possible, all these elements make the organoleptic assessment fundamental. One of the most important stages in assessing the quality of fine fur skins is in the raw stage when they are offered for sale in auctions. The Scandinavian classification system is one of the most appreciated systems from this point of view alongside other grading systems like Nafa, Kopenhagen Fur and Sojuzpushnina [12]. General characteristics like skin size, sex, colour shade and the quality of fur cover are important. The most important quality evaluating criteria for fur skins refer preponderantly to the fur cover: underfur density, guard hair length, the ratio of underfur to guard hairs, guard hair density, gloss, softness and resilience and finally the colour clarity. Sorting the fur skins and evaluating their quality when selling by auction are done by highly experienced persons. In this case, only the fur skin size can be measured by instrumental methods.

But this quality evaluation does not ensure the preservation of this level of quality after processing the fur skins. Therefore, it is recognized that hidden defects play an important role in the risks of fur processing. The processing of fine fur skins is made largely (95% of total fine fur skins) for assortments with fur outward and less for those with fur inward (nappalan, etc.). The native look of fur cover is generally kept by performing white tannages (without basic chrome salts imparting an unpleasant green shade) and a severe selection of fatliquoring and degreasing materials.

However, some unavoidable faults of fur cover, also the fashion requirements impose an elevation of their quality by dyeing or shearing. In this case, the rubbing, light and weather fastness of dyeing are very important properties for the wearer. A system for evaluating the fur skin quality must involve a minimum of physical-mechanical and chemical tests attesting and assuring the quality both for clothes manufacturers and wearers.

Thus, beside the organoleptic assessment which remains very important, we consider the following tests to be important:

- load at tensile break, longitudinal and transversal (ISO 3376) with recommended values of 14-50 N;
- shrinkage temperature (ISO 3380) with recommended values of 55-80°C;
- chromium oxide content (ISO 5398-1) with recommended values of 0.5-1.5%;
- volatile matter (ISO 4684) with recommended values of max. 14%;
- pH of aqueous extract (ISO 4045) with recommended values of min. 3.5;
- dichloromethane extractable substances from leather and hair (ISO 4048) with recommended values of 10-25% and maximum 2%, respectively;
- colour fastness to to-and-fro rubbing (ISO 11640) with recommended values of min. 3;
- artificial light and weather fastness (ISO 105:B02) with recommended values of min. 4.

For every type of fur skin, the allowable limits must be specified.

The requirements related to the wearer's health assurance have imposed a series of allowable limits for possibly hazardous substances. These characteristics and their limits are shown in Table 4.

Table 4: Hazardous substances in fur skin and their limits

Critical chemical substances	Admissible value	Method
Azo dyes (aryl amines), mg/kg, max	20	ISO 17234-1 and ISO 17234-2
Chlorophenols content, including isomers as applicable, mg/kg, max	0.5	ISO 17070
Formaldehyde, mg/kg, max	20	ISO 17226-1 and ISO 17226-2
Chromium VI, mg/kg, max	3	ISO 17075
Extractible heavy metals, mg/kg, max		ISO 17072-1
Cr	100	
Pb	0.8	
Cd	0.1	
As	0.2	
Hg	0.02	
Sb	5	
Co	1	
Cu	50	
Ni	4	
Zn	100	
Ba	100	
Se	100	
Dimethyl fumarate, mg/kg, max	0.1	ISO 16186
Organotin compounds, mg/kg, max	0.1	ISO 16179

MATERIALS AND METHODS

Four kinds of fur skins were purchased from fur skins producers: polar fox, dyed mink, woolen sheepskin processed on wool (Pannofix type) and suede shearling sheepskin. The fur skins were analyzed by standard methods for physical-chemical, physical-mechanical and fastness tests according to methods in force. Scanning electron microscopy was performed using a FEI Quanta 200 microscope for showing the features of wool and hair morphologies for sheep skins and fine fur skins in correlation to wearing characteristics. Wearing tests were performed according to a method described in STAS 13134 based on the initial and final weighing of fur skins rubbed on the fur. The results are expressed in percentages and the higher the value, the lower the wear resistance. These results were correlated with cystinic sulfur content of wool or hair analyzed according to SR 13206. Other tests were performed for water vapour absorption behavior determination and light resistance of fur skins at 96% relative humidity (Binder MKF 56) in order to evaluate the wearing performances of different fur skins, according to ISO 4684 and ISO 105-B02 methods.

RESULTS AND DISCUSSIONS

Research on Fur Skin Quality Control – A Case Study

Four kinds of fur skins were assessed with the final aim of understanding the correlation of structural and processing characteristics with chemical, mechanical, and wear properties. The selected furs skins were: polar fox, mink, suede sheepskin, and wool-on sheepskin. The main physical-chemical characteristics presented in Table 5 show the high diversity of structural and processing-specific properties. The high content of ash suggests that the fur skins have a high weight.

Table 5: Chemical characteristics of fine fur skins, suede, and wool-on sheepskins

Fur skin	Moisture	Ash	Fat extract	Metal oxides	Cr_2O_3	pH of extract
Polar fox	11.16	6.78	18.08	3.00	1.04	4.1
Mink	16.60	5.53	22.08	2.60	0.74	4.8
Wool on sheepskin	11.26	5.23	14.88	4.82	3.08	4.1
Suede Fur sheepskin	13.53	3.82	10.02	3.42	2.21	4.1

Table 6 shows that wool on sheepskin has values for most of the physical-mechanical tests under admissible limits, while mink and fox fur skins show very high values (with exception of tear load of polar fox fur skin).

Table 6: Physical-mechanical characteristics of fine fur skins, suede, and wool-on sheepskins

Fur skin	Load at tensile break, longitudinal, N	Load at tensile break, transversal, N	Tear load, N	Stitch-tear resistance, N
Polar fox	140	55	5	25
Mink	192	72	17	33.7
Wool on sheepskin	30	14	6	32.2
Suede Fur sheepskin	100	144	19.5	46.7

The correlation of cystinic sulphur of wool and hair [13] with wear fastness [14] (STAS 13134) of different processed woolen sheepskins and fine furskins (mink, polar fox) showed a good correlation as it can be seen in Figure 2. Higher value of wear fastness means low wear resistance and higher wool/hair deterioration. In Figure 3 a comparison of sulfur cystinic of degreased raw wool, woolen-sheepskin and suede sheepskin demonstrates that the higher the degree of wool processing, the lower the cystinic sulfur content. It is known that the woolen sheepskins are intensely processed on wool with acid, aldehydes, alcohol solution at high temperatures for getting a very straight, defibrated and bright wool. These kinds of fur skins are made from fine wool breeds of sheep such as merinos. Scanning electronic microscopy images of wool/hair of studied fur skins confirm the more degraded state of woolen sheepskins as compared to the other fur skins (Images 1- 4).

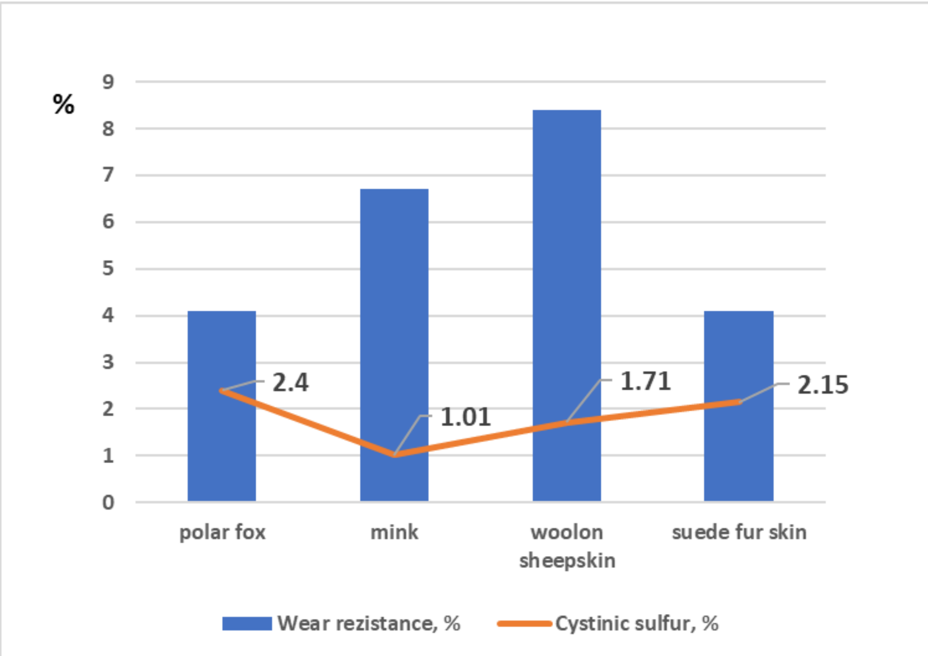


Figure 2. The correlation of wear resistance with cystinic content of wool/hair

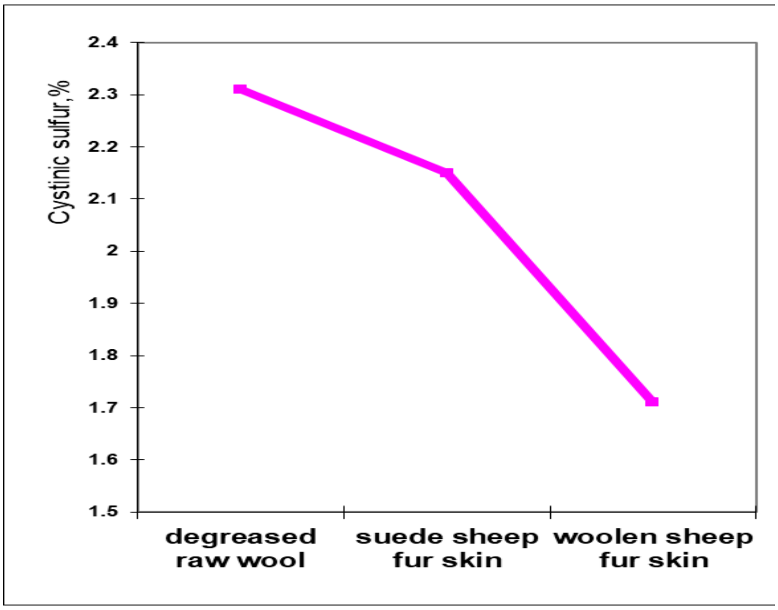
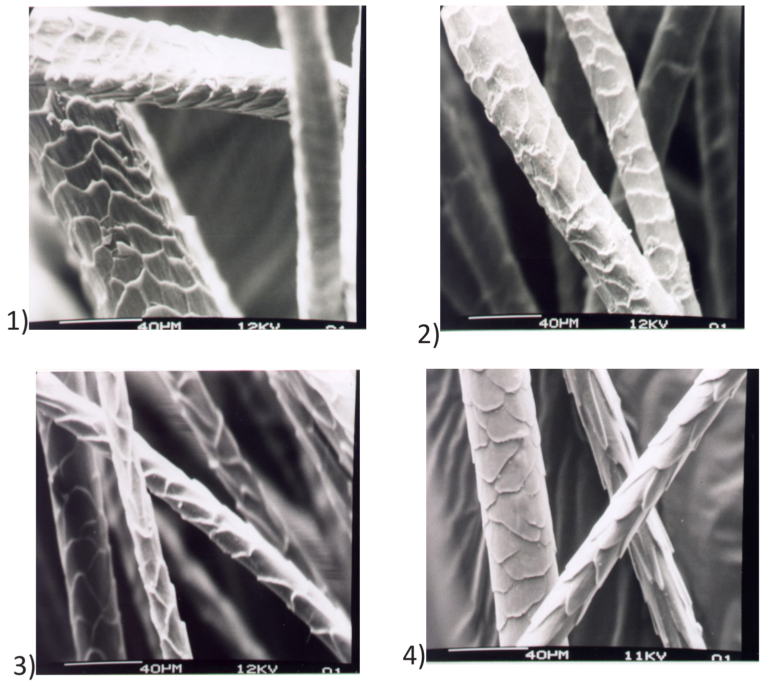


Figure 3. Cystinic sulfur as a function of wool processing degree

A study on the behavior of fur skins in humid atmosphere revealed that the fur skins with long hair can protect the wearer from moisture absorption and cold feeling. It is recognized that collagen, the main protein of dermal layer has the native property to absorb 50% water of its weight, as compared to keratin, with 30% water absorption ability. Different chemical auxiliary materials can change this native property. In Figure 4 it can be seen that the polar fox fur skin has the behavior against moisture very similar with hair behavior and dermis layer shows to be more hydrophilic. Figure 5 shows the woolen sheepskins behavior against moisture which is more stable due to the processing chemical materials with hydrophobic properties. Woolen sheepskin behavior is closer to dermis than to the wool due to the more consistent dermis layer and short wool length (15 mm).



Images 1-4. Wool of: 1 – suede sheep fur skins; 2 – woolen sheepskin. Guard and down hair of: 3 – polar fox; 4 – mink

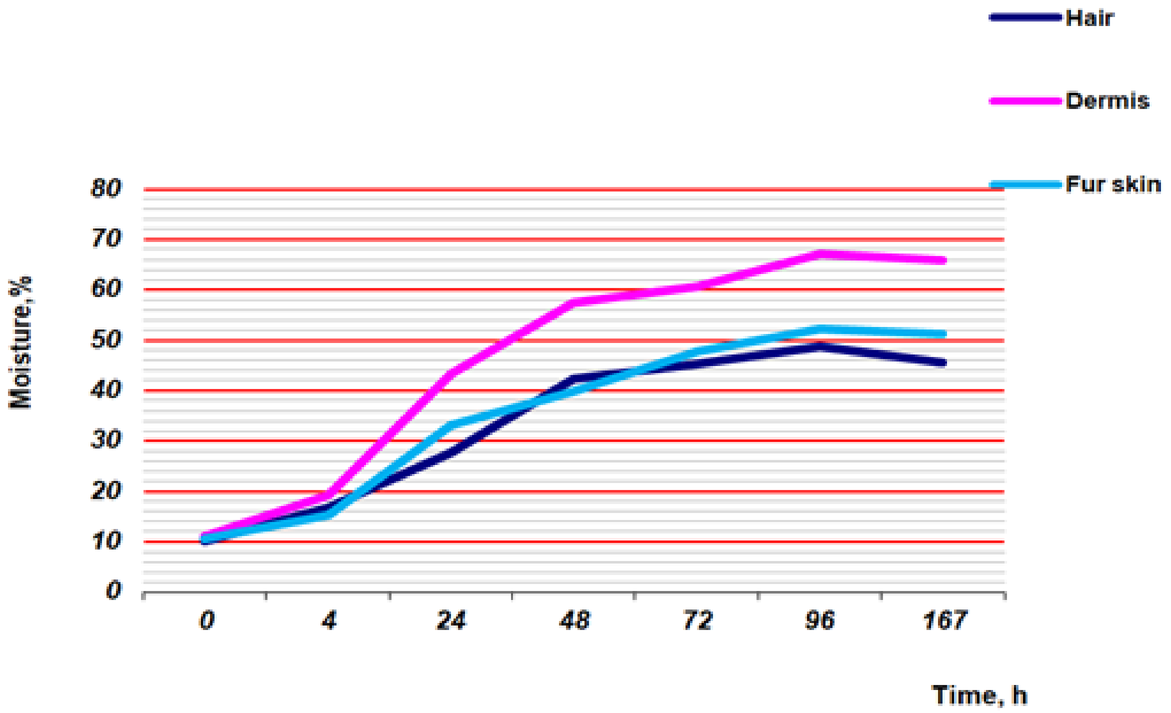


Figure 4. Polar fox hair, dermis and fur skin behavior against moisture (96% RH), over time

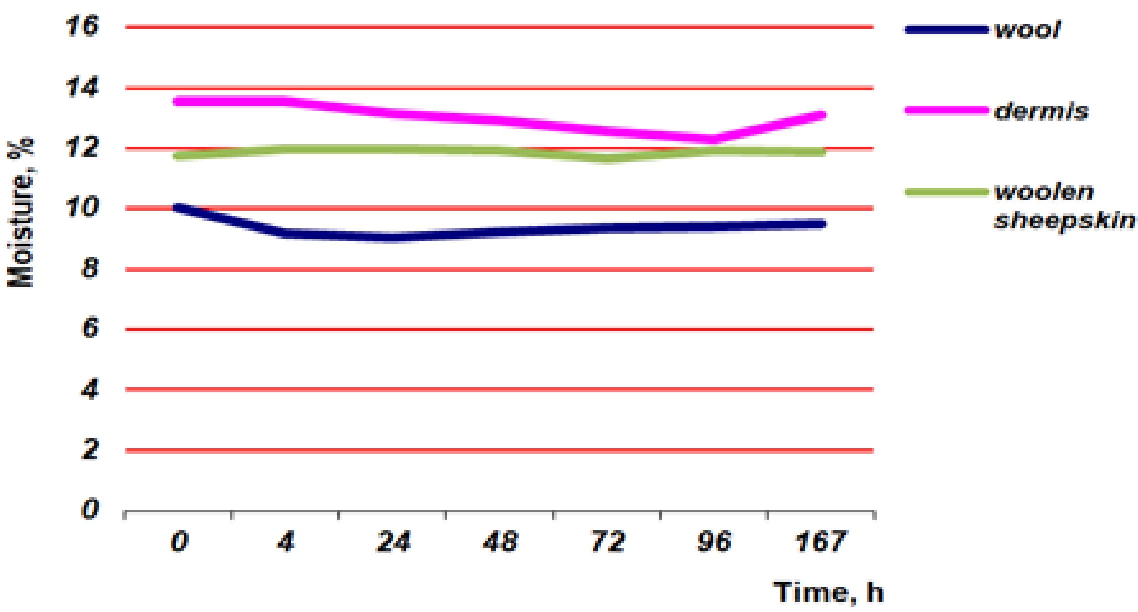


Figure 5. Wool, dermis and woolen sheepskin behavior against moisture (96% RH), over time

The influence of moisture on light fastness of fur skins is very important for clothes durability and quality. The polar fox furs are more sensitive to light when the moisture is high because they are not dyed as other fur skins whose resistance is excellent or very good (Figure 6).

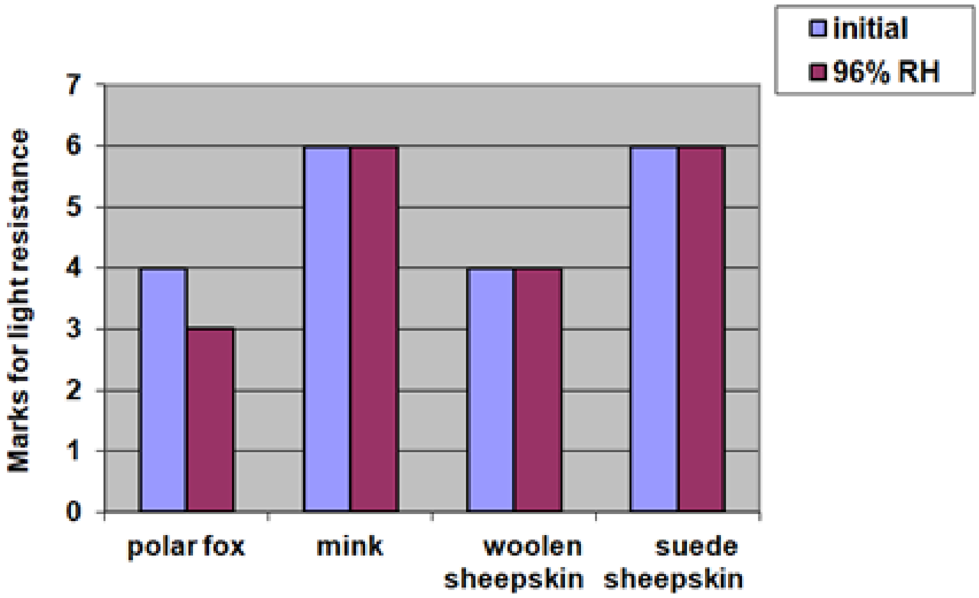


Figure 6. Light fastness of fur skins under the influence of moisture (96% RH)

Fine furs or sheep skins represent smart and versatile materials, a source of beauty and elegance, protection and durability. The responsible exploitation of fur skins from hunting, farming, slaughtering, tanning to post end of life represents an ecological activity, contributing to environmental and human protection and evolution. The scarce information on fur skin properties and research data on this material demands for more knowledge generation with the final benefits for legislative authorities, economical entities, consumers and science.

CONCLUSIONS

Investigations for quantification of fur skins quality will contribute prevailing empiric knowledge with modern requirements of quality assurance, consumer and environmental protection. A data base on fur skins quality assessment and values is needed for international standards elaboration, technical specifications in commercial transaction and consumer protection. The elaboration of commercial specifications will benefit from a recognized data base of information which can be useful for fur skin producers and traders.

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MECHANICAL PROPERTIES OF WET BLUE FOLLOWING ACID BATING PROCESS TREATED WITH CRUDE ENZYME FROM *Rhizopus oligosporus*

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MECHANICAL PROPERTIES OF WET BLUE FOLLOWING ACID BATING PROCESS TREATED WITH CRUDE ENZYME FROM *Rhizopus oligosporus*

ABSTRACT. Enzyme is one of the ingredients used in several tanning processes such as bating. Bating is generally conducted under alkaline conditions, but it can also be carried out under acidic conditions. This study aimed to evaluate mechanical and physical properties of wet blue following bating conducted under acidic conditions treated with enzymes synthesized by *R. oligosporus* compared to those of the conventional process. This study was arranged on a completely randomized design with a single factor, i.e., five levels of enzyme activity (0, 2.5, 5, 7.5, and 10 U mL^{-1}). The results showed that the use of enzymes from *R. oligosporus* influenced positively mechanical properties of the wet blue produced. Increasing levels of enzyme activity produced significantly higher tear strength and lower elongation at break values. No significant differences in tensile strength were observed following applications of both the synthesized and conventional enzymes. The synthesized enzyme was best applied at an activity level of 5 U mL^{-1} producing leather with good tear strength (55.40 ± 5.49 N mm^{-1}) and elongation at break (45.63 ± 4.17 %) values, which were significantly different from those of 0 U mL^{-1}, but not significantly different from those of 10 U mL^{-1} and the conventional process. Mechanical properties of the leather produced met the standard values for goat/sheep leather jacket as specified in Indonesian National Standard (SNI) 4593:2011. The results suggest that the synthesized enzyme from *R. oligosporus* can potentially replace the conventional enzyme commonly used in the bating process.

KEY WORDS: mechanical properties, wet blue, enzyme, *R. oligosporus*, bating

PROPRIETĂȚILE MECANICE ALE PIELII WET BLUE DUPĂ PROCESUL DE SĂMĂLUIRE CU ACIZI ȘI TRATARE CU ENZIMĂ BRUTĂ DIN *Rhizopus oligosporus*

REZUMAT. Enzima este unul dintre ingredientele folosite în mai multe procese de tăbăcire, cum ar fi sămăluirea. Sămăluirea se desfășoară în general în condiții alcaline, dar poate fi efectuată și în condiții acide. Acest studiu și-a propus să evalueze proprietățile mecanice și fizice ale pielii wet blue în urma sămăluirii efectuate în condiții acide și tratate cu enzime sintetizate din *R. oligosporus* comparativ cu cele obținute în urma procedeului convențional. Acest studiu a fost organizat ca design experimental complet randomizat cu un singur factor, adică cinci niveluri de activitate enzimatică (0, 2,5, 5, 7,5 și 10 U mL^{-1}). Rezultatele au arătat că utilizarea enzimelor din *R. oligosporus* a influențat pozitiv proprietățile mecanice ale pielii wet blue obținute. Creșterea nivelurilor de activitate enzimatică a condus la valori semnificativ mai mari ale rezistenței la sfâșiere și valori mai mici ale alungirii la rupere. Nu s-au observat diferențe semnificative în ceea ce privește rezistența la rupere în urma aplicării atât a enzimelor sintetizate, cât și a celor convenționale. Enzima sintetizată a fost aplicată cel mai bine la un nivel de activitate de 5 U mL^{-1} obținându-se piele cu o rezistență la sfâșiere (55,40 ± 5,49 N mm^{-1}) și valori de alungire la rupere (45,63 ± 4,17 %) bune, care au fost semnificativ diferite de cele obținute la 0 U mL^{-1}, dar nu semnificativ diferite de cele obținute la 10 U mL^{-1} și prin procedeul convențional. Proprietățile mecanice ale pielii obținute au îndeplinit valorile standard pentru o jachetă din piele de capră/oaie, așa cum este specificat în Standardul Național Indonezian (SNI) 4593:2011. Rezultatele sugerează că enzima sintetizată din *R. oligosporus* poate înlocui enzima convențională utilizată în mod obișnuit în procesul de sămăluire.

CUVINTE CHEIE: proprietăți mecanice, wet blue, enzimă, *R. oligosporus*, sămăluire

LES PROPRIÉTÉS MÉCANIQUES DE LA PEAU WET BLUE APRÈS LE PROCESSUS DE CONFITAGE ACIDE ET LE TRAITEMENT À L'ENZYME BRUT DE *Rhizopus oligosporus*

RÉSUMÉ. L'enzyme est l'un des ingrédients utilisés dans de nombreux processus de tannage, comme le confitage. Le confitage est généralement réalisé dans des conditions alcalines, mais peut également être réalisé dans des conditions acides. Cette étude visait à évaluer les propriétés mécaniques et physiques de la peau wet blue après acidification et traitement avec des enzymes synthétisées à partir de *R. oligosporus* par rapport à celles obtenues par la procédure conventionnelle. Cette étude a été organisée comme une conception expérimentale entièrement randomisée avec un seul facteur, soit cinq niveaux d'activité enzymatique (0, 2,5, 5, 7,5 et 10 U mL^{-1}). Les résultats ont montré que l'utilisation d'enzymes de *R. oligosporus* influence positivement les propriétés mécaniques de la peau wet blue obtenue. Des niveaux accrus d'activité enzymatique ont conduit à des valeurs significativement plus élevées de résistance à la déchirure et à des valeurs plus faibles d'allongement à la rupture. Aucune différence significative dans la résistance à la traction n'a été observée après l'application d'enzymes synthétisées et conventionnelles. L'enzyme synthétisée a été mieux appliquée à un niveau d'activité de 5 U mL^{-1} pour obtenir une peau avec une résistance à la déchirure (55,40 ± 5,49 N mm^{-1}) et des valeurs d'allongement à la rupture (45, 63 ± 4,17 %)

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bonnes, significativement différents de ceux obtenus à 0 U mL^{-1}, mais pas significativement différents de ceux obtenus à 10 U mL^{-1} et par la procédure conventionnelle. Les propriétés mécaniques du cuir obtenu ont respecté les valeurs standard pour une veste en cuir de chèvre/mouton, telles que spécifiées dans la Norme Nationale Indonésienne (SNI) 4593 : 2011. Les résultats suggèrent que l'enzyme synthétisée à partir de *R. oligosporus* peut remplacer l'enzyme conventionnelle couramment utilisée dans le processus de confitage.
MOTS CLÉS : propriétés mécaniques, peau wet blue, enzyme, *R. oligosporus*, confitage

INTRODUCTION

Tanning is one of the oldest technologies that can improve physical, chemical, biological, and mechanical properties of skin. The skin quality improvement technology involves many stages, which are grouped into beamhouse/pre-tanning, tanning, and post-tanning processes [1]. In each process, there are several stages that need to be passed to obtain tanned leather, some of which are deliming and bating [2]. Deliming and bating are different, but they are integrated processes. Deliming is a process that aims to remove lime left on the skin after the liming process is carried out [1]. It needs to be done so that the resulting skin is not hard and stiff.

According to [3, 4], deliming can be carried out in two conditions, namely slightly acidic and slightly alkaline. The bating stage will adjust the conditions carried out at the time of deliming. This shows the connection between the deliming and bating processes. In general, both deliming and bating processes are carried out under alkaline conditions. Some studies have shown that deliming can also be carried out under acidic conditions. However, only few studies have shown bating in acidic conditions.

Bating aims to remove non-collagen components remaining in the skin [2]. In addition, it helps perfect the opening of collagen fibers [5]. To achieve this goal, a bating agent, which contains enzymes, is required in the process. There are many different types and brands of bating agents (enzymes) that could be used for leather tanning. They could be obtained from bacteria, fungi, or yeast. This certainly provides an opportunity to synthesize enzymes from certain species that can also be used in bating, such as *Rhizopus oligosporus*.

R. oligosporus is a mold used in the production of tempeh (a traditional Indonesian food made from fermented soybeans). *R. oligosporus* is an enzyme-producing mold that can work optimally in acidic conditions. Enzymes from *R. oligosporus* can be synthesized at pH 3-7 and temperatures of 25-30°C [6, 7]. Previous studies have shown that *R. oligosporus* can synthesize proteases and lipases [6–9]. According to [7], the resulting enzyme can work optimally at pH 5.5. This condition is in line with our previous findings [10], where the deliming process works optimally at the same pH. Therefore, in this study, bating was carried out under acidic conditions using enzymes produced from *R. oligosporus* and the characteristics of the resulting skin was evaluated.

EXPERIMENTAL

Materials and Methods

The study was conducted by using a crude enzyme (acid protease) from *Rhizopus oligosporus*, liming pelt sized 30 x 15 cm from the krupon of goatskin, lime, sodium sulfide, degreasing agent, tartaric acid, sodium chloride, sulfuric acid, formic acid, chromium sulfate, and sodium bicarbonate. The main equipment used includes an autoclave and HACH UV-Vis spectrophotometer. This study was conducted in three stages; (1) enzyme production, (2) enzyme application to the pelt (as a bating agent), and (3) pelt and wet blue properties evaluation.

This study was arranged on a completely randomized design with a single factor, which was enzyme activity, and four replications. Data were analyzed using ANOVA and Duncan's advanced test with a confidence level of 5%. The research procedure, from soaking to deliming, followed [10], and then continued as described in Table 1.

Table 1: Procedure of experiment

Processes	Materials	Doses	Remarks
Bating	Alkaline bate[a] Enzyme from *R. oligosporus*[b]	0.2%	[a]enzyme that was used in the conventional process. [b]prepared enzyme that was used in the experiment. Bating was done for 45 min. Then, the pelt was drained and washed.
Pickling	Water	100%	Water and NaCl were added and mixed for 20 min in a drum. Then, other materials were added periodically with an interval of 15 min. The pH of pelt was 3.
	NaCl	10%	
	Formic acid	0.6%	
	Sulfuric acid	0.4%	
Tanning	Chromium sulphate	8%	Chromium Sulphate was added and mixed for 1 h. Then, sodium bicarbonate was added and mixed for 6 h. The wet blue was drained and washed, followed by aging and toggling.

Enzyme Production

Rhizopus oligosporus from yeast was incubated on potato dextrose agar (PDA) media for 3 days. Then, the spores were propagated into skim media for 3 days. The propagated filtrate was used as a starter to produce the enzyme. The substrate used for enzyme production was sterilized tofu wastewater. The starter and substrate were mixed and shaken at 210 rpm, room temperature (28-30°C) for 72 h. The mixture was filtered to obtain an enzyme filtrate. Then, the filtrate was centrifuged at 4°C, 4,000 rpm for 20 min to obtain a clear enzyme solution [11].

Enzyme Application in Bating Stage

The enzyme solution (crude enzyme) obtained was applied to the pelt with different levels of enzyme activity. In general, skin, deliming, and bating processes were conducted according to the method developed by Nugraha *et al.* 2020. The crude enzyme dosages were 0 U mL^{-1}, 2.5 U mL^{-1}, 5 U mL^{-1}, 7.5 U $mL^{-1,}$ and 10 U mL^{-1} in the bating solution. Then, the pelt was tanned using chromium sulfate. The procedures are presented in Table 1.

Tear Strength

Tear strength of wet blue were tested using the INSTRON 3383 Universal Testing Machine. In this test, a hook was attached to the tool in order to pull the sample in the opposite direction so that the sample was torn. The tear strength value was measured when the sample started to tear and the needle indicated the tear strength value on the test tool stops [12]. The value of the tear strength was calculated using the following equation:

$$\text{Tear strength (kgf/mm)} = F/t \tag{1}$$

F = the value read on the test equipment (kgf)
t = skin thickness (mm)

Tensile Strength

Tensile strength test of wet blue was carried out using the INSTRON 3383 Universal Testing Machine. The sample was placed on the test equipment by means of both ends of the sample being clamped on the test equipment. The distance between the clamps was 5 cm. After the sample was ready, the tool was turned on and off when the sample broke [12]. The value of tensile strength was calculated using the following equation:

$$\text{Tensile strength (kgf/mm}^2\text{)} = F/\ (t \times l) \tag{2}$$

F = value read on the device (kgf)
l = width of skin under test (mm)
t = thickness of skin under test (mm)

Elongation at Break

Elongation is a measurement of the elongation of the stretched skin from the initial condition to the final condition, i.e., when the skin is broken during tensile strength testing. Skin elongation was calculated by comparing the length of the skin before and after the test. The elongation at break was calculated using the following equation:

$$\text{Elongation at break} = (L_1 - L_0)/L_0 \tag{3}$$

L_1 = length at break time (mm)
L_0 = initial length (mm)

RESULTS AND DISCUSSIONS

Tear Strength

Tear strength is the force required to tear the resulting leather sample. Based on the analysis carried out (Table 2), enzyme activity influenced tear strength of the tanned leather produced. It is shown that increasing enzyme activity from 0 U mL^{-1} and 2.5 U mL^{-1} did not increase significantly tear strength of the tanned leather. Tear strength of the leather started increasing significantly (55.40 ± 5.49 N mm^{-1}) when the enzyme activity applied was 5 U mL^{-1}. However, further increases in enzyme activity up to 10 U mL^{-1} did not result in significantly higher tear strength values, which were also insignificantly different from that of the tanned leather produced by the conventional treatment. The tear strength values of the tanned leather produced by the all treatments complied with that of Indonesian National Standard (SNI) 4593:2011, i.e., minimum 12.5 N mm^{-1} [13].

Table 2: Mechanical wet blue properties in various enzyme activity

Enzyme Activity	Tear Strength (N mm^{-1})	Tensile Strength (N mm^{-2})	Elongation at break (%)
0 U mL^{-1}	47.66 ± 2.91^{a}	23.31 ± 2.22^{a}	51.65 ± 4.89 ^{d}
2.5 U mL^{-1}	52.20 ± 4.63^{ab}	26.13 ± 2.59^{a}	48.63 ± 2.29^{cd}
5 U mL^{-1}	55.40 ± 5.4^{9b}C	26.94 ± 2.13^{a}	45.63 ± 4.1^{7b}C
7.5 U mL^{-1}	57.74 ± 1.61^{bc}	27.85 ± 2.41^{a}	41.50 ± 1.65^{b}
10 U mL^{-1}	59.99 ± 4.13^{c}	26.63 ± 2.38^{a}	34.42 ± 2.59^{a}
Conventional	56.54 ± 3.01^{bc}	26.22 ± 2.46^{a}	46.17 ± 3.63^{bc}

Note: Means in the same column with different superscripts differ significantly ($p<0.05$)

In general, there was a consistent trend showing that the higher the enzyme activity was, the higher the tear strength of the tanned leather was. This is because the enzymes used in skin peeling degrade non-collagen compounds. This condition facilitates chromium to penetrate the skin and bind to collagen. As a result, this increases tear strength of the leather. According to [14], the amount of tanning material contained in the leather affects tear strength of the leather after treated by an enzyme. Figure 1 shows that the tear strength of the leather was influenced by the concentration of chromium oxide in the leather. It is shown that increasing concentrations of chromium oxide increased the tear strength of the leather. Meanwhile, according to [15], fiber direction, skin thickness, and the angle of collagen fibers to the grain layer affect the tear strength of the skin.

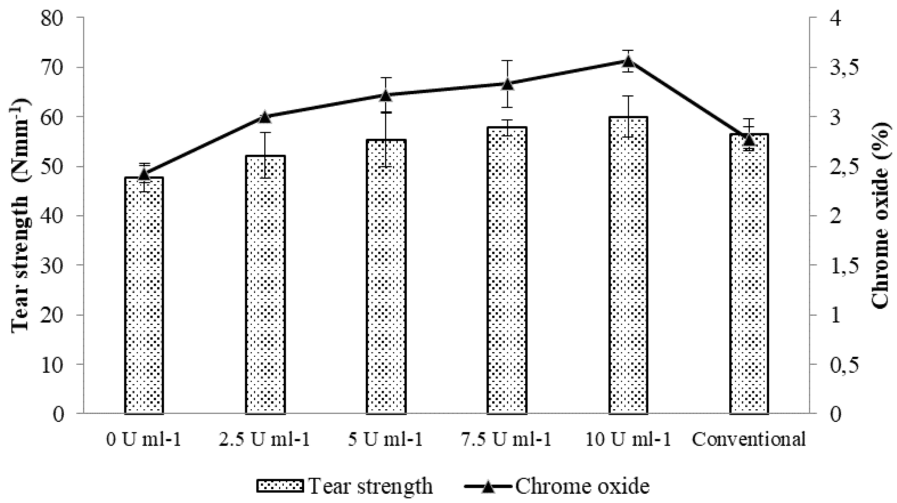

Figure 1. Correlation between concentration of chromium oxide in the leather and the resulting tear strength of the leather (wet blue)

Tensile Strength

Tensile strength is the force required to pull the leather to break per unit length times the thickness of the leather. The results showed that the levels of enzyme activity applied in leather peeling affected tensile strength of the leather. Table 2 shows that increasing enzyme activity up to 7.5 U mL^{-1} tended to increase tensile strength of the leather. However, at enzyme activity of 10 U mL^{-1}, there was a decrease in tensile strength of the leather produced. The highest tensile strength was 27.85 ± 2.41 N mm^{-2} found at 7.5 U mL^{-1} treatment. On the other hand, the lowest tensile strength (23.31 ± 2.22 N mm^{-2}) was observed in the leather treated with 0 U mL^{-1} enzyme activity. However, statistical analysis showed that the differences in the tensile strength values as influenced by the different levels of enzyme activity as well as the conventional treatment were insignificant (Table 2). Based on the Indonesian National Standard (SNI) issued by [13], the tensile strength values of the leather produced by the all treatments met the standard value determined in SNI 4593:2011, i.e., above 14 N mm^{-2}.

The results from this study were in agreement with those reported by [16] indicating that tensile strength of the leather decreases at a certain concentration of bating agent. This phenomenon could be due to decreased levels of elastin in the leather. According to [17], when an enzyme used for pulverizing the skin works more effectively, the collagen fibers are largely exposed to tanning materials. This facilitates strong interactions, and thus increases tensile strength. Previous studies showed that degradation of elastin in leather influences the quality/properties of the final leather product [18, 19]. However, excessive elastin degradation may cause a decrease in the quality of the skin itself [20]. This is also supported by [21] reporting that the use of proteases results in skin damage due to excessive elastin degradation. Furthermore, [22] found that proteases exhibit a hydrolytic activity against elastin, and the affecting factors are pH and temperature.

Elongation at Break

Elongation is the increase in length of the leather pulled up to break divided by the initial length of the leather. Table 2 shows that the levels of enzyme activity influenced elongation of the leather produced significantly. The higher the enzyme activity was, the lower the elongation of the leather was. It is shown that the highest elongation of the leather (51.65 ± 4.89 %) was obtained when the enzyme activity applied was 0 U mL^{-1}. Conversely, the lowest elongation of the leather (34.42 ± 2.59 %) was found at the enzyme activity of 10 U mL^{-1}.

Further analysis shows that the elongation at 0 U mL^{-1} was not significantly different from that at 2.5 U mL^{-1}. The elongation started decreasing significantly when enzyme activity was applied at 5 U mL^{-1}, and it was not significantly different from those of 2.5 U mL^{-1}, 7.5 U mL^{-1}, and the conventional treatment. Further significant decrease in elongation of the leather occurred at enzyme activity of 10 U mL^{-1}, which was significantly lower than that of the conventional treatment (Table 2). The elongation values of the leather produced by the all treatments met the standard elongation value for goat/sheep leather jacket as specified in SNI 4593:2011, which is below 60 % [13].

The decrease in the elongation value of the tanned leather was in line with the increased level of enzyme activity used during leather pulverization. The presence of elastinolytic activity in enzymes causes a decrease in elastin levels in the skin, so that skin elasticity is lower. This is also supported by previous findings from [23]. However, the decrease in elastin levels does not only occur in skin crushing, but also liming, which is the stage before removing lime and peeling the skin. According to [22], elastin plays an important role in skin elasticity and affects the quality of the final skin product.

CONCLUSIONS

Crude enzyme extract from *R. oligosporus* applied in the bating process influenced positively mechanical properties of the leather. In this study, the best treatment was application of the synthesized enzyme with an activity level of 5 U mL^{-1}. The best treatment produced tanned leather with good tear strength and elongation at break values, which were significantly different from those of 0 U mL^{-1}, but not significantly different from those of 10 U mL^{-1} and the conventional treatment. No significant differences were observed in the tensile strength values of all treatments. All mechanical properties of the leather produced in this study met the standard values for goat/sheep leather jacket as specified in Indonesian National Standard (SNI) 4593:2011. The results suggest that the crude enzyme extract from *R. oligosporus* can potentially be used to replace the conventional enzymes in the bating process.

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PRELIMINARY STUDY ON THE ADHESIVENESS PROPERTIES OF HIDE GLUE

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PRELIMINARY STUDY ON THE ADHESIVENESS PROPERTIES OF HIDE GLUE

ABSTRACT. Adhesives based on collagen extracted from the skin, tendons, cartilage, bones of animals and fish have been widely used over time as binders and consolidating agents for various organic and inorganic materials. The use of these types of adhesives or glues or gelatins in the restoration activity has been practiced by restorers due to their properties, but also to the special requirements of reversibility of treatments that are stipulated in national and international norms specific to the restoration of heritage objects. For this purpose, the technology of gelatin extraction from bovine hide was studied, and then gelatins were characterized in terms of chemical properties (ash, total nitrogen, amino nitrogen, dermal substance, pH of analytical solution) and gluing experiments were conducted on collagen substrates (leather and parchment, newly made specifically for the restoration activity and some samples of historical parchment, pieces of parchment detached from the back of a heritage object) and characterized in terms of physical-mechanical properties (shear strength and peel strength). Gelatins made from bovine hide were also compared to a commercial product, rabbit glue, a material frequently used in the field of restoration.
KEY WORDS: raw hide, glue/adhesive, gelatin, wood restoration, book restoration, parchment restoration

STUDIU PRELIMINAR PRIVIND PROPRIETAȚILE DE ADEZIVITATE ALE CLEIULUI DIN PIELE

REZUMAT. Adezivii pe bază de colagen extras din piele, tendoane, cartilagii, oase ale animalelor și peștilor au fost utilizați de-a lungul timpului, pe scară largă, ca lianți și consolidanți pentru diverse materiale organice și anorganice. Folosirea acestor tipuri de adezivi sau cleiuri sau gelatine în activitatea de restaurare a fost și este practicată de către restauratori datorită proprietăților acestora, dar și cerințelor speciale de reversibilitate a tratamentelor care sunt stipulate în normele naționale și internaționale specifice restaurării obiectelor de patrimoniu. În acest scop s-a studiat tehnologia de extracție a gelatinelor din piei bovine, s-au caracterizat din punct de vedere al proprietăților chimice (cenușă, azot total, azot aminic, substanță dermică, pH soluție analitică) și s-au experimentat probe de lipire pe suporturi colagenice (piele și pergament, nou realizate special pentru activitatea de restaurare și niște probe de pergament istoric, bucăți de pergament detașate de pe partea de verso a unui obiect de patrimoniu), apoi s-au caracterizat proprietățile fizico-mecanice (rezistența la forfecare și rezistența la dezlipire). Gelatinele realizate din piele bovină s-au testat comparativ și cu un produs comercial, clei de iepure, material utilizat frecvent în domeniul restaurării.
CUVINTE CHEIE: piele crudă, clei/adeziv, gelatină, restaurare lemn, restaurare carte, restaurare pergament

ÉTUDE PRÉLIMINAIRE SUR LES PROPRIÉTÉS ADHÉSIVES DE LA COLLE DE PEAU

RÉSUMÉ. Les adhésifs à base de collagène extrait de la peau, des tendons, du cartilage, des os d'animaux et de poissons ont été utilisés au fil du temps, à grande échelle, comme liants et consolidants pour divers matériaux organiques et inorganiques. L'utilisation de ces types d'adhésifs ou de colles ou de gélatines dans l'activité de restauration était et est encore pratiquée par les restaurateurs en raison de leurs propriétés, mais aussi des exigences particulières de réversibilité des traitements qui sont stipulées dans les normes nationales et internationales spécifiques à la restauration des objets de patrimoine. À cet effet, la technologie d'extraction des gélatines de peaux de bovins a été étudiée, et on a caractérisé les gélatines en termes de propriétés chimiques (cendres, azote total, azote aminé, substance dermique, pH de la solution analytique) et on a testé leur adhérence sur des substrats en collagène (cuir et parchemin, nouvellement fabriqués spécialement pour l'activité de restauration et quelques échantillons de parchemin historique, morceaux de parchemin détachés du dos d'un objet patrimonial) et on a caractérisé leurs propriétés physico-mécaniques (résistance au cisaillement et au détachement). Les gélatines à base de peau bovine ont également été testées en comparaison avec un produit commercial, la colle de peau de lapin, un matériau fréquemment utilisé dans le domaine de la restauration.
MOTS CLÉS : cuir brut, colle / adhésif, gélatine, restauration de bois, restauration de livres, restauration de parchemins

INTRODUCTION

Adhesives or glues of animal origin are the adhesives with the oldest historical record of use [1]. Evidence of their use dates back to at least 1350 BCE and, indeed, animal glue was used to glue wood decorations to the tomb of King Tutankhamun [2]. Animal glues are water-based adhesives that use gelatin protein as an adhesive polymer. Gelatin is the same material used in the food and pharmaceutical industry. It is produced industrially by the chemical treatment (hydrolysis) of collagen protein contained in the skins and bones of mammals [1, 2].

Simple mixtures of gelatin (skin glue) and water have been used in woodworking for thousands of years and in binding books for at least 200 years. Modern animal glues contain not only gelatin and water, but also performance modifiers such as plasticizers, sugars, salts, surfactants, antifoams and biocides. The performance of modern formulations of these

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glues is much improved compared to the simple gelatin/water mixtures of the past [3-5]. The formulations are adapted to each application by changing parameters such as adhesion level, bonding time and viscosity.

In bookbinding, animal glues are used in the manufacture of cardboard cases, covers and flyleaves. The major advantage of animal glues and the reason why they are still used 70 years after the appearance of synthetic adhesives, is that they have an excellent initial wet adhesion. In other words, when applying an adhesive film with animal glue, it is immediately very sticky. This property allows the use of animal glues when a glued turned edge is needed, in bookbinding and in restoration. Animal glues form only mechanical bonds. This means that they adhere well to porous surfaces, but poorly to some coatings and all non-porous substrates, such as metals and plastics. Glues of animal origin are obsolete in certain situations by synthetic adhesives, but persist due to their unique property of excellent moisture adhesion and reversibility of gluing (specific requirement of the restoration-conservation activity), which remains unmatched by any aqueous synthetic adhesive.

EXPERIMENTAL

Materials and Methods

For this study, raw bovine hides were processed to obtain leather tanned with vegetable tannins and raw calfskins and goatskins to obtain parchments. For this, raw materials specific to the leather industry were used, but the special requirements of the restoration field were also taken into account. Bonding tests were also performed on historical parchment samples.

Gelatin or adhesive made and tested in this study was obtained from bovine hide waste.

The investigation methods included chemical methods to characterize the resulting gelatins and physico-mechanical methods to characterize the adhesive strength of these gelatins when gluing leather and parchment.

The technology for gelatin extraction from bovine hide was experimented (protected by patent application) in acid medium, at a pH lower than the isoelectric point:

Stage I: Weighing residual fragments of untanned bovine hide. Dosing the float, water at 70°C, solid/liquid ratio of 1:3, thermostating at 70°C, stirring for 60 min. at 70°C.

pH adjustment with formic acid 25%, to 4.5-5.0, stirring for 4 hours at 70°C.

Quantitative filtration at 70°C, decanting, cooling to 4-6°C for 14-16 hours.

Collection of the first gelatin fraction, with homogenous appearance: GC1

Stage II: Dispersion of residue from the first stage (filtration and decantation residue), in water at 85°C, solid/liquid ratio of 1:2, thermostating at 85°C, stirring for 3 hours.

Quantitative filtration at 85°C, decanting, cooling to 4-6°C for 14-16 hours.

Collection of the second homogenous gelatin fraction: GC2.

Stage III: Dispersion of residue from the second stage (filtration and decantation residue), in water at 95°C, solid/liquid ratio of 1:1, stirring for 3 hours at 95°C. Quantitative filtration at 95°C, decanting, cooling to 4-6°C for 14-16 hours, collection of the third gelatin fraction: GC3.

Figure 1. The extraction process in progress

For this study, leather and parchments were prepared using technologies inspired from ancient recipes at the Leather and Footwear Research Institute (ICPI) of the National Research and Development Institute for Textiles and Leather (INCDTP), Bucharest. In the current technological process of parchment manufacturing, skins preserved by salting [6-8] were used.

Characterisation of Gelatins Extracted from Solid Waste Generated during Wet Hide Processing

Table 1: Chemical characteristics of gelatin from bovine hide

No.	Characteristics, UM	Standard method	GC1
1	Dry substance, %	SR EN ISO 4684 :2006	7.03
2	Total ash, %	SR EN ISO 4047 :2002	1.00
3	Total nitrogen, %	SR EN ISO 5397 :1996	16.22
4	Amino nitrogen, %	ICPI method, not standardized	undetectable
5	Dermal substance, %	SR EN ISO 5397 :1996	98.29
6	pH of analytical solution	STAS 8619/3 :1990	5.86

The lack of amino nitrogen confirms a high average molecular weight, over 40 kDa, specific to gelatins and a high degree of cohesion of gelatin, as suggested by the strength of gelatin.

The better quality of GC1 gelatin as adhesive compared to GC2 and GC3 gelatins was confirmed by the effectiveness of gluing the parchment on wood; the adhesion of the parchment glued with GC1 gelatin compared to GC2 and GC3 gelatins is much better, requiring a greater peel force.

The gelatin strength was determined by a compression test using Texture Analyzer TEX'AN TOUCH 20 N. The gelatin strength is expressed by the maximum force (Fmax. in grams) required for a cylindrical probe (Bloom cylinder) to compress the gel located in a standard glass container with a diameter of 59 mm (+/- 1 mm) and a height of 85 mm, under standard conditions: sensor with a diameter of 0.5 inches, penetration speed 0.5 mm/s, penetration distance 4 mm, Figure 2.

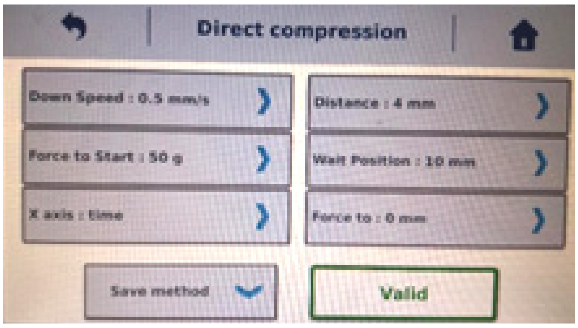
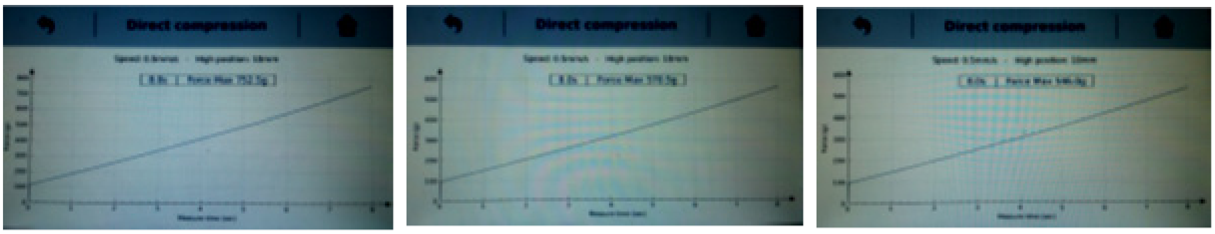


Figure 2. Standard parameters to determine gelatin strength (Bloom test)

Results of tests conducted on raw gelatins are presented in Figure 3.



(a) (b) (c)

Figure 3. Determination of gelatin strength: (a) GC1, (b) GC2, (c) GC3

The analytical data indicate that the strength of GC1 gelatin, extracted at 70°C, expressed in grams, is about 25% higher than GC2 and GC3 gelatins which were extracted at 85°C and 95°C, respectively, which is natural, because at high temperatures, more intense hydrolytic processes take place, leading to the breaking of peptide chains and the appearance of oligopeptides and even free amino acids, which weaken the cohesion of gelatin.

CG1 and CG2 gelatins were selected for testing as adhesives for joining collagenous materials in order to promote them in the field of heritage restoration, following the request for gluing pieces of parchment taken from the back of the inventory item titled Original Transylvanian Medieval Shield from the 15th century. The shield is registered as a "Hussar shield" and it is specific to the cavalry, with a characteristic curved shape, and the upper part cut obliquely to protect the shoulder and back. The Hussars were soldiers of the Hungarian cavalry in the Middle Ages. It has been used in Eastern Europe since the first quarter of the 15th century and has been in use for over 100 years. This type of shield was used by the Ottomans, Poles, Romanians, Croats, Austrians, but especially by Hungarian hussars (riders), a name with which it remained associated. In museums and literature, this shield is called the “Hungarian shield”. The shield is a donation from 1926 of the City Hall of Sibiu.

The description of this heritage object from the restoration file is very brief and incomplete due to the impossibility on the part of the restorer to understand the complexity of its composition (materials, form, historical contextualization, technique, etc.).

The wooden shield is covered/glued with calf parchment both on the outside and inside. The outer part, i.e. the convex part, consists of a whole piece of parchment, painted in red and yellow oblique stripes (according to Image 1), and on the concave part (Image 2) the parchment on the front is continued, but because there was not enough material, several pieces were glued which are now totally or partially detached (Images 3 and 4). The technique of assembling the parchment on wood was made with wet parchment by gluing with a gelatin on the wooden substrate. Calf parchment is a compact, dense, lightweight material, very resistant to tearing and breaking, and its use in this assembly (parchment glued to wood) makes it a special object suitable for the purpose created: light, resistant to piercing arrows and spears, resistant to cutting.

Image 1. Front view of the shield

Image 2. Back view of the shield

Image 3. Detail from the back side of the shield

Image 4. Detail from the back side of the shield

RESULTS AND DISCUSSIONS

Tests for Adhesiveness

The adhesive capacity of the following gelatins or glues was studied:

- rabbit skin (granules, commercial product of the “Divolo” company) – 5g in 50 mL distilled water was brought into fluid state by heating in a water bath at 40°C;
- GC1 - from bovine hide (70°C);
- GC2 - from bovine hide (85°C).

The materials used were vegetable tanned bovine hide, vegetable tanned sheep hide, dry calf parchment, wet calf parchment, fir wood. Gluing experiments were prepared in duplicate for each sample: a and b.

The work method involved brushing the glue onto the two contact surfaces (wood and flesh side of parchment) of 3x3 cm^2 and pressing the glued surface using 3 kg weights for 72 h.

Experiment I-1 consisted of gluing wet parchment to wood (fir) (Image 5). After 72 h from gluing, it is noticed that the contact surface of the parchment and wood is consistent (Image 6), which indicates an appropriate behaviour of the parchment, typical for the parchment-wood ensemble from the heritage object: Transylvanian medieval shield.

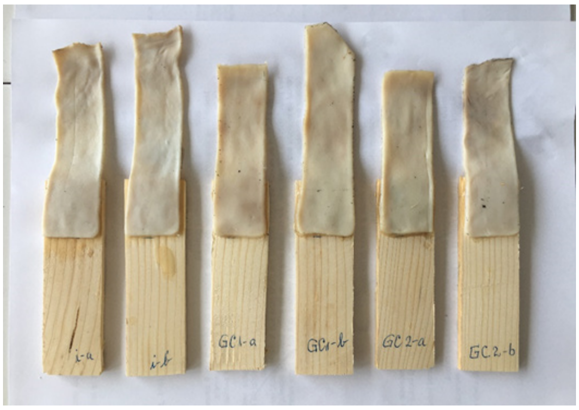


Image 5. Gluing wet parchment to wood

Image 6. Gluing wet parchment to wood after 72 h

Experiment I-2 consisted in gluing leather to leather (bovine and sheep), and experiment I-3 involved gluing bovine leather to wet parchment (Image 7), both following a work method similar to that of experiment I-1.

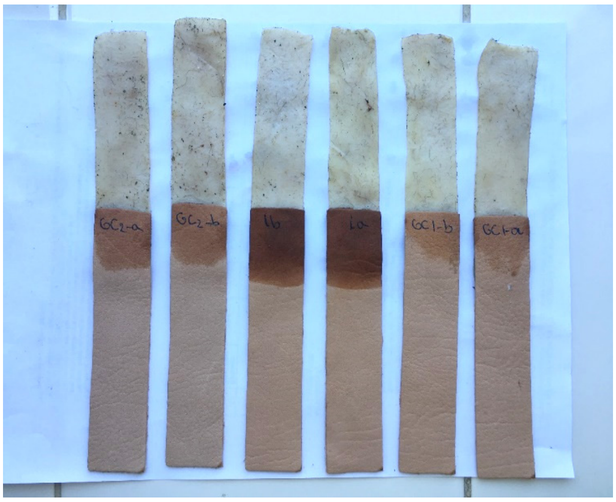


Image 7. Gluing vegetable tanned bovine leather to wet parchment

Image 8 presents samples from experiment I-4, gluing bovine leather to new dry parchment, with a similar work method to that of experiment I-1.

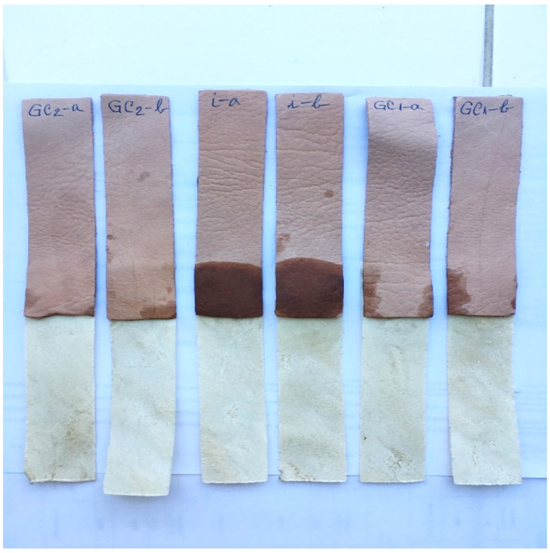


Image 8. Gluing vegetable tanned bovine leather to new dry parchment

In experiments I-3 and I-4 a wetting of the leather samples was noticed on the grain side due to water migration from the rabbit skin gelatin, samples i-a and i-b, compared to leather samples treated with gelatins GC1 and GC2.

In order to determine the rehydration ability of new parchment intended for the restoration activity that involves the gluing operation, rehydration experiments were conducted to study and assess behaviour to various ways of gluing in operations characteristic to restoration such as adding materials, replacing or doubling, experiments I-5 and I-6.

Experiment I-5 consisted of 4 samples of new dry parchment rehydrated by immersion in distilled water and alcohol, 1:1 (2 thin samples: 1-2 and two thick samples: 3, 4, Image 9).

Image 9. New parchment rehydrated in water, after 72 h

Image 10. New parchment rehydrated in urea, after 72 h

Experiment I-6 involved rehydrating 4 samples of new dry parchment by immersion in 2% hydroalcoholic solution, urea and salt (2 thin samples: 1b-2b and two thick samples: 3b-4b, Image 10).

Results of physical-mechanical analyses of gluing samples using the three 3 gelatins/glues [rabbit skin glue (i); bovine hide glue 70°C (GC1); bovine hide glue 85°C (GC2)] are presented in the following tables:

Table 2: Shear strength of samples I-1 when gluing wet parchment to wood

Characteristics	UM	Sample code/ Determined values 148/1			*Uncertainty	Standard method
Shear strength	N/mm^2	i	GC1	GC2	$\pm$ 0.34	**STAS 6651:1991
		a – 0.52 b – sample detached average: 0.52	a – 1.42 b – 1.35 average: 1.39	Samples detached		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Table 3: Shear strength of samples I-2 when gluing wet parchment to bovine leather

Characteristics	UM	Sample code/ Determined values 148/2			*Uncertainty	Standard method
Shear strength	N/mm^2	i	GC1	GC2	$\pm$ 0.34	**STAS 6651:1991
		a – 5.74 b – 6.96 average: 6.35	a – 10.06 b – 11.10 average: 10.58	a – 11.00 – leather breaks b – 11.10 average: 11.05		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Table 4: Shear strength of samples I-3 when gluing dry parchment hydrated in distilled water to bovine leather

Characteristics	UM	Sample code/ Determined values 148/3			*Uncertainty	Standard method
Shear strength	N/mm^2	i	GC1	GC2	$\pm$ 0.34	**STAS 6651:1991
		a – 9.77 b – 3.94 average: 6.86	a – 5.72 b – 8.03 average: 6.88	a – 7.34 b – 5.54 average: 6.44		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Table 5: Shear strength of samples I-4 when gluing dry parchment hydrated in urea to bovine leather

Characteristics	UM	Sample code/ Determined values 148/4	*Uncertainty	Standard method
Shear strength	N/mm^2	GC1	$\pm$ 0.34	**STAS 6651:1991
		a – sample detached b – 5.72 c – 6.38 d – 4.97 average: 5.69		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Table 6: Shear strength of samples I-5 when gluing dry parchment samples hydrated in distilled water to bovine leather - code 148/5

Characteristics	UM	Sample code/ Determined values 148/5		*Uncertainty	Standard method
Shear strength	N/mm^2	i	GC2	$\pm$ 0.34	**STAS 6651:1991
		a – 5.50 b – 3.47 c – 4.10 average: 4.36	a – 3.43		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Results of shear strength presented in tables 2-4 show that the best values were obtained for gluing with gelatin GC1.

Historical parchment samples were hydrated for 72h and marked as follows:

- **., .., ..., iv, v, vi** hydrated in hydroalcoholic solution (1:1),
- **., .., ..., iv, v, vi**, hydrated in urea solution

Experiment I-6: Gluing on a surface of 3x3 cm^2, wood (fir)-wet historical parchment

- **., .., ..., iv, v, vi**, hydrated in hydroalcoholic solution

Experiment I-7: Gluing on a surface of 3x3 cm^2, wood (fir)-wet historical parchment

- **., .., ..., iv, v, vi**, hydrated in urea solution

Images of gluing tests for experiments I-6 and I-7 are shown below:

Image 11. Gluing wood and historical parchment hydrated for 72 h in hydroalcoholic solution

Image 12. Gluing wood and historical parchment hydrated for 72 h in urea solution

Table 7: Shear strength of samples I-6 when gluing historical parchment hydrated in ethyl alcohol to wood

Characteristics	UM	Sample code/ Determined values 148/6			*Uncertainty	Standard method
Shear strength	N/mm²	i	GC1	GC2	± 0.34	**STAS 6651:1991
		a – 7.40 – parchment breaks b – 7.24 average: 7.32	a – 7.01 – parchment breaks b – 6.98 – parchment breaks average: 6.99	a – 6.72 b – 6.21 – parchment breaks average: 6.47		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Table 8: Shear strength of samples I-7 when gluing historical parchment hydrated in urea to wood

Characteristics	UM	Sample code/ Determined values 148/7			*Uncertainty	Standard method
Shear strength	N/mm²	i	GC1	GC2	± 0.34	**STAS 6651:1991
		a – 7.14 b – 6.56 average: 6.85	a – 5.41 b – 6.71 average: 6.06	a – 1.53 b – 2.44 average: 1.99		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
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In order to study the overall gluing process, experiments were conducted regarding the peel strength of leather samples, as follows: experiment I-8 – gluing two bovine leather samples on a surface of 3x10 cm^2 and experiment I-9 – gluing two lambskin samples on a surface of 3x10 cm^2.

Results of physical-mechanical analyses of gluing samples I-8 and I-9 using the three gelatins/glues [rabbit skin glue (i); bovine hide glue 70°C (GC1); bovine hide glue 85°C (GC2)] are included in the test report and presented in the following tables:

Table 9: Peel strength of samples I-10 when gluing bovine leather

Characteristics	UM	Sample code/ Determined values 149/1			*Uncertainty	Standard method
Peel strength	N/mm	Bi	BGC1	BGC2	± 0.14	**STAS 6651:1991
		a – 0.26 b – 0.32 average: 0.29	a – 0.44 b – 0.71 average: 0.58	a – 0.85 b – 0.97 average: 0.91		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

Table 10: Peel strength of samples I-11 when gluing lambskin

Characteristics	UM	Sample code/ Determined values 149/2			*Uncertainty	Standard method
Peel strength	N/mm	Ci	CGC1	CGC2	± 0.14	**STAS 6651:1991
		a – 0.24 b – 0.18 average: 0.21	a – 1.14 b – 1.19 average: 1.17	a – 0.69 b – 0,78 average: 0.74		

* Uncertainty for a 95% confidence interval; k=2 and is expressed in the measuring unit of the measured property
** Test not accredited by RENAR

CONCLUSIONS

The gelatins or glues studied were: i - rabbit glue (commercial product), GC1 - bovine hide glue (extracted at 70°C) and GC2 - bovine hide glue (extracted at 85°C).

Gluing tests were performed on vegetable tanned bovine hide, vegetable tanned goatskin, dry calfskin parchment, wet calfskin parchment, fir wood, historical parchment, while gluing experiments were prepared in duplicate for each sample: a and b. The working method for performing the tests was the same for the 3 types of glues studied. The physical-mechanical properties of shear strength and peel strength were characterized, and the bovine hide glue, CG1 extracted at 70°C had the best results, which recommends it for use in the restoration activity.

Acknowledgement

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EUROPEAN RESEARCH AREA

COTANCE NEWSLETTERS

Starting with January 2019, the COTANCE Council will issue a monthly ***COTANCE Newsletter*** with the purpose of **promoting an improved image of leather** to relevant decision makers and domestic stakeholders including Members of the European and National Parliament, Governmental authorities, Ministerial officers, Customers of the leather industry, Brands, Retail chains, Relevant NGOs, Designers, etc. The monthly newsletters present topics that tell the truth about a controversial aspect or a fact that is not well known by the general public to bring about a better understanding of leather and the European leather industry, as well as a positive predisposition to legislate in favor of the leather industry. The newsletters are available in seven languages at https://www.euroleather.com/index.php/newsletter, and were also published in the 2019-2021 issues of *Leather and Footwear Journal*. Newsletters 4 and 5 of 2022 are given below.

NEWS 4/2022

European Tanners are Modern Alchemists

Some encounters are real experiences; they make us feel good and won't be forgotten. Among them is the experience of leather. Soft, firm, supple, and strong, leather comes with special sensory properties. It has an appearance, a smell, a touch, a sound that appeals to our senses.

These sensations, so specific to leather, would not be possible without the transformation of an animal skin into a noble material. Through the tanning process, the skin is transformed into the rot-proof and highly durable material, with many properties we know as leather. This process has always fascinated man. Surrounded by poetry and mystery, it has been the subject of much research so that it could be better understood and improved. This research is the prerogative of tanners, the true alchemists of tanning.

Let's consider the current processes for tanning leather:

Chrome Tanning:

Thanks to this type of tanning, using chromium III salts, leathers become supple, very resistant to friction and heat, and waterproof, and can be dyed in a wide range of colours. This is why 85% of leathers tanned in the world are chrome tanned.

In recent years, due to the risk of the formation of chromium VI, a skin-sensitising contaminant, chrome tanning has had a bad press. However, this is only an issue in poorly made leather. The risk can be well managed by the implementation of measures and controls to ensure consumer safety, as is the case in Europe. This is enforced by the restriction on chromium VI in leather, implemented in the EU REACH regulations.

Synthetic Tanning:

Some tanners practice chrome-free tanning, called synthetic tanning. This process is based on chemistry, using substances that interact with the collagen of the skin and tan it. The resulting leather has different properties to chrome-tanned leather. This means that leather made using these tanning materials may not be suitable for all the applications of chrome-tanned leather. Conversely, synthetically-tanned leathers may perform better in some applications. European tanners are working with their technical centres and chemicals suppliers to expand the options and applications of synthetic tanning.

Vegetable Tanning:

Vegetable tanning was already introduced in our previous Newsletter.

With its characteristic smell, its warm colours that deepen over time and this beautiful ageing effect called “patina”, vegetable-tanned leather embodies the very essence of this age-old material.

In conclusion, there is no competition between the different types of tanning; they offer a range of possibilities underlining the complexity and subtlety of the tanners' know-how. Giving leather special properties shows the true skills of a tanner!

European tanners are modern alchemists. They know how to mix these tanning and retanning processes to make unique leathers, but also work ethically and responsibly while respecting people and the environment.

edited bv

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NEWS 5/2022

Avoid Microplastics, Choose Leather

Leather imitations, such so-called 'vegan leather' and 'eco-leather', are usually made from polyurethane (PU) or polyvinyl chloride (PVC). Recently they include plastics incorporating plant-based materials.

As long as these fossil fuel-based derivatives remain cheap, their use will increase, with the associated environmental impacts during their use and at the end of their life. Unlike leather, synthetic polymers are prone to shedding microfibres and do not degrade easily in nature. When disposed of, typically after a short use period, they tend to remain in the environment for a very long time, emitting harmful substances and polluting the environment. A growing amount of microplastics (fragments of plastics less than 5 mm) in marine and freshwaters has been shown by numerous studies (link1, link2).

Are our clothes and footwear also responsible for microplastics?

The most common sources of microplastic pollution are polypropylene in packaging materials and PET used in the manufacture of bottles. PET from plastic bottles is increasingly recycled into clothing and footwear, but recycling after this second lifecycle has not yet been solved.

Animals and people may inhale microparticles or ingest them with food and water. Tiny particles of microplastics have been found in crustaceans and other aquatic life at the very start of the food chain. Scientist analysed blood samples of healthy adult donors and found microplastic particles in most samples (link3).

Microplastics get into nature when plastic waste breaks down, from tyre abrasion, but also when clothing made of synthetic fibres is washed. The proportion of microplastics from clothing might be small, but it goes straight into wastewater and the environment. Abrasion of plastic soles can also cause microplastic pollution.

So what does microplastic have to do with leather? Leather is the natural alternative to plastic!

Choosing leather in consumer products make them not only more appealing, but helps curb microplastic pollution. Indeed, leather is the result of the recycling of a residue of meat production, it does not cause microplastics shedding and at the end of a very long lifecycle, is capable of biodegrading providing micronutrients for plant growth.

You do a good thing for the planet if you chose leather shoes, leather clothes or bags. You reduce a waste from livestock, support a recycling industry that creates beauty and wealth, and you can be confident that you will not pollute rivers and seas with microplastics.

edited by COTANCE

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News Release from the IULTCS

07 July 2022

Tyson Foods to Sponsor IULTCS Young Leather Scientist Grant in 2023

The Executive Committee of the IULTCS is delighted to announce that Tyson Foods will be joining the prestigious list of sponsors who support our young leather research community. This will be the 9th year that grants have been awarded and each year the research projects aim to address industry needs.

“Tyson Foods is proud to sponsor the 2023 IULTCS grant for young scientists,” states Mike Larson, VP Hides & Tannery, Tyson Foods. “We’re involved in the first step in the leather-making process and committed to producing high quality hides in a sustainable and responsible way. We’re pleased to support innovative leather research to help the industry continue to learn, understand and advance.”

IULTCS President Jean-Pierre Gualino expressed his thanks saying “We are very happy to have the support of Tyson Foods as they represent the starting point of leather production. Without good quality raw material we cannot produce good quality leather. We hope that we can attract young scientists who will direct their studies at understanding more fully, how best to utilise this valuable commodity in a sustainable way. We really appreciate the grant being provided.”

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