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Resistance of Nile tilapia leather subjected to pickling with different salt concentrations and exposure times of skins in salt-free water

Resistência do couro de tilápia-do-Nilo submetido ao píquel com diferentes concentrações de sal e tempos de exposição das peles em água sem sal

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ABSTRACT: The skins discarded during the filleting process, when subjected to an appropriate tanning process, can be transformed into high-quality leather. Thus, the objective of this study was to analyze the effect of salt concentration and acid swelling as a function of the exposure time of Nile tilapia skins acidified in salt-free water. To this end, three tests were conducted: Test I – Evaluation of pickled skins; Test II – Evaluation of the effect of salt concentration in the pickling stage; and Test III – Evaluation of the effect of pickled skins at different exposure times in salt-free water. The removal of acidified skins to salt-free water promotes acid swelling, which can significantly reduce the leather's resistance, tear strength, elongation, and tensile strength. A salt concentration below the recommended level leads to acid swelling, while an excessive concentration prevents proper tanning, resulting in collagen fiber densification. Regarding the exposure time of skins in salt-free water, significant losses in their properties were already observed after ten minutes. It is concluded that salt is essential in the pickling stage, requiring a Baumé degree of 7. Skins should not be exposed to a salt-free solution to prevent acid swelling, which compromises their resistance quality. Exposure to salt-free water can reduce elasticity, tensile strength, and progressive tearing, regardless of the exposure duration.

Keywords: collagen fibers; acid swelling; *Oreochromis niloticus*; progressive tearing; tensile strength.

RESUMO: As peles descartadas no processo de filetagem, quando submetidas a um processo de curtimento adequado, podem ser transformadas em couros de alta qualidade. Dessa forma, o objetivo deste estudo foi analisar o efeito da concentração do sal e do intumescimento ácido em função do tempo de exposição das peles de tilápia-do-Nilo, acidificadas em água sem sal. Para tanto, foram realizados três ensaios. Ensaio I – Avaliação das peles piqueladas; Ensaio II - Avaliação do efeito da concentração de sal na etapa de píquel; e Ensaio III - Avaliação do efeito das peles piqueladas em diferentes tempos de exposição em água sem o sal. A remoção das peles acidificadas para água sem a presença do sal, promove um intumescimento ácido podendo reduzir muito a resistência, o rasgo, alongamento e tração dos couros, a concentração de sal abaixo do indicado causa intumescimento ácido e quando acima do indicado, não ocorre o curtimento das peles, causando adensamento das fibras colágenas. Em relação ao tempo de exposição das peles em água sem sal, após dez minutos já causam perdas consideráveis nos seus atributos. Conclui-se que o sal é importante na etapa do píquel, necessitando de um Baumé 7. As peles não devem ser expostas a solução sem sal, para evitar o intumescimento ácido e prejudicar sua qualidade de resistência. A exposição das peles na água sem sal, poderá reduzir a elasticidade, tração, e rasgamento progressivo, independente do tempo de exposição.

Palavras-chave: fibras colágenas; intumescimento ácido; *Oreochromis niloticus*; rasgamento progressivo; tração.

INTRODUCTION

The production of Nile tilapia (*Oreochromis niloticus*) has progressively established itself in Brazil as the most widely farmed species, which may be attributed to its strong adaptation to confinement, robustness, and rapid growth. In 2023, tilapia farming in Brazil reached a production of 579.080 tons, representing a 5.28% increase compared to 2022, when 550.060 tons were produced. These figures highlight the significance of tilapia, which accounted for 65.3% of the country's total farmed fish production (PeixeBR, 2024).

The versatility of Nile tilapia in culinary applications is one of the main factors driving its increasing demand (Araujo; Reynol, 2017). However, the rising supply of this species is directly linked to a greater generation of waste, which can account for up to 70% of the fish's total weight (Monteiro *et al.*, 2014). Among the waste generated during tilapia processing, the skin stands out, representing up to 10% (Corrêa *et al.*, 2024). However, these skins acquire significant market value when subjected to the tanning process. Transforming the skins into leather not only mitigates the environmental impact caused by waste from the tilapia processing industry but also results in a high-quality product with sufficient durability for applications in the fashion and textile industry (Oliveira *et al.*, 2022).

The tanning process directly influences the physicochemical characteristics of leather, impacting its quality and properties, which must align with the requirements of the final product (Cavali *et al.*, 2022). The stages of the tanning process are closely related to the chemicals used, their concentrations, the duration of

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each step, and proper pH control (Souza, 2007; Franco, 2011). Critical factors for ensuring the effective incorporation of tanning agents, dyes, and oils into the leather during processing include the enzymes applied during the bating stage, the method of acid application during the pickling stage, the tanning agents used, the oils applied in fatliquoring (including the type of oil and solution temperature), and the required pH for fixation in the final stage. Additionally, each stage of the process is influenced by the mechanical action applied, which also plays a crucial role in determining the quality of the final product (Neu *et al.*, 2015).

The pickling stage precedes the application of the tanning agent and is essential for reducing the pH through the addition of acids. However, if not performed correctly, this process can compromise leather quality, leading to acid swelling. Additionally, to protect the collagen fibers of the skins intended for leather production, the use of salt during this stage is necessary. The salt concentration (sodium chloride) should be maintained between 6 and 8 °Baumé to ensure the effectiveness of the process (dos Anjos Vilhena *et al.*, 2022).

Thus, the objective of this study was to evaluate the physicomechanical and histological characteristics of the skins under different exposure times in salt-free water (brine at 7° Baumé) and under different saline concentrations (brine at 4. 7. and 10° Baumé).

MATERIAL AND METHODS

Raw Material

For this study, 30 kg of tilapia skins were obtained as a donation from Smart Fish, located in Rolândia, Paraná, Brazil. The skins were removed from Nile tilapia fillets using a specialized machine (Fengxiang brand, model FGB-400. Guangdong, China). After removal, the skins were packaged, frozen, and transported in isothermal boxes to the Skin Processing Laboratory at the Iguatemi Experimental Farm, Paraná, Brazil.

Tanning Process

For the experiments, the methodology proposed by Souza (2007) was followed. The skins were thawed, subjected to soaking (re-moisturizing) with 1% MK IV® surfactant, rinsed, weighed, and then proceeded to the liming stage, using a solution composed of 2% lime, 3% soda ash, and 1% MK IV® surfactant, for 60 minutes. After this stage, the skins were washed and underwent the deliming process with 1% MK IV® surfactant, 2% ammonium sulfate, and 1.5% Kalplex MK®, for 20 minutes, followed by another rinse with running water. For the bating stage, 0.5% Koropon MKSC® and 1% MK IV® surfactant were used for 40 minutes. The second deliming was carried out with 2% Kalplex MK® and 1% MK IV® surfactant, for 20 minutes, followed by the degreasing step, using 1% MK IV® surfactant and water for 30 minutes, with this step being repeated.

The pickling stage was carried out after the degreasing step, during which the skins were acidified to allow the addition of the tanning agent. For this purpose, a saline-acid solution with 7° Baumé was prepared, and the pH of the solution was adjusted according to the type of tanning agent used in the process: pH 3.0 for tanning with chromium salts and pH 4.0 for tanning with tannins.

After adding water, salt (sodium chloride) was incorporated, and the salinity level was verified using an aerometer (densimeter). The skins were then immersed in the saline solution, where they remained for 10 minutes. This step serves to protect collagen fibers from acid swelling (Souza, 2007). After this period, acid was added in a diluted form and in three portions, with 15-minute intervals between each addition. The acid concentration was adjusted according to the tanning agent used, to establish the desired pH of the saline-acid solution.

For the pickling stage, the skins were separated for three tests. The objective of these tests was to analyze the effect of salt presence, salt concentration, and the impact of acid swelling at different exposure times of acidified skins in salt-free water.

Test I – Evaluation of Pickled Skins (saline-acid solution, 7° Baumé, and pH = 3.0) Exposed to Salt-Free Water

The objective of this test was to evaluate the resistance of chrome-tanned leather subjected to the pickling stage using a saline-acid solution at 7° Baumé. Two treatments were conducted: Treatment 1 (Trat 1), in which the skins remained in the pickling solution, and Treatment 2 (Trat 2), in which the skins were removed from the pickling solution and exposed to salt-free water for 15 minutes.

For the preparation of the pickling solution, water was added to the drum (a cylindrical, rotating equipment used to agitate the leather with chemical substances) in an amount equivalent to 100% of the skin volume, followed by the addition of salt to reach 7° Baumé. The skins were then placed in the drum and subjected to 10 minutes of rotation.

After this period, 3% diluted formic acid (1:10) was added to the solution in three increments, every 15 minutes, to reach pH 3.0. The skins remained in motion inside the drum for 60 minutes, with continuous monitoring of the pH of both the skins and the solution. To verify the skin pH, a drop of bromocresol green indicator was applied to the flesh side (the side that remains in contact with the meat). At pH 3.0. the indicator should turn yellow. The solution pH was monitored using pH test strips. After verifying the pH of the skins and solution, 30 skins (n=30) from Treatment 1 (Trat 1) were removed and identified by making a hole at the end of the caudal fin region. These skins were then placed in salt-free water for 15 minutes (Treatment 2 – Trat 2). The removal of the skins from the saline-acid solution and their exposure to salt-free water induced acid swelling (Trat 2). This procedure was carried out to evaluate the importance of salt presence in the pickling stage, as the skins underwent acid swelling (salt-free water exposure) for 15 minutes to assess their resistance quality at the end of the tanning process.

After 15 minutes in salt-free water, the acid-swollen skins were returned to the drum, where the Trat 1 skins remained, so that both groups could proceed together in the tanning process. In this test, 10% chromium salts were added during the tanning stage.

Test II – Evaluation of the Effect of Salt Concentration in the Pickling Stage (4° Baumé, 7° Baumé, and 10° Baumé, pH = 4.0) and Assessment of Salt Protection on Collagen Fibers

In this test, the skins were subjected to a saline-acid solution with different salt concentrations during the pickling stage. The objective was to evaluate how the salt concentration in the solution would influence the leather's resistance at the end of the tanning process. To achieve this, the skins were divided into three batches and placed in saline-acid solutions with salt concentrations of 4° Baumé (Treatment 1), 7° Baumé (Treatment 2), and 10° Baumé (Treatment 3). Each batch was properly identified to ensure that, after the pickling stage, the skins would proceed together in the tanning process. During pickling, after preparing the solutions with different salt concentrations, the skins were immersed in their respective solutions and agitated in the drum for 10 minutes. After this period, 2% diluted formic acid (1:10) was added to the solution in three increments, every 15 minutes, to reach a pH of 4.0. as the tanning process would be carried out using vegetable tannin (12% Weibull®). The skins remained in motion within the saline-acid solution for 60 minutes, with continuous monitoring of the pH of both the solution and the skins. The skin pH was verified by applying a drop of bromocresol green indicator, which should turn apple green, indicating the correct pH of 4.0. The solution pH was also monitored using pH test strips to ensure that the desired value was reached. After achieving the appropriate pH in all skins, the three treatments were transferred to a single drum, allowing them to proceed together in the tanning process. Subsequently, 12% vegetable tannin was added, and the skins remained in motion for 60 minutes.

Test III – Evaluation of the Effect of Pickled Skins (7° Baumé and pH = 4.0) at Different Exposure Times in Salt-Free Water

In this test, the skins were immersed in a saline-acid solution (7° Baumé and pH = 4.0) for 60 minutes. They were then divided into two batches: one remained in the saline-acid solution, while the other was washed to evaluate the effect of acid swelling as a function of exposure time in salt-free water. The washed skins were placed in salt-free water, and every 10 minutes, they were removed, identified, and returned to the saline solution for 5 minutes, followed by reimmersion in the saline-acid solution (7° Baumé) to continue the tanning process. The experiment was structured as follows: Treatment 1 (Trat 1) consisted of skins that were not exposed to salt-free water (time 0); Treatment 2 (Trat 2) involved 10 minutes of exposure; Treatment 3 (Trat 3) had 20 minutes of exposure; and Treatment 4 (Trat 4) involved 30 minutes of exposure in salt-free water. After the pickling stage, 12% vegetable tannin was added, and the skins remained in motion in the drum for 60 minutes.

After the tanning process in Tests I, II, and III, the skins underwent a 24-hour resting period, followed by the neutralization stage using 2% sodium bicarbonate. Next, the retanning process was carried out with 4% vegetable tannin, followed by the fatliquoring stage, in which 10% oils were applied. To complete the process, the skins were subjected to fixation with 2% diluted formic acid for 30 minutes. After these steps, the leather was washed and left to dry.

After the leather was dried, test specimens were extracted from the central region, aligned longitudinally to the length of the leather, corresponding to the body length of the animal. These samples were used to assess the resistance and elasticity quality of the leather through tensile strength, elongation, and progressive tearing analyses. Additionally, samples from the central region of the leather were also collected for histological analyses.

Leather Resistance Tests

The test specimens were stored in a climate-controlled environment at approximately 23°C and 50% relative humidity for 24 hours, in accordance with ABNT-NBR 10455 (2021). After this period, the thickness of each sample was measured following ABNT-NBR 11114 (2020) to calculate tensile strength, elongation, and progressive tearing resistance. For the tensile and elongation tests (ABNT-NBR ISO 3376. 2014) and the progressive tearing test (ABNT-NBR ISO 3377-2. 2014), the test specimens were extracted from the leather according to ABNT-NBR 2418 (2015) using a clicker press. The cuts were made longitudinally, following the length of the tilapia leather. The tests were conducted using an EMIC dynamometer, with a separation speed between loads of 100 ± 20 mm/min, and a load cell capacity of 200 kgf.

Histological Analysis of Leather

Samples from the central region of five leather pieces per treatment were collected after the tanning process and complete air drying for histological analysis. The samples were dehydrated in a graded ethanol series of increasing concentrations, cleared in xylene, and embedded in paraffin. Longitudinal sections in the craniocaudal direction, with a thickness of 5 µm, were obtained using a microtome (RM2125 RTS, Leica Biosystems Nussloch GmbH, Germany). The histological sections were stained with Hematoxylin and Eosin (HE), and the images were captured using a digital Olympus PMC 35B camera (Japan).

Statistical Analysis Applied in the Experiments

Test I followed a completely randomized design with two treatments: Treatment 1 (with salt) and Treatment 2 (exposure for 15 minutes in salt-free water). The parameters were analyzed using the F-test at a 5% probability level.

Test II followed a completely randomized design with three treatments or pickling techniques: Treatment 1 (4° Baumé), Treatment 2 (7° Baumé), and Treatment 3 (10° Baumé). Analysis of variance (ANOVA) was applied, followed by Tukey's test at a 5% probability level, with 10 replications, using leather as the experimental unit.

Test III followed a completely randomized design with four treatments, based on the exposure time of the skin in salt-free water: Treatment 1 (0 minutes – control), Treatment 2 (10 minutes), Treatment 3 (20 minutes), and Treatment 4 (30 minutes). Statistical analysis was performed using Dunnett's test at a 5% probability level, with 10 replications, using leather as the experimental unit.

RESULTS AND DISCUSSIONS

Test I – Evaluation of Pickled Skins (Saline-Acid Solution, 7° Baumé) Exposed to Salt-Free Water

During the tanning process, the pickling stage requires the use of salt to protect collagen fibers from acid swelling. To assess the importance of this practice, a batch of skins that had been in the saline-acid solution was removed and left to rest for ten minutes. After this period, salt was added again, and the skins were returned to the saline-acid solution. During the washing process and 15-minute exposure to salt-free water, the skins without salt protection experienced acid swelling, which was evidenced by transparency and an increase in thickness (0.91 mm). This swelling caused collagen fiber weakening, leading to a thickness increase that did not return to its original value due to the denaturation of protein bonds (collagen fibers). Although no statistically significant difference was observed in thickness, the acid-swollen leather showed a significant difference in resistance, making it more vulnerable to progressive tearing (16.21 N/mm) and requiring less applied force (15.44 N) compared to leather that did not undergo acid swelling (Table 1). With 15 minutes of swelling, the leather lost 65.22% of its strength compared to skins not exposed to this condition. This result highlights the importance of preventing acid swelling after the pickling stage to maintain the quality and resistance of the leather.

Oliveira *et al.* (2022), when working with tilapia skins tanned with chromium salts, obtained leather with a thickness of 1.00 mm, which was slightly higher than the "with salt" treatment in this study. However, the applied force reported by Oliveira *et al.* (2022) was higher, reaching 54.08 N, resulting in a higher tearing resistance of 53.85 N/mm. On the other hand, Souza *et al*. (2021), when evaluating chromium-tanned tilapia skins in the longitudinal direction, found lower values, with a thickness of 0.69 mm, a maximum force of 22.88 N, and a tearing resistance of 12.56 N/mm. In contrast, Corrêa *et al.* (2024) reported that chromium-tanned tilapia skins with a thickness of 1.01 mm exhibited higher force and tearing resistance than those obtained in this experiment, with values of 59.14 N and 61.17 N/mm, respectively.

Acid swelling resulted in skin thickening (0.90 mm) due to collagen fiber denaturation (Table 2). Although this swelling occurred for a short period, it was sufficient to significantly affect the resistance of chrome-tanned leather (10%). The tensile strength (10.08 N/mm^2) was significantly lower, as were deformation, elasticity, and the force required for the test, compared to leathers that did not undergo acid swelling, meaning those that retained salt in the pickling solution (Table 2). Acid swelling caused a 24.91% reduction in leather elasticity, leading to a 24.46% decrease in deformation and requiring 48.99% less force to perform the tensile strength test. These data indicate that the leather became more fragile after exposure to salt-free water, confirming that acid swelling compromised collagen fibers. As shown in Table 2. In the tensile strength test, acid-swollen leathers exhibited 56.31% lower resistance compared to those that did not undergo this condition.

Regarding tensile strength and elongation, the values obtained in this study for salt-treated skins were higher than those reported by Oliveira *et al.* (2022), who recorded a force of 140.58 N, deformation of 47.83 mm, elongation of 78.97%, and tensile strength of 13.52 N/mm^2. Additionally, Souza *et al.* (2021) found lower values, with leather thickness of 0.64 mm, a force of 60.06 N, tensile strength of 9.13 N/mm^2, and elongation of 61.65%. Similarly, Corrêa *et al*. (2024) obtained lower values for force, deformation, elongation, and tensile strength, reporting 118.52 N, 42.86 mm, 71.40%, and 13.45 N/mm^2, respectively, indicating that the skins exhibited lower elasticity. These results reinforce the superiority of the values observed in this study compared to those reported by other authors.

Table 1 – Progressive Tearing of Nile Tilapia Leather Subjected to Acid Swelling During the Pickling Stage Compared to Leather That Remained in the Saline-Acid Solution Throughout the Entire Pickling Stage.

Treatment	Thickness (mm)	Force (N)	Tearing (N/mm)
With Salt	0.86±0.081	40.66±17.31	46.58±18.84
Without Salt	0.91±0.16	15.44±7.81	16.21±4.82
p-value	0.4871	0.0011	0.002
C.V.[2] (%)	14.86	47.87	43.79

[1] Medians ± standard deviation analyzed by the F-test. [2]C.V.= Coefficient of Variation.

Table 2 – Tensile Strength and Elongation of Nile Tilapia Leather Subjected to Acid Swelling During the Pickling Stage Compared to Leather That Remained in the Saline-Acid Solution.

Treatment	Thickness (mm)	Force (N)	Deformation (mm)	Elongation (%)	Tensile Strength (N/mm^2)
With Salt	0.81±0.07[1]	186.88±47.92	71.33±5.04	119.11±8.72	22.89±4.63
Without Salt	0.90±0.15	95.33±52.75	53.88±10.27	89.44±16.83	10.08±4.65
p-value	0.1143	0.0014	0.0003	0.0002	0.0001
C.V.[2] (%)	14.36	35.71	12.93	12.85	28.18

[1] Medians ± standard deviation analyzed by the F-test. [2]C.V.= Coefficient of Variation.

Test II – Evaluation of the Effect of Salt Concentration in the Pickling Stage (4° Baumé, 7° Baumé, and 10° Baumé) and Assessment of Salt Protection on Collagen Fibers

In the pickling stage, skins are immersed in a water and salt solution to protect collagen fibers before the application of acid, preventing acid swelling, which could compromise fiber integrity. However, using the correct amount of salt is essential to ensure this protection, with a recommended concentration between 6 and 8° Baumé (Franco *et al.*, 2013). In this study, no statistical difference was observed among the treatments for thickness and elongation. However, regarding tensile strength, the 7° Baumé treatment provided the highest leather resistance, reaching 17.80 N/mm^2, while the 10° Baumé treatment showed no significant difference compared to the other treatments, achieving 15.02 N/mm^2 (Table 3).

The 7° Baumé and 10° Baumé treatments did not show a significant influence on tear resistance, whereas the 4° Baumé treatment resulted in significantly lower resistance (13.84 N/mm^2), although it did not differ statistically from 10° Baumé. It is important to highlight that reaching 10° Baumé requires a higher amount of salt, which, from an economic perspective, becomes unfeasible, as 7° Baumé provides similar characteristics. Corrêa *et al.* (2021) conducted experiments with pickling at 6° Baumé, obtaining leather with a thickness of 1.36 mm, with tensile strength (11.42 N/mm^2) and elongation (80.00%) close to the results found in this study for 4° Baumé. Additionally, the tear strength observed was 45.44 N/mm, suggesting that using 6° Baumé may produce similar characteristics to 4° Baumé, potentially leading to inferior results.

Matiucci *et al.* (2021) conducted the tanning of tilapia skins using 8° Baumé, obtaining a leather thickness of 0.86 mm. Regarding tensile strength and elongation, the values were higher than those observed in this study, with tensile strength of 18.81 N/mm^2 and elongation of 114.75%. However, the tear strength was lower, with an average of 69.36 N, when compared to the values of skins tanned at 7° and 10° Baumé.

This indicates that a low salt concentration (4° Baumé) compromises collagen fibers due to swelling, as they are not sufficiently protected by the salt added during the pickling stage. On the other hand, a high salt concentration (10° Baumé) results in "overprotection" of the fibers, preventing the acid from properly preparing them for tanning, which hinders the penetration and absorption of tannin. For this reason, the tensile strength results showed no significant difference in the resistance of leather tanned with pickling at 10° Baumé compared to those tanned at 4° Baumé (Table 3).

Test III – Evaluation of the Effect of Pickled Skins (7° Baumé and pH = 4.0) at Different Exposure Times in Salt-Free Water

In Test III, it was possible to assess the importance of salt in the pickling stage and the impact of exposing collagen fibers to acid swelling. A significant difference was observed for all evaluated parameters, as shown in Table 4. These findings reinforce the crucial role of salt in protecting collagen fibers during pickling, preventing structural damage and maintaining leather integrity.

Table 3 – Resistance Test of Leather Subjected to Different Salt Concentrations in the Pickling Stage.

Treatment	Thickness (mm)	Tearing (N/mm)	Elongation (%)	Tensile Strength (N/mm^2)
4° Baumé	1.03±0.13[1]	55.41±9.37b	83.50±11.44	13.84±2.04b[3]
7° Baumé	1.02 ±0.34	69.36±11.85a	92.50±8.48	17.80±4.75a
10° Baumé	0.96±0.11	69.15±13.41a	92.50±9.71	15.02±2.78ab
p-value	0.4000	0.0178	0.8267	0.0410
C.V.[2] (%)	12.69	18.04	10.55	21.77

[1]Medians ± standard deviation; [2]C.V.= Coefficient of Variation; [3]Different letters in the same column indicate significant differences according to Tukey's test (5%).

Table 4 – Resistance of Leather Subjected to Different Exposure Times in a Salt-Free Solution to Assess the Effect of Acid Swelling.

Exposure Time (min)	Thickness (mm)	Tearing (N/mm)	Elongation (%)	Tensile Strength (N/mm^2)
0	0.98±0.17b[1]	67.80±13.28a	92.50±8.48a	17.80±4.75a
10	1.04±0.10a	47.40±11.40b	82.10±9.26b	10.28±2.21b
20	0.91± 0.10b	48.57± 5.92b	79.70±12.24b	11.86±2.81b
30	1.17± 0.28a	36.37 ± 8.07b	67.40±16.85b	8.25±3.26b
p-value	0.0196	<0.0001	0.0007	<0.0001
C.V.[2] (%)	17.55	20.14	15.12	28.11

[1]Medians ± standard deviation; [2]C.V.= Coefficient of Variation; [3]Different letters in the same column indicate significant differences according to Dunnett's test (5%).

When the skins were exposed to acid swelling (without the presence of salt), the results were inferior compared to the leather that remained with salt throughout the entire pickling process (Time 0, Table 4). The exposure of collagen fibers to a salt-free solution had a significant influence on the evaluated parameters.

Regarding thickness, 20 minutes of exposure did not cause a significant increase, likely due to the residual presence of salt in the solution, which may have prevented the expected thickening. However, this exposure led to collagen disorganization, resulting in lower tear resistance, elongation, and tensile strength, as shown in Table 4.

The tensile strength of the leather was 17.80 N/mm^2 with the optimal Baumé concentration (7°Bé), while resistance decreased, ranging from 10.28 N/mm^2 to 8.25 N/mm^2 as the exposure time to salt-free water increased (from 10 to 30 minutes). For progressive tearing resistance, the initial value was 67.80 N/mm, decreasing from 47.40 N/mm to 36.37 N/mm over 30 minutes. Leather elasticity was immediately affected after 10 minutes of exposure to a salt-free environment, with significantly lower values and a gradual decline over time (from 82.10% to 67.40% between 20 and 30 minutes), when compared to the initial value (92.80%). These results indicate that exposure of the skins to salt-free water, or a low salt concentration, caused a significant reduction in tear resistance (46.46%), elasticity (27.14%), and tensile strength (53.65%) after 30 minutes of exposure. This underscores the importance of salt in the pickling solution, as it protects collagen fibers from acid swelling, preventing fiber expansion (swelling) and structural rupture.

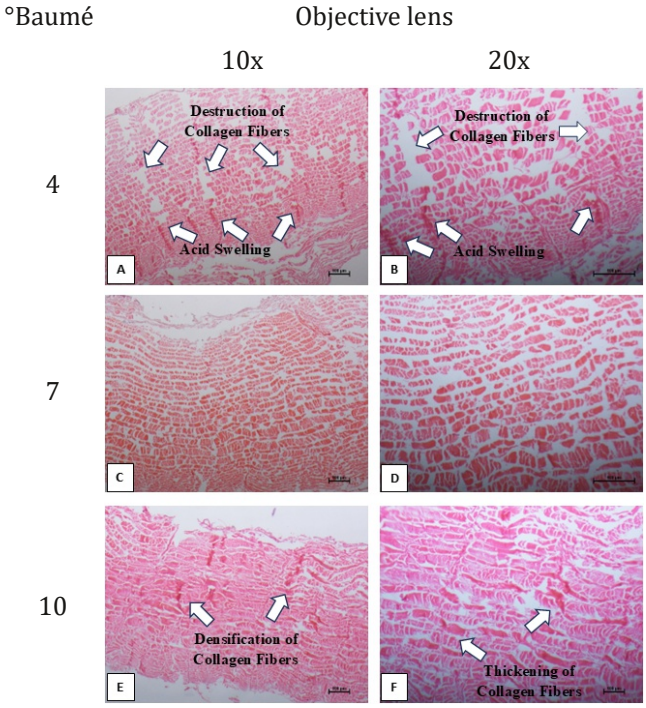
Corrêa *et al.* (2024) evaluated Nile tilapia skins tanned with vegetable tannin and obtained tear resistance values similar to those in the present study, with a thickness of 1.01 mm and 68.11 N/mm. However, the average elongation (69.65%) and tensile strength (14.91 N/mm^2) were lower compared to the results obtained in this study, which considered the initial condition (without exposure to salt-free water).

Histological Analyses of Leather

Test II – Evaluation of the Effect of Salt Concentration in the Pickling Stage (4° Baumé, 7° Baumé, and 10° Baumé) and Assessment of Salt Protection on Collagen Fibers

According to Franco *et al.* (2013), the resistance of Nile tilapia leather is primarily related to its histological structure, specifically how the collagen fiber bundles in the deep dermis overlap. Souza (2007) explains that salt usage in the pickling solution is crucial to protect collagen fibers and prevent acid swelling. The author also suggests that the salinity level should be maintained between 6° and 8° Baumé. In this context, the 4° Baumé treatment showed lower values in tensile strength and progressive tearing tests compared to the other treatments (Table 3), due to acid swelling of collagen fibers (Figure 1. A and B), which led to denaturation (gelatinization) or destruction of these fibers. On the other hand, the 10° Baumé treatment (Figure 1. E and F) did not show significant statistical differences in tensile strength compared to the other treatments. This result can be explained by the overprotection of collagen fibers due to the high salt concentration in the pickling solution. The excess salt hindered the acid's action on collagen fibers, preventing their proper acidification for the tanning agent to penetrate effectively, ultimately resulting in collagen structure densification.

Figure 1 – Histological Analysis of Nile Tilapia Leather at Different Baumé Levels During the Pickling Stage.



A and B – 4° Baumé, C and D – 7° Baumé, E and F – 10° Baumé.

Source: Author's Collection.

The 4° Baumé treatment indicated that the amount of salt present was insufficient to adequately protect collagen fibers, resulting in acid swelling. As a consequence, the collagen fibers in the central layer of the dermis became visibly thicker. The reduced salt concentration failed to protect the deeper fibers of the dermis, limiting its protective effect to only the more superficial fibers. In image B, it is possible to observe highly swollen fibers, and in the central region, the destruction of fiber bundles, indicating failure in protection and the subsequent denaturation of collagen fibers.

In contrast, in the 10° Baumé treatment, the high salt concentration that penetrated the collagen fibers hindered their proper preparation for receiving the tanning agent. The excess salt prevented efficient acidification of the fibers, impairing acid action and compromising fiber preparation for tannin absorption. As a result, the fibers were not properly treated, making it difficult for the tanning agent to act effectively, ultimately negatively impacting the final leather quality.

Test III – Evaluation of the Effect of Pickled Skins (7° Baumé and pH = 4.0) at Different Exposure Times in Salt-Free Water

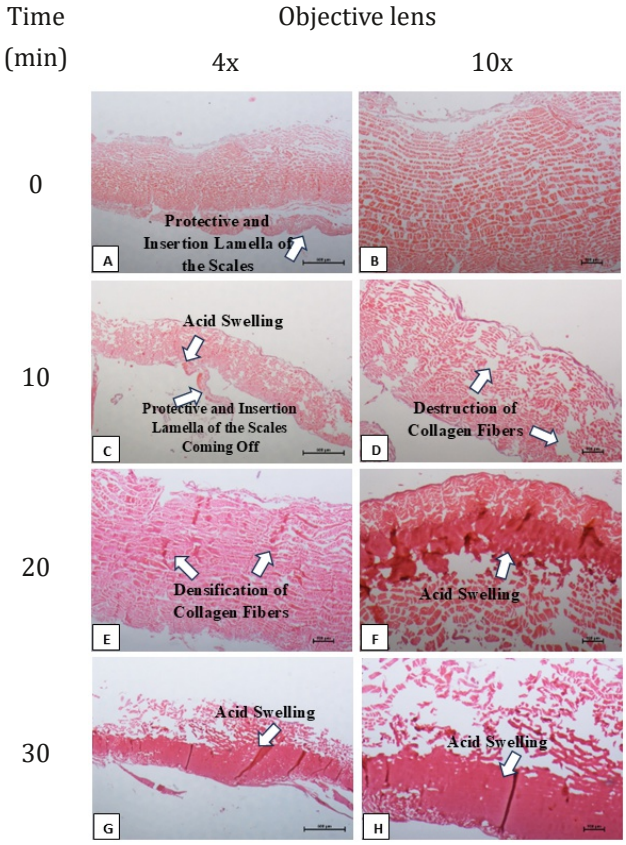
Pickling, as highlighted by Souza (2007), is a crucial step in the tanning process, especially for vegetable-tanned leathers. During this stage, the solution's pH is maintained around 4. which promotes protein denaturation, particularly collagen fibers. Additionally, skin dehydration occurs, which, along with the pH alteration, contributes to changes in protein charge. This charge shift generates electrostatic repulsion between molecules, leading to the breakage of hydrogen bonds that stabilize protein structures. Nelson and Cox (2022) explain that protein denaturation, induced by low pH, exposes hydrophobic regions in proteins, causing protein aggregate formation, where molecules cluster in a less organized manner. In the context of leather skins, this process significantly impacts mechanical and structural properties, such as resistance and elasticity, by disrupting the collagen fiber organization.

The exposure of skins to an acidic solution without the protective role of salt in the pickling stage leads to detrimental effects on collagen fiber structure, ultimately compromising leather quality. The destruction of collagen fibers results in a significant reduction in tensile strength and elasticity, as evidenced in the tests performed (Table 1). The absence of salt to protect the fibers prevents their proper preparation, leading to swollen, hardened, or stiffened leather. Additionally, the insertion lamellae and the protective layer of the scales key elements that contribute to leather texture and its characteristic fish leather pattern are damaged. The structural alteration of scales affects the visual appearance and integrity of the leather, making it of inferior quality. These structural damages emphasize the importance of rigorous control in the pickling process, including correct salt concentration, to ensure that the leather retains good mechanical and aesthetic properties.

In Figure 2 the behavior of leather during the induced acid swelling process can be observed, allowing for a detailed analysis of the effects on the histological structure of fish skins.

Images A and B represent the ideal histological characteristics for fish leather, which are essential for ensuring optimal resistance test results. These images show the structural integrity of collagen fibers, which directly contribute to the leather's strength and elasticity. The treatment shown in these images produced superior results compared to the other treatments (Table 1), as determined by Dunnett's test. Images C and D display the occurrence of acid swelling, where an increase in thickness and modification of collagen fibers due to the acid's action without salt protection are visible. This process compromises the leather's structure, as seen in the fiber alterations. Images E and F show partial destruction of collagen fibers, with the structure beginning to detach from the fiber bundles. The acid swelling process is already more evident, affecting the leather's integrity. Images G and H present a fully swollen histological section, where the collagen fiber bundles are almost entirely destroyed due to the absence of salt and prolonged exposure to acid. These images highlight the damage caused by the lack of fiber protection during the pickling process, resulting in structurally compromised leather. These histological observations are fundamental for understanding the adverse effects of acid swelling on leather quality, emphasizing the importance of maintaining proper fiber protection throughout the pickling process.

Figure 2 – Histological Analysis of Nile Tilapia Leather at Different Exposure Times to Acid Swelling. (A and B – 0 min, C and D – 10 min, E and F – 20 min, G and H – 30 min).



Source: Author's Collection.

According to Rhein (1956), the salt concentration in the pickling bath should be maintained between 6% and 10%, corresponding to a Baumé range of 6° to 10°. This interval is crucial for protecting collagen fibers during the pickling process, preventing acid swelling, which can negatively impact leather quality. Additionally, the choice of acid type used in the tanning process (formic, sulfuric, lactic, or acetic acid) should be based on its concentration and the required pH for the specific tanning method (chrome salt or vegetable tannin). This decision depends on acid availability and process requirements. Salt plays a protective role for collagen fibers during the pickling stage, directly influencing the physical properties of the leather, such as resistance, elasticity, and flexibility. Its presence in the solution helps prevent acid swelling, which could compromise fiber structure, resulting in weaker, less elastic leather that is more prone to damage. The salt concentration (expressed in °Baumé) and the exposure time of skins to acid are critical factors that must be carefully controlled to ensure high-quality leather. Therefore, the proper use of salt, in terms of both quantity and exposure time, is essential for the success of the tanning process. Strict control of these parameters ensures that the leather maintains its desired physical properties and is not compromised during processing.

CONCLUSIONS

It is concluded that salt plays a fundamental role in the pickling stage, being crucial for the protection of collagen fibers and the preservation of leather properties. The optimal salt concentration is the one that maintains a Baumé of 7. as lower values, such as 4° Baumé, significantly compromise tear and tensile strength, affecting the overall quality of the leather. Exposure of skins to a salt-free solution should be avoided, as it causes acid swelling, leading to structural damage to collagen fibers, which compromises the physical properties of the leather. Additionally, prolonged exposure to salt-free water (between 10 and 30 minutes) further intensifies the damage, reducing elasticity, tensile strength, and progressive tearing resistance. Regardless of exposure time, salt is responsible for protecting collagen fibers from acid action, ensuring that the leather maintains its quality and remains suitable for industrial and commercial applications. Therefore, the presence and correct dosage of salt in the pickling process are essential for obtaining high-strength and durable leather.

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