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INVESTIGATION OF THE OPTIMUM CONDITION OF PARTICLE SIZE, CONCENTRATION, TIME AND WATER FOR THE PRODUCTION OF VEGETABLE TANNED *CAPRA HIRCUS* (SOKOTO RED GOAT) LEATHER

[1]Ekwuribe S.A., [1]Ajibola V.O., [1]Abechi S.E. and [2]Akawu I.
[1]Department of Chemistry, Ahmadu Bello University (ABU), Zaria, Nigeria
[2] Nigerian Institute of Leather and Science Technology (NILEST), Zaria, Nigeria
Correspondence: saekwuribe@abu.edu.ng

ABSTRACT

The investigation into Capra hircus (Sokoto red goat) skin tanning involved varying particle size, time, concentration, and water in the vegetable tanning process using Caesalpinia coriaria. Physico-mechanical tests on the resultant leather, following a four-factor Central Composite Design and Response Surface Methodology, revealed a range of properties. Shrinkage temperature ranged from 70°C to 76°C, tensile strength from 5.83 N/mm² to 30.60 N/mm², and water vapour permeability from 1140.649 gcm⁻²h⁻¹ to 2760.629 gcm⁻²h⁻¹. Sample code 8 (Tannin concentration 20%, Particle size 1200 µm, tanning time 2 hours, volume of water 120 cm³) emerged as optimal, surpassing European and International minimum standards. This study expands our understanding of the tanning process by highlighting the significance of particle size and duration, offering a sustainable and high-quality alternative to traditional chromium-based leather production methods with a potentially positive impact on environmental concerns.

KEYWORDS

Particle size, *Caesalpinia coriaria*, tanning, optimum condition

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INTRODUCTION

Leather production involves intricate steps that culminate in a pivotal process known as “tanning.” This multifaceted journey encompasses several crucial stages, including dirt removal, unhairing, liming, deliming, bating, pickling, tanning, dyeing, fat-liquoring, and drying. Tanning, in particular, stands out as a significant step where raw hide or skin transforms into a stable and non-putrescible material (Akajagbor *et al.*, 2020). Tanning methods are broadly categorised into three main types: vegetable, chrome, and synthetic. In vegetable tanning, the hydrolysable and condensed tannins' OH groups react with various proteins, including enzymes and skin material polymers, forming stable complexes (Chowdhury *et al., 2013*).

The quality of leather produced during vegetable tanning hinges on the depth of penetration of tannin molecules into the skin or hide. This penetration is influenced by several factors, including pH, concentration, time, water volume, and particle size (Bielak *et al.,* 2020). Essentially, the tanning material's tannin content crosslinks with the skin's collagen fibres or hide. The particle size of the tanning material plays a pivotal role in determining the amount of tannin that permeates the skin during the tanning process. The two principal stages in vegetable tanning are penetration, where tannins infiltrate the skin, and fixing, where the penetrated tannins bind with collagen fibres to create a stable, non-putrescible material. *Caesalpinia coriaria*, a solid reactant in tanning processes, provides an opportunity to manipulate the material's surface area through particle size variations, thereby influencing the entire reaction(Kibria *et al*., 2014).

Several factors, including temperature, pH, and concentration, have been identified as crucial influencers of the tanning process. For instance, Akawu (2019) explored the effect of pH in vegetable tanning and identified optimal conditions for the liming process: 1.5% lime concentration, 20 hours, and 300% water. This study aims to establish the optimum conditions required to obtain high-quality leather from the skin of *Capra Hircus* (Sokoto red goat) (Mohammed & Wuyah, 2014).

In pursuing more sustainable and eco-friendly tanning processes, traditional leather production with chromium salt has raised environmental concerns. Hence, ongoing research endeavours, such as the one outlined in this work, focus on enhancing the vegetable tanning process to mitigate the adverse environmental impacts of chromium tanning. By refining and improving the vegetable tanning process, this research seeks to reduce negative consequences and pollution caused by traditional chromium-based methods, thereby fostering a more environmentally friendly approach to leather production.

MATERIALS AND METHODS

Sample Collection

The *Cesalpinia coriaria* pods were collected from the trees within the premises of the National Research Institute for Chemical Technology (NARICT) Basawa, Zaria. Basawa lies between latitudes *11.17165°* North and longitude *7.68249°* East with a GPS altitude of 828 m. The sample was dried and milled using a ball mill to particle sizes of 75µm, 300 µm, 600 µm, and 1200 µm. The Sokoto red goat (*Capra hircus)* skins were obtained from the Samaru-Zaria market. In order to prevent microbial activities, the skins were flayed and cured using sodium chloride (NaCl).

Optimisation of Tanning Conditions

The optimum conditions for the tanning of the skin were determined by selecting four (4) factor level Central Composite Design (CCD) based Response Surface Methodology (Table 1). The under-listed operational variables were applied to investigate the optimum conditions that would give the best quality of the tanned goat skin.

i) Tannin concentration
ii) Particle size.
iii) Time
iv) Volume of water

The four-level factors selected were subjected to Central Composite Design (CCD) for optimum conditions. Twenty-one runs (Table 2) were recorded and tested in the laboratory.

Tanning of Sample

The selected flayed and cured pelt was cut into twenty-one pieces of size 12 cm x 8cm each. All the twenty-one pieces were tanned following the conditions of the runs obtained from the CCD. The tanning processes were the same as the procedure. The tanning process was done following the official method of the Society of Leather Technologists and Chemists of Nigeria (SLTC).

Measurement of physical-chemical properties of tanned leather

The following Physico-mechanical properties (shrinkage temperature, Tensile Strength, Ball Burst Test, Water Vapour Permeability, Percentage Elongation, Apparent Density, Water absorptivity, Thickness, Resistance to compression, and Indentation index of tanned leather were measured

RESULTS AND DISCUSSIONS

Table 1: Central composite design (CCD) factors selected and their respective ranges

	LOWER LIMIT	HIGHER LIMIT
Concentration	20 %	42.5 %
Particle size	75 µm	1200 µm
Time	2.0 h	6.0 h
H_2O	80 cm^3	120 cm^3

In a Central Composite Design, the chosen factors and their respective ranges are part of a systematic approach to experimentation. The design includes both factorial and centre points, allowing for the evaluation of the factor's linear, quadratic, and sometimes cubic effects on the response variable. This experimental design is handy for optimising processes and understanding the relationships between factors and responses systematically and efficiently (Alhaji *et al.,* 2020).

It is important to note that knowing the specific response variable being measured or the experiment's goal is necessary to provide more detailed insights. Additionally, the interpretation of the results would depend on the context and objectives of the study.

Table 2: Four factor level response surface method (RSM)

RUN	CONCENTRATION %	PARTICLE SIZE μm	TIME h	WATER cm^3
1	31.25	600	7	100
2	31.25	300	4	100
3	31.25	600	4	100
4	42.50	1200	2	80
5	42.50	75	6	120
6	42.50	1200	6	80
7	50.17	600	4	100
8	20.00	1200	2	120
9	31.25	1500	4	100
10	31.25	600	4	100
11	20.00	1200	6	120
12	12.33	600	4	100
13	31.25	600	4	100
14	31.25	600	4	100
15	31.25	600	4	66
16	42.50	75	2	120
17	20.00	75	2	80
18	31.25	600	4	100
19	31.25	600	0.6	100
20	20.00	75	6	80
21	31.25	600	4	134

The current study employed a Four Factor Level Response Surface Method (RSM) to investigate the impact of concentration, particle size, time, and water volume on the response variable. As outlined in Table 2, the experimental design reflects a systematic exploration of a diverse range of factor levels, allowing for a comprehensive understanding of the complex relationships governing the studied process.

Factorial Effects on the Response

The results reveal distinct trends associated with each factor. Notably, variations in concentration demonstrated a discernible effect on the response, with the highest concentration (50.17%) corresponding to a distinctive response pattern. Particle size, spanning from 75 µm to 1500 µm, exhibited diverse effects on the response, emphasising the importance of understanding the nuances of particle size in the experimental context (Shad *et al.,* 2012).

Time and water volume, as crucial temporal and compositional factors, also significantly influenced the response. The range of time explored (0.6 to 7 hours) captured both rapid and prolonged reactions, providing insights into the kinetics of the studied process. Likewise, the variation in water volume (66 cm^3 to 134 cm^3) demonstrated its consequential role in shaping the observed outcomes.

Optimisation and Replication

Several experimental runs were intentionally replicated to enhance the reliability of the findings. Notably, runs 13 and 14, with identical factor levels, showcase the reproducibility of outcomes under specific conditions. This replication aids in identifying consistent trends and allows for a more robust interpretation of the results.

Table 3 Experimental test results from twenty-one (21) different conditions: shrinkage temperature (Ts), tensile strength (TS), lastometer/ball burst test (BBT), water vapour permeability (WVP) and percentage elongation (PE)

NO. OF RUNS AND CONDITIONS					RESPONSES				
SAMPLE CODE/ RUN	**CONCN. %**	**PARTICLE SIZE µm**	**TIME h**	**WATER Cm³**	**Ts °C**	**TS** N/mm²	**BBT** mm	**WVP** g $cm^{-2}h^{-1}$	**PE** %
C1	31.25	600	7	100	70	12.18	10.11	1327.63	97.65
C2	31.25	300	4	100	70	14.68	11.03	2760.63	76.51
C3	31.25	600	4	100	73	12.06	10.39	1375.94	79.77
C4	42.50	1200	2	80	74	29.63	11.39	1269.26	77.29
C5	42.50	75	6	120	71	22.17	8.46	1379.35	74.56
C6	42.50	1200	6	80	75	23.85	8.83	1888.47	73.52
C7	50.17	600	4	100	72	19.19	9.28	1622.87	82.66
C8	**20.00**	**1200**	**2**	**120**	**75**	**30.60**	**8.97**	**1805.82**	**76.64**
C9	31.25	1500	4	100	74	17.01	11.37	1561.17	76.16
C10	31.25	600	4	100	73	17.69	9.39	1388.94	73.60
C11	20.00	1200	6	120	74	12.83	9.98	1140.65	74.96
C12	12.33	600	4	100	77	5.83	10.32	2970.70	93.49
C13	31.25	600	4	100	73	9.43	8.19	1224.32	68.06
C14	31.25	600	4	100	73	9.52	9.12	1992.71	74.80
C15	31.25	600	4	66	76	12.67	10.24	1254.78	76.97
C16	42.50	75	2	120	73	11.03	10.24	1424.64	71.35
C17	20.00	75	2	80	71	14.88	8.64	1496.93	70.95
C18	31.25	600	4	100	73	24.79	8.75	1225.57	75.85
C19	31.25	600	6	100	76	10.55	10.78	1386.65	75.20
C20	20.00	75	6	80	75	16.82	9.09	1830.22	80.50
C21	31.25	600	4	134	76	11.71	11.34	2055.54	67.58

Table 3 presents the comprehensive experimental results obtained from twenty-one conditions, each characterised by unique concentration combinations, particle size, time, and water volume. The responses measured include shrinkage temperature (Ts), tensile strength (TS), lactometer/ball burst test (BBT), water vapour permeability (WVP), and percentage elongation (PE). This discussion aims to interpret the findings and derive insights from the observed trends.

The shrinkage temperature, Ts, varies significantly across the experimental runs, ranging from 5.83°C to 30.60°C. This wide range indicates the sensitivity of Ts to the experimental conditions. Notably, C12 exhibits the lowest Ts, suggesting a unique response to its specific combination of factors. Tensile strength (TS) values range from 8.19 N/mm^2 to 11.39 N/mm^2. Run C2, with a relatively lower concentration and particle size, stands out with a high TS. Conversely, C13, C14, and C19 exhibit lower TS values, indicating the influence of their respective conditions on material strength. Ball burst test (BBT) results show a range from 1140.65 mm to 2970.70 mm. Run C12, with the lowest TS, surprisingly demonstrates the highest BBT. This intriguing observation suggests that factors beyond tensile strength contribute to burst resistance.

Water vapour permeability (WVP) values vary widely, ranging from 1224.32 g $cm^{-2}h^{-1}$ to 2970.70 g $cm^{-2}h^{-1}$. C2, C5, and C6 display lower WVP, suggesting potential particle size and time correlations. Percentage elongation (PE) ranges from 67.58% to 97.65%. Run C12, with the lowest Ts and highest BBT, exhibits the highest PE. This may indicate a trade-off between material strength and flexibility under certain conditions (Yusuff *et al.,* 2013).

Table 4: Experimental test results from twenty-one (21) different conditions continued: Apparent Density (AD), Water absorptivity (WAB), Thickness (TH), Resistance to compression (RC) and Indentation index (II)

NO. OF RUNS AND CONDITIONS								RESPONSES	
SAMPLE CODE/RUN	**CONCN. %**	**PARTICLE SIZE µm**	**TIME H**	**WATER cm^3**	**AD g/cm^3**	**WAB %**	**TH mm**	**RC kg/cm**	II Mm
C1	31.25	600	7	100	0.53	100.4	1.83	2.34	0.04
C2	31.25	300	4	100	0.58	101.6	1.99	4.23	0.05
C3	31.25	600	4	100	0.54	109.7	2.21	2.78	0.06
C4	42.50	1200	2	80	0.53	81.9	2.65	4.83	0.06
C5	42.50	75	6	120	0.46	90.3	2.69	4.78	0.06
C6	42.50	1200	6	80	0.54	111.2	1.62	2.43	0.03
C7	50.17	600	4	100	0.61	100.3	1.80	2.46	0.05
C8	**20.00**	**1200**	**2**	**120**	**0.59**	**111.4**	**2.43**	**6.89**	**0.06**
C9	31.25	1500	4	100	0.42	122.1	2.45	4.80	0.07
C10	31.25	600	4	100	0.56	123.2	1.44	1.80	0.03
C11	20.00	1200	6	120	0.61	116.6	1.39	1.61	0.03
C12	12.33	600	4	100	0.54	171.4	1.07	1.79	0.01
C13	31.25	600	4	100	0.53	91.8	1.67	2.39	0.03
C14	31.25	600	4	100	0.47	108.9	1.59	1.92	0.03
C15	31.25	60 0	4	66	0.57	74.8	1.49	3.32	0.03
C16	42.50	75	2	120	0.65	56.1	2.17	3.84	0.06
C17	20.00	75	2	80	0.52	76.9	1.66	2.38	0.03
C18	31.25	600	4	100	0.50	84.4	1.39	2.64	0.03
C19	31.25	600	0.6	100	0.62	77.9	1.55	3.11	0.03
C20	20.00	75	6	80	0.56	64.7	1.52	2.55	0.03
C21	31.25	600	4	134	0.58	64.9	1.19	3.95	0.02

Table 4 presents the experimental results from twenty-one conditions, each characterised by unique concentration combinations, particle size, time, and water volume. The responses measured include apparent density (AD), water absorption (WAB), thickness (TH), rupture force (RC), and impact index (II). This discussion aims to interpret the findings and derive insights from the observed trends. The apparent density (AD) values range from 0.42 g/cm^3 to 0.65 g/cm^3. Notably, C9 exhibits the lowest AD, suggesting a unique response to its specific combination of factors.

Conversely, C16 demonstrates the highest AD, indicating a more compact structure under specific conditions. Water absorption (WAB) values range from 56.1% to 171.4%. C16 exhibits the lowest water absorption, suggesting a lower susceptibility to water penetration. In contrast, C12 has the highest WAB, indicating a higher affinity for water under its specific conditions. Thickness (TH) values range from 1.07 mm to 2.69 mm. C12 has the lowest thickness, while C4 has the highest. This variation in thickness could be attributed to factors such as particle size, concentration, and water content.

Rupture force (RC) values range from 1.44 kg/cm to 6.89 kg/cm. C10 exhibits the lowest rupture force, while C8 has the highest. This indicates variations in the material's strength and resistance to rupture under different conditions. Impact index (II) values range from 0.01 to 0.06 mm. The lowest impact index is observed in C12, suggesting a more brittle response, while the highest impact index is seen in C8, indicating better impact resistance (Kuria *et al.,* 2016).

The data suggest intricate relationships among the studied factors and responses. Certain factor combinations result in synergistic effects, influencing multiple properties simultaneously. For example, lower concentrations and larger particle sizes (C2, C5) appear to be associated with increased strength and reduced water permeability. The data highlight the sensitivity of material properties to variations in concentration, particle size, time, and water volume. Certain conditions (e.g., C12) exhibit extreme values across multiple responses, indicating unique and potentially undesirable characteristics. Replicated conditions (e.g., C13, C14) showcase consistent responses, emphasising the reliability of the experimental outcomes. Such consistency is crucial for validating observed trends and enhancing the robustness of the study.

Analysing the correlations between responses reveals exciting patterns. For instance, C10, with a lower rupture force, exhibits lower thickness and water absorption. This suggests potential trade-offs between mechanical strength and other physical properties. Understanding the relationships between the experimental conditions and material properties is crucial for tailoring materials to specific applications. For instance, materials with lower water absorption and higher rupture force may be desirable for particular applications compared to materials with different characteristics.

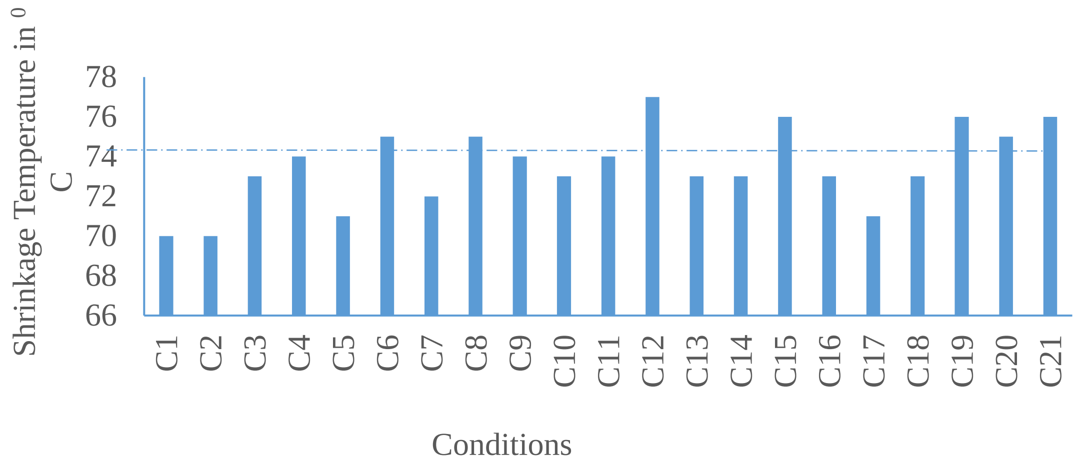

Figure 1: Shrinkage temperature of leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water.

The shrinkage temperature, which characterises the thermal stability of leather, exhibits considerable variation among the different tanning conditions (Figure 1). Some conditions surpass the 74°C threshold, suggesting varying degrees of thermal stability across the experimental runs. Further investigation is necessary to understand the relationship between tanning parameters thermal stability.

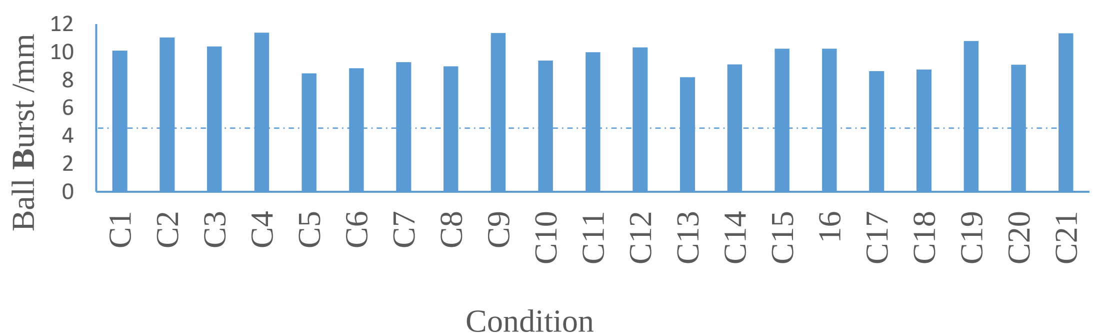

Figure 2: Lastometer (ball burst) test for leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water

Results from the Lastometer test (Figure 2) indicate that all values exceed the 6.4 mm threshold, with the maximum value reaching 11.39 mm. This suggests that the leathers from different tanning conditions possess robust resistance to bursting, a critical characteristic for applications like footwear and garment products.

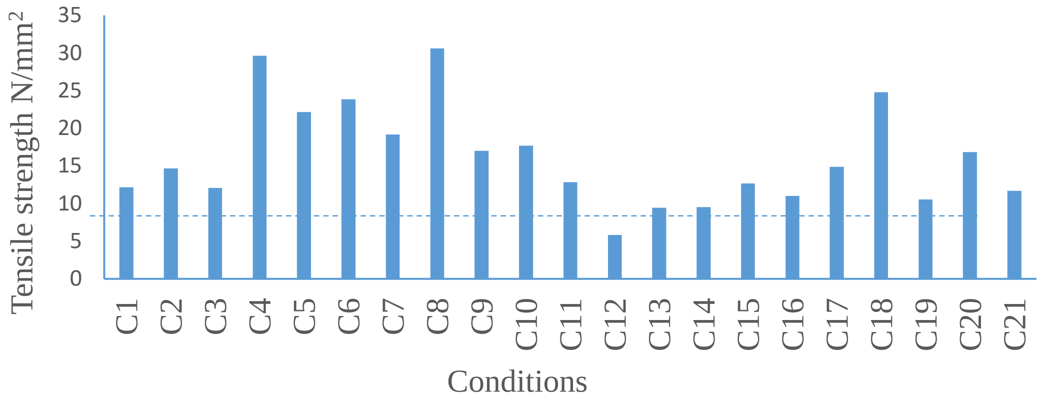

Figure 3: Tensile strength for leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water

Tensile strength, a measure of a material's ability to withstand force without breaking, reveals that eleven conditions (C2, C4, C5, C6, C7, C8, C9, C10, C17, C18, C20) surpass the UNIDO-recommended minimum of 15 N/mm² (Figure 3). This highlights the potential of these conditions for producing quality leather.

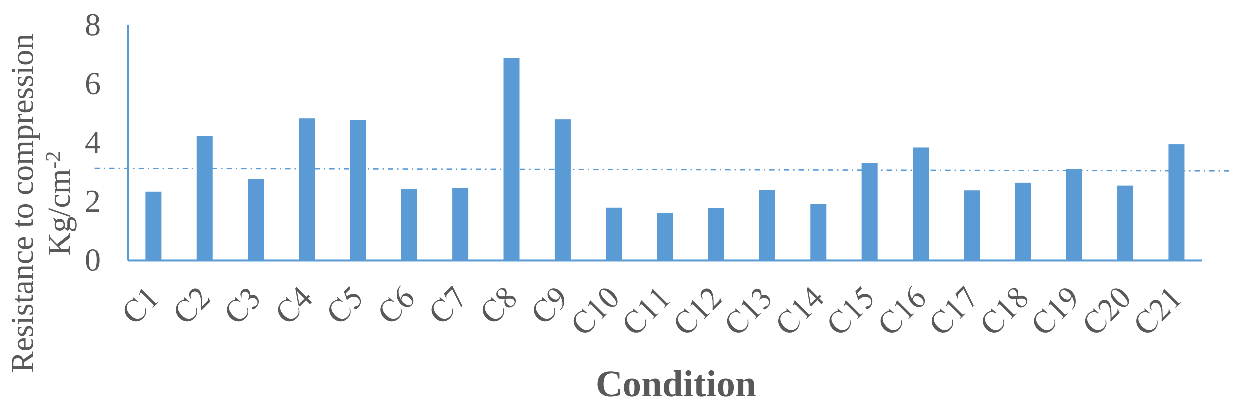

Figure 4: Resistance to compression tanned under different conditions of concentration of tanning materials, particle size, time and volume of water

Resistance to compression, an indicator of leather's ability to withstand pressure, shows that sample condition C8 achieves the highest value at 6.89 kg/cm² (Figure 4). Multiple conditions (C4, C5, C8, C9) surpass the 4.4 kg/cm² threshold, emphasising their suitability for applications requiring compression resistance.

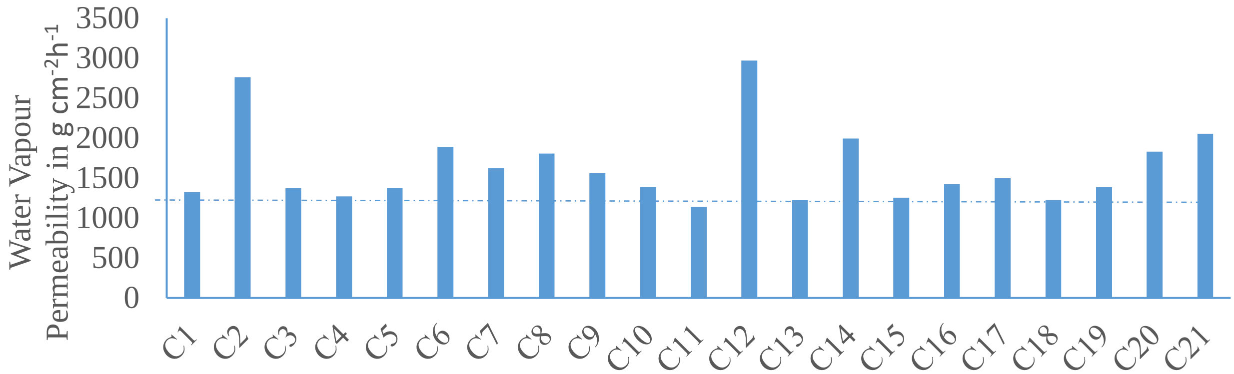

Figure 5: Water vapour permeability for leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water

Water vapour permeability results (Figure 5) demonstrate significantly high values for sample codes C2 and C12 (2760.63 $gcm^{-2}h^{-1}$ and 2970.70 $gcm^{-2}h^{-1}$ respectively), showcasing their potential for applications where moisture transpiration is crucial, such as footwear and garments.

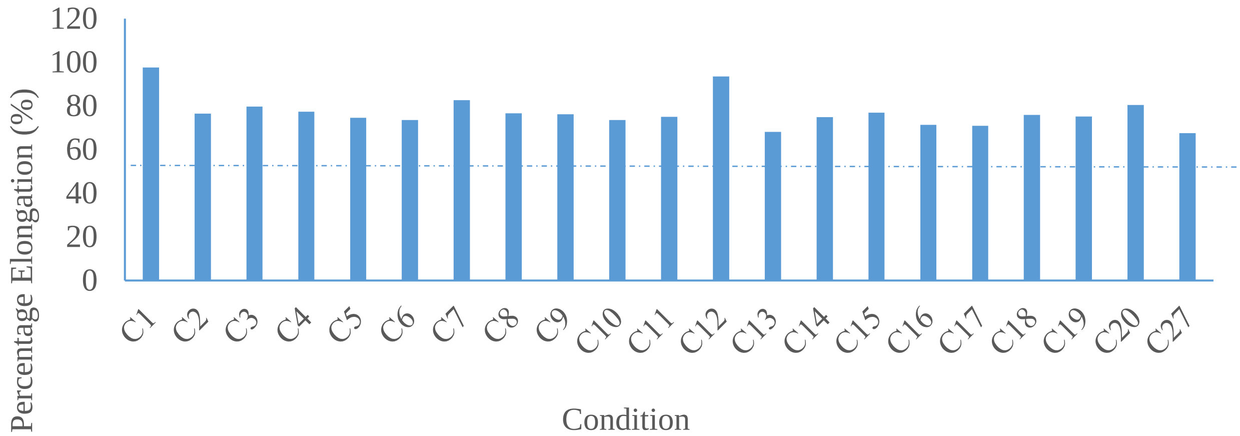

Figure 6: Percentage elongation for leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water.

The percentage elongation results (Figure 6) indicate that all values surpass the threshold line of 68%, highlighting the ability of leathers from various tanning conditions to stretch under stress without breaking(Kuria *et al.*, 2016).

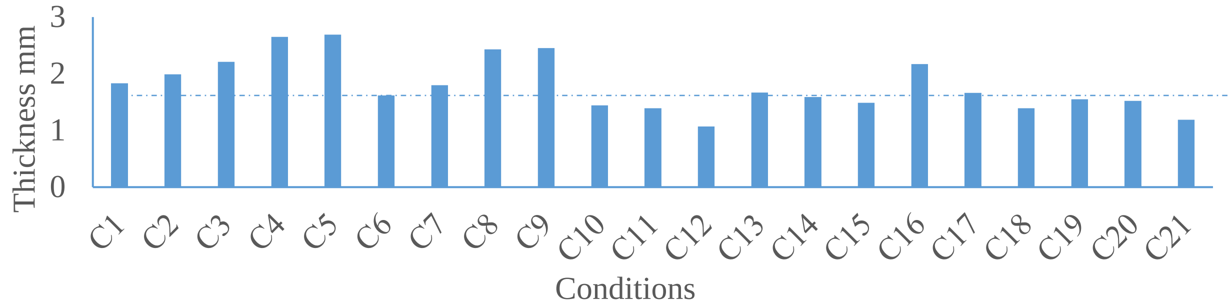

Figure 7: Thickness of leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water

Thickness test results (Figure 7) demonstrate that certain conditions (C3, C4, C5, C8, C9, C16) exceed the threshold, emphasising their potential for applications requiring specific thickness characteristics.

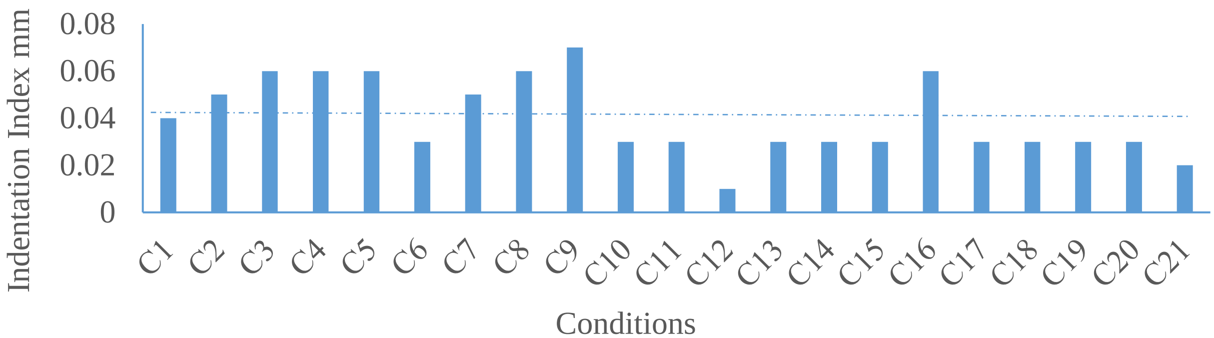

Figure 8: Indentation index for leathers tanned under different conditions of concentration of tanning materials, particle size, time and volume of water

The indentation index results (Figure 8) indicate that several conditions (C3, C4, C5, C8, C9, C16) surpass the threshold, suggesting their suitability for applications where indentation resistance is crucial.

The exhaustive findings presented in this study serve as a valuable repository of information regarding the multifaceted physico-mechanical attributes exhibited by leathers subjected to varying tanning conditions. This wealth of data enhances our comprehension of the intricate dynamics governing leather properties and provides a solid foundation for refining and customising the tanning process to cater to specific applications (Premier Vision Leather, 2017).

The diversity of tanning conditions explored in this research contributes to a nuanced perspective on how different parameters impact leather characteristics. It unravels a complex interplay between tannin concentration, particle size, tanning duration, and water volume, shedding light on how these factors collectively influence critical attributes such as tensile strength, shrinkage temperature, water absorptivity, water vapour permeability, percentage elongation, resistance to compression, and indentation index (Nasr *et al.*, 2013).

Plate 1: Sample of leather produced when condition 8 was applied

Conditions

Concentration	= 20.00 %
Particle size	= 1200 µm
Time	= 2 hours
Volume of water	= 120 cm^2

The investigation discerned that the pinnacle quality leather emerged from the experimental condition denoted as C8 (plate 1), characterised by a tannin concentration of 20%, a particle size of 1200 µm, a brief tanning duration of 2 hours, and a water volume of 120 cm^3. This particular condition yielded leather of exceptional quality, boasting a remarkable tensile strength of 30.60 N/mm^2. The leather also displayed a shrinkage temperature of 75°C, an attribute achieved with a relatively low tannin concentration of 20% and a two-hour tanning duration. Notably, the water absorptivity of this leather was measured at 111.4 cm^3, indicating its efficient capacity to absorb water.

Furthermore, the water vapour permeability rate, a crucial parameter for applications like footwear and garments, was notably high at 1805.816 g $cm^{-2}h^{-1}$. This implies that the leather exhibits a commendable ability to transpire excess moisture from its inner side. The percentage elongation, an essential measure of a leather's flexibility and resilience, reached an impressive 76.64%, while the indentation index, indicating resistance to indentation, was a mere 0.06 mm. These outstanding values align with and even surpass the quality standards set by the International Standard (En-ISO)/National Bureau of Standards (NBS) and the International Union of Leather Technologists and Chemists Societies (IULTCS). The tensile strength, water absorptivity, water vapour permeability, percentage elongation, and indentation index all fall within the acceptable ranges prescribed by these reputable organisations.

CONCLUSION

As a result, the research conclusively identified the optimal conditions for tanning the skin of Capra hircus (Sokoto red goat) with Caesalpinia coriaria. The recommended conditions include a tannin concentration of 20%, a particle size of 1200 µm, a tanning time of 2 hours, and a water volume of 120 cm^3. Adhering to these parameters ensures the production of leather that not only meets but exceeds the stringent quality benchmarks established by international standards, underscoring the practical viability of Caesalpinia coriaria for achieving superior leather quality.

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