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TANNING EFFICACY OF *PILIOSTIGMA THONNINGII* PODS FOR SUSTAINABLE LEATHER PRODUCTION

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ABSTRACT

This study investigated the tanning efficacy of tannins extracted from *Piliostigma thonningii* pods for sustainable leather production. This was necessary to harness locally sourced, viable vegetable tannins to meet local demand, thereby reducing the overdependence of local leather industries on imported tannins. The extraction of tannins was carried out using the Procter extraction method. Analyses were carried out on the tannin extract to determine the tannin class, physicochemical properties and tanning strength of the sample extract. The analysis for the physicochemical (pH, total solids and moisture content) properties and the tanning strength (total soluble matter, tannin content, and non-tannin content) was carried out using the official method of tannins analysis described by the Society of Leather Technologists and Chemists (SLTC). The tannin class of *Piliostigma thonningii* pods and other analyses (tannin strength, tannin purity and ultraviolet visible) were carried out using standard laboratory methods. The analysis showed that the tannins from *Piliostigma thonningii* pods belong to the hydrolysable class of vegetable tannins, with a pH of 4.6. Other results from the analysis include tannin content (24.4 %), the non-tannin content (9.8 %), total soluble matter (35.2 %), tannin strength (2.5 %) and tannin purity (0.7 %). These results indicate that tannins from *P. thonningii* pods possess characteristics typical of effective vegetable tanning materials, making them a viable and sustainable alternative for leather production.

KEYWORDS

Piliostigma thonningii Pods; Stabilising Collagen; Vegetable Tannins; Tanning

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INTRODUCTION

Animal hide and skins are subject to biodegradation the moment they are separated from the animal carcass. To permanently preserve the skin and impart a supple effect, tannins are applied (Akawu, 2017; Leticia *et al.*, 2015). These tannins, most of which are either of mineral or plant origin, help modify the skin, keeping it permanently preserved through chemical reactions (Covington, 2009; Sarkar & Sorcar, 2005). The product of this chemical reaction (tanning) is what is known as leather. The choice between one tannin and another depends on several factors, including the end user's desired properties, the availability of the tanning material, cost-effectiveness, and the material's impact on the user and the environment, among others (China *et al.*, 2020).

Tanning materials from mineral sources, the major of which are chromium (III) salts, have a negative impact on the environment, and hence the search for alternatives with a positive environmental impact (China *et al.*, 2019). Researchers have settled on tannins from plant sources, among all the other options, because of their eco-friendliness (Mahdi and Gurshi, 2009). Vegetable tannins, as they are often called, are both user and environmentally friendly. Besides, they give the leather produced from them a feel that tannage materials of mineral origin do not (Musa *et al.*, 2019). Although the tanner will settle for this, it needs to be harnessed and made available. Also, since these tannins are obtained from plant parts such as roots, stems, leaves, fruits, and pods, their ability to stabilise collagen must first be established (Sai *et al.*, 2019). Studies have shown that any plant material with a tannin content of 10%, a tannin strength of 1.5%, and a tannin purity of 0.5% can effectively stabilise collagen (Kuria *et al.*, 2016). Tannins obtained from different parts of a plant also belong to different classifications. They are either hydrolysable or condensed in nature,

depending on whether their root material is gallo/ellagi tannins (hydrolysable) or catechin moiety (condensed tannins) (Hagerman 1997, cited in Akawu, 2014).

Vegetable tannins are polyphenolic in nature, but not all polyphenols can stabilise collagen. Any polyphenol that will stabilise collagen must have the normal phenolic reaction with ferric chloride, evident by a change in colour to blue-black, and must have a molecular weight between 500 and 3000Da (Akawu, 2014; Griffiths, 1991). Another way to determine whether a plant contains tannable tannins is to combine it with collagen and observe its reaction by performing physical and/or chemical analyses (Bate-Smith & Swan, 1962; Griffiths, 1991). Several countries have been able to harness their naturally endowed resources to a greater extent into a material that can stabilise collagen (Leather International Magazine, 2011). In Nigeria, the search is ongoing, keeping the tanning industry largely dependent on the importation of vegetable tanning materials despite the country's abundant natural plant resources (Akawu, 2014; Iliya *et al.*, 2018; Jimoh & Oladiji, 2005).

Piliostigma thonningii is a common plant in Nigeria, growing as a wild plant in almost all the country's geopolitical zones, with its pods maturing and falling to the ground (Iliyasu *et al.*, 2019). Studies have shown that the stem has good tannin content (34%) (Gimba *et al.*, 2009). Harvesting the stem means the plant will go extinct in no time. Therefore, a study of its pod became imperative, as it matures and falls off with little or no use (Iliyasu *et al.*, 2020). A preliminary investigation to determine its ability to combine with collagen was positive, with a shrinkage temperature of <80, typical of hydrolysable tannins (Iliya *et al.*, 2018; Covington, 2009). This preliminary study was limited to applying the plant pod crude powder to animal skin and conducting physical tests to determine its ability to stabilise collagen, without necessarily establishing the tannin class, soluble

matter, or tanning strength of the material. It is on this note that this research was undertaken.

EXPERIMENTAL PROCEDURES

Sample Collection

Dried *Piliostigma thonningii* pods were collected from trees around Bassawa in the Sabon Gari Local Government Area (LGA) of Kaduna State, Nigeria.

Sample Preparation

The sample was prepared in accordance with SLC 112 (1996). The dried pods were crushed using a Thomas Wiley laboratory mill model 4. The powdered pods were passed through sieves of different mesh sizes to produce particles of different sizes. These were kept in accordance with ALCA (1957), as cited in Mahdi and Gurshi (2009).

Characterization

A qualitative test was performed to determine the tannin class using a standard laboratory method. The quantitative analysis was conducted to determine moisture, total solids, total soluble, pH, tannin content, non-tannin content, tannin purity, tannin strength, etc., using standard laboratory methods. Analyses on the extract were carried out in replicates.

Procter Extraction of Tannins

Procter extraction was carried out in accordance with SLC 112 (1996). The ground and sieved sample (40g) was weighed and soaked overnight in cold distilled water in a conical flask. This was inserted into a Procter extractor, and the fittings were well-connected to the conical flask for water extraction of the tannins. The tannin was first extracted with cold water at 25 °C in the extractor, then warmed at 40 °C for warm-water extraction, and finally heated at 80 °C for hot-water extraction. This was done to maximise extraction output. The extraction continued until a clear solution was obtained.

Determination of Total Solids

The determination of total solids was done in accordance with SLC 114 (1996). The method is intended for determining the total solid material in any sample of a solid or liquid tannin extract. The

procedure is as follows: 50 cm³ of the uniformly turbid tannin infusion was transferred into a pre-weighed conical flask, which was then weighed. This was transferred into a pre-weighed Petri dish and evaporated to dryness in a water bath. The residues were dried at 98.5 - 100 °C in a vacuum to constant weight, and the total solid was evaluated using the equation:

$$\% \text{Total solid} = \frac{W_2}{W_1} \times 100$$

Where:

W₁= Weight of material taken for test.

W₂= Weight of residue after drying.

Determination of Total Soluble Matter

The determination of total soluble matter was done in accordance with SLC 115 (1996). This method is intended for use in the determination of the total soluble matter in any sample of either ground material or a solid or liquid extract. The procedure is as follows:

The extract was filtered using a dry filter cloth. The first 250 cm³ filtrate was rejected, and the filtration continued. The filtrate (50 cm³) was evaporated on a petri dish and dried to constant weight.

Thus:

$$\% \text{ Total soluble} = \frac{\text{average weight of residue} \times A \times 100}{B \times C}$$

Where:

A = Volume of extract.

B = Volume of extract pipetted.

C = Weight of ground sample used for extraction.

Determination of Tannins content (modified shakes method)

The modified shakes method is intended for use in the determination of the matters not absorbable by hide powder, in a sample of a material, or a solid or liquid extract. The procedure is as follows. The ground sample (2g/50 cm³) was weighed, dissolved in distilled water and shaken for 30 minutes. The mixture was filtered, and the resulting filtrate was used for the analysis. The filtrate (0.05 cm³) was diluted with distilled water (10 cm³), and iron (III)

chloride solution (0.2 cm³) was added. The mixture was allowed to stand for 5 minutes, then scanned on a UV-Vis spectrophotometer (Jenway 6305) at 570 nm, the maximum absorbance wavelength of tannins. Hide powder (2 g) was placed into 50 cm³ of distilled water, and the extract (0.05 cm³) was added, shaken for 30 min, and filtered. Iron (III) chloride (0.2 cm³) was added to the filtrate, which was then analysed by UV-Vis spectrophotometry at 570 nm.

Calculation:

$$\text{Percentage of tannin} = \frac{\text{Abs}_s - \text{Abs}_c \times 100}{Wt}$$

Where:

Abs_s = Absorbance of sample.

Abs_c = Absorbance of the sample after treatment with hide powder.

Wt = Weight of the original sample (Akawu, 2017).

Determination of Non-Tannin Content

The non-tannin content was determined by subtracting the tannin content from the total soluble matter.

Thus: % non-tannins = % total soluble – % tannin content (Akawu, 2017).

Determination of pH

This was done in accordance with SLC 120 (1996). A solid extract was prepared by dissolving a specified amount in boiling water and adjusting the solution to a specific gravity of 1.05 at 18.20 °C with cold water. The pH was determined using a pH meter (Jenway 3505).

Determination of Tannin Strength

This was done by dividing the % tannin by the % total soluble, thus:

$$\text{Tannins strength} = \frac{\% \text{ tannin}}{\% \text{ total soluble}} \quad (\text{Kuria et al., 2016}).$$

Determination of Tannin Purity

This was determined by dividing the % tannin by the % non-tannin, thus:

$$\text{Tannins purity} = \frac{\% \text{ tannin}}{\% \text{ non-tannin}} \quad (\text{Kuria et al., 2016}).$$

RESULTS AND DISCUSSION

Table 1: Qualitative Analysis for Tannin Class of Sample Extract

TEST	OBSERVATION	INFERENCE
Extract + FeCl ₃	Bluish-black precipitate	Hydrolysable Tannins present
Extract + a drop of formaldehyde + 10% HCl	Pinkish precipitate formed	Hydrolysable Tannin confirmed

The analysis of the sample extract (*P. thonningii*) for the tannin class is presented in Table 1. The qualitative analysis shows that tannins from *Piliostigma thonningii* pods belong to the hydrolysable tannin class. This is evident from the change in colour to blue-black on reaction with ferric chloride, and is further confirmed by the formation of a pinkish precipitate on reaction with formaldehyde and hydrochloric acid. This result agrees with the work of Akawu (2017), which shows the same reaction for hydrolysable vegetable tannins. Hydrolysable tannins can undergo hydrolysis at temperatures above 60 °C, unlike the condensed tannins, which are liable to oxidation and polymerisation but do not efficiently hydrolyse. These tannins are complex materials composed of various polyphenolic moieties linked by ester linkages. This means that any tanning using tannins from *Piliostigma thonningii* pods must be carried out under controlled conditions. The temperature of the tanning liquor must not exceed 60 °C so as not to have the problem of hydrolysis ('Bloom'), evident by the deposition of an aggregate on the surface of the leather, thereby impeding penetration, and rendering the material unstable without proper cross-linking (Covington, 2009). It is also expected that leathers produced from these tannins should have their shrinkage

temperature between 70 and 80 °C (Covington, 2009).

Table 2: Physicochemical Properties of Sample Extract

PARAMETERS	SAMPLE
Tannin pH	4.6
Moisture Content, %	4.7
Total Solid, %	95.3

Physicochemical analysis of the sample extract was performed to determine its pH, moisture content, and total solids. The results are presented in Table 2. The physicochemical properties of the tannins indicate that those from *Piliostigma thonningii* pods have a pH of 4.6. This is within the acidic pH range typical of vegetable tannins. The pH (4.6) is within the recommended range of 4-6 for vegetable tannins (SLTC, 1996). Being acidic means that the pelt (skin) will not be affected by plumping (acidic swelling) during processing, since collagen swells markedly in a strong acidic medium (Akawu, 2017). The ability to manipulate the pH value is what separates one tanner from another. The pH of a tan liquor indicates whether the leather will be properly tanned. Vegetable tanning is carried out at a weakly acidic pH to maximise the rate of tannin diffusion into the substrate. This is why every tanner aims to achieve tannin penetration into the substrate before fixation. The pH of these vegetable tannins is close to the isoelectric point (5.5) of the raw skin. At this pH, the net charge of collagen is zero, with a high diffusion rate. This means that these tannins from *Piliostigma thonningii* will penetrate with ease without fixing at the surface, which could lead to poor tannage (China *et al.*, 2020).

The tannins' moisture content (4.7 %) indicates their ability to retain water when not in use. This moisture content result is within the recommended moisture content (≤ 14 %) for vegetable tanning materials. With this moisture content, storage of these tannins will not be

difficult if it is adequately carried out. It can be stored for a long time without risk of hydrolysis (ALCA, 1957). The total solid content of *Piliostigma thonningii* pods was 95.7%. The material constituting this total solid could be harnessed to impart the properties of the tanning materials, as demonstrated in the findings of this work.

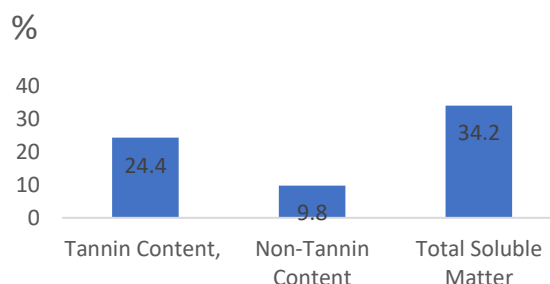


Figure 1: Soluble Matter of Sample Extract

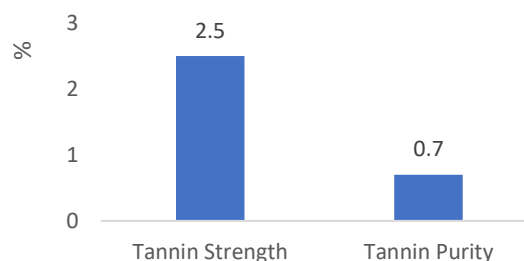


Figure 2: Strength Properties of Sample Extract

Analyses were carried out on the sample extract to determine the soluble matter and the strength properties of the tannin extract. The results obtained are presented in Figures 1 and 2. The analysis of soluble matter (Figure 1) shows that *Piliostigma thonningii* pods have a significant tannin content (24.4 %), which helps convert animal skin into a stable material called leather. The tannin content is above the minimum recommended (10 %) necessary to stabilise collagen into leather (Kuria *et al.*, 2016).

The non-tannin content of the sample extract (9.6 %) shows that the extract is suitable for tanning. It is expected that the non-tannin content of

vegetable tannins should be less than the tannin content for proper tanning to take place. The non-tannin content of the extract (Figure 1) is less than the tannin content of the extract, making it a good material for tanning animal skin. Non-tannin contents of vegetable tannins are sometimes called pseudo-tannins because of their role in aiding the penetration of the tannins in the substrate, giving it the advantage that other tannins (mineral) do not have (Sarkar & Sorcar, 2005). However, this classification may not be considered explicit, as non-tannins do not play an active role in stabilising collagen and converting it into leather. Any material whose molecular weight is not between 500 and 2000 and does not possess enough binding sites to form a stable bond with a substrate cannot be termed tannin (Jaime, 1999).

The analysis for strength properties of the tannins is a function of the ratio of tannins content to that of non-tannins content for tannins strength, and the ratio of tannins content to that of total soluble content for tannins purity ratio. The sample extract has a strength ratio of 2.5% and a purity ratio of 0.7% (Figure 2). These further confirm the tanning strength of the sample extract as both are above the minimum required standard of 1.5 and 0.5, respectively, for tannable tannins (Kuria *et al.*, 2016). The tanning ability of the *Piliostigma thonningii* pod goes beyond the average.

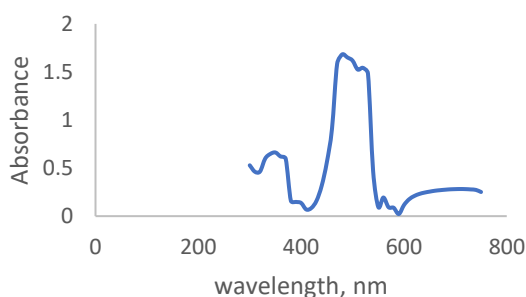


Figure 3: UV-Visible Spectra of *P. thonningii*

The extract from *Piliostigma thonningii* pods was analysed using a UV-Vis spectrophotometer. The spectra are presented in Figure 3. This spectrum shows the lander max of the tannin extract at 480 nm, the wavelength at which the absorbance was maximum. The ultraviolet-visible (UV) (Figure 3) analysis of the sample extract is typical of tannable vegetable tannins. Tannable vegetable tannins absorb at a wavelength of between 400 and 750 nm. The sample extract shows a prominent peak at a wavelength above 400 nm (480 nm), making it suitable for the intended purpose of stabilising collagen. This result agrees with the work of Akawu (2017), which shows that tannable tannins of the hydrolysable class absorb at 470 nm.

CONCLUSIONS

This study analysed the sample extract both qualitatively and quantitatively to determine the tannin class and the ability of the tannins to stabilise collagen into leather. The qualitative test showed that the tannins from *Piliostigma thonningii* pods belong to the hydrolysable class. The quantitative tests showed that the tannin extract has sufficient strength to bind collagen effectively, thereby converting it into a stable material called leather. The synergistic tanning properties of these tannins can be investigated in combination with other tannins of either the same or a different class for improved tanning performance.

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