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PHYSICOCHEMICAL CHARACTERIZATION OF *CITRULLUS LANATUS* SEED OIL COLLECTED IN DIABA, NORTHERN SENEGAL: COMPARISON OF SOXHLET EXTRACTION AND HEXANE MACERATION

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ABSTRACT

Citrullus lanatus seeds are an oil-bearing plant material that may be valorized as a local source of vegetable oil in Senegal. This study investigated the extraction and physicochemical characterization of oil from *C. lanatus* seeds collected in December 2025 in Diaba, northern Senegal. After drying away from direct sunlight, the seeds were subjected to two hexane-based extraction procedures: continuous Soxhlet extraction and maceration. Extraction yields were 31.4% and 24.2%, respectively. The oils were characterized by determining pH, density, calorific value, unsaponifiable matter, volatile matter, as well as acid, saponification, ester, iodine and peroxide values. Both oils were light yellow and had a pleasant odour. The Soxhlet oil showed acid, saponification, ester, iodine and peroxide values of 3.93 mg KOH/g, 179.52 mg KOH/g, 175.59 mg KOH/g, 39.84 g I_2/100 g and 10 meq O_2/kg, respectively. For maceration, the corresponding values were 6.17, 223.28, 217.11, 105.32 and 8. Unsaponifiable matter contents were 0.50% and 0.85%, while volatile matter contents were 1.11% and 0.98%. These findings demonstrate an effect of extraction procedure on yield and several physicochemical characteristics.

Keywords: *Citrullus lanatus*; Seed oil; Soxhlet; Maceration; Physicochemical characterization.

1 INTRODUCTION

Vegetable oils play an important role in human nutrition, the cosmetics industry, traditional pharmacopoeia and various agro-industrial sectors[1,2,3]. Beyond their energy role, they are an essential source of fatty acids, bioactive compounds and molecules of technological interest[4,5,6]. In a context marked by the search for local, sustainable and high-value natural resources, exploring new oilseeds resources from cultivated or wild plants appears to be a promising way to strengthen food security and support the economic development of rural regions.

Among plant species of interest as oil sources, *Citrullus lanatus* (better known as "beref" in Senegal or "egusi" in West Africa), which belongs to the Cucurbitaceae family, holds a special place among unconventional oilseeds resources. Its seeds are a valuable by-product rich in lipids, proteins and minerals[7]. In several regions of Africa, notably in Senegal, *Citrullus lanatus* seeds are traditionally roasted and eaten as a snack, ground into flour, used in sauce formulations, or exploited as a source of oil[7,8]. This oil is attracting growing interest because of its potential nutritional characteristics and its prospects for use in the food, pharmaceutical and cosmetic fields for its moisturizing properties.

The composition and quality of a vegetable oil depend on many factors, including the geographical origin of the plant, soil and climate conditions, the ripening stage of the fruit, post-harvest conditions, and the extraction method used[9,10]. In particular, the choice of extraction process can influence not only the oil yield but also its physicochemical properties, oxidative stability and overall composition[11,12,13]. Soxhlet extraction, widely used in the laboratory, generally gives good yields thanks to continuous extraction of the plant material with hot solvent. Conversely, maceration is a simpler and often less energy-intensive method, but its efficiency can be influenced by contact time, sample particle size and the nature of the solvent used[11,12,13].

The quality of a vegetable oil is classically assessed by determining several physicochemical parameters. The acid value reflects the degree of triglyceride hydrolysis and the free fatty acid content, while the peroxide value is an indicator of the primary oxidation of the oil. The iodine value is used to estimate the degree of unsaturation, whereas the saponification value gives information on the average molar mass of the fatty acids making up the triglycerides. The ester value, density, and unsaponifiable and volatile matter contents also provide useful information on the purity, stability and technological properties of the oil. Together, these parameters make it possible to assess the suitability of the oil for various uses and to compare its profile with that of other vegetable oils already exploited[14,15,16].

In Senegal, and particularly in the Sahelian and Sudano-Sahelian zones, local oil-bearing plant resources remain insufficiently characterized. The valorization of *Citrullus lanatus* seeds could nevertheless represent an interesting opportunity, both nutritionally and economically, especially in rural areas where the cultivation and consumption of the seeds of this species are widespread. However, the available data on the physicochemical properties of oil obtained from locally harvested seeds remain limited, particularly for the northern regions of the country.

It is in this context that the present study was undertaken. Its objective is, on the one hand, to characterize the oil extracted from *Citrullus lanatus* seeds collected in Diaba, in the Saint-Louis region of northern Senegal, and, on the other hand, to compare the influence of two extraction processes, Soxhlet extraction and maceration, on the yield and physicochemical characteristics of the oil obtained. More specifically, the aim is to determine the extraction yields and to evaluate various oil quality parameters, namely the acid, peroxide, iodine, saponification and ester values, as well as density, unsaponifiable matter content and volatile matter content. The results will provide new data on the potential of this local oilseeds resource and contribute to its valorization.

2 MATERIALS AND METHODS

2.1 Plant material

The biological material (Figure 1) was collected in December 2025 in Diaba, a locality situated at 15°55′0″ N and 13°30′0″ W, in the Saint-Louis region of northern Senegal. After harvesting and identification at IFAN (Institut Fondamental d'Afrique Noire), the seeds were separated from the pulp (Figure 1), cleaned to remove visible impurities, then dried away from direct sunlight until their moisture content stabilized. This drying mode was chosen to limit thermal and photo-oxidative alterations likely to affect the quality of the lipid constituents.

After drying, the seeds (Figure 1) were ground to obtain a powder suitable for contact with the extraction solvent.



Figure 1: Photographs of the plant, pulp and seeds of Citrullus lanatus

2.2 Oil extraction

2.2.1 Soxhlet extraction

A 50 g mass of seed powder was placed in a cellulose extraction thimble, then placed in the Soxhlet extractor. Extraction was carried out for 1 h 30 min, corresponding to 10 cycles, to ensure exhaustion of the plant matrix. At the end of the extraction, the resulting miscella was concentrated by solvent evaporation, then the recovered oil was dried to remove residual traces of hexane before weighing (Figure 2).

2.2.2 Extraction by maceration

For maceration, the same quantity of 50 g of powder was brought into contact with hexane in an Erlenmeyer flask for 18 h under continuous stirring at room temperature. After maceration, the mixture was filtered to separate the marc from the filtrate. The solvent was then removed by evaporation, and the oil obtained was dried before weighing (Figure 2).

2.3 Extraction yield

The oil extraction yield was calculated using the following relationship:

$$\%\ oil\ obtained = \frac{mass\ of\ oil\ obtained\ in\ grams}{mass\ of\ powder\ extracted\ in\ grams} \times 100$$

2.4 Physicochemical characterization of the oils

The oils obtained by Soxhlet extraction and by maceration were subjected to a series of physicochemical analyses to assess their quality, stability and valorization potential. The determinations were carried out according to the usual methods of fat and oil analysis, based on recognized standards (AFNOR, ISO or AOAC).

2.4.1 Acid value (I_A)

In an Erlenmeyer flask, a 0.5 g oil sample was dissolved in 20 mL of a 1:1 (v/v) ether/EtOH mixture in the presence of three drops of phenolphthalein. The mixture was then titrated with a 0.1 N potassium ethanoate (KOEt) solution until a pink color appeared. A blank test was carried out with 20 mL of the 1:1 (v/v) ether/EtOH mixture following the same experimental protocol.

$$I_A = \frac{56.1 \times N \times (V - V')}{w}$$

- $V = volume\ of\ de\ KOEt\ for\ the\ assay, in\ mL$
- $V' = volume\ of\ KOEt\ for\ the\ blank, in\ mL$
- $N = titer\ of\ KOEt$
- $w = mass\ of\ the\ oil\ in\ grams$

The acidity is deduced from the acid value and is a conventional expression of the percentage of free fatty acids. These two chemical indicators were determined according to standard NF V 03-906 (AFNOR, 1984).

2.4.2 Saponification value (I_S)

A 0.5 g mass of oil was placed in a flask and dissolved in 20 mL of a 1:1 (v/v) ether/EtOH mixture and 20 mL of 0.5 M KOEt solution. The mixture was refluxed for one hour, then cooled to room temperature. The excess KOEt was titrated with a 0.5 N HCl solution in the presence of three drops of phenolphthalein. A blank test was carried out with 20 mL of the 1:1 (v/v) ether/EtOH mixture and 20 mL of 0.5 M KOEt solution following the same experimental protocol. This parameter was determined following the protocol described in standard NF T 60-206 (AFNOR, 1984).

$$\boldsymbol{I_S = \frac{56.1 \times N_H \times (V' - V)}{w}}$$

- $V = volume\ of\ de\ HCl\ for\ the\ assay, in\ mL$
- $V' = volume\ of\ HCl\ for\ the\ blank, in\ mL$
- $N_H = equivalent\ concentration\ of\ HCl$
- $w = mass\ of\ the\ oil\ in\ grams$

2.4.3 Iodine value (I_I)

In an Erlenmeyer flask, a 0.5 g mass of oil is dissolved in 25 mL of CCl_4, and 10 mL of Wijs solution is added. The mixture is placed in the dark for 45 minutes. 100 mL of water and 15 mL of 10% KI solution are then added, and the flask is returned to the dark for 10 minutes, giving a yellow color. The excess iodine is titrated with a 0.1 N $Na_2S_2O_3$ solution until decolorization in the presence of starch paste. A blank test is carried out with 25 mL of CCl_4 following the same protocol.

$$I_I = \frac{12.69 \times N \times (V' - V)}{w}$$

- $V = volume\ of\ de\ Na_2S_2O_3\ 0.1\ N\ for\ the\ assay, in\ mL$
- $V' = volume\ of\ Na_2S_2O_3\ 0.1\ N\ for\ the\ blank, in\ mL$
- $N = equivalent\ concentration\ of\ Na_2S_2O_3$
- $w = mass\ of\ the\ oil\ in\ grams$

2.4.4 Peroxide value (I_P)

In an Erlenmeyer flask, a 0.5 g mass of oil, 10 mL of chloroform, 15 mL of acetic acid solution and 1 mL of potassium iodide solution are mixed. The mixture is shaken vigorously for 1 minute, then placed in the dark for 5 minutes, after which 15 mL of distilled water are added. The mixture is finally titrated with a 0.1 N $Na_2S_2O_3$ solution in the presence of 1% starch paste until decolorization. The same protocol was used to carry out a blank test with 10 mL of chloroform.

$$I_P = \frac{1000 \times N \times (V - V')}{w}$$

- $V = volume\ of\ de\ Na_2S_2O_3\ 0.1\ N\ for\ the\ assay, in\ mL$
- $V' = volume\ of\ Na_2S_2O_3\ 0.1\ N\ for\ the\ blank, in\ mL$
- $N = equivalent\ concentration\ of\ Na_2S_2O_3$

- $w = mass\ of\ the\ oil\ in\ grams$

2.4.5 Ester value (I_E)

The ester value was calculated from the difference between the saponification value and the acid value using the following relationship:

$$I_E = I_s - I_A$$

It is used to determine the fraction of fatty acids engaged in esterified form in the triglycerides.

2.4.6 Unsaponifiable matter content (m_i)

The unsaponifiable matter content was determined after saponification of a 2 g oil sample. The unsaponified fraction was extracted with diethyl ether. After evaporation of the ether, the unsaponifiable fraction was dried and weighed. The result was expressed as a percentage of the mass of the oil sample. This analysis generally follows the official method of standard ISO 3596.

$$m_i = \frac{mass\ of\ the\ diethyl\ ether\ extract}{mass\ of\ the\ oil} \times 100$$

2.4.7 Volatile matter and water content (T)

The determination of volatile matter and water was carried out by the gravimetric method. A 1 g oil sample was placed in an oven at 105 °C. The mass loss was monitored at 10-minute intervals until a constant mass was obtained.

$$T = \frac{m - m'}{m}\ x\ 100$$

- **m=** initial mass of the sample
- **m'=** constant mass of the sample after time in the oven

2.4.8 Calorific value (CV)

The calorific value (CV), expressed in kilojoules per kilogram, was calculated using formula 3 of Batel *et al.*[17]:

$$CV = 47645 - 4.187I_i - 38.31I_S$$

2.4.9 Determination of pH

A 2 g sample of each oil was placed in a beaker containing 13 mL of hot distilled water, and the mixture was stirred at room temperature. The pH of the mixture was determined using a pH meter.

2.4.10 Density (d)

Density was determined at room temperature using a graduated cylinder and a balance. A 1 mL sample of each oil was weighed and the density was calculated using the relationship:

$$d = \frac{mass\ of\ the\ oil\ sample}{volume\ of\ the\ oil\ sample}$$

3 RESULTS AND DISCUSSION

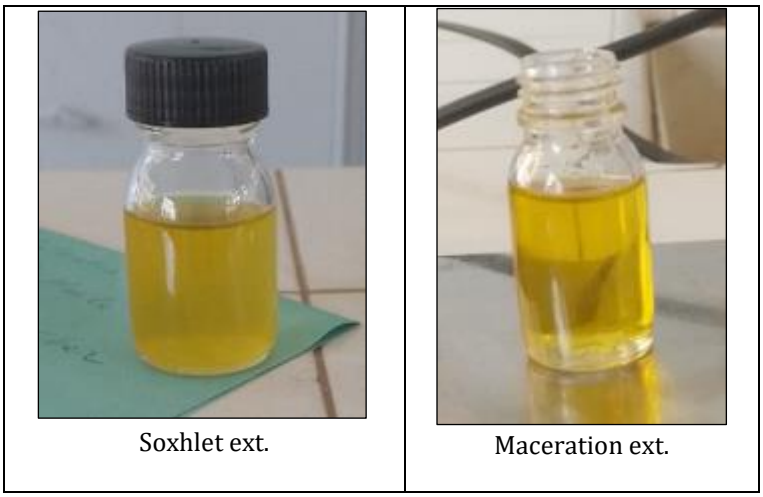
The physicochemical properties (pH, density, calorific value, and acid, saponification, ester, iodine and peroxide values) are presented in Table 1. The comparison of the acid, saponification, iodine and peroxide values of *Citrullus lanatus* oil with those of olive and peanut oils is shown in Table 2. Figures 3 and 4 show, respectively, the change in mass of the oils extracted by Soxhlet and by maceration as a function of time at 105 °C.

Table 1: Physicochemical parameters of *Citrullus lanatus* seed oil

Parameters	Soxhlet extraction	Maceration
% yielded	31.4	24.2
pH	6.70	6.20
Density (g/cm3)	0.80	0.77
Calorific value (Kj/Kg)	40600.78	39162.80
Unsaponifiables (%)	0.50	0.85
Volatile matter (%)	1.11	0.98
Colour	Light yellow	Light yellow
Odour	Pleasant	Pleasant
Iodine value (gI_2/100g)	39.84 ± 0.01	105.32 ± 0.01
Acid value (mgKOH/g)	3.93 ± 0.43	6.17 ± 0.14
Acidity (%)	1.96 ± 0.26	3.08 ± 0.07
Saponification value (mgKOH/g)	179.52 ± 0.05	223.28 ± 0.04
Ester value	175.59 ± 0.48	217.11 ± 0.18
Peroxide value ($mEqO_2$/Kg)	10.00 ± 0.30	8.00 ± 0.38

Table 2: Comparison of the values of *Citrullus lanatus* seed oil with those of olive and peanut oils

Indices	Soxhlet	Maceration	Olive oil	Peanut oil
Iodine value (gI_2/100g)	39.84 ± 0.01	105.32 ± 0.01	75 − 94	77 − 107
Acid value (mgKOH/g)	3.93 ± 0.43	6.17 ± 0.14	0.6 − 4	0.6 − 4
Saponification value (mgKOH/g)	179.52 ± 0.05	2232.8 ± 0.04	184 − 196	187 − 196
Peroxide value ($mEqO_2$/Kg)	10.00 ± 0.30	8.00 ± 0.38	5 − 20	10 − 15



Soxhlet ext. Maceration ext.

Figure 2: *Citrullus lanatus* oil extracts

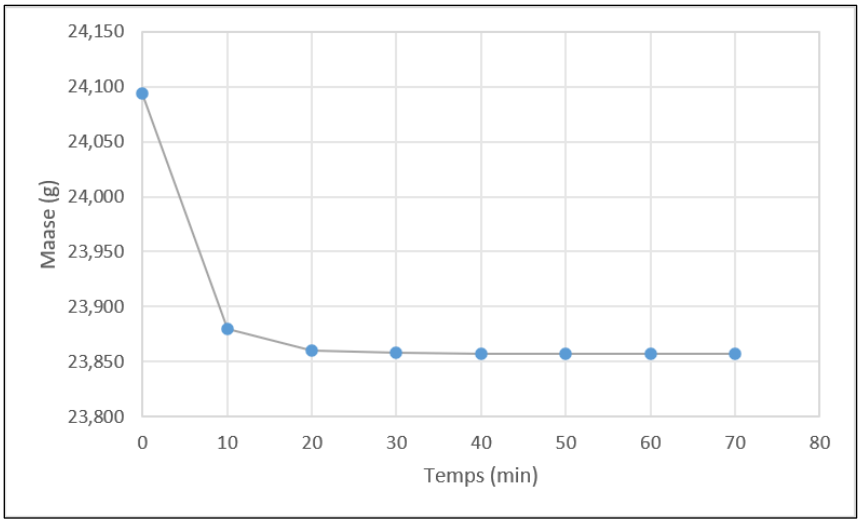


Figure 3: Curve showing the change in oil mass over time at 105°C (Soxhlet extraction)

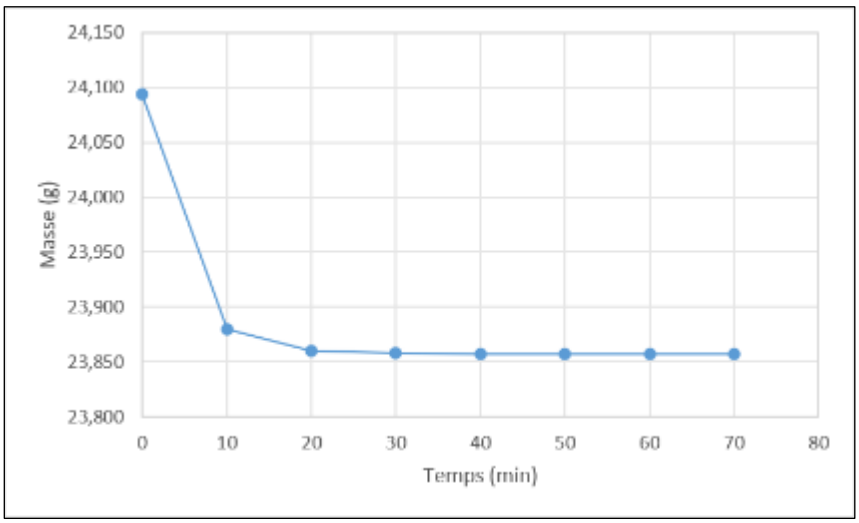


Figure 4: Curve showing the change in oil mass over time at 105°C (maceration)

Citrullus lanatus seeds oil is light yellow (Figure 1). The yields of Soxhlet extraction and maceration are 31.4% and 24.2%, respectively. The difference observed shows that continuous hot extraction favors a greater recovery of the lipid fraction of the seeds. It is also comparable to the yields of 24.8–30.0% reported by Mabaleha *et al.* for several oils extracted from melon seeds by Soxhlet[18].

The acid value, lower for the Soxhlet oil (3.93 mg KOH/g) than for the macerated oil (6.17 mg KOH/g), indicates a difference in the amount of free fatty acids measured. The values remain within a range already observed for melon seed oils, but origin and storage conditions can strongly influence this parameter.

The peroxide value is 10 meq O_2/kg for Soxhlet and 8 meq O_2/kg for maceration. These values are close to the upper limit of the range reported for several melon seed oils by Mabaleha *et al.* (1.1–10.9 meq/kg) [18]. The slightly higher level in the case of Soxhlet may be discussed in relation to exposure to heat and atmospheric oxygen during extraction.

The iodine values obtained by Soxhlet and by maceration are 39.84 and 105.32 g I_2/100 g, respectively, which shows that the oil obtained by maceration has a markedly higher degree of unsaturation than the oil obtained by Soxhlet. The value obtained for maceration is comparable to those frequently reported for *C. lanatus* oils, often above 90 g I_2/100 g, whereas the value obtained for Soxhlet is markedly lower[19,20,21]. This difference warrants a verification of the method, the units and the calculations. A fatty acid analysis by GC-FID or GC-MS would be particularly important.

The saponification value of the oil extracted by Soxhlet (179.52 mg KOH/g) is close to values already published for *C. lanatus* seed oils[19,20,21]. The value of 223.28 mg KOH/g obtained by maceration is, however, higher. Since the saponification value is related to the average molar mass of the fatty acids, this difference could reflect a difference in

the extracted fraction or analytical variability. Replicates and statistical analysis would help consolidate this observation.

Compared with commonly consumed vegetable oils such as olive and peanut oils (Table 2), the oils from *Citrullus lanatus* seeds obtained from fruits collected in Diaba show contrasting characteristics. The acid, saponification and peroxide values of the oil extracted by Soxhlet are comparable to those of these vegetable oils. However, the iodine value of this oil is markedly lower than those of olive and peanut oils. The oil obtained by maceration has acid and saponification values higher than the values reported for peanut and olive oils. The iodine and peroxide values of the oil obtained by maceration are comparable to the values reported for olive and peanut oils[22,23].

The unsaponifiable matter contents are low (0.50–0.85%). This fraction may contain sterols, tocopherols, hydrocarbons, pigments and other minor constituents. Its detailed characterization would be an interesting perspective for assessing the functional value of the oil.

The water and volatile matter contents, 1.11 and 0.98% for Soxhlet and maceration respectively, are relatively close. These values are, however, higher than the standard, which sets the maximum content at 0.2% at 105 °C[24].

The measured densities (0.79–0.77) are lower than several published values for *C. lanatus*, often around 0.90 [18,19,20,21]. The measurement conditions, the temperature and the definition of density used should therefore be checked before any direct comparison.

The pH values of the oils, 6.70 and 6.20 for Soxhlet extraction and hexane maceration respectively, should be interpreted with caution because pH is not a standard indicator for an anhydrous oil. The calorific values of the oils are 40,600.78 and 39,162.80 kJ/kg for Soxhlet extraction and maceration, respectively. In human nutrition, since this oil is consumed by local populations, this parameter gives positive information on the caloric value of this fat.

Overall, the results show that the extraction method influences the yield and certain quality parameters. These results already obtained justify further investigation of the molecular composition of the oil from *C. lanatus* seeds collected in Diaba.

4 CONCLUSION

The oil from *Citrullus lanatus* seeds collected in Diaba shows interesting valorization potential. Soxhlet extraction gave the highest yield, 31.4%, compared with 24.2% for maceration. Both oils are light yellow in color with a pleasant odor, but differences were observed for the acid value, saponification value, ester value, unsaponifiable matter and calorific value, while for the iodine values the difference is very significant.

These results constitute a first basis for characterizing this local resource. Before drawing a conclusion on food or cosmetic use, it is recommended to complete the study with the fatty acid profile, tocopherols, phytosterols, phenolic compounds, UV extinction coefficients and oxidative stability. The iodine, density and saponification values should also be verified and documented with the exact methods and units.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

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Disclosure of conflict of interest

The authors declare no conflict of interest.

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