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Physicochemical Analysis and Characterization of the Lipid Fraction of the Tubers of *Cyperus esculentus* L. var. Sativus from Côte d'Ivoire

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Aims: this work consists in determining the physical, physico-chemical parameters, the composition in fatty acids and unsaponifiables of the oil of tuber of *Cyperus esculentus* of Côte d'Ivoire**.**

Methodology: After extraction of the fat by the Soxhlet method with hexane, the physico-chemical parameters were determined by colorimetric assay. The analysis of the fatty acid and unsaponifiable acid composition was carried out by GC-MS.

Results: The fat content ranged from 23.64 to 27.66%, those of fatty and unsaponifiable acids were from 90.41 to 95.20% and 1.59 to 1.60%, respectively. As for the values of the acid, peroxide, saponification, iodine, ester, refraction and density indices, they varied respectively from 2.81 to 10.95 mg KOH/g, 1 to 2 meq of O_2/Kg oil, 198.72 to 216.78 mg KOH/g, 54.64 to 73.86 g I_2/100g oil, 193.11 to 205.83 mg KOH/g, 1.453 to 1.461, and 0.76 to 0.83. In the fatty acid composition of oils, the predominance of unsaturated fatty acids (70.7-92.14%) was revealed, mainly oleic acid prevails (51.04-82.83%). Additionally, petroselinic (0.23-10.15%,) and 2,6-di-O-palmitoyl-L-ascorbic (8.58-8.84%) acids known for their medicinal properties (anti-inflammatory, antimicrobial, antioxidants), were first discovered in the oil of *Cyperus esculentus*. As for the unsaponifiables, they are mainly dominated by phytosterols (22.30-44.03%) and hydrocarbons

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(27.70-50.8%).
Conclusion: These results suggest that oil from *Cyperus esculentus* tubers from Côte d'Ivoire can be used as an excellent source of edible and medicinal oil.

Keywords: Cyperus esculentus; oil; physico-chemical parameters; GC-MS; Côte d'Ivoire.

1. INTRODUCTION

Vegetable oils also occupy an important place in the diet of men, in cosmetics or in certain soft medicines. They have pharmaceutical properties and are very important energy reserves [1]. Their properties are determined by the plurality of fatty acids and unsaponifiables which they contain. Essential fatty acids (arachidonic, linoleic, linolenic) have an antioxidant effect, their consumption prevents oxidative stress and slows down the aging process. We know that the human need for essential fatty acids is 2 g/day [2,3]. In addition, oleic (monounsaturated) and linoleic (polyunsaturated) acids increase HDL cholesterol, which reduces the risk of cardiovascular disease, diabetes, asthma and cancer [4]. As for the constituents of the unsaponifiables of vegetable oils, they have many interesting properties. Indeed, polyphenols have antioxidant, antibacterial and anti-inflammatory properties. Phytosterols, good healing and regenerating agent, improve the "barrier" function of the skin and microcirculation, slow down skin aging. As powerful natural antioxidants, carotenoids regenerate and repair the skin by stimulating the synthesis of collagen and photoprotectors [2,5]. There are limited sources of linoleic and linolenic acids in the plant kingdom, but almost nothing has been found for arachidonic acid. But the African continent is full of oilseeds, most of which are unused or little used. However, they may be important new sources of these mono- and polyunsaturated fatty acids.

In recent years, *Cyperus esculentus* L. var. sativus (nutsedge, tiger nut, chufa) has been the subject of many studies. The increased interest in this crop is explained by the nutritional value of its tubers [6,7,8,9]. *Cyperus esculentus* is a weed plant of tropical and Mediterranean regions. It is a monocotyledonous root vegetable that grows in humid places and belongs to the Cyperaceae family [10]. Tigernut tubers are one of the oldest cultivated plants [11]. In ancient Egypt, nuts have been found in archaeological excavations where its tubers have been used as a source of food, medicine and incense for fumigating homes and clothes, as well as incense for myrrh. From Egypt, Arab traders spread tiger nuts to northern and western Africa, Sicily and Spain [12]. At present, given the composition of lipids, starch, proteins, sugars, vitamins E, C, as well as trace elements (magnesium, calcium, phosphorus, iron, etc.), tiger nut is very popular. Several works have shown that the tubers of *C. esculentus* produce edible oil (up to 33%) rich in unsaturated fatty acids [7,8,9]. Moreover, the unsaponifiable part of *C. esculentus* oil is rich in vitamin E and phenolic compounds [13]. As a result of research conducted by doctors, it was found that 100 g of tiger nut cover the daily norm of useful vitamins and microelements [14]. Such consumption contributes to effective weight loss and improvement of metabolic disorders in obese diabetics [14]. In Côte d'Ivoire, tiger nut is sold everywhere, commonly called tchongon in the Malinké, Bété, Dida and Bambara languages; atadjo in apolo; shop in koulan go; maguélé in Sénoufo (Korhogo) and consumed after soaking in water or blanched as in the traditional case, and also used in powder form in drinks [15].

Despite these advantages, the oil from the tubers of *Cyperus esculentus* is still not part of the local diet. However, in the face of changing demographics and the complexity of health issues and trends, the high cost of living is increasingly felt. From this point of view, tiger nut can contribute to poverty reduction among vulnerable populations, especially rural African women. Thus, the aim of this work is to determine the physico-chemical parameters (refractive indices, acid, peroxide, saponification, ester and iodine) and the fatty acid and unsaponifiable acid composition of the tuber oil of *Cyperus esculentus* from Côte d'Ivoire.

2. MATERIAL AND METHODS

2.1 Plant Material

The yellow tubers of *Cyperus esculentus* were collected in April 2020 in three villages in Côte d'Ivoire: Klolékaha (in the department of Sinematiali, 9° 35' North 5° 23' West), Lélékaha (in the department of Korhogo, 9° 27' 41" North 5° 38' 19" West) and Sokala Sobara (in the department of Dabakala, 8° 21' North 4° 31' West). They have been authenticated at the

Center National de Floristique (CNF) of the University FELIX HOUPHOUËT-BOIGNY (herbarium N°UCJ004623). Tubers are sampled according to harvest locations and designated TKS, TLK and TSD for tubers from Klolékaha, Lélékaha and Sokala Sobara respectively. Arrived at the laboratory, the tubers are cleaned with running water, dried in an oven (50°C) for 24 hours then pulverized and stored in glass bottles.

2. 2 Methods

2.2.1 Physical analysis of *Cyperus esculentus* tubers

TSK, TLK and TSD tubers were characterized by the shape (round, oval or elongated), the size (small, medium or large) and mass. Tuber size was determined by measuring the length (L, cm) and diameter (thickness, T, cm) of 300 tubers from each batch using an electronic digital caliper (Mitutoyo Body, accuracy 0.01mm). When the L/E ratio ≤ 1.24 the tubers are said to be round; oval if L/T < 1.25 and elongated if L/T ≥ 1.45 [16]. Concerning the size, the tubers are said to be small if E ≤ 0.5 cm; medium if 0.5< T ≤ 1.0 cm and large if T>1.0 cm [17]. The mass (M, g) of each tuber was determined using an electronic balance (Adventure Pro, precision 0.001 g).

2.2.2 Fat extraction

The tuber oil was obtained by continuous Soxhlet extraction for 2 h from 15 g of powder mixed with 3 g of anhydrous Na_2SO_4 using hexane as extraction solvent. The hexane was removed after extraction using a rotary evaporator (BÜCHI). The yield (Yield) of fat matter extraction (FM) was calculated according to the formula (1):

Yield (%) = (m0/m1) × 100 (1)
m0 is mass of the extracted fat (g), m1 is mass of the initial vegetable powder

2.2.3 Determination of physical and physicochemical parameters

For each parameter measured, three tests were carried out, and the average of the three tests was taken into account.

- The refractive index (Ir) was determined according to the ISO 6320: 2000 standard at 20° C. with a refractometer (Leica AR 200 Barolworld brand).
- The density (D) was determined by calculating the ratio of the weight of oil and the weight of water taken in the same volumes according to the protocol described by NF ISO 6883.
- The saponification index (Is) was determined according to the method described by Bamba et al., 2015 [18] and standard NF T 60-206 [19].
- The acid index (Ia) was determined according to standard NFV 03-906 [19].
- The peroxide index (Ip) was determined following the protocol described by the AOAC standard, 1981 [20].
- The iodine value (Ii) was determined according to the method described by Bamba, 2016 [21] and the AOAC standard, 1981.[20]
- The Ester Index (Ie) value is equal to the Saponification Index (Is) for pure glycerides and it was calculated based on the analytical data using the formula Ie = Is - Ia.
- The calorific value (CP) was calculated using the following expression:
  CP = 47645-4.187Ii-38.31Is (kJ/kg) [22; 23]

2.2.4 Determination of the content of unsaponifiables

The fat (5 g) was dissolved in a solution (2N) of KOH in ethanol (50 mL). The resulting mixture was boiled and heated for 1 hour under reflux. After cooling, add 100 ml of distilled water. The organic part was extracted with diethyl ether (3×60 mL) then washed with distilled water (5×50 mL) until neutral pH. The organic phase was then dried with $MgSO_4$ anhydrous, filtered and evaporated to dryness under reduced pressure using a rotary evaporator after distillation of the solvent [24]. The residue obtained, constituting the unsaponifiable (Ins) fraction, is weighed and its content is calculated according to the following formula (2):

Ins (%) = (m1/m2) × 100 (2)
Ins is the content of unsaponifiable; m1 is mass of the unsaponifiable fraction (g); m2 is mass of the FM (g).

2.2.5 Preparation of methyl esters (ME) of fatty acids

After extraction of the unsaponifiable, the aqueous phase is treated by 5 mL of HCl (5N). Fatty acids (FA) were extracted with ethyl acetate (3 × 25 mL). The organic fractions were collected and dried over anhydrous $MgSO_4$. The fatty acids were obtained after removal of the solvent using a rotary evaporator [25]. The extraction yield is calculated using the formula (3) below.

FA (%) = (m'1/m'2) × 100 (3)
FA is fatty acid content; m'1 is mass of fatty acids (g); m'2 is mass of FM (g)

To facilitate GC/MS analysis, fatty acids have been methylated to form methyl esters (ME). To do this, 1 mL of hydrochloric methanol (2N) was added to the fatty acids. The whole is brought to the boil and heated for 1 hour on a heating plate with slow stirring at a temperature of 45 to 50°C. After cooling, 20 mL of distilled water was added to the reaction mass, then the EMs were extracted with 25 mL ethyl acetate. After solvent removal using a rotary evaporator, EMs were obtained for analysis.

2.2.6 GC/MS analysis of the unsaponifiable fraction and methylated fatty acids

1 mg of methylated fatty acid and 1 mg of unsaponifiable to be analyzed are dissolved in 2 ml of hexane and 2 ml of a pyridine/ acetic anhydride mixture (50/50, v/v), respectively. The analyzes were carried out on a GC/MS (brand SHIMADZU, model QP2010SE), equipped with a column with a Zebron ZB-5 ms, 20 m long, with an internal diameter (0.18 mm) and a film thickness of the stationary phase (0.18 µm). Helium (He) was the carrier gas at linear velocity (0.9 ml/s). The temperature of the oven (50°C) is then increased in gradient at the rate of 4°C /min up to 250°C and maintained for 15 min. The injector and detector temperatures were set at 250°C and 280°C, respectively. Split mode injection was performed for 30 min at an ionization energy of 70 eV, scan rate (50 scans/s) and acquisition rate (10 000 amu/s). The phytocomponents were identified by comparing their retention time (TR) with the retention time of authentic compounds and the spectral data from the internal library spectrometer.

3. RESULTS AND DISCUSSION

3.1 Physical Characteristics of Tubers of *Cyperus esculentus* (*C. esculentus*)

The mass, length, and thickness of the tubercles of *C. esculentus* were measured and the linear dimensions obtained were used to classify the tubers according to shape and size (Table 1). The results show that the length of the TKS, TLK and TSD tubers does not differ significantly ($p>0.05$) on the other hand their thickness differs markedly ($p<0.05$). Tubers TKS (0.83 ± 0.009 cm) and TLK (0.824 ± 0.007 cm) have almost the same thickness, however TSD tubers are significantly thicker (1.025 ± 0.011cm).

In addition, TKS and TLK tubers are respectively medium and elongated-rounded while those TSD are large and elongated. Regarding the mass, variability is observed, TKS (0.32 ± 0.007 g) has the smallest mass while TLK has the highest mass (0.76 ± 0.007 g). Also note that the mass of the tubers depends on their shape. Indeed, the elongated tubers TSD (0.76 ± 0.007g) and TKS (0.52 ± 0.03g) are heavier than the TLK tubers round (0.32 ± 0.007g). The evolution of the mass of the tubers depends on the thickness, but at equal thickness, the increase in mass is consistent with the increase in length. In addition, the physical parameters of TKS, TLK and TSD tubers (length 1.009-1.453 cm; thickness 0.82-1.025 cm and mass 0.32-0.76 g) are similar to those of Spanish tubers (length 1.0-1.437; thickness 0.821-0.905; mass 0.3-0.51g). However, they are higher than those of Burkina Faso and Nigeria, whose parameters fluctuate respectively between 0.762-0.1203 and 0.649-0.806 cm in length; 0.77-1.119 and 0.645-0.788 cm for the thicknesses; 0.23-0.29 and 0.24-0.3g for the masses [16].

3.2 Fat, fatty Acid and Unsaponifiable Content of *Cyperus esculentus* tubers

The oils extracted from tubers (TKS, TLK and TSD) are golden yellow in color with a pleasant smell and have a content of 23.64 ± 0.25; 24.72 ± 0.05 and 27.66 ± 0.17% respectively (Table 2I). these oil contents are lower than those of some conventional oils such as olive (29-50%) [26], peanut (45-50%) [27], copra and palm kernel (48%) [28,29]. The results show that the tubers TSD contained more FM (27.66±0.17%) than TKS (23.64 ± 0.25%) and TLK (24.72 ± 0.05 %) tubers. The fat content obtained is close to that of the tubers of *C. esculentus* studied by the Agricultural Institute of Turkey (22.8 to 32.8%) [30]. Variability in fat content can be linked to the climatic conditions in which the species evolves [31].

Table 1. Physical parameters of *C. esculentus* tuber

Parameters	Samples		
	TKS	TLK	TSD
Mass (g)	0.52 ± 0.03[a]	0.32 ± 0.007[b]	0.76 ± 0.007[c]
Thickness (T) (cm)	0.83 ± 0.009[a]	0.824 ± 0.007[a]	1.025 ± 0.011[b]
Length (L) (cm)	1.371 ± 0.012[a]	1.009 ± 0.012[a]	1.453 ±0.032[a]
(L/T)	2.56 ± 0.85[a]	1.24 ± 0.017[a]	1.48 ± 0.038[a]
Shape	Elongate Medium	Round Medium	Elongate Wholesale

For each tuber, values in the same row followed by superscripts (a–c) differed significantly (P < 0.05). TKS: tubers from Klolékaha department of Sinematiali; TLK: tubers from Lèlèkaha department of Korhogo; TSD tubers from Sokala Sobara department of Dabakala

Table 2. Contents of fat, fatty acids and unsaponifiables

Content (%)	Samples		
	TKS	TLK	TSD
Fat	23.64 ± 0.25	24.72 ± 0.05	27.66 ± 0.17
Unsaponifiable	1.59 ± 0.01	1.60 ± 0.01	1.60 ± 0.005
fatty acids	95.20 ± 0.4	92.07 ± 0.23	90.41 ± 1.04

TKS: tubers from Klolékaha department of Sinematiali; TLK: tubers from Lèlèkaha department of Korhogo; TSD tubers from Sokala Sobara department of Dabakala

TKS, TLK and TSD tubers showed no significant difference in content between the unsaponifiable fractions (1.59 ± 0.01; 1.6 ± 0.01 and 1.60 ± 0.005 %) and fatty acids (95.20±0.4; 92.07 ± 0.23 and 90.41 ± 1.04%). The average content of unsaponifiable (Ins) obtained in this study (1.56% or 15.6 g/kg) was superior to those of conventional oils such as peanut and palm kernel oil Ins≤ 10g/Kg, palm oil Ins≤12g/Kg, coconut, cotton and soybean Ins≤15g/Kg [32]. Moreover, this average is higher than that of *C. esculentus* tubers from Nigeria (0.50-0.82%) [9] and from Turkey (0.5-0.59%) [33]. Furthermore, the average percentage of unsaponifiable is 0.2 to 2%, as reported in the literature [34]. These high values of unsaponifiable can indicate the presence of large amounts of polyphenols, phytosterols, vitamins or pigments in the tubers, which can give *C. esculentus* oil stability and interesting biological properties.

3.3 Physical and Physico-chemical Characteristics by FM

The physical and physico-chemical parameters of fat (FM) are listed in Table 3. The refractive index is an indicator of oil purity and the degree of fatty acid unsaturation. An oil with a high content of unsaturated fatty acids has the highest refractive index. It also provides information on the degree of oil degradation, increased free fatty acids, reduced the refractive index considerably [34]. For most oils, it ranges from 1.44 to 1.48.

Table 3. Physical and physico - chemical characteristics of *Cyperus esculentus* FM

Parameters	Samples		
	TKS	TLK	TSD
Refractive index (25°C)	1.461 ± 0.001	1.457 ± 0.001	1.453 ± 0.001
Density	0.8 ± 0.002	0.76 ± 0.0012	0.83 ± 0.0011
Acid Value (mg KOH/g)	2.81 ± 0.01	5.61 ± 0.001	10.95 ± 0.001
Peroxide value (m $_{eq}$ of O_2 /Kg)	2 ± 0	2 ± 0	1 ± 0
Iodine index (g of I_2 /100g)	54.64 ± 3.40	62.96 ± 1.59	73.8 6 ± 2.13
Saponification index (mg KOH/g)	208.63 ± 1.54	198.72 ± 4.51	216.78 ± 2.51
Ester number (mg KOH/g)	205.82 ± 1.54	193.11 ± 4.51	205.83 ± 2.51
Calorific value (KJ/Kg)	39422.96 ± 63.94	39768.88 ± 178.01	39058.50 ± 77.61

TKS: tubers from Klolékaha department of Sinematiali; TLK: tubers from Lèlèkaha department of Korhogo; TSD tubers from Sokala Sobara department of Dabakala

The value of the oil refractive index of *C. esculentus* (1.453-1.461) (Table 3) is more or less close to the refractive index of palm oils (1.454-1.456), cottonseed (1.458-1.466), peanut oil (1.460-1.465) [35], but lower than corn (1.458-1.466), sunflower (1.461-1.468) [35] and olive oil (1.469-1.470) [36] and higher than that of coconut oil (1.448-1.450) and palm kernel oil (1.448-1.452) [35]. They are also comparable to the refractive indices of tuber oils of *C. esculentus* from Egypt (1.466) [37]. However, they are lower than those of Ghana (1.47) [11].

The density of an oil is also considered as a physical criterion that allows its quality to be controlled. The values obtained in this study varying from 0.76 to 0.83 are lower than that of most vegetable oils (palm, coconut, cotton, peanut) which varies from 0.891 to 0.926. However, they are close to those of *C. esculentus* oil from Turkey (0.896) [33] and lower than those found for tubers from Ghana (0.912) [11].

Acid number is a measure of the amount of free acids in an oil. This indicator helps to determine the degree of oil change. Thereby, it is an important quality indicator in the production of edible oils [38]. The results obtained show that the acid values of the samples TKS, TLK and TSD range from 2.81 to 10.95 mg KOH/g. TKS tubers had the lowest acid number (2.81 mg KOH/g) and below the recommended range ($Ia < 4$ mg KOH/g) for edible oils [39]. This low acid number indicates a low content of free fatty acids, characterizing the purity and stability at room temperature of the TKS sample [40]. The acid value of the TSD tuber oil (10.95 mg KOH/g) was the highest and far exceeded the standard. However, this value is comparable to that obtained for the black variety (9.12 mg KOH/g) of *C. esculentus* tubers from Nigeria [9]. The acid index of TLK tubers (5.61 mg KOH/g), although higher than the standard, is close to the values of conventional oils: cottonseed ($Ia<7$), palm kernel ($Ia=4.7$) and peanut ($Ia=6$)] [41].

As it concerns the peroxide index, the values varied from 1 to 2 meq of O_2 /Kg. These values were of the same order of magnitude and less than 10 meq of O_2 /Kg of oil found in most conventional oils [42]. This classifies the oils of the TKS, TLK and TSD tubers among the oils indicating an acceptable level of oxidation [43]. These oils can therefore be used in gastronomy [41].

As for the saponification index, the results show that the values varied from 198.72 ± 4.51 to 216.78 ± 2.51 mg KOH/g. The lowest values in the range of 198.72 ± 4.51 to 208.63 ± 1.54 mg KOH/g corresponded to TKS and TLK tuber oils, and the highest value (216.78 ± 2. 51 mg KOH/g) to TSD tuber oil. These values are slightly higher than those of *C. esculentus* tuber oil from Africa: 180.30 mg KOH/g (Nigeria; [9]), 192.88 mg KOH/g (Egypt; [8]) and 180.24 mg KOH/g (Ghana, [11]) and from Europe 164.76 (Turkey, [33]). Because, according to Mohammad, 2011 [38] oils with a high saponification index would contain a high proportion of lower fatty acids. However, the saponification indices of oils presented in our study were much lower than those of coconut (248-265) and palm kernel (230-254) oils commonly used in soap making [32]. Therefore, TKS, TKL and TSD oils are not recommended for soap making. Nevertheless, the saponification index of TKL (198.72 ± 4.51 mg of KOH/g) approaches that of conventional oils such as soybean (189-195), peanut (187-196) and cottonseed (189-198) [42]. While the saponification indices of TKS (208.63 ± 1.54 mg KOH/g) and TSD (216.78 ± 2.51 mg KOH/g) were comparable to that of palm oil (190-209).

The iodine value of the oils studied varied from one collection site to another. The highest value (73.86 ± 2.13 g of I_2 /100g) was observed in TSD tubers, and the lowest value was observed in tubers in TKS tubers (54.64 ± 3.40 g of I_2 /100 g of oil). The iodine numbers of TKS, TLK and TSD oils between 54.64 ± 3.40 and 73.86 ± 2.13 g of I_2/100g of oil are close to the iodine numbers of the oil of *C. esculentus* from Nigeria (57.30-83.3 g of I_2/100g of oil) rich in monounsaturated fatty acids [9; 44]. So, these oils would be rich in monounsaturated fatty acids. The iodine value is the most useful constant because it highlights the important division of vegetable oils into drying, semi-drying, and non-drying oils. Thus, the oil is said to be non-drying if $Ii<95$; semi -drying if $95 \leq Ii < 130$ and drying if $130 \leq Ii < 200$ [45]. With regard to these values, the oils of the TKS, TLK and TSD tubers are non-drying.

Whatever the sample (TKS, TLK and TSD), the ester indices were of the same order and all slightly lower than their saponification index with the exception of TSD (Table 3). This means that the TKS and TLK samples contained a moderate amount of free acids.

The calorific value of oils from TKS, TLK and TSD tubers (39422.96, 39768.88 and 39058.50

KJ/Kg, respectively) is more than 3500 KJ/Kg. This would mean that these oils could be used as a bio-lubricant for engines.

3.4 Fatty Acid Composition of *Cyperus esculentus* oil

The analysis of fatty acid and unsaponifiable composition is one of the important ways to determine the nutritional value of edible vegetable oils. Fatty acids, especially unsaturated and polyunsaturated, are of great importance for the normal functioning of the human body [13,46]. Acids such as oleic, linoleic, linolenic belong to the group of biologically active vitamin like compounds. Many unsaturated fatty acids are essential, i.e. are not synthesized in the human body and must therefore be provided by food. Currently, many methods are used for the analysis of fatty acids, the most common of which is GC/MS [47,48,49]. Thus, the fatty acid composition of TKS, TLK and TSD tuber oils determined by GC/MS is listed in Table 4. This analysis revealed the presence of several saturated and unsaturated fatty acids, mainly monounsaturated fatty acids whose values are 61.74; 92.14 and 86.97% respectively for TKS, TLK and TSD. TLK oil contained the highest value (92.14%), while TKS oil (61.74 %) had the lowest. The main unsaturated fatty acids identified were oleic acid, petroselinic acid, linoleic acid, and 2,6-di-O-palmitoyl L-ascorbic acid. Oleic acid is the most abundant fatty acid with a content of 51.04 to 82.83%. This is consistent with literature data that oils from the tubers of *C. esculentus* from different countries contain more oleic acid from 50.85 to 77.20% [8,9,11,50,51]. This fatty acid was found in all the oils with the highest values observed in those of the tubers harvested at Lèlèkaha (TLK: 82.83%) and Sokala Sobara (TSD: 77.86%) and the lowest values observed in that of Klolékaha (TKS: 51.04%). However, petroselinic acid was found with a high content in TKS (10.15%) and a low content in TLK (0.23%) and then in TDS (0.29%). This compound is known for its anti-inflammatory [52] and antimicrobial [53,54] activity in various food, cosmetic and pharmaceutical formulations. Also, in the tubers of *C. esculentus*, 2,6-di-O-palmitoyl-L-ascorbic acid was found for the first time in significant quantities (8.84% in TLK and 8.58% in TSD). Its presence can improve the quality of the oil, since vitamin C esters (palmitate or stearate) are powerful antioxidants, often used as food additives, and also as a source of vitamin C. They confer certain functional properties in cosmetology [55] and in pharmacy [56] to stabilize oil-in-water emulsions. In addition, the data in Table 4 show the absence of linoleic acid in TLK while this was observed in TKS (8.96%) and TSD (4.47%), which is comparable to literature data (4-9.35%) [8,37,44].

Moreover, saturated fatty acids were identified with contents ranging from 7.86 to 20.26% with the highest value observed in TKS. The main saturated fatty acids identified were palmitic, stearic and pentadecyl. Palmitic and stearic acids were found in the oil of all samples, but pentadecyl acid (1.08%) was only found in TKS. Palmitic (14.87%) and stearic (3.33%) acid contents in TKS were the highest. They are approximately equal to 12.96-15.4% reported in the literature for palmitic acid [8,9,37] and 3.0-3.3% for stearic acid [44]. On the other hand, their content of 6.29-6.60% (palmitic acid) and 1-1.55% (stearic acid) were very low in the TLK and TSD samples. The variability in fatty acid composition of oils from one harvest location to another may be due in part to climatic or environmental factors [13].

Thus, the content in oleic acid (51.04-82.83%) found in this study shows similarity with olive oil [13]. This fatty acid will prevent the deposition of cholesterol in the vessels (antiatherogenic) and lower the level of lipids in the blood (hypocholesterolemia). We also note the presence of linoleic acid in TKS and TSD, an essential fatty acid of the ω-6 types, which only enters the body with food and plays a structural role as the main component of membrane phospholipids [2]. From the above, it can be said that the presence of the polyunsaturated acids (oleic, linoleic and petroselinic acid) in the oils of *C. esculentus* is indicative of its high nutritional potential and, therefore, of its benefits for human health.

3.5 Composition of *Cyperus esculentus* oil Unsaponifiable

According to El-Naggar (2016) [8], unsaponifiable play a decisive role in the stability of oils. GC/MS analysis of unsaponifiable fractions yielded chromatograms (Fig. 1,2 and 3) representing the spectra of identified compounds in TKS, TLK and TSD tuber oil. The data in Table 5, for their part, show the retention times and the compounds identified for these unsaponifiable fractions. The numbered peaks correspond to the compounds identified in the samples.

Table 4. Results of GC/MS fatty acid analysis of *Cyperus esculentus* tuber oil

Compositions	Samples					
	MG TKS		MG TLK		MG TSD	
	TR (min)	%	TR (min)	%	TR (min)	%
Butyl acetate					2.891	0.36
myristic acid , C14:0	9,532	0.13			9,538	0.05
palmitoleic acid , C16:1 ω-6	10,427	0.29	10,432	0.11	10.431	0.11
palmitic acid , C16:0	10.533	14.87	10.534	6.60	10,529	6.29
pentadecyl acid , C15:0	10.695	1.08				
2,6-Di-O-palmitoyl of L-ascorbic acid			10.762	8.84	10.726	8.58
(Z)-Heptadec-10-enoic acid	10.879	0.07			10.883	0.04
margaric acid , C17:0	10.974	0.08	10.987	0.04		
14-Methylhexadecanoic acid					10.983	0.03
linoleic acid , C18:2 ω-6	11,299	8.96			11.301	4.47
stearic acid C18:0	11.423	3.33	11.428	1.00	11.424	1.55
oleic acid , C18:1 ω-9	11.354	51.04	11.352	82.83	11.570	77.86
nonadecylic acid , C19:0	11.696	0.30				
godoic acid, C20:1, ω- 9	12.145	0.19	12.159	0.07		
18-methylnonadecanoic acid	12.238	0.42	12.247	0.13		
paullinic acid, C20:1, ω- 7					12.154	0.09
behenic acid , C22:0	13.062	0.05				
Cis -vaccenic, C18:1, ω- 7			12.313	0.06		
petroselinic acid, C18:1, ω- 12	11.518	10.15	12.393	0.23	12.393	0.29
arachidic acid, C:20 :0			12.507	0.09	12.50	0.04
lignocérique,acid , C24:0					14.145	0.05
Saturated fatty acids		**20.26**		**7.86**		**8.37**
Monounsaturated fatty acids		**61.74**		**92.14**		**86.97**
Polyunsaturated fatty acids		**8.96**		**0**		**4.47**
Total unsaturated fatty acid		**70.7**		**92.14**		**91.44**

TKS: tubers from Klolékaha department of Sinematiali ; TLK: tubers from Lèlèkaha department of Korhogo; TSD tubers from Sokala Sobara department of Dabakala

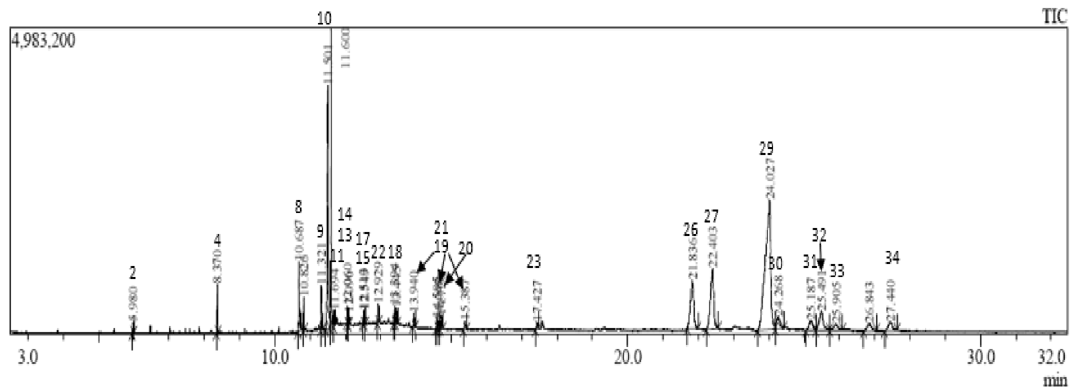

Fig. 1. GC chromatogram of the unsaponifiable fraction TKS

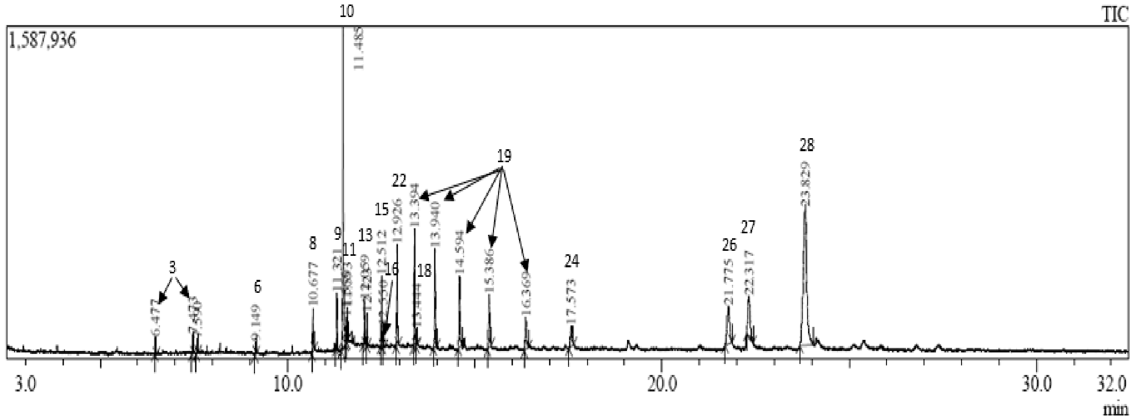

Fig. 2. GC chromatogram of the unsaponifiable fraction TLK

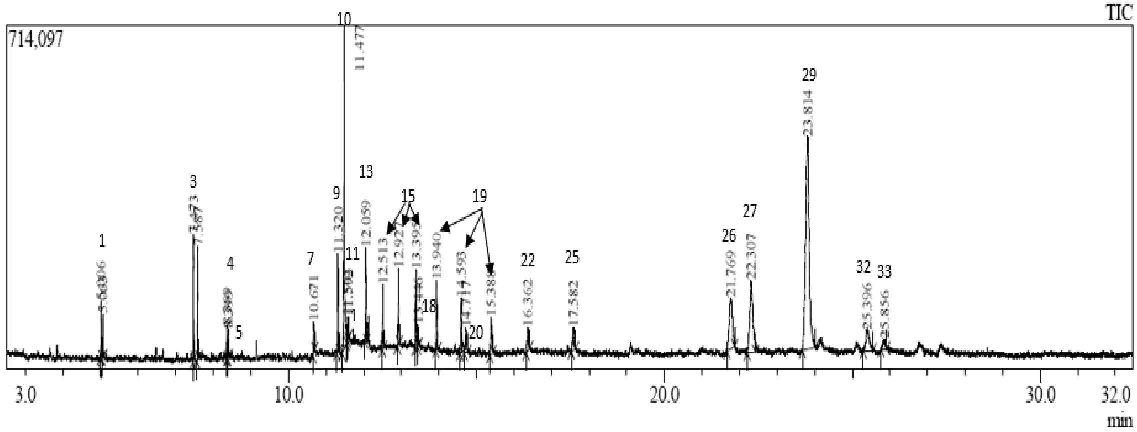

Fig. 3. GC chromatogram of the unsaponifiable fraction TDS

Most of the components identified are hydrocarbons (40.67; 27.7 and 50.8% in TKS, TLK and TSD, respectively). Stigmastane-3,5-diene [29], the main identified hydrocarbon (37.12 and 34.24% for TKS and TSD) is formed by dehydration of β-sitosterol [57]. Indeed, this dehydration may be due to the high temperatures during GC analysis and the reducing environment in the chromatographic system. It should be noted that this high hydrocarbon content is higher than that of tuber oil from Nigeria (20.57%) [50], but lower than that of *C. esculentus* from Egypt (77.795 %) [58].

In addition, the presence of phytosterols to also manifests in the unsaponifiable fractions studied (30.68; 44.03 and 22.30% in TKS, TLK and TSD, respectively), in which β-sitosterol acetate (28) predominates in TLK (31.33%) and (3β)-3-acetyloxycholest-5-en-24-one (26) present in high rate (8.18, 6.68 and 7.60% in TKS, TLK and TSD, respectively). Only the TKS sample contained tocopherol (0.48%) (23) and terpene esters (0.23%). It should be noted that tocopherol (vitamin E) also plays an important role in maintaining good health of human organism [8]. The vitamin E content recorded in this study is higher than the content (0.016-0.018%) found for tubers from Turkey [33]. On the other hand, it is lower than the content reported (5.39%) for tubers from Nigeria [50]. According to the work of Tao et al. (2012) [59], the presence of β-sitosterol acetate may confer *C. esculentus* oil from bacterial properties. As for the phenolic compounds, they were found in notable quantity (1.8; 1.38 and 2.92% in TKS, TLK and TSD, respectively), which is higher than those of *C. esculentus* from Turkey (0.20-0.27%) [33].

Table 5. Results of GC/MS analysis of the unsaponifiable fraction of *Cyperus esculentus* tuber oil

Compositions		Samples					
		Ins TKS		Ins TLK		Ins TSD	
		TR (min)	%	TR (min)	%	TR (min)	%
1	(E)-4-oxopen-2-en-2-yl acetate					5.006	2.28
2	2-Ethylhexyl acetate	5.98	0.24				
3	3-acetylpentane-2,4-dione			6.477	1.53	7.473	4.75
4	(2,6-bis (1,1- dimethylethyl)- 4-methyl) phenol (polyphenol)	8.370	1.02			8.370	0.65
5	4a,8-dimethyl-2-(prop-1-en-2- yl)-1,2,3,4,4a,5,6,8a-octahydronaphthalene (hydrocarbon)					8.399	0.64
6	3,8-Dimethyl-4-(1-methylethylidene)-2,4,6,7,8,8a-hexahydro-5(1H)-azulenone (Aromadendrane sesquiterpenoids)			9.149	0.43		
7	2,6-Di-O-palmitoyl of L-ascorbic acid					10.671	0.76
8	Hexadecanoic acid (C16:0)	10.687	3.00	10.677	1.56		
9	2-O-acetoxytetradecane	11.321	1.05	11.321	1.76	11.320	2.11
10	(Z) octadec-9-enoic acid	11.501	20.47	11.485	15.28	11.477	10.44
11	Octadecanoic acid (C18:0)	11.694	0.52	11.565	2.13	11.564	0.86
12	oleic acid (C18:1 ω-9)					11.592	0.54
13	8-Hydroxy-2-methyl-7-O-triflioroacetyloctadecane (polyphenol)	12.060	0.37	12.059	1.38	12.059	2.27
14	Farnesyl acetate (terpene)	12.096	0.23				
15	Eicosane (hydrocarbon)	12.513	0.32	12.123	3.06	12.513	5.83
16	Octadecanoic acid (C18:0))			12.550	0.70		
17	Docosanoic acid (behenic acid, C 22:0)	12.549	0.34				
18	Eicosanoic acid (C20:0)	13.445	0.52	13.444	0.72	13.446	0.65
19	Hexatriacontane (hydrocarbon)	13.394	2.05	13.394	21.27	13,940	7.32
20	4,4'-thiobis [2-(1,1-dimethyl) -5-methyl] phenol (polypheneol)	14.648	0.41				
21	Squalene (terpene-hydrocarbon)	14.720	0.61			14.717	1.26
22	Tetratetracontane (hydrocarbon)	12.929	0.57	12.926	3.57	16.362	1.51
23	β-tocopherol (Vitamin E)	17.427	0.48				
24	1-Bromotriacontane ($C_{30}H_{61}Br$)			17.573	2.58		
25	1-Octadecanesulphonyl chloride ($C_{18}H_{37}ClO_2S$)					17.582	1.59
26	(3β)-3-acetyloxycholest-5-en-24-one (phytosterol)	21.836	8.18	21.775	6.68	21.769	7.60
27	(3β, 22Z) -3-O-acetylstigmasta-5,22-diene (phytosterol)	22.403	9.90	22.317	6.02	22.307	9.85
28	(3β)-3-O-acetylstigmas-5-ene (β-Sitosterol acetate) (phytosterol)			23.829	31.33		
29	Stigmastan-3,5-diene (hydrocarbon)	24.027	37.12			23.814	34.24
30	3-(acetyloxy)-17-hydroxypregn-5-en-20-one (phytosterol)	24.268	2.07				
31	(3β)-9,19-cyclolanost-24-en-3-ol (phytosterol)	25.187	3.36				
32	(3β, 5α)-3-O-acetylstigmast-7-ene (phytosterol)	25.491	3.91			25.596	3.72

Compositions		Samples					
		Ins TKS		Ins TLK		Ins TSD	
		TR (min)	%	TR (min)	%	TR (min)	%
33	(3β, 5α) -3-O-acetylstigmasta-7,16,25-triene (phytosterol)	25.905	1.29			25.856	1.13
34	(6α, 16α) -6-methyl-16-(1-methylethyl) pregn - 4 -ene-3,20-dione (phytosterol)	27.440	1.97				
	Totals		**100**		**100**		**100**
	Tocopherol		**0.48**				
	Ester terpenes		**0.23**				
	Hydrocarbons		**40.67**		**27.7**		**50.8**
	Phenolic compounds		**1.8**		**1.38**		**2.92**
	Phytosterols		**30.68**		**44.03**		**22.30**

TKS: tubers from Klolékaha department of Sinematiali; TLK: tubers from Lèlèkaha department of Korhogo; TSD tubers from Sokala Sobara department of Dabakala

4. CONCLUSION

During this study, the physico-chemical characteristics, the composition of fatty acids and unsaponifiable of the oil of tubers of *Cyperus esculentus* from Côte d'Ivoire have been determined. The values of the physico-chemical parameters show that this oil as a whole has physical and physico-chemical properties that meet the standards of edible oils. It can be an excellent source of oil nutritious due to its richness in omega-9, unsaturated fatty acids (oleic and linoleic). Its consumption may reduce the risk of cardiovascular disease by lowering total cholesterol and LDL ("bad" cholesterol) levels in the blood.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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