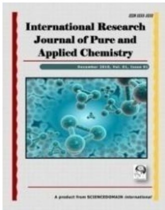

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Oil Extraction from Edible Oilseeds; *Irvingia gabonensis, Citrullus lanatus and Telferia occidentalis* and Evaluation in Metallic Soap Preparation

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Author's contributions

This work was carried out in collaboration among all authors. Authors EAE, AUA and IUU designed the study and wrote the protocol. Author EAE preformed the statistical analysis, managed the literature search and wrote the first draft of the manuscript with constructive assistance from author UJE. All authors read and approved the final manuscript.

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ABSTRACT

The extraction of oils from *Irvingia gabonensis, Citrullus lanatus* and *Telferia occidentalis* in organic solvents was carried out. Oil compositional analysis was done with Gas Chromatography (GC), results revealed varying compositions of fatty acids as major constituents of the produced oils. Oil of *Irvingia gabonensis* was mainly saturated whereas those of *Citrullus lanatus* and *Telferia occidentalis* were mainly unsaturated. The produced oils were applied in the production of metallic soaps. Oils of *Citrullus lanalus* and *Telferia occidentalis* showed high affinity to copper over zinc and

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nickel as indicated in high metallic content of the metal in the soap. *Irvingia gabonensis* seeds oil showed low affinity for metallic soap production which could be due to high saturation of fatty acid. Comprehensive and comparable analysis of the physiochemical properties of the oils from individual seeds showed different characteristics which is indicative of potential applications. From results of analysis extracted oils of *Citrullus lanatus* and *Telferia occidentalis* seeds have potentials for metallic soap preparation while *Irvingia gabonensis* have potential for other applications.

Keywords: Oilseeds; extraction; physiochemical properties; metallic soap.

1. INTRODUCTION

The search for new resources for production of goods useful to man continues to grow globally. Nigeria with its growing population is in dire need of other resources base to diversity its economy from overdependence on crude oil derived products [1]. Agriculture and agro-based produce offer promising and useful products to complement and/or replace most of the petrochemicals derived from petroleum. Oil seeds are one of such agro-based products which find diverse applications. The production and use of vegetable oils (lipids) in different applications has necessitated the exploitation of mostly, seeds which are not consumed directly as food for alternative uses [2,3]. Lipids are obtained from plants and animals, of which sources have claimed abundance of 68 and 32 % respectively [4,5]. Like most organic materials, oils and fats are made up of three elements; carbon, hydrogen and oxygen. These elements combine together to form chains known as fatty acids, three of these chains then join together to form a molecule known as a triacylglycerol, the basis of all oils and fats. Oils and fats vary in both their appearance and functionality due to differences in the types of fatty acid chain which join together to form the triglyceride molecule [6,7]. A number of oils from oilseeds including corn, sunflower, palm kernel, peanut, olive, soyabean, rapeseed, coconut, cashew, cotton and have been characterized and used for various application which include cooking, frying, hydrogenation (margarine production), baking fats, couverture, confectionery, ice cream, plastics, cosmetics, soap making, pharmaceuticals and for chemical and technical industrial applications as vanish and lacquer [8]. In order to exploit the unconventional seeds for their oil production potentials and new applications, edible seeds: bush mango - *Irvingia gabonensis,* fluted pumkin- *Telferia occidentalis* and melon- *Citrullus lanatus* seeds (Fig. 1) were utilizes for oil extraction and subsequent evaluation for metallic soap production.

2. MATERIALS AND METHODS

The oilseeds were purchased from a local market in Uyo, Akwa Ibom State, Nigeria. The seeds were shelled and selected, sun dried and then oven dried at 45ºC to a constant weight and blended into fine powder with a mechanical blender. The drying temperature was chosen to regulate moisture content; higher temperatures may result to burnt seeds and higher free fatty acid levels. The milled samples were packed in airtight plastic containers and stored in a refrigerator at 4ºC prior to analysis. All reagents including n-hexane, petroleum ether, Nickel nitrate, $Ni(NO_3)_2.6H_2O$; Copper sulphate, $CuSO_4.5H_2O$; Zinc sulphate, $ZnSO_4.7H_2O$; Aluminium nitrate $Al(NO_3)_3.9H_2O$; Calcium chloride, $CaCl_2$ and Magnesium sulphate, $MgSO_4.5H_2O$ were analytical grade products of British Drug House, England.

2.1 Oil Extraction

Oils were extracted from the seeds using different solvents according to literature [9]. n-Hexane was used for the extraction of *Telferia occidentalis* seeds oil using soxhlet extraction method at 80ºC, *Citrullus lanatus* and *Irvingia gabonensis* seeds oils were extracted using the same method with petroleum ether (40-60ºC) as the solvent [10]. Briefly, measured quantities of ground seeds samples were wrapped with filter paper and carefully placed inside the extraction thimble connected to a round bottom flask.

The soxhlet extractor was then placed in the heating mantle and fitted with a condenser connected to tap water supply (inlet) which was properly clamped to a retort stand. The apparatus was then heated with the mantle at 80ºC and the extraction was allowed to continue for six hours during which time the sample refluxed until extraction was completed. After this time, the sample residue was removed from the extraction thimble and the extracted oil was poured in to an open beaker and heated in an oven at 100ºC for 24 hours to evaporate any water in the matrix of the oil. The obtained oil was degummed by treatment with 0.2% of phosphoric acid (H_3PO_4). The mixture was heated in a water-bath at 60ºC for 15 minutes. The gum was separated by centrifugation operated at a speed of 3000 rpm for 20 minutes and the oil was decanted. The oil was then filtered, neutralized with caustic soda to remove the residual acid and tested with litmus paper followed by washing out of soap and drying [11,12].

2.2 Preparation of Metallic Soap

Metallic soaps of the seeds oils were prepared following steps shown in Fig. 2. *Telferia occidentalis* and *Citrullus lanatus* seeds oils were used for nickel, copper and zinc soaps while *Irvingia gabonensis* seeds oil was applied for production of aluminum, calcium and magnesium soaps. The general equations of reactions for the formation of the soaps are presented below.

$$\underset{\text{Fatty acid}}{RCOOH_{(aq)}} + \underset{\text{Sodium hydroxide}}{NaOH_{(aq)}} \longrightarrow \underset{\text{Sodium salt of fatty acid}}{RCOONa_{(aq)}} + \underset{\text{water}}{H_2O_{(l)}}$$

$$\underset{\text{Sodium salt of fatty acid}}{2RCOONa_{(aq)}} + \underset{\text{Metal salt solution}}{M^{2+}2X^{-}_{(aq)}} \longrightarrow \underset{\text{Metallic soap (insoluble)}}{(RCOO^{-})_2M^{2+}_{(s)}} + \underset{\text{water soluble salt}}{2Na^{+}X^{-}_{(aq)}}$$

Citrullus lanatus seeds   *Irivingia gabonensis* seeds   *Telferia occidentallis* seeds

Fig. 1. Photographs of oilseeds used in the present study

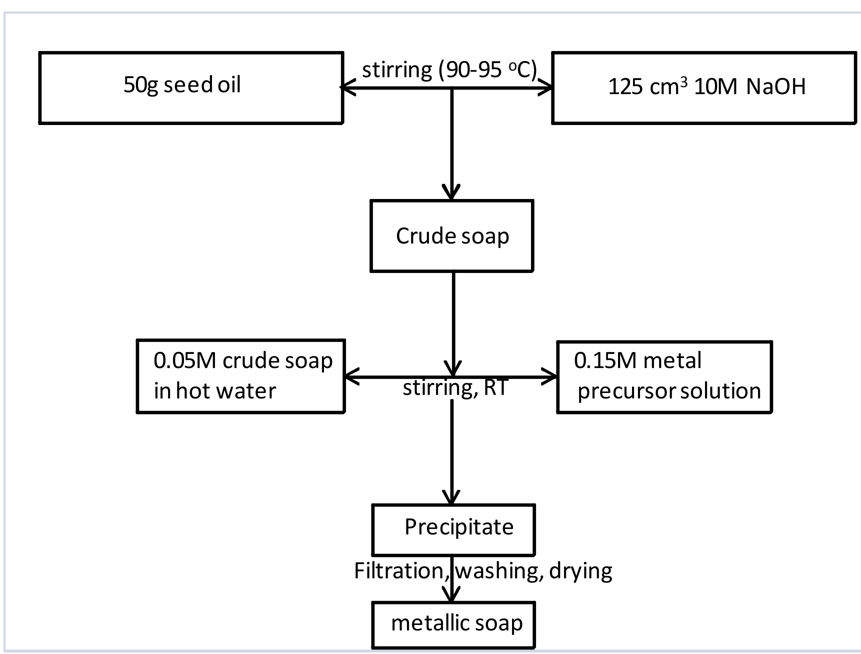

Fig. 2. Schematic diagram of metallic soap preparation

2.3 Characterization

The fatty acid composition was characterized using Gas chromatograph with flame ionization detector (GC-FID), Hewlett Packard 6890 series GC system equipped with a HP-5 (30m × 0.22 mm i.d., 5% dimethylsiloxane; film thickness, 0.25µm capillary column using helium as a carrier gas (1mL/min flow rate) at injection and detection temperatures, 250ºC. The column was maintained at an initial temperature of 129ºC for 5min and then programmed at 2ºC/min to a final temperature of 220ºC and was maintained for 18 min. Fatty acids were identified by retention time relative to internal standard. The quantification of fatty acid methyl esters (FAMEs) composition was realized by integration of the FID peak area with the correction factor (internal normalization method). The extracted oils were characterized for their physicochemical properties following methods in literature [9,13]. Metal contents in the as prepared soaps were measured using atomic absorption Spectrophotometer (AAS).

3. RESULTS AND DISCUSSION

3.1 Physiochemical Properties of Extracted Oils

The physiochemical properties of seeds oils are summarized in Table 1. The total lipid content of *Telferia occidentalis, Citrullus lanatus* and *Irvingia gabonensis* seeds are 47.21%, 57.26% and 67.60% respectively. From the results *Irvingia gabonensis* has the highest lipid content followed by *Citrullus lanatus* and *Telferia occidentalis*. Iodine value of *Telferia occidentalis* and *Citrullus lanatus* are 42.9 and 114.94 respectively, while that of *Irvingia gabonensis* was 7.11. The results indicate that *Citrullus lanatus* oil is semi-drying oil consisting predominately polyunsaturated fatty acids mainly oleic and linoleic fatty acids while *Irvingia gabonensis* and *Telferia occidentalis* are non-drying oil with saturated fatty acids mainly myristic and lauric fatty acids. The class of oils whose iodine value is between 100-150 possesses the property of absorbing oxygen on exposure to the atmosphere; though do not do so sufficiently to qualify them as drying oils. They become thicken and remain sticky but do not form a hard dry film. They find possible applications in the production of paints, soap, and margarine [8]. The saponification value of *Citrullus lanatus* oil is 220.19; that of *Telferia occidentalis* oil is 117.8 and *Irvingia gabonensis* oil is 221.7. ANOVA analysis ($p < 0.05$) shows significant difference in values [3]. Saponification value is an indication of the average molecular weight of oil. A high saponification value implies greater proportion of fatty acids of low molecular weight. Therefore, these results indicate that all the oils contained higher proportions of low molecular weight fatty acids. The values agree reasonably with the saponification values of olive oil (185 – 196), soya bean oil (193), and linseed oil (193 – 195) mg KOH/g oil [14,15].

C. lanatus, *T. occidentalis* and *I. gabonensis* acid values are 6.09, 1.70 and 5.49 (mg/kg) respectively. The acid value is a measure of the free fatty acid group present and is used as an indicator for edibility of oil and suitability for use in the paint industry. A low acidity value of oil characterizes rather stable oil [16,17]. The acid values of the seeds oils are in conformity with the specification of edible oil 15 mg KOH/g oil [18]. Oils with lower values of acidity containing (1-7% of oleic acid), are more acceptable for edible applications, hence these oils can be certify fit for consumption. The acids that are usually formed include free fatty acid, acid phosphates and amino acids. Free fatty acids are formed at a faster rate than the other types of acids. The low acid values also indicate that the oils could be stored for a long time without deterioration. The free fatty acid values are 4.51 %, 1.30 %, and 2.76% for *C. lanatus*, *T. occidentalis* and *I. gabonensis* respectively. A high free fatty acid (FFA) value is associated with a high deterioration rate of oil and thus resulting in the development of objectionable flavor and odor. In the present study, the values are below the 5.00% FFA content recommended as the maximum for non-rancid oil [19], which implies that the oils in the present work are not rancid and oil could be stored for a longer time without being deteriorated [7]. The peroxide values of *C. lanatus*, *T. occidentalis* and *I. gabonensis* seed oils are 12.00, 0.01, and 1.20 meq/kg respectively. This index measures the hydro-peroxide and the primary oxidation products of the oil which contains mostly polyunsaturated acids and easily undergoes oxidation [12]. It also associates with oxidative rancidity. Oxidative rancidity is the addition of oxygen across the double bonds in unsaturated fatty acids in the presence of enzyme or certain chemical compounds. The odor and flavor associated with rancidity are due to liberation of short chain carboxylic acids [20,21]. High peroxide values are associated with higher rate of rancidity whereas low peroxide values of the oils indicate that they are less liable to oxidative rancidity at room temperature [22,23]. Variation of peroxide values in this study could therefore be due to difference in the number of unsaturated fatty acid content, since rate of autoxidation of fats and oils increases with increasing level of unsaturation and hence, *T. occidentalis* and *I. gabonensis* seeds oils are more stable to oxidation. All seeds oils are relatively acidic as reflected in their pH values. The specific gravities of the oils are 0.91, 0.9 and 0.89 for *C. lanatus*, *T. occidentalis* and *I. gabonensis* seeds oils respectively. Therefore the three seeds oils are slightly less dense than water and their products are insoluble in cool water and other polar solvents (Table 4).

The refractive index for *C. lanatus*, *T. occidentalis* and *I. gabonensis* are 1.35, 1.42 and 1.24 respectively. Refractive index has been shown as one of the most important aids for classifying fatty oils in terms of its purity and application. It is closely related to the nature of the product (i.e. molecular weight and degree of unsaturation). The refractive index of *C. lanatus* falls within the range for oils that can be used in paint production [9,24]. The colour of oil is used in judging quality and in determining the degree of bleaching of the oil, the darker the colour, the poorer the quality [25,26].

3.2 Fatty Acids Composition of the Oils

Nine fatty acids were investigated and results are presented (Fig. 3). In *Irvingia gabonensis* oil, eight different types were detected and four are saturated which dominant. The distribution of the fatty acids is shown, with 93.2% saturated and 6.8% unsaturated types. The unsaturated consists of about 92.6% monounsaturated fatty acids. The most abundant fatty acid is myristic acid ($C_{14:0}$; 54.39%) followed by Lauric acid ($C_{12:0}$; 36.83%) and unsaturated acid myristoleic acid ($C_{14:1}$; 4.93%). This result is in agreement with previous reports; [1] which authors found myristic acid ($C_{14:0}$; 52%) and lauric acid ($C_{12:0}$; 38%), [16], myristic acid ($C_{14:0}$) ranging (50.92-53.71%) and lauric acid ($C_{12:0}$) ranging 36.60-39.37%) and Mackey and Ingle [27] reported myristic acid ($_{C14:0}$; 39.6%) and lauric acid ($C_{12:0}$; 52.4%) for the same seed. The other two seeds oils *C. lanatus* and *T. occidentalis* comprise five fatty acids: palmitic, stearic, oleic, linoleic, and linolenic acids. The fatty acid composition of the *C. lanatus* is in close agreement with values obtained in previous studies [1,9,22]. Of the five fatty acids, linoleic acid is the most abundant (62.14) %. The total saturated and unsaturated fatty acids contents of the *C. lanatus* seeds oil are 18.9 and 81.1 % respectively, with palmitic acid (10.57 %) the predominant saturated fatty acid. *T. occidentalis* oil has five main fatty acids namely: palmitic (C_{16}), stearic (C_{18}), oleic ($C_{18:1}$n-9), linoleic ($C_{18:2}$n-6) and linolenic ($C_{18:3}$n-3) acids, the most abundant being oleic acid (41.08%) and the least being linolenic acid (0.14%). The fatty acid profile is as follows: $C_{18:1}$> $C_{18:2}$> C_{16}> C_{18}> $C_{18:3}$. The major saturated fatty acids in the oils were palmitic and stearic acids and the main unsaturated fatty acids were oleic and linoleic acids. The Fatty acid composition shows that the oils of *C. lanatus* and *T. occidentalis* seeds are rich in polyunsaturated fatty acids. The distribution is comparable with other seeds from literature [28] (Table 2). It is reported that polyunsaturated fatty acids have stimulative effects on the cognitive function in mammals and are major components of the membranes neurales; they improve vision, neurotransmission and the faculty of learning in children [29]. Therefore, children who consume the seeds of *C. lanatus* could boast their retentive memory capacity. Though linolenic acid is an omega-3 fatty acid with positive health effects, it easily oxidises and is undesirable in edible oils because of the off-flavors and potentially harmful oxidation products formed [30,31]. Warner and Gupta [30], showed that decrease in linolenic acid from 2 to 0.8% in oils improved flavor quality and oxidative stability of fried foods. According to the authors, for oils to be very good for frying, its linolenic acid level should be less that 1%.This was obtained for *T. occidentalis* seed oil in this study. The linolenic acid level makes it good oil for the fight against cardiovascular illnesses [1]. The studied oils samples contain oleic and linoleic acids, and can be classified in the oleic-linoleic acid group. Linoleic acid is one of the most important polyunsaturated fatty acids in human food because of its prevention of distinct heart vascular diseases [2]. The Fatty acid composition suggests that *T. occidentalis* seed oil can be good for table, cooking and frying oils and for making mayonnaise [24,32]. In comparison with other oils (Table 2) it is noted that, *Irvingia gabonensis* seeds oil is more saturated than conventional oils which are a major constituent of food frying oil. The unsaturated fatty acid to saturated fatty acid ratio of 0.07 is lower than butter and unsaturated soft margarine and thus convenient for use in confectionaries [16].

3.3 Characterization of Prepared Soaps

Tables 3 and 4 show the characteristic properties of the prepared metallic soaps. Oils of *C. lanatus* and *T. occidentalis* seeds were applied for the preparation of nickel, copper and zinc soaps whereas *I. gabonensis* seeds oil was evaluated for aluminum, magnesium and calcium soaps. The pH of nickel, copper, zinc and aluminum soaps are slightly acid whereas those of calcium and magnesium are basic. The yields of *T. occidentalis* seeds oil soaps are 43.85% for copper, 45.50% for zinc and 49.28% for nickel and 42, 51 and 53% for nickel, copper and zinc respectively with *C. lanatus* seeds oil. However, the yields of soap produced with *I. gabonensis* seeds oil (not reported) are poor compared with the other oils. This may be due to high saturation of the oil of the later seeds oil. Copper shows highest affinity for formation of soap with the prepared oils over other metals as evidenced with a high concentration of the metals in the final soap (14.21 and 21.57% copper content) for *C. lanatus* and *T. occidentalis* seed oils respectively.

Table 1. Physiochemical properties of the three seeds oil: *Citrullus lanatus, Telferia occidentalis and Irvingia gabonensis* seed oils

Test	Unit	*C. lanatus*	*T. occidentalis*	*I. gabonensis*
Specific gravity (27 °C)	Kg/m^3	0.9129	0.911	0.895
Refractive index (27 °C)		1.35	1.42	1.25
Moisture index	%	6.30	6.40	5.44
pH		4.45	5.00	4.43
Colour		Pale yellow	Pale yellow	Light yellow
Saponification value	mg KOH/g oil	220.19	117.80	221.80
Acid value	mg KOH/g oil	6.09	1.70	5.49
Free fatty acid	mg KOH/g oil	4.51	1.30	2.76
Iodine value	gI_2/100g	114.94	42.9	7.11
Unsaponification matter	%	4.5	4.4	0.12
Peroxide value	meg/kg	12.0	2.0	1.2
Lipid content	%	57.26	47.21	67.60
State at room temperature		Liquid	Liquid	Solid

Values are means of 3 determinations

Table 2. Literature comparison of types of fatty acids from seeds oils

Oil seeds	Oil yield (%)	Class of oil/fats		
		Monounsaturated	Polyunsaturated	Saturated
Sunflower	40-45	22	65	13
Soya bean	18-20	24	61	15
Rape seed	40	58	33	9
Palm kernel	45	18	2	80
Peanut	40-50	48	34	18
Coconut	65	8	1	91
Corn	30-40	25	62	13
Olive	-	72	16	12
Canola	-	62	32	6
Grape	-	17	71	12
Cotton seed	-	19	52	28
Safflower	-	13	17	10
**C. lanatus*	57.26	13.65	67.43	18.90
**T. occidentalis*	47.21	41.08	32.18	26.74
**I. gabonensis*	67.60	6.27	0.54	93.17

*[16,28]; *results from present work*

Table 3. Physiochemical characteristics of prepared metallic soaps (nickel, copper and zinc)

Test	Nickel soap		Copper soap		Zinc soap	
	C. lanatus	*T. occidentalis*	*C. lanatus*	*T. occidentallis*	*C. lanatus*	*T. occidentalis*
pH	6.72-6.87	6.77-6.84	4.97-6.81	5.18-5.20	5.30-6.70	6.09-6.24
Metal content, %	6.20	0.12	14.21	21.57	11.20	0.06
Colour	Green	Green	Blue-green	Blue-green	White	White
Texture	Powder	Powder	Powder	Powder	Powder	powder
Moisture content, %	0.51	25.28	0.40	7.80	0.45	13.15
Melting point, ^{o}C	114	60-88	110	100-112	120	80-180
Bulk density, g/cm^3	0.81	1.50	0.77	0.60	0.77	0.04
Total ash content, %	17.82	29.84	14.46	40.42	17.30	33.69
Yield, %	42	49.28	51	43.85	53	45.50
Foaming property	None	None	None	None	None	None

Table 4. Physiochemical characteristics of prepared metallic soaps from *Irvingia gabonensis seeds oil* (aluminum, calcium and magnesium)

Metallic soap	Free fatty acid, %	pH	Metal content, %	Moisture content, %	Melting point	Total ash, content %	Colour
Aluminum	10.55	5.62-6.84	8.10	89.54	178-198	4.15	White
Calcium	0.73	7.62-9.06	0.03	3.58	118-120	26.35	White
Magnesium	5.33	8.25-8.37	0.02	90.08	100-115	3.15	white

The relative high ash contents in the as prepared soaps mean that the soaps contain much organic residue. The soaps have melting points range 100 – 112°C for copper, 100-180°C for zinc and 60 – 88°C for nickel with *T. occidentalis* seed oil. These values agree with the values obtained by Nene [13] (112°C for Cu; 80°C for Ni and 120°C for Zn) implying that these can also be used as driers. On the other hand *C. lanatus* seeds oil metallic soaps of nickel, copper and zinc soaps have melting points of 114, 110 and 120°C respectively which corresponds to data of *T. occidentalis.* Melting points of aluminum, calcium and magnesium soaps range between 178-198°C, 118-120 °C and 100-115°C respectively. The high melting points are higher than those of other soaps, possible due to high saturation of its oil precursor. However the high melting points make them suitable for high temperature applications. Solubility properties (Table 5) reveal the as prepared soaps are insoluble in cool water but partially soluble in kerosene [33].

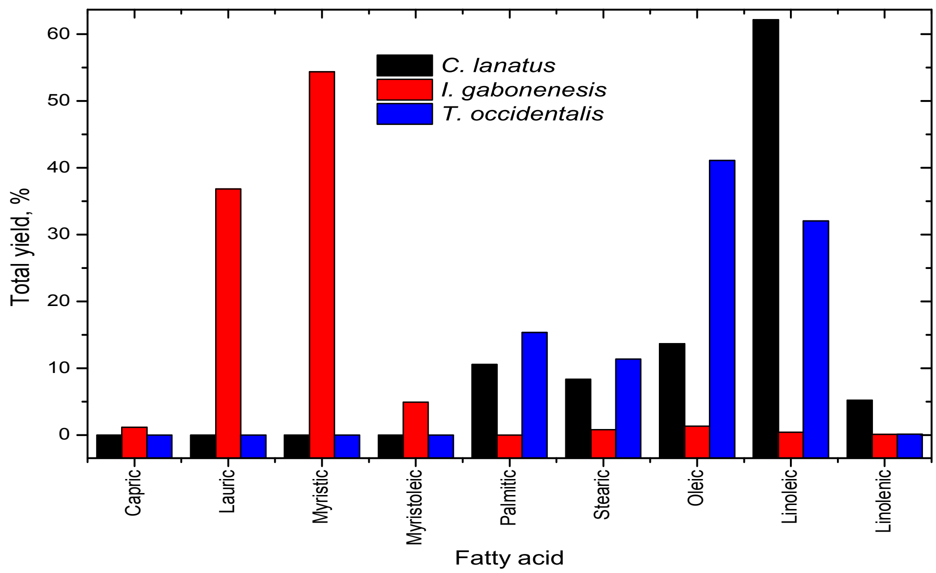

Fig. 3. Fatty acid composition of the three oil extract from the oilseeds

Table 5. Solubility properties of metallic soap in different solvents

Soap	Methanol	Benzene	Acetone	Kerosene	Hot water	Cool water
			C. lanatus			
Nickel	Sparingly soluble	Insoluble	Insoluble	Sparingly soluble	soluble	Insoluble
Copper	Sparingly soluble	Insoluble	Insoluble	Sparingly soluble	soluble	Insoluble
Zinc	Sparingly soluble	Insoluble	Insoluble	Sparingly soluble	soluble	Insoluble
			T. occidentalis			
Nickel	Sparingly soluble	Insoluble	Insoluble	Sparingly soluble	Not tested	Insoluble
Copper	Sparingly soluble	Insoluble	Insoluble	Sparingly soluble	Not tested	Insoluble
Zinc	Sparingly soluble	Insoluble	Insoluble	Sparingly soluble	Not tested	Insoluble
			I. gabnensis			
Aluminum	Soluble	Soluble	Soluble	Partially soluble	Soluble	Insoluble
Magnesium	Insoluble	Soluble	Soluble	Partially soluble	Soluble	Insoluble
Calcium	Soluble	Soluble	Soluble	Partially soluble	Soluble	insoluble

4. CONCLUSION

Oil have been successfully extracted from some edible oilseeds and applied in the preparation of metallic soaps. Two oilseeds, *C. lanatus and T. occidentalis* contain high proportion of unsaturated fatty acid whereas *I. gabonensis* oilseed produced mainly saturated fatty acid consisting of predominantly single carbon carbon bond. Oil of *C. lanatus* seeds is classified as semi-dying whereas *T. occidentalis* and *I. gabonensis* seeds oils are non-drying oils with drying capabilities. These results suggest different applications of the oils such as for industrial and technical use as well as foods. Metallic soap production was feasible as shown with good yields of metallic soap having high organic residue (from oil). From this work the potentials of applying edible seeds oil for metallic soap production have been clearly demonstrated and it is expected future work is feasible to exploit non-edible seeds for economical use.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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