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Chemical Composition on the Seeds and Oil of Sesame (*Sesamum indicum* L.) Grown in Congo-Brazzaville

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Abstract : Proximate composition and physicochemical analyses were carried out on the seed and Sesame oil (*Sesamum indicum* L.). The results showed that the seed contained 5.7% moisture, 20% crude protein, 3.7% ash, 3.2% crude fiber, 54% fat and 13.4% carbohydrate. The seeds were found to be good sources of minerals. Potassium (851. 35 ± 3.44 mg/100g) was the highest, followed in descending order by Phosphorus (647.25± 3.52 mg/100g), Magnesium (579.53 ± 0.42 mg/100g), Calcium (415.38 ± 3.14 mg/100g) and Sodium (122.50 ± 4.21 mg/100g).The physical properties of the oil extracts showed the state to be liquid at room temperature. The oil was found to contain high levels of unsaturated fatty acids, especially oleic (up to 38.84%) and linoleic (up to 46.26%). *Sesamum indicum* L. oil can be classified in the oleic-linoleic acid group. The dominant saturated acids were palmitic (up to 8.58%) and stearic (up to 5.44%). The oil extracts exhibited good physicochemical properties and could be useful as edible oils and for industrial applications.

Key words: *Sesamum indicum* L., oil yield, physicochemical properties, proximate composition, essential fatty acid and activation energy

INTRODUCTION

Sesame (*Sesamum indicum* L.*)* is one of the most important oilseed crops worldwide, and has been cultivated in Korea since ancient times for use as a traditional health food. Sesame seeds are used in the making of tahin (sesame butter) and halva, and for the preparation of rolls, crackers, cakes and pastry products in commercial bakeries. There are numerous varieties and ecotypes of sesame adapted to various ecological conditions. However, the cultivation of modern varieties is limited due to insufficient genetic information. Many farmers continue to grow local sesame (Souza *et al*., 1991), bean (Phaseolus vulgaris L.) (Singh *et al*., 1991), cotton (Gossypium hirsutum L.) (Brown, 1991), Triticales (Royo *et al*., 1995), soybean (Glycine max L.) (Perry *et al*., 1991) and biserrula (*Biserrula pelecinus* L.) (Loi *et al*., 1997). Two studies that used morphological characters to group genotypes into clusters (Ganesh *et al*., 1995; Patil *et al*., 1994) found a wide genetic diversity in Indian sesame genotypes. Multivariate analysis based on morphological characters provides genetic information that will allow the breeder to improve populations by selecting from specific geographic regions (Souza *et al*., 1991). So, aim of the present study was to exmain the proximate composition and physicochemical analyses on the seed and Sesame oil (*Sesamum indicum* L.).

MATERIALS AND METHODS

This study was led to the Laboratory of Engineering and Biomolecule of the ENSAIA-INPL, Vandoeuvre-lès-Nancy (France) for the period of Jan. 5, 2009 to Feb. 27, 2009.

Materials: Sesame seed (*Sesamum indicum* L.), grown in Mbama, Northern Congo, were bought from Tsiémé market, Brazzaville. Only seeds that were not damaged were chosen and stored under cool dry storage conditions until needed.

Methods: Proximate analysis of *Sesamum indicum* L. seed Moisture, crude protein (micro-Kjeldahl), crude fiber and oil (Soxhlet) contents were determined using the methods described by Pearson (1976), whereas the ash content was determined using the method of Pomeranz *et al.* (1994) and total carbohydrate was determined by difference. All determinations were done in triplicate.

Oil extraction: Dried *Sesamum indicum* L. seeds were ground in a Moulinex Model SeB PREP'LINE 850 (Moulin cafe). For solvent extraction (soxlhet method), 50g of ground seeds were placed into a cellulose paper cone and extracted using light petroleum ether (b.p 40-60°C) in a 5-l Soxhlet extractor for 8 h (Pena *et al*., 1992).

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The oil was then recovered by evaporating of the solvent using rotary evaporator Model N-1 (Eyela, Tokyo Rikakikal Co., Ltd., Japan) and residual solvent was removed by drying in an oven at 60°C for 1 h and flushing with 99.9% nitrogen. For methanol/chloroform extraction (Bligh and Dyer, 1959), 100g of the ground seeds were homogenised with a chloroform mixture methanol (1:1) and water. Two phases was obtained, aqueous layer (methanol-water) and organic layer (chloroform). Oil was recovered by evaporating of the solvent (chloroform) using rotary evaporator Model N-1 (Eyela, Tokyo Rikakikal Co., Ltd., Japan) and residual solvent was removed by drying in an oven at 60 °C for 1 h and flushing with 99.9% nitrogen All experiments were done in triplicates and the mean and standard deviations were calculated.

Physical and chemical analysis of crude oil:

Thermal behaviour: The thermal property of the oil samples was investigated by differential scanning calorimetry using a Perkin–Elmer Diamond DSC (Norwalk, USA). The instrument was calibrated using indium and zinc. The purge gas used was 99.99% nitrogen with a flow rate of 100 ml/min and a pressure of 20 psi. Sample weights ranged from 5-7 mg and were subjected to the following temperature program: Frozen oil sample was heated at 50 °C in an oven until completely melted. Oil sample was placed in an aluminium volatile pan and was cooled to -50 °C and held for 2 min, it was then heated from - 50 to 50°C at the rate of 5 °C.min^{-1} (normal rate) (Che Man *et al*., 1995), and held - 50°C isothermally for 2 min and cooled from -50 to 50°C at the rate of 5°C per minute. The heating and cooling thermograms for the normal and the fast (hyper DSC) scan rates were recorded and the onset, peak, and offset temperatures were tabulated. These values provide information on the temperature at which the melting process starts, the temperature at which most of the TAG have melted, and the complete melting temperature of the oil, respectively.

Viscosity measurements: A rheometer as described by Nzikou *et al*. (2007) was used to measure the different oil viscosities. By this procedure, a concentric cylinder system is submerged in the oil and the force necessary to overcome the resistance of the viscosity to the rotation is measured. The viscosity value, in mPa.s, is automatically calculated on the basis of the speed and the geometry of the probe. Temperature (20 °C) was controlled with a water bath connected to the rheometer. The experiment was carried out by putting 3 ml of sample in a concentric cylinder system using 100 s^{-1} as shear rate.

Chemical analysis: Determinations for peroxide, iodine, and saponification values, unsaponifiable matter and free fatty acid contents were carried out using Pena *et al*. (1992) standard analytical methods. The fatty acid composition was determined by conversion of oil to fatty acid methyl esters prepared by adding 950 µl of n-hexane 50 mg of oil followed by 50 µl of sodium methoxide using the method of Cocks *et al*. (1966). The mixtures were vortex for 5 s and allowed to settle for 5 min. The top layer (1 µl) was injected into a gas chromatograph (Model GC - 14A, Shimadzu Corporation, Kyoto, Japan) equipped with a flame-ionisation detector and a polar capillary column (BPX70 0.25), 0.32 mm internal diameter, 60 m length and 0.25 µm film thickness (SGE Incorporated, USA) to obtain individual peaks of fatty acid methyl esters. The detector temperature was 240 °C and column temperature was 110°C held for one minute and increased at the rate of 8 °C/min to 220 °C and held for one minute. The run time was 32 min. The fatty acid methyl esters peaks were identified by comparing their retention time with those of standards. Percent relative fatty acid was calculated based on the peak area of a fatty acid species to the total peak area of all the fatty acids in the oil sample. The minerals were determined by atomicabsorption spectrophotometry. One gram samples, in triplicate, were dry ashed in a muffle furnace at 550°C for 8 h until a white residue of constant weight was obtained. The minerals were extracted from ash by adding 20.0 ml of 2.5% HCl, heated in a steam bath to reduce the volume to about 7.0 ml, and this was transferred quantitatively to a 50 ml volumetric flask. It was diluted to volume (50 ml) with deionised water, stored in clean polyethylene bottles and mineral contents determined using an atomic absorption spectrophotometer (Perkin-Elmer, Model 2380, USA). These bottles and flasks were rinsed in dilute hydrochloric acid (0.10 M HCl) to arrest microbial action which may affect the concentrations of the anions and cations in the samples. The instrument was calibrated with standard solutions.

Statistical analysis: Values represented are the means and standard deviations for three replicates. Statistical analysis was carried out by Excel Version 8.0 software. Significance was defined at $P < 0.05$.

RESULTS AND DISCUSSION

Proximate analysis of *Sesamum indicum* L seed oil: Results obtained showed that the seeds contained 5.7% moisture, 48.5% crude oil, 20% crude proteins, 7.78% carbohydrate (by difference), 9.4% crude fiber and 4.2% ash (Table 1). The high percentage of oil makes this seed a distinct potential for the oil industry. According to Egbekun and Ehieze (1997). Variation in oil yield may be due to the differences in variety of plant, cultivation climate, ripening stage, the harvesting time of the seeds and the extraction method used.

Minerals: The *Sesamum indicum* L seeds contained significant amount of important minerals (Table 2). The Potassium concentration (851. 35 ± 3.44 mg/100g dry

mater) was the highest, followed in descending order by Phosphorus (647.25± 3.52 mg/100g dry mater), Magnesium (579.53 ± 0.42 mg/100g dry mater), Calcium (415.38 ± 3.14 mg/100g dry mater) and Sodium (122.50 ± 4.21 mg/100g dry mater). Potassium is an essential nutrient and has an important role is the synthesis of amino acids and proteins (Malik, 1982). Calcium and Magnesium plays a significant role in photosynthesis, carbohydrate metabolism, nucleic acids and binding agents of cell walls (Russel, 1973). Calcium assists in teeth development (Brody, 1994). Magnesium is essential mineral for enzyme activity, like calcium and chloride; magnesium also plays a role in regulating the acid-alkaline balance in the body. Phosphorus is needed for bone growth, kidney function and cell growth. It also plays a role in maintaining the body's acid-alkaline balance (Fallon, 2001).

Oil extraction: Characteristics of the oil were compared with *Sesamum indicum* L varieties described by Egbekun and Ehieze (1997). The extracted oils were liquid at room temperature. The oil content of *Sesamum indicum* L "Congo-Brazzaville" seeds for the two methods utilised and the level at which the differences are significant are shown in Table 3. The oil extraction with the Soxlhet method had the highest yield, due to the increased ability of the solvent to overcome forces that bind lipids within the sample matrix (Lumley *et al*., 1991). The Blye and Dyer method, showed the low yield due to losses during the separation of the two phases, aqueous layer (methanol-water) and organic layer (chloroform). The results of the above authors agree with those of the present work.

Physical and chemical properties of oil:

Physical properties:

Differential Scanning Calorimetry (DSC): DSC is suitable to determine these physical properties. The results of thermal analysis of oils are presented in Table 4. The obtained peaks were asymmetries and may indicate the presence of three components in oil extracted from the two methods. The first peaks at low melting points appear at – 46.47 °C (H_f = +1.77 J.g^{-1}) for Blye and Dyer method and – 47.71 °C (H_f = +2.11 J.g^{-1}) for Soxlhet method. These peaks correspond to triglycerides formed by poly unsaturated acids (PUFA). The second melting points are at – 27.71 °C (H_f = +48.02 J.g^{-1}) for Blye and Dyer method and – 29.69 °C (H_f = +40.75 J.g^{-1}) for Soxlhet method. This is a characteristic of mono unsaturated acids (MUFA). The last peaks appear to – 6.12 °C (H_f = +0.89 J.g^{-1}) for Blye and Dyer method and – 6.61 °C (H_f = +0.54 J.g^{-1}) for Soxlhet method, suggest the presence of mixed triglycerides groups with different melting points.

Viscosity: Viscosity is a measure of resistance of a fluid to deform under shear stress. It is commonly perceived as thickness, or resistance to pouring. Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction. In optics to know the rheological proprieties of these oils, we studied the influence of temperature on viscosity. Activation energies of the various classes of fatty acids contained in these oils

Table 1: Proximate analysis (g/100 g dry weight) of *Sesamum indicum* L. oil seed

Characteristic	Obtained values[a] (M ± S.D.)	Reported values[b]
		1
Moisture content (%)	5.7 ± 0,24	7.0
Crude protein[c](%)	20 ± 0.12	19.1
Ether extract (%)	54 ± 0.16	48.2
Crude fiber (%)	3.2 ± 0.22	3.6
Ash content (%)	3.7 ± 0,92	5.2
Total carbohydrate[d] (%)	13.4	17.9

[a] M ± S.D. mean ± standard deviation, [b] (1) Egbekun and Ehieze (1997). [c] Crude protein = N (%) x 6.25, [d] Non-fiber carbohydrate was estimated by difference of mean values i.e 100-(sum of percentages of moisture, ash, protein and lipid)

Table 2: Mineral elemental Composition of *Sesamum indicum* L. seeds

Mineral elements	Composition (mg/100g) of Seed
Calcium, Ca	415.38 ± 3.14
Phosphorus, P	647.25± 3.52
Magnesium, Mg	579.53 ± 0.42
Potassium, K	851. 35 ± 3.44
Sodium, Na	122.50 ± 4.21

Values are mean ± S.D of triplicate determinations

Table 3: Physical and chemical properties of *Sesamum indicum* L. seed oil extracted using solvent process

Properties	Obtained values	
	Blye & Dyer	Soxlhet
Oil[a] (%)	52.0 ± 2.25^{B}	57.0 ± 2.35^{A}
PV	0.04 ± 0.32^{A}	
FFA (as %		0.06 ± 0.75^{A}
oleic acid)	1.35 ± 0.24^{A}	1.80 ± 0.10^{A}
IV (wijs)	112.4 ± 0.35^{A}	117.2 ± 1.42^{A}
Saponification value	192 ± 1.42^{A}	197 ± 0. 21^{A}
Unsaponifiable matter Content (%)	1.65 ± 0.12^{A}	1.87 ± 0.27^{B}
Viscosity (mPa.s) at 20°C	40.60 ± 0.12^{B}	29.10 ± 0.17^{B}
Ea (KJ. mol^{-1})	19.79	13.69

Means for the determined values in the same row followed by the same superscript letter are not significantly different (P < 0.05), [a] Oil = weight of extracted oil x 100/weight of seed, Abbreviations: PV: Peroxide Value, FFA: Free Fatty Acid, IV: Iodine Value.

Table 4: Melting behaviour of *Sesamum indicum* L. seed oil using different scan rates. Experimental conditions: temperature program set at -50 °C for 10 min, rising to 50 °C at rate of 5°C.min^{-1}

Thermogram	5 °C.min^{-1}	
	Blye and Dyer	Soxlhet
Peak 1 [°C]	-46.47	-47.71
ΔH_f [J.g^{-1}]	+1.77	+2.11
Peak 2 [C]	-27.71	-29.69
ΔH_f [J.g^{-1}]	+48.02	+40.75
Peak 3 [°C]	-6.12	-6.61
ΔH_f [J.g^{-1}]	+0.89	+0.54

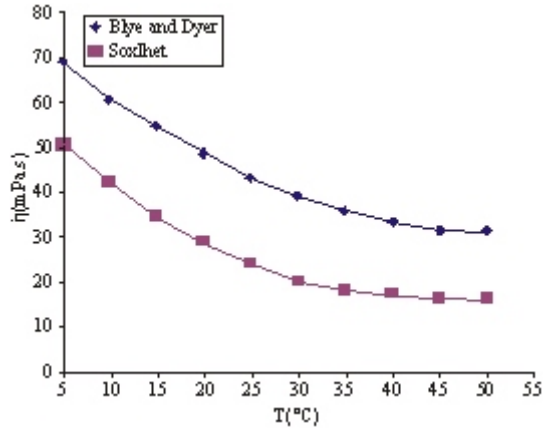

Fig. 1: Effet of *Sesamum indicum* L. temperature on seed oil

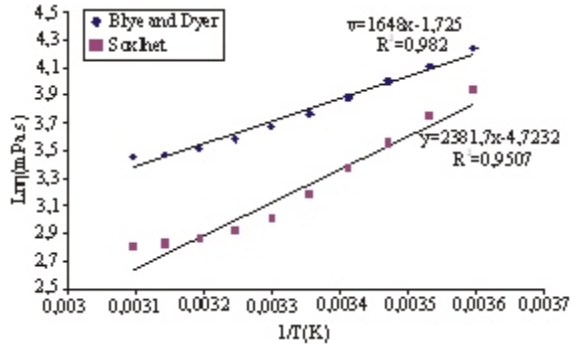

Fig. 2: Relationship between viscosity and temperature for *Sesamum indicum* L. seed oil extracted by Blye and Dyer and Soxlhet. Solid line Arrhenius model

Table 5 : Oil viscosity at various temperature in degree celsuis

T (°C)	η (mPa.s)	
	Blye and Dyer	Soxlhet
5	69.70	51.40
10	60.90	42.40
15	54.90	34.95
20	48.90	29.00
25	43.30	24.15
30	39.40	20.30
35	36.20	18.40
40	33.70	17.50
45	32.10	16.80
50	31.60	16.60

Table 6: Energie plot derived from the Arrhenius equation

1/T (K^{-1})	Lnη (mPa.s)	
	Blye and Dyer	Soxlhet
0.00359712	4.24420032	3.93963817
0.00353357	4.10923317	3.74714836
0.00347222	4.00551335	3.55391847
0.00341297	3.8897774	3.36729583
0.0033557	3.76815264	3.18428438
0.00330033	3.67376582	3.01062089
0.00324675	3.58905912	2.91235066
0.00319489	3.51749784	2.86220088
0.00314465	3.46885603	2.82137889
0.00309598	3.45315712	2.8094027

were given Table 3. When the temperature increases, viscosity decreases exponentially (Fig. 1) some is the extraction method (Arslan *et al*., 2005; Nzikou *et al*., 2007). Viscosity varies between 51.40 and 16..60 mPa.s when temperature decreases of 50 to 5 °C by Soxlhet method. By Blye and Dyer method, the viscosity of oil decreases of 69.70 to 31.60 mPa.s (Table 5). The viscosity of the oil obtained by Blye and Dyer method was highest, possibly because of the water that was absorbed by the gums (phospholipids) during extraction. This calculator calculates the effect of temperature on reaction rates using the Arrhenius equation.

$$\eta = A*\exp^{(-Ea/R*T)}$$

Where η is the viscosity, A is constant, Ea is the activation energy (in KJ mol−1), R is the universal gas constant and T is the temperature (in degrees Kelvin). R has the value of 8.314 x 10^{-3} KJ mol^{-1} K−1. We should use this calculator to investigate the influence of temperature on viscosity. Linear regression analysis was applied to the logarithmic form of Arrhenius equation in order to determine the parameters of the relation (Fig. 2, Table 6). lnη against 1/T, −Ea/RT is the slope from which Ea was evaluated. Activation energies of oils are given in Table 3. The highest value of activation energy is obtained by Blye and Dyer method (19.59 KJ mol−1) and 13.69 KJ mol^{-1} by Soxlhet method. The higher the activation energy, the more stable the fatty acid is.

Chemical properties: The chemical properties of oil are amongst the most important properties that determines the present condition of the oil. Free fatty acid and peroxide values are valuable measures of oil quality. The iodine value is the measure of the degree of unsaturation of the oil. The free fatty acid and the unsaponifiable matter content of the Soxlhet method were significantly higher ($P < 0.05$) than those of the Blye and dyer method (Table 3). There was no significant difference in the iodine and saponification values, in the two extraction methods ($P > 0.05$). The slightly higher value of unsaponifiable matter in the Soxlhet method may be due to the ability of the solvent to extract other lipid associated substances like, sterols, fat soluble vitamins, hydrocarbons and pigments (Bastic *et al*., 1978; Salunke *et al*., 1992).

Fatty acid composition: The major saturated fatty acids in *Sesamum indicum* L seed oil were palmitic (8.58%), stearic (5.44%) acids with small arachidic acid (0.9%). The main unsaturated fatty acids are linoleic (46.26%) and oleic (38.84%) acids (Table 7). There was no significant difference ($P > 0.05$) in the amounts of the major fatty acids in the two oil samples. The two oil samples of *Sesamum indicum* L contained saturated and unsaturated acids (14.90% and 85.10%) respectively. *Sesamum indicum* L oil can be classified in the

Table 7: Relative percent composition of fatty acid in *Sesamum indicum* L. seed oil

Fatty acid	Determined values		Reported values[a]
	Blye and Dyer	Soxlhet	1
C14 :0	–	–	–
C16 :0	8.49 ± 1.23[A]	8.66 ± 1.32[A]	8 - 11
C16 :1	–	–	Max 0.3
C18 :0	5.43 ± 0.18[A]	5.45 ± 0.20[B]	4-6
C18 : 1	38.81 ± 0.35[B]	38.86 ± 0.31[A]	37-42
C18 :2	46.34 ± 0.10[A]	46.18 ± 0.12[A]	39-47
C18 :3	–	–	Max 0.6
C20 :0	0.94 ± 0.18[A]	0.85 ± 0.15[A]	Max 1
C20 :1	–	–	Max 0.4
C22/0	–	–	Max 0.5
Saturated	14.85	14.96	–
Unsaturated	85.15	85.04	–

Means for the determined values in the same row followed by the same superscript letter are not, significantly different (P < 0.05), [a](1) (Teco, 2005).

oleic-linoleic acid group. Linoleic acid which is one of the most important polyunsaturated fatty acids in human food because of its prevention of distinct heart vascular diseases (Boelhouwer, 1983). *Sesamum indicum* L oil is predominantly made up of oleic and linoleic acids (38.84% and 46.26%) respectively. One notes 0 % of linolenic acid C18:3 in the two methods Blye and Dyer and Soxlhet (Table 7). The results obtained are in agreement with those of the literature Teco Finance Export (2005).

CONCLUSION

This study showed that the sesame seed is a good source rich in protein, minerals and oil. *Sesamum indicum* L. seed oil is of unsaturated type and contains mainly the fatty acids oleic C18:1(38.84 %) and linoleic C18:2 (46.26%). The oil can be classified in the oleic-linoleic acid group. High unsaponifiable matters content (1.76%) guarantees the use the oils in cosmetics industry. The oil extracts exhibited good physicochemical properties and could be useful for industrial applications.

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