African Journal of Biotechnology Vol. 5 (10), pp. 987-991, 16 May 2006
Available online at http://www.academicjournals.org/AJB
ISSN 1684–5315 © 2006 Academic Journals

Full Length Research Paper

Extraction and nutritional properties of *Solanum nigrum* L seed oil

J. R. Dhellot[1]*, E.Matouba[1, 2], M.G. Maloumbi[1], J.M Nzikou[1], M.G. Dzondo[1, 2], M. Linder[2], M.Parmentier[2], S. Desobry[2]

[1]ENSP- Laboratoire de Valorisation des Agro ressources (LVAR) BP 69 Brazzaville – République du CONGO.
[2]ENSAIA – Laboratoire de Physico-chimie et Génie Alimentaires 2, avenue de la forêt de Haye, 54505 Vandoeuvre-lès-Nancy,

Accepted 19 April, 2006

With the aim of diversifying the lipids sources eaten by the African populations and those of Congo Brazzaville in particular, a physicochemical study of *Solanum nigrum* L seeds was carried out and three chemical methods (Soxhlet, Bligh and Dyer, and Folch) were used to extract the oil. The dry matter content of the seeds is 94.22%. Average lipids content varies between 34.5 and 37.5% dry matter, proteins content is 17% dry matter and crude ash content averages 7.18% dry matter and the principal mineral element is Mg (180 mg/100g). The acid value of the oil is about 2.5, saponification value varies between 157.3 and 190.1, peroxide value is low at 5.13 and iodine is 102.33.The fatty acid compositions of *S. nigrum* seeds oil shows that it has 67.9% of linoleic acid, indicating its high unsaturation. Apart from linoleic acid, other prominent fatty acids were palmitic, stearic and oleic acids. The following average profile is: 18: 2n-6 > 18: 1 n-9 > 16: 0 > 18: 0. The oil is liquid at room temperature and green in colour. Oil viscosity varies between 20 and 35 mPa.s at 25°C. Three activation energies which vary between 0.8 to 26.58 kJ.mol-1 were determined using Arrhenius's equation. The melting points estimated by Differential Scanning Calorimetry were found to be between -22.0 and -12.0°C for the Soxhlet and Folch-extracted oils. Bligh and Dyer oil have three melting points at -36.2, -15.2 and 33.7°C.

Key words: Oil, *Solanum nigrum* L, essential fatty acid, oil extraction, activation energy.

INTRODUCTION

Solanum nigrum L named "black morelle" is an annual herbaceous plant of 10 to 60 cm high with a green, smooth and semi climbing stem. The opposite leaves, with whole limb, oval and diamond shaped are slightly cogged. It is a rather common species in wet woods, near rivers and old walls. It grows nearly everywhere in Africa and America. In India *S. nigrum* mixed with other herbal medicines has hepatoprotective effect in cirrhotic patients. This protective effect can be attributed to the diuretic, anti-inflammatory, anti-oxidative, and immuno-modulating properties of the component herbs (Fallah, 2005). It also protects against hepatitis B virus infection B (De Silva, 2003; Galitskii, 1997; Kalab, 1997). *S. nigrum* is especially known for its toxicity because it contains solanine, a neurotoxic glyco alkaloid (Abbas, 1998). The extract of its fruits have anti-tumor and neuropharmaco-logical properties and it can be used as an anti-oxidant and cancer chemo-preventive matter (Son, 2003; Perez, 1998).

S . nigrum leaves are eaten in Congo as green

*Corresponding authors E-mail: dhellotjoce@yahoo.fr.

vegetables. The objective of this study is to contribute to the *S. nigrum* seed oil extraction by determining seeds and oils physicochemical properties in order to deduce its dietetic and nutritional advantages.

MATERIALS AND METHODS

Lipid extraction

S. nigrum seeds were obtained from the market of Brazzaville. Lipid extraction was carried out by three methods; Soxhlet extraction (Horowitz, 1984), Bligh and Dyer extraction (Bligh and Dyer, 1959) and Folch extraction (Omoti , 1987; Folch, 1957). After removing solvent, using a Rotavapor apparatus, the seed oil obtained was drained under some nitrogen stream and then stored in a freezer (−20°C) for subsequent physicochemical analyses.

Analytical methods

All analytical determinations were performed at least in triplicate. Values of different parameters were expressed as the mean ± standard deviation. Dry matter was determined according to AOAC (1990). The weight of fat extracted from 40 g of seed powder was determined to calculate the lipid content. Result was expressed as the percentage of lipids in the dry matter of seed powder. Total protein was determined by the Kjeldahl method. Protein was calculated using the general factor (6.25) (AOAC, 1990). Data were expressed in percentage of dry weight. To remove carbon, about 2 g (seed powder), in a porcelain container, was ignited and incinerated in a muffle furnace at about 550°C for 8 h. The total ash was expressed in percentage of dry weight. The mineral constituents (Ca, Na, K and Mg) present in the *S. nigrum* seeds were analysed separately, using an atomic absorption spectrophotometer (Hitachi Z6100, Japan).

Oil analysis

Standard procedures of the American Oil Chemist Society were used for indices values (AOAC, 1997): acid value (standard 969.17, 1997); iodine value (standard 993.20, 1997); peroxide value (standard 965.33, 1997); and saponification value (standard 920.160, 1997).

Fatty acid composition

The oils were converted into methyl esters using a KOH in methanol solution (2 mol l^{-1}) (Norme FIL, 182: 1999). Then, the extracted fatty acid methyl esters (FAME) were dissolved in pure hexane (Merck) for GC analyses. GC analyses were performed on a Perichrom™ 2000 system gas chromatograph (Saulx-les-Chartreux, France), equipped with a flame hydrogen ionization detector and a capillary column (BPX70 SGE Australia Pty Ltd, 25 m x 0.25 mm x 0.5 µm film). The injector and detector temperature were set at 260°C. The column temperature was programmed from 70 to 180°C at 39.9 °C min^{-1}, and kept at this temperature during 8 min. It then underwent a second heating up to 220°C (3°C min^{-1}). Nitrogen was the carrier gas (1.1 bars). The Winilab software (Périchrom, Saulx-les-Chartreux) was used to integrate the chromatograms; the peak areas of triplicate injections were measured. The identification of the peaks was achieved by retention times and by comparing them with authentic standards analysed under the same conditions.

Differential scanning calorimetry (DSC)

Thermal properties were determined with a Perkin-Elmer differential scanning calorimeter, DSC -7 equiped with a thermal analysis data station (Perkin-Elmer Corp., Norwalk, CT). Nitrogen was the purging gas at 20 ml/min. The calorimeter was calibrated according to standard procedures set in the manufacturer's book using indium and distilled water. The sample (~15 g weighed into an aluminium pan) was quickly cooled to −60°C at the speed of 15°C min^{-1}, maintained at this temperature for 10 min, and heated to 60°C at a speed of 5°C min^{-1}. Melting point and enthalpies ΔH (J/g) were calculated for each peak by Pyris software (Perkin-Elmer Corp., Norwalk, CT). The DSC measurements were carried out in triplicate.

Viscosity determination

Viscosity was measured at 25°C or at other temperatures with a Stress Tech Rheologica Rheometer (Rheologica Instruments AB, Lund, Sweden) with a steel cone-plate (C40/4) under a shear stress increased from 7 to 20 Pa.

RESULTS AND DISCUSSION

Oil physicochemical properties

Oil content of *S. nigrum* seeds is between 34.5 and 37.5% (Table 1). It's also richer in oil than some other tropical plants such as *Canarium schwenfurthii* (36.1%) and cotton (16-28%) (Kapseu, 1997a), but have less oil compared to *Balanites aegyptiaca* (48.3%) and *Dacryodes edulis* pulp (43.2%) (Dzondo, 2005). Maximum oil extraction is obtained with Folch method (37.5%).

The ash content in the seeds studied is 7% (Table 1). Thus, they are more or less pure. The macroelements analysis gives the following profile: Mg > K> Ca >Na (Table 1).

With a protein content of 17%, the *S. nigrum* seeds are inferior protein source compared to *Dacryodes edulis* pulp (34%; Dzondo, 2005) and usual oilseeds such as peanut or soybean. The protein content is higher those of major cereals (corn, sorghum, rice etc), which does not generally exceed 13%. The dry matter content of 94% indicates that the seeds are dry.

Oils obtained have a low acidity ranging between 2 and 3% of oleic acid. The saponification value varies, depending on extraction method, from 157 to 190 (Table 1). Oil extracted by Folch method has the highest saponification value 190. The average value (170) is lower than that of *D. edulis* pulp oil (201) (Kapseu, 1997b) and usual oils such as soybean (189-195), peanut (187-196) and cotton (189-198) (Codex Alimentairus, 1993). Whatever the extraction method, iodine

Table 1. *Solanum nigrum* L seed oil physicochemicals properties using different extraction methods[a]

Parameter	Sox[a]	B&D	Folch	Mean
Lipids (%)	34.5 ± 2.4	35.82 ± 3	37. 52 ± 1.6	36
AV	3.07±0.12	2.07±0.3	1.92±0.3	2.35
SV	157.3±3.7	164.66±8.4	190.1±6.5	170.69
I V	102.8±1.91	100.94±0.93	103.25±0.76	102.33
PV	7.4±0.1	4.3±0.1	3.7±0.02	5.13
Viscosity	21.51	19.93	35.49	25.64
Fatty acids				
16:0	10.26	10.23	10.07	10.19
18:0	4.63	4.51	4.71	4.62
SFA	14.97	14.81	14.87	14.88
18:1n-9	15.89	16.19	16.27	16.12
MUFA	16.00	16.39	16.39	16.26
18:2n-6	67.8	67.75	67.4	67.65
18:3n-3	0.83	0.85	0.87	0.85
18:4n-3	0.3	0.28	0.31	0.30
PUFA	68.93	68.88	68.64	68.82
n-3	1.13	1.13	1.18	1.15
n-6	67.80	67.75	67.46	67.67
PUFA / SFA	4.55	4.55	4.55	4.55
n-6/n-3	60.00	59.96	57.17	59.04
Crude ash		7.18 ± 0.97		
K		37.21		
Na		7.12		
Mg		179.3		
Ca		11.1		
Proteins (%)		17.04 ± 0.67		
Dry matter (%)		94.22 ± 2.27		

[a]Sox (Soxhlet), B&D (Bligh and Dyer), and Folch extraction methods.
AV: acid value in % oleic acid. SV: Saponification value in mg KOH g^{-1}. IV: Iodine value. PV: Peroxide value in meq O_2/kg. Viscosity is at 25°C in mPa.s. Minerals in mg/100 g.

value remains unchanged at 102. These oils are unsaturated in comparison with that of *D. edulis* pulp oil (60-85) (Omoti, 1987, Kapseu, 1997b), *Coula edulis* (90-95), *Canarium schwenfurthii* (71-95) (Kapseu, 1999; Abayeh, 1999). The Peroxide value of these oils ranges between 3 and 5. These values are below 10 which characterizes the majority of conventional oils (Codex Alimentairus, 1993).

Viscosity

At 25°C the viscosity of these oils varies between 20 and 35 mPa.s indicating rather fluid oils. Oil obtained by Folch method is more viscous (35.5 mPa.s). With a view to evaluating activation energies of various fatty acids classes contained in these oils, we studied the temperature influence on viscosity. When the temperature increases, viscosity decreases exponentially (Igwe, 2004; Varshi, 1958). Arrhenius's equation was used to determine the activation energy from viscosity results:

$$\eta = A\, e^{-Ea/RT}$$

A = frequency factor called also exponential pre factor energy, Ea = level crossing before elementary flow can begin in kJ mol^{-1}, R = 8.31 Jmol-1K-1 (perfect gases constant) and T = absolute temperature (K).

In a plot of Ln η against 1/T (Figure 1), -Ea/R is the slope from which Ea was evaluated. Activation energies

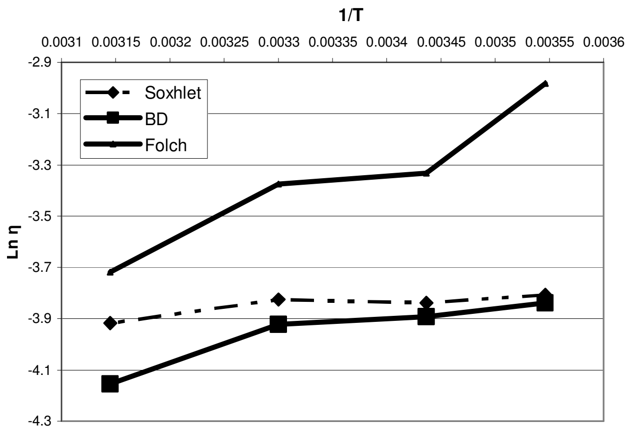

Figure 1. Arrhénius plot of *Solanum nigrum* L seed oil.

Table 2. Activation energy in KJ mol^{-1} for fatty acids classes in *Solanum nigrum* L seed oils.

Activation energy	*S. nigrum* oil			Mean
	Folch	B&D	Soxhlet	
E_{a1}	-26.58	-4.08	-2.39	-11.02
E_{a2}	-2.62	-1.82	+0.79	-1.22
E_{a3}	-18.24	-12.39	-4.87	-11.83

Table 3. Linoleic and linolenic acid contents of usual oils and fats.

Oil /Fat	Linoleic acid (%)	Linolenic acid (%)
Peanut oil	30.5	0
Rapeseed oil	21.2	9.6
Corn oil	55.9	0.9
Walnut oil	56.7	12.3
Grapeseed oil	67.3	0.3
Soybean oil	52.6	7.3
Sunflower oil	64.1	0.05
Olive oil	12.9	0.85
Mixed oil	47	1.2
S. Nigrum L oil	67.65	0.85
Butter	1.16	0.46
Cream	0.52	0.12
African sheabutter	5.98	Nd
Goose fat	12	Nd
Palmkernel fat	2.7	Nd
Cooking butter	12.4	1.24
Pig fat	8.1	Nd

Source: CIQUAL.databases, AFSSA report (2003).

of oils are given in Table 2. Three values for each oil are generally observed. These oils contain three fatty acids classes. Activation energies of Folch oil are higher than those of Soxhlet oil, activation energy of second fatty acids class of Soxhlet oil is endothermic (- 0,8 kJ mol-1) but other activation energies are exothermic, varying between 2.5 and 27 kJ mol^{-1}.

Oil fatty acids composition

Fatty acids composition of these oils indicates the existence of four major peaks: palmitic acid (10.19%), stearic acid (4.6%), oleic acid (16%) and linoleic acid (67.6%). The average profile is: 18:2 n-6 > 18:1 n-9 > 16: 0 > 18:0. Linolenic acid (0.85%) is also present. Linoleic acid content of *S. nigrum* oil is similar to that of sunflower (67.9%) and grapeseed oil (64%) (Table 3). The ratio, PUFA/SFA, is 4.55; therefore these oils are unsaturated. Fatty acids composition is independent of extraction method. *S. nigrum* seed oil is a good source of linoleic acid and it can be used as frying oil.

Table 4. Thermal analysis of *Solanum nigrum* L seed oil.

Peak/ΔH	Melting points and enthalpies		
	Soxhlet	B&D	Folch
Peak 1 [°C]	-22.5	-36.23	-23.23
ΔH [J/g]	6.1	20	6.2
Peak 2 [°C]	-11.6	-15.22	-12.81
ΔH [J/g]	1.31	0.19	0.87
Peak 3 [°C]		33.71	
ΔH [J/g]		0.08	

Thermal analysis

Thermal analysis of *S. nigrum* oil shows a similar profile in Folch and Soxhlet extractions; two melting points at –23 and -12°C. Bligh and Dyer oil thermal profile is different; the first peak is at a very low melting point, -36°C corresponding to the poly unsaturated fatty acids, the second at -15°C and a third at a high melting point of 34°C which corresponds to saturated acids (Table 4).

Conclusion

S. nigrum seeds have a high lipid content. Their protein content and minerals elements (Mg being prominent) are considerable. *S. nigrum* oil is an important source of linoleic acid. Linoleic and linolenic acid contents also give these oil nutritional and dietetic properties. *S. nigrum* seeds can be relied on by the economies of central Africa because it contains large quantities of unsaponifiable matter (green color) thus opening good prospects for its exploitation by the cosmetic industry.

ACKNOWLEDGEMENTS

Analyses were carried out at ENSAIA, the LPGA Laboratory of Nancy. We are grateful to the personnel and the persons in charge of the Laboratory for their collaboration and reception.

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