Research Journal of Applied Sciences, Engineering and Technology, 2(3): 227-232, 2010
ISSN: 2040-7467

Submitted Date: February 28, 2010 Accepted Date: March 09, 2010 Published Date: May 10, 2010

Characterization of Seeds and Oil of Sesame (*Sesamum indicum* L.) and the Kinetics of Degradation of the Oil During Heating

[1,3]J.M. Nzikou, [1]M. Mvoula-tsiéri, [1]C.B. Ndangui, [1]N.P.G. Pambou-Tobi, [1]A. Kimbonguila,
[2]B. Loumouamou, [2]Th. Silou and [3]S. Desobry
[1]ENSP-UMNG, Laboratory of Food Physicochemistry and Biotechnology,
Pole of Excellence in Nutrition and Food, P.O. Box 69 Brazzaville-Congo
[2]Equipe Pluridisciplinaire de Recherche en Alimentation et Nutrition,
Centre IRD. P.O. Box 1286 Pointe-Noire Congo
[3]ENSAIA-INPL, Laboratory of Engineering and Biomolecule, 2,
Avenue de la forêt de Haye, 54505 Vandoeuvre-lès-Nancy, France

Abstract: The aim of this study to characterization and the kinetics of degradation of the oil during heating 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 degradation kinetic of the oil was also investigated. The thermal oxidation of the double bonds of the oil showed a first-order thermal oxidation kinetic and the Arrhenius plot yielded a straight line with a slope equivalent to activation energy of 12.428 KJ.mol^{-1}.There is the possibility of considering the seed as feed supplement and its oil for industrial application.

Key words: Activation energy, degradation, DSC, kinetics, oil yield, *Sesamum indicum* L

INTRODUCTION

Seed oils are important sources of nutritional oils, industrial and pharmaceutical importance.

The characteristics of oils from different sources depend mainly on their compositions and no oil from a single source can be suitable for all purposes (Mohammed and Jorf Thomas, 2003). The study of these constituents is important for their effective uses.

Seed oils are known to deteriorate when processed inadequately with the principal decomposition reaction being oxidation. Oxidation of seed oil occurs through a free radical mechanism, initially characterised by the emergence of a sweetish and unpleasant odour which becomes progressively worse until it attains a characteristic smell of rancid fat (Gouveia *et al*., 2004). Heating is one of the most commonly used methods of food preparation in the home and industries and prolong use of oil for this purpose causes change in its physical and chemical properties (Morette and Fett, 1998).

Under the influence of temperature, fat and oils are susceptible to oxidation primarily leading to the formation of hydroperoxides. Due to their high reactivity, these hydroperoxides especially at high temperatures rapidly react with secondary oxidative products e.g. aldehydes, ketones, peroxides, hydrocarbons as well as cyclic compounds that exhibit very different possible toxic or carcinogenic properties (Kowalki, 1995). The products formed during this oxidative process can be determined by chemical analysis and one of the frequently used tests employed to predict the quality of seed oils is the determination of peroxide value and iodine value. A number of seed oils have been characterised but the vast majority have not been adequately evaluated.

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

Corresponding Author: J.M. Nzikou, ENSP-UMNG, Laboratory of Food Physicochemistry and Biotechnology, Pole of Excellence in Nutrition and Food, P.O. Box 69 Brazzaville-Congo

farmers continue to grow local sesame (Souza and Sorrels, 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 and McIntosh, 1991) and biserrula (*Biserrula pelecinus* L.) (Loi *et al*., 1997). No study has been conducted in these areas. Even, there are limited informations on the physicochemical and proximate composition of the seed and seed oil of this plant. In view of this, the aim of this work is to characterize, correlate parameters such as peroxide value and iodine value in quality assurance and analyze the stability of this oil during thermal treatment.

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. 15, 2010 to Feb. 29, 2010. 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. 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 and Meloan, (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 (Moulinex coffee). For solvent extraction (soxlhet method), 50 g of ground seeds were placed into a cellulose paper cone and extracted using light petroleum ether (b.p 40-60ºC) in a 5-l Soxhlet extractor for 8 h (Pena *et al*., 1992). 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 60ºC 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^{-1} 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 50ºC in an oven until completely melted. Oil sample was placed in an aluminium volatile pan and was cooled to -50ºC and held for 2 min, it was then heated from -50 to 50ºC at the rate of 5ºC.min^{-1} (normal rate) (Che Man and Swe, 1995), and held -50 ºC isothermally for 2 min and cooled from -50 to 50ºC at the rate of 5ºC.min^{-1}. 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 (20ºC) was controlled with a water bath connected to the rheometer. The experiment was carried out by putting 2 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 and Van Rede, (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 240ºC and column temperature was 110ºC held for 1 min and increased at the rate of 8ºC.min^{-1} to 220ºC 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 550ºC 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.

Proximate analysis: Proximate analysis was carried out as described by the Association of Official Analytical Chemists (AOAC, 1995).

Heat treatement: Thermal degradation of *Sesamum indicum* L oil was carried out by heating the oil up to 200ºC for a period of 0-240 min. The peroxide and the iodine values were determined respectively at 100, 150 and 200ºC using the Association of Official Analytical Chemists method.

Kinetic calculations: A general reaction rate expression for the deterioration kinetic can be written as follows (Ramaswami *et al.*, 1989; Van Boeke, 1996):

$$-d[C]/dt = k[C]^m$$

Where 'C' is the quantitative value of the concentration of the molecule under consideration, 'k' is the reaction rate constant and 'm' is the order of the reaction. For first order reaction where m = 1 the equation can be written as:

$$\ln ([C_t] / [C_o]) = -kt$$

Where;

$[C_o]$: The concentration of the reactants under consideration at time zero

$[C_t]$: The concentration of the reactants at time't'

Arrhenius relationship of the reaction rate to temperature is generally given as:

$$K = Ao \exp (-Ea/RT)$$

Where;

Ea : The activation energy of the reaction

R : The gas constant

T : Absolute temperature

A_o : A pre-exponential constant

Each experiment was performed in triplicates and the average values were taken for the parameters determined. Kinetic data were analysed by regression analysis using MS Excel 8.

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

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 and Srivastava, 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

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

Properties	Obtained values Soxlhet Process
[a]Oil (%)	57.0±1.27[A]
PV	0.60±0.75[A]
FFA (as % oleic acid)	1.80±0.10[A]
IV (wijs)	117.2±1.42[A]
Saponification value	197±0. 21[A]
Unsaponifiable matter Content (%)	1.87±0.27[B]
Viscosity (mPa.s) at 20ºC	29.10±0.17[B]
E_a (KJ. mol^{-1})	12.428

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, PV: Peroxide Value, FFA: Free Fatty Acid, IV: Iodine Value.

Table 4: Melting behaviour of *Sesamum indicum* L. seed oil. Experimental conditions: temperature program set at -50ºC for 2 min, rising to 50ºC at rate of 5ºC.min^{-1}

Thermogram	5ºC.min^{-1} Soxlhet Process
Peak 1 [ºC]	-47.71
H [J.g^{-1}]	+2.11
Peak 2 [°C]	-29.69
H [J.g^{-1}]	+40.75
Peak 3 [°C]	-6.61
H [J.g^{-1}]	+0.54

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 and Enig, 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 is high, it was found to be 54±0.16% which shows that the processing of the oil for industrial or edible purposes would be economical. This value is higher than the value reported for the seed of *Moringa oleifera* (40.0±1.34%), Nzikou *et al*., (2009).

Physical properties of oil:

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 by Soxlhet method. The first peak at low melting point appears at -47.71ºC with H_f = +2.11 J.g^{-1}. This peak corresponds to triglycerides formed by Poly Unsaturated Acids (PUFA). The second melting point is at -29.69ºC with H_f = +40.75 J.g^{-1}. This is a characteristic of Mono Unsaturated Acids (MUFA). The last peak appears to -6.61ºC with H_f = +0.54 J.g^{-1}, suggests 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. Measurement value of viscosity at 20ºC is represented in Table 3. The value obtained is of 29.10±0.17 mPa.s.

Chemical properties of oil: Iodine value is a measure of the degree of unsaturation in oil and it is an identity characteristic of native oil. It indicates the degree of unsaturation in the fatty acids of triacyglycerol. This value could be used to quantify the amount of double bonds present in the oil, which reflects the susceptibility of oil to oxidation. The iodine value obtained is high which suggest the presence of unsaturated fatty acid and this places the oil in the drying groups. This oil may find application as a raw material in industries for the manufacture of vegetable oil-based ice cream (Ibiyemi *et al*., 1992). The free fatty acid value is on the low side (1.80±0.10 as % oleic acid). This value shows that this oil is stable. The saponification value is high and this suggests the use of the oil in production of liquid soap, shampoos and lather shaving creams. The peroxide value is 0.6±0.75 mg.g^{-1} oil, this value is lower than that expected of rancid oil, which ranges from 20.00 to 40.00 mg.g^{-1} oil (Oderinde and Ajayi, 1998). This shows that the oil is not rancid and considered stable (Ajayi *et al*., 2002).

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 5). The oil sample of *Sesamum indicum* L. contained saturated and unsaturated acids (14.96 and 85.04%) respectively. *Sesamum indicum* L. oil can be classified in the 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 have oleic and linoleic acids (38.86 and 46.18%) respectively. One notes 0% of linolenic acid C18:3. The results obtained are in agreement with those of the literature Tecco Finance Export (2005).

Kinetic data for the degradation of *Terminalia capatta* L. oil: The rate of production of peroxide in *Sesamum indicum* L. oil increases as the temperature increases as shown in Table 6. This shows that the prolong heating of this oil makes it to undergo thermal degradation resulting in oxidative rancidity, formation of hydroperoxides and other products of degradation that can liberate volatile compounds.

Table 5: Relative percent composition of fatty acid in *Sesamum indicum* L. seed oil

Fatty acid	Determined values Soxlhet Process	Reported values[a]
C14 :0	–	–
C16 :0	8.66 ±1.32 ^{A}	8 - 11
C16 :1	–	Max 0.3
C18 :0	5.45±0.20 ^{B}	4-6
C18 : 1	38.86±0.31 ^{A}	37-42
C18 :2	46.18±0.12 ^{A}	39-47
C18 :3	–	Max 0.6
C20 :0	0.85±0.15 ^{A}	Max 1
C20 :1	–	Max 0.4
C22:0	–	Max 0.5
Saturated	14.96	–
Unsaturated	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]: (Tecco, 2005).

Table 6: Effect of heating on PV and IV of *Sesamum indicum* L. oil

Temp. (°C)	Time (mins)	PV ($mg.g^{-1}$)	IV (mg/iodine)
100	30	0.95±0.12	117.01±0.71
	60	1.36±0.40	116.76±0.18
	120	1.76±0.11	116.45±0.36
	180	2.19±1.43	116.24±0.42
	240	2.65±0.32	116.01±0.12
150	30	1.24±0.23	116.89±0.32
	60	1.56±0.18	116.16±0.18
	120	1.94±0.23	115.99±0.05
	180	2.26±0.15	114.96±0.38
	240	2.61±0.05	114.46±0.47
200	30	1.84±0.14	116.40±0.42
	60	2.05±0.71	115.92±0.28
	120	2.24±0.28	115.31±0.54
	180	2.47±1.24	114.96±0.32
	240	2.71±0.48	114.46±0.71

Values are mean ± standard deviation of triplicate determinations
PV: Peroxide Value, IV: Iodine Value

Table 7: Kinetics paramaters for degradation of *Sesamum indicum* L. oil

Kinetics paramaters	100°C	150°C	200°C
$k_{1\,(S\,-1)}$	0.00004	0.0001	0.00009
$t_{½\,(S)}$	17325	6930	7700

k: reaction rate constant, t½: time of half-reaction or half life

Table 6 also shows that the iodine value of the oil decreases as it was heated over a period of time. This suggests the loss of unsaturation in the fatty acids of the triacylglycerols.

In order to obtain the reaction rate constant ('k'), a first order degradation of the oil was presumed (Labuza and Riboh, 1982). Accordingly 'ln $[C]_t/[C]_o$ was plotted against 't'(time) from which rate constant 'k' was calculated from the slope of the line (Anthon and Barrett, 2002) as shown in Fig 1. A correlation coefficient > 0.9 in all the cases confirmed the assumption of the degradation (loss of unsaturation) to follow the first order kinetic. The half-life for the degradation was calculated from the rate constant as '0.693/k' and is given Table 7.

Figure 2 shows the Arrhenus plot of ln k versus 1/T for the reduction of unsaturation (Iodine value) in *Sesamum indicum* L. oil. The linear nature of the plot obtained gave the activation energy of the reaction to be 12.428 $KJ.mol^{-1}$ (Table 3).

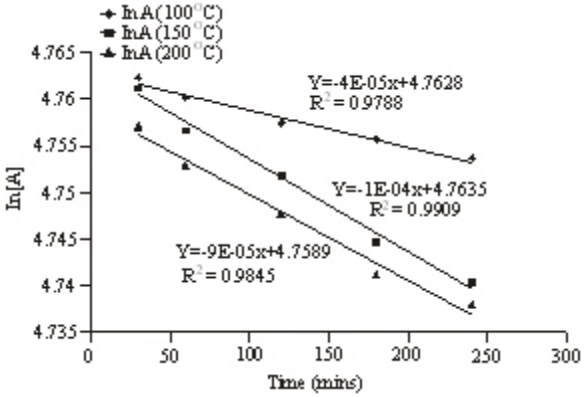

Fig. 1: Graph of rate of change in concentration of iodine value against time, A: Represents iodine value

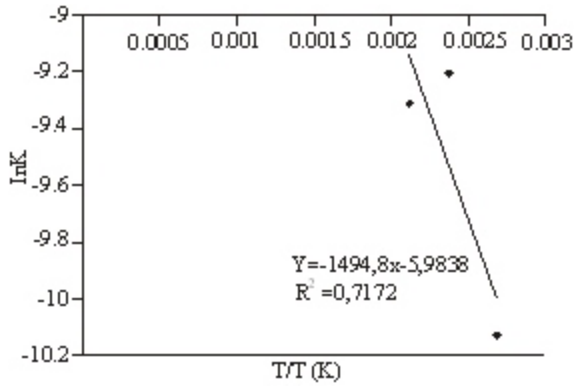

Fig. 2: Arrhenius plot for oxidation rate of the double bonds of fatty acids in *Sesamum indicum* L. oil

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 (1.87%) guarantees the use the oils in cosmetics industry. The oil extracts exhibited good physicochemical properties and could be useful for industrial applications. The thermal oxidation of *Sesamum indicum* L. oil follows a first order reaction. This oxidation is temperature and time dependent. The process quality assurance of this oil can be monitored using iodine value and peroxide value.

ACKNOWLEDGMENT

We would like to thank Carole Jandel and Carole Perroud for their assistance in conducting chemical analyses.

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