



## ORIGINAL ARTICLE

# Impact of incorporating sesame oil (*Sesamum indicum* L.) in an Algerian frying oil and margarine formulation

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## ABSTRACT

**Background and aims:** This study, carried out in collaboration with the research and development department of the "Cevital spa" agri-food unit, aimed to incorporate sesame oil into the formulation of two fatty food products: a frying oil and margarine to improve their physicochemical and organoleptic qualities. **Methods:** The sesame oil was obtained from the sesame seed by cold pressing to preserve its nutritional characteristics. The frying oil was elaborated with a mixture of three oils (sunflower, soybean, and non-roasted sesame). The margarine was enriched with 2 % of roasted sesame and then, the quality of the products was assessed. Gas chromatography (GC-FID) profile indicated that sesame oil is an oleic-linoleic rich oil with saturated fatty acid (SFA) / unsaturated fatty acid (USFA) ratio from of 0.11. **Results:** The organoleptic tests and physicochemical analyses, including the oil, showed that the resulting recipe is a combined oil rich in n-6 and n-9, offering an interesting ratio of MUFA /PUFA and with a SFA content of 11.49 % for an appropriate utilization in frying and cooking. Enriched margarine showed compliance with the standards set by the *Codex Alimentarius* and has a characteristic taste, smell and appearance, color and spread ability to the product with a sesame note. **Conclusion:** The results of the current study support the sesame oil supplementation to conventional frying oil and to commercial margarine. Sesame oil may therefore be an alternative source of fatty acids that could contribute to the diversification of combined oils.

## ARTICLE INFORMATION

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## 1 Introduction

Vegetable oils and fats have a major contribution to our diet; they are consumed directly in the form of refined or virgin oil, or indirectly via several products of the food industry. Consumers are becoming increasingly demanding in terms of quality; safety and nutritional food are at the center of societal concerns <sup>1</sup>. Vegetable oils offer a large choice in terms of taste, use, price and quality, when each oil possesses its flavor, aroma, nutritional value, and the variety of the fatty acids that distinguishes it from the other oils. Depending on their nature, vegetable oils are more or less rich in certain essential polyunsaturated fatty acids <sup>2</sup>. Furthermore, vegetable oils are also a substantial source of vitamin E, known for its

antioxidant properties; Therefore, vegetable oils are considered as important foods which provide essential and healthy nutrients <sup>3-5</sup>. These oils are different in terms of taste and nutritional quality. Several authors proved that mixing them could be an appropriate alternative to take advantage of their health benefits <sup>6,7</sup>.

Vegetable oils have multiple uses including frying that is one of the most popular processes used by consumers and food industries for various applications, which provides tasty food in a relatively short period of time <sup>8</sup>. However, the repeated use of frying oils at high temperatures can produce constituents that not only compromise the quality of the food but can also promote the formation of a variety of decomposing elements

with adverse nutritional implications for human health<sup>9</sup>. The lipids degradation of edible oils during frying depends on the composition of the starting oil, the nature of the food, as well as the frying conditions such as temperature, duration of use, surface area of the oil exposed to the air in relation to the volume, periodic use, frying capacity (kg of fried food/hour) and mode of heat transformation (gas or electric)<sup>10</sup>.

In general, many oils rich in polyunsaturated fatty acids (PUFA), especially linoleic acid (omega 6) and alpha linolenic acid (omega 3), are indeed more sensitive to oxidation and must hence be renewed more often<sup>11</sup>. In order to increase the shelf life of frying baths, antioxidants for instance essential oils or natural extracts can be added, or mixture of vegetable oils can be made with a linolenic acid (omega 3) content of no more than 2 %<sup>12</sup>.

Among the oil-rich plants, sesame (*Sesamum indicum* L.); cultivated for its edible seeds and for oil production with a content varying from 35% to 57%<sup>13</sup>. Sesame is a herbaceous plant of the *Pedaliaceae* family and the seeds are enclosed in the fruit, consisting of a capsule<sup>14</sup>. This plant has a cycle of 75 to 135 days depending on the variety and a development of 0.70 to 2.30 m. Sesame has been cultivated for a long time in the hot and moderately humid areas of the world, especially in West Africa. There are white, brown, and black seeds; light-colored seeds have a higher oil content, while black seeds have thicker shells<sup>15</sup>. Sesame oil is rich in PUFA, iron, magnesium, manganese, copper, and calcium and contains vitamins B1 and E<sup>16</sup>. This oil belongs to the oleic-linoleic, it has a low amount of linolenic acid of the omega 3 family and a balanced ratio of omega-fatty acids (omega 6 and omega 9)<sup>17</sup>.

Sesame seeds are often used whole in cooking for their strong nutty flavor, incorporated into a wide range of sweet and savory breads, flours, biscuits and snacks, or simply sprinkled on hamburger buns<sup>18</sup>. Part of the sesame production (about 20 %) is reserved for the consumption of the seeds, which are consumed roasted or for the preparation of traditional "sesame pure" dishes, for these outlets, the seeds are dehulled by chemical or physical methods (soaking, flotation, drying)<sup>19</sup>. The paste obtained after grinding the seeds is used in the preparation of varied local dishes, cakes, and chocolate. In addition, the paste obtained from filtering the oil is used in the preparation of soap and massage cream<sup>20</sup>. In recent times, sesame oil has become remarkably stable thanks to specific natural antioxidants (sesamol, sesamin, and sesamolol). It is recommended to use in agri-food industries by several researchers aiming to enhance foods stability with good nutritional value<sup>21</sup>.

In the current study, sesame oil was incorporated either roasted or not roasted, to improve the organoleptic quality of the margarine while also stabilizing the formulation of frying oils. This work was carried out in collaboration with the

research and development department of the "Cevital" agro-food Company in Bejaia, Algeria.

In this regard, we carried out in collaboration to enrich their products by blending them with others protected naturally edible oil without adding synthetic chemicals antioxidants. Thus, the objective was to blend cold pressed oil of both roasted and not roasted sesame seed with the refined vegetables oils, and to exploit their effect to improve the organoleptic quality of the margarine while also stabilizing the formulation of frying oils.

## 2 Materiel and Methods

### 2.1 Plant material and oil extraction

Indian sesame seeds (*Sesamum indicum* L.) were purchased on local markets. The processing is relatively simple and involves the following operations, First, the sesame seeds were spread out on preheated trays in thin layers of about 2-3 mm. Then, the sesame seeds were roasted in a preheated electric oven (GmbH 56, Binder, Germany) at  $200 \pm 2$  °C for 20 minutes according to the method described previously<sup>21</sup>. The roasted and unroasted seeds were then passed through a fixed hammer mill for about one hour; pressing was done with the help of scorers for 30min, separating the liquid phase from the solid phase, resulting in cold extracted crude oil directed to storage tanks, and cake bagged manually. After decanting, the oils are collected in shaded glass bottles, filled, labelled, and stored at a temperature of 6 °C.

### 2.2 Formulation of combined oil

The elaboration of a combined oil for frying and/or cooking was done at the level of the research and development department of the "Cevital" agri-food Company with a mixture of three oils: soya, sunflower and sesame, the elaboration of the formula is done at the laboratory level by respecting the recommendations of Hamitri-Guerfi et al.<sup>22</sup> and the quality indications used as a tool of follow-up and selection at the level of the company which are:

- Saturated fatty acids < 10%.
- Omega 3 < 2 %.

For this reason, the best formulation according to the company's laboratory experts for good frying and cooking use is a configuration with: 71 % sunflower, 27 % soya and 2 % sesame.

### 2.3 Analyses performed on roasted and unroasted sesame oil

#### 2.3.1 Refractive index

The refractometer (ATAGO N°54918.1T) is the device used to measure the refractive index. After the regulation system was turned on, the refractometer has been directed to the

**Table 1.** Development of a frying oil formula

Oil	Sunflower	Soybean	Sesame	Elio recipe			Total
				% Sunflower	% Soybean	% Sesame	
FA (%)				71	27	2	100
C12 :0	0.3	0	/	0.21	0.00	0.00	<b>0.21</b>
C14 :0	0.2	0	/	0.14	0.00	0.00	<b>0.14</b>
C16 :0	0.35	10.59	10.1	4.51	2.86	0.2	<b>7.57</b>
C18 :0	3.19	4.13	5.56	2.26	1.012	0.11	<b>3.49</b>
C18 :1	32.42	23.53	41	23.02	6.37	0.82	<b>30.13</b>
C18 :2	56.69	53.22	42.35	40.02	14.37	0.85	<b>55.47</b>
C18 :3	0	7.66	0.32	0.00	2.07	0.01	<b>2.07</b>
C20 :0	0.1		0.6	0.07	0.00	0.01	<b>0.08</b>
Total	99.25	99.13	99.93	70.47	26.77	2.00	<b>99.23</b>
% Saturated	10.14	14.72	16.26	7.2	3.97	0.33	<b>11.50</b>
% Double bonds	89.11	84.41	83.67	63.27	22.79	1.67	<b>87.73</b>
% EFA ( $\omega 3 + \omega 6$ )	56.69	60.88	42.67	40.25	16.44	0.85	<b>57.54</b>
$\omega 6 / \omega 3$	56.69	6.95	132.34	/	6.95	132.34	<b>26.74</b>
MUFA/PUFA ( $\omega 9 / \omega 3 + \omega 6$ )	0.57	0.39	0.96	0.57	0.39	0.96	<b>0.52</b>
PUFA/SFA	5.59	4.14	2.62	5.59	4.14	2.69	<b>5.00</b>

EFA: Essential fatty acids; MUFA: Monounsaturated fatty acids; PUFA: Polyunsaturated fatty acids; SFA: Saturated fatty acids.

light and the eyepiece has been well cleaned for a clear vision. With the help of a pipette, the sesame oil extract was placed, in sufficient quantity, on the horizontal face of the refractometer prism P. Once the adjustment operations have been carried out, it is enough to look into the eyepiece O' (the one on top) and to read the value of the refractive index for the sample <sup>23</sup>. A quantity of oil (10 g) was placed between the prisms of a refractometer completely fill the space between the prisms. After a few minutes, the oil reached the desired temperature, and the measurement was taken according to the following formula:

$$Ri = (T - T_1) \times F + Ri'$$

Where: Ri: Refractive index; Ri': Refractive index read on the refractometer; T: Reference temperature: 40 °C for Sunflower and Soybean oils; T1: Measurement temperature; F: Correction factor = 0.00035 for T= 20 °C.

### 2.3.2 Peroxide value

The peroxide value (Pv) is the amount of product in the sample expressed as milliequivalent gram of active oxygen per kilogram of fat oxidizing potassium iodide <sup>23</sup>.

A quantity of 5 g of oil was placed in an Erlenmeyer, 12 mL of chloroform and 18 mL of acetic acid and 1 mL of saturated solutions of potassium iodide (KI) were added. The flask was shaken for 1 min in obscurity. 75 mL of distilled water were added then the mixture and titrated with sodium thiosulfate solution (0.01 N) and were subjected to iodometric determination of peroxide value according to AOCS official method.

The results were then expressed as follows:

$$Pv(\text{meqg} / \text{kg}) = N \times (V_1 - V_0) \times 1000 / P$$

Where: Pv: Peroxide value expressed in milliequivalent grams per kilogram; V0: Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution used in the blank test in ml; V1: Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> used in the titration in ml; N: Normality of the Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution 0.01N; P: Test sample in grams.

### 2.3.3 Saponification value

The saponification value "SV" is the amount of potassium hydroxide (KOH) in mg required to saponify the free fatty acids which provides information on the chain length and allows determining the average molecular weight of the fatty acid <sup>23</sup>. In brief, in a 100 mL flask, 2 g of oil were placed with 25 mL of KOH 0.5 N, the mixture was allowed to saponify for one hour in the cold, then a few drops of phenolphthalein were added, after the blank test, a titration with 0.5 N KOH was carried out on the sample as well as the blank test. The expression of the results was made according to the formula:

$$Sn(\text{mg} / \text{g}) = 56.1 \times N \times (V_0 - V) / M$$

Where: Sn: Saponification value in milligrams per gram; V0: Volume of 0.5 N HCl for the blank test in ml; V: Volume of 0.5 N HCl for the sample taken in ml; M: Test sample in grams; N: Exact normality of the HCl solution used (0.5 N).

### 2.3.4 Iodine value

The iodine value "IV" was also used to quantitatively determine the unsaturation of fats. IV is the number in grams of iodine fixed per 100 g of fat. A quantity of oil (0.15 g) was dissolved in 15 mL of carbon tetrachloride (CCl<sub>4</sub>) in an Erlenmeyer flask. A volume of 25 mL of Wijs' reagent was then added. The mixture was stirred and placed in the dark for one hour. After that, 20 mL of potassium iodide (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) solution (10 %) and 150 mL of water were added to the previous mixture. The iodine released is titrated with sodium thiosulfate (0.1

N) in the presence of starch as an indicator until the medium turns colorless. A blank test was carried out under the same conditions using all elements of the mixture with the exception of oil<sup>24</sup>. The results were calculated as follows:

$$I_v = N(V_0 - V) \times 12.69 / P$$

Where:  $I_v$ : Iodine value (g/I<sub>2</sub>/100 g oil);  $V_0$ : Volume of sodium thiosulfate solution used for the blank test in mL;  $V$ : Volume of sodium thiosulfate solution used (mL, 0.1 N) for the oil; 12.69: P: weight of oil aliquot (g); (Concentration conversion coefficient).

### 2.3.5 Chromatographic profile

#### *Gaz chromatography analysis*

The fatty acid profile roasted, and unroasted sesame oil and their blend was carried out by gas chromatography (GC)-FID technique according to ISO 5508 (1990). Gas liquid chromatography was carried out on a 30 m 0.25 mm FAMEWAX capillary column with a 0.25-mm thick polar polyethylene coating (Restek, Bad Homburg, Germany) equipped with a 5 m 0.25 mm Guard Column (Restek). Separation was performed with the following temperature gradient on a 6890 series GC instrument (Agilent, Weilbronn, Germany): 140 1C to 210 1C with 10 1C/min, 210 1C to 230 1C with 2 1C/min and 230 1C for 7 min (total run time 24 min). Helium was used as carrier gas at a constant flow of 1.5 mL/min. One microliter of the sample was injected into an injector kept at 275 1C, and the split was set to 1:50. The flame ionization detector was operated at 300 1C with 45 mL/min hydrogen flow, 450 mL/min air flow and a make-up flow of 45 mL/min helium.

#### *Fatty acids analysis*

Prior, fatty acid methyl esters (FAME) were prepared by methanolic boron trifluoride (13-15%). The characteristics of the GC ((Chrompack CP 9002) were as follow: Capillary column: DB 23; (50% Cyanopropyl). 30 m long, 0.32 mm internal diameter and 0.25 µm film thickness. The carrier gas: Hydrogen (H<sub>2</sub>). Injector: SPLIT1/1100 (250°C). The amount injected: 0.1 µL. The oven temperature: 150 °C (+3 °C/min). Detector: FID (Flame Ionization Detector), temperature 250°C). Fatty acids were identified by comparison with relative retention times of standards and calculation of equivalent chain length values.

## 2.4 Analysis performed on the combined oil

In addition to the peroxide value which has been described above, the following parameters were measured:

### 2.4.1 Free fatty acids

Ten (10 g) of product was added after introducing phenolphthalein into 75 mL of neutralized ethyl ethanol until a pink color appeared, the mixture was stirred followed by

heating for a few seconds to improve the reaction; titration was carried out with sodium hydroxide solution (NaOH, 0.1 N) until a persistent pink color appears; the volume of the doughnut drop was noted. The results were expressed as follows:

$$A(\%) = N \times V \times M / 10 \times M$$

Where: A: Product acidity; N: NaOH normality (0.1N); V: Volume of NaOH burette drop (ml); M: Molar mass of the acid suitable for expression; M = 282 g/mol for oleic acid and M = 256 g/mol for palmitic acid; m: the mass in grams (g) of the test sample.

### 2.4.2 Polar compounds

The measurement of polar compounds in oil was performed with an "OPTIFRY Tester". This device allows an accurate measurement of the polar compounds present in the oil which is an approach of the polymers responsible of the degradation of frying oils. The method entails completely covering the air holes on the tester probe with oil before holding it in the oil at an angle of about 45 °C to allow the air to escape. The result is shown within 5 s.

### 2.4.3 Accelerated oxidation test or "Rancimat test"

This test is widely used to assess the quality of edible oils and fats by evaluating the stability and durability of the products in a rapid and simple way. The apparatus used is the "Rancimat" (CH 9100 Methrom, Switzerland). The principle of the test is to age the fat prematurely by thermal decomposition at a specific temperature under intensive air bubbling. The induction time specification for the accelerated oxidation test, expressed in hours (h), is the time during which the material has resisted to oxidation. In brief an aliquot of oil (3 g) was placed in a "Rancimat" heating block, the temperature was maintained at 98 °C at an airflow rate of 10 L/h. The measurement was based on the conductimetric detection of volatile acids, so, the volatile compounds released during the degradation process were collected in a cell containing 60 mL of distilled water in which an electrode for measuring electrical conductivity was immersed. The degradation products of this extensive oxidation were carried away by an air stream and collected in a measuring cell filled with distilled water<sup>25</sup>.

### 2.4.4 Frying test

The material used was the potato because of its simple composition, it is the most used in frying and it is free of fat<sup>26</sup>. The potatoes were peeled and cut into chips of more or less equal size and then dried with absorbent paper. The test was carried out using an electric fryer with a removable lid, with a capacity of 1 kg of frying product and 2.5 L of oil, equipped with a thermostat and a timer. After heating the oil bath to

180 °C, the frying process was started by introducing the first weight of chips while setting the frying time at 4 min, then the second weight was introduced while timing again and repeated until the tenth weight. After heating the oil bath to 180 °C, frying was started by introducing the first weight of chips while setting the frying time at 4 min and then the second weight was introduced while timing again and this was repeated until the tenth weight is reached. The ratio of the quantity of fries to the quantity of oil in the fryer is displayed in Table 2. Three samples were considered: a control (E0), taken from our mixture before frying, the other two samples, taken from the fifth (E5) and the tenth (E10) frying respectively, filtered, cooled, and then put into oil bottle preforms (120 mL).

The organoleptic quality of the oil mixture, during the frying process, was assessed by a group of specialists of the food-processing complex "Cevital". The tests were carried out on the state of the fry including the evaluation of its crispness and its color, as well as the state of the frying bath including the evaluation of its color and the nature of the smoke.

**Table 2.** Ratio of fries to oil in the fryer

Number of fry	Quantity of oil (g)	(Potatoes / oil = 100/kg)
1 <sup>st</sup>	2380	238
2 <sup>nd</sup>	2380	238
3 <sup>rd</sup>	2380	238
4 <sup>th</sup>	2380	238
5 <sup>th</sup>	2380	238
6 <sup>th</sup>	2260	226
7 <sup>th</sup>	2260	226
9 <sup>th</sup>	2260	226
10 <sup>th</sup>	2260	226

## 2.5 Analyses carried out on margarine

A scaled-down margarine recipe was developed with 2 % roasted sesame oil. The analyses performed on the enriched margarine are:

### Moisture content

The moisture was determined by the method described by the standard ISO 662 (1998). The heating was carried out for the margarine in a water bath, to have the separation of the two phases (aqueous and fat); the aqueous phase (water) was then evaporated, to obtain the dry product, the weights of the empty beaker  $P_0$ , of the all-salt product  $P_1$  and of the beaker with the dry product  $P_2$  were taken. The moisture content was determined on 3 g of margarine brought to 100 °C until constant weight and the following formula was applied to assess the water content which was expressed in percentage:

$$MC = (P_0 + P_1 - P_2 / P_1) \times 100$$

### Salt content

This consists of measuring the salt concentration in the margarine, in principle and according to Bentayeb Ait Lounis et al. <sup>27</sup>, it is the determination of chlorides (Cl<sup>-</sup>). Briefly, 5 g of the sample to be tested was put in an Erlenmeyer flask, then 100 mL of boiling distilled water was added; the mixture was stirred for a few minutes to melt the sample and then proceeded to cooling to -50°C, a few drops of potassium chromate (K<sub>2</sub>CrO<sub>4</sub>) were added to the mixture. The color of the mixture before titration was yellow. The titration was done with a solution of silver nitrate AgNO<sub>3</sub> at (0.1 N) until a brick red color was obtained. The salt content was determined by the following equation:

$$S(\%) = V \times N \times M / 100 \times m$$

Where: V: Volume of AgNO<sub>3</sub> burette drop (ml); N: Normality of AgNO<sub>3</sub> (0.1N); M: Molecular mass of NaCl salt, M = 58.5 g/mol; m: Mass in grams of the test sample.

### Melting point

The melting point is the temperature at which a fat change from a solid state to a liquid. In brief, the margarine was introduced into two glass capillary tubes on a height of 1 cm, the refrigeration was then carried out with the refrigerator for a duration of 20 mins. The two capillaries were then fixed to a thermometer with a rubber ring so that the bottom of the capillary tubes was at the same level as the bottom of the mercury ball of the thermometer; the whole was immersed in a beaker containing osmosis water followed by a slow heating of about 0.5 °C/min in a water bath filled with water, the temperature noted corresponds to the melting point of the margarine (oil) expressed in degrees Celsius.

### pH

The two phases, fat, and water must be separated, and the water phase used to determine the pH. Its value was indicated by the pH-meter in pH units on the scale of the instrument (NE.1.2.430/1989).

### Solid fat content

To determine the level of solids in margarine, each sample was melted in an oven at 100 °C and then filtered. The obtained filtrate was then poured into three tubes up to 2 cm. The tubes were incubated at three different temperatures: 20 °C/ 20 min, 30 °C/20 min and 40 °C/ 20 min. Value were read by nuclear magnetic resonance (NMR) spectrometer (minispec mq20, Germany), processed and the final results were given in percentage of solid according to the method described in international standard (ISO, 1995).

**Table 3.** Chromatographic profile of studied oils

Fatty Acids	SOR	SONR	According to (ISO 5508, 2000) (%)
Palmitic acid (C16:0)	9.54	9.3	8-11
Stearic acid (C18:0)	6.27	6.1	4-6
Oleic acid (C18:1)	42.59	42.0	37-42
Linoleic acid (C18:2)	40.91	40.74	39-47
Linolenic acid (C18:3)	Traces	0.28	<0,6
Arachidonic acid (C20:0)	0.68	0.7	<1
Eicosenoic acid (C20:1)	00	00	<0.4
SAF (%)	10.22	10	
USFA (%)	89.77	89.12	
SFA/USFA	0.113	0.101	

SFA: Saturated fatty acids; USFA: Unsaturated fatty acids; SOR: sesame oil roasted; SONR: sesame oil not roasted

## 2.6 Statistical analysis

Data were reported as the mean and standard deviation (SD) of the triplet determinations. Two software packages were used for the presentation and analysis of the results; Excel stat v. 2019 and Minitab 17 which was used for the one-way analysis of variance (ANOVA). Mean values were compared at a significant level of  $p < 0.05$ .

## 3 Results and Discussion

### 3.1 Fatty acids profile of the oils

The fatty acids were identified by gas chromatography (GC) by comparing their retention times with a reference chromatogram of a standard mixture of methyl esters of known composition and concentration. The results of the profile determination by chromatography of roasted and unroasted sesame oil are reported in Table 3.

The GC-FID analysis showed the presence of four fatty acids: palmitic, stearic, oleic, and linoleic fatty acids. The observation of the relative proportions of the identified and measured fatty acids confirms that they are in accordance with the values given by ISO 5508, 2000, so the studied cold extracted oil is (oleic-linoleic) in nature.

**Table 4.** Physicochemical characteristics of roasted and unroasted sesame oil

Parameters	RSO	URSO	Reference values
Saponification index (mg KOH/g)	190.0 ± 2.64	191.0 ± 2	187 - 195 (Codex Alimentarius, 1983)
Iodine Index (g Iodine/100g)	109.0 ± 0.63	108.0 ± 1.22	104 - 120 (Codex Alimentarius 1983)
Refractive Index at 40°C	1.4650 ± 0.049	1.4648 ± 0.045	1,474 - 1,477 (ISO 6320, 2000)
Peroxide value (meq O <sub>2</sub> / kg)	9.6 ± 0.6	7.6 ± 0.22	Max 10 (ISO 3960 4th edition, 2007)

RSO: Roasted sesame oil; URSO: Unroasted sesame oil.

### 3.2 Sesame oil physicochemical characteristics

The iodine index (Iv) is an indicator of unsaturation of oils and fats. IV is the amount of iodine fixed by 100 g of oil in grams, indicating the overall state of oil unsaturation. The IV increased by the increase of the oil unsaturation. The roasted and unroasted sesame oils have acceptable (Ii) values compared to the range established by the Codex Alimentarius 1983 (104 - 120 /100 g oil) which were  $109 \pm 0.6$  and  $108 \pm 1.2$  (g Iodine/100 g), respectively for roasted and unroasted sesame. Moreover, these values are in accordance with those suggested by Pocklington<sup>28</sup> for edible oils of good quality.

Fats are principally deteriorated by oxidation, a chemical phenomenon involving very different reaction mechanisms fat deteriorates leading to oxidative or hydrolytic rancidity<sup>29</sup>. The peroxide value of an oil or fat is used to measure the degree of rancidity reactions that have developed during storage, and it represents the early stages of oxidation. The values obtained were low ( $9.6 \pm 0.6$  for sesame oil roasted (SOR) and  $7.6 \pm 0.2$  for sesame oil not roasted (SONR) compared to the maximum acceptable value ISO 3960 4<sup>th</sup> edition, 2007 which is 10 meq KOH/g for oilseeds. The oils studied were analyzed the same week of their extraction which explains the low values obtained that confirm the good quality of the product. The low peroxide values indicate that the oils have low amounts of oxidative rancidity. In contrast to oils sold in Bulgaria, where Yanishlieva and Marinova<sup>30</sup> reported values of 8.8 and 4 meq for sunflower and soybean oil, respectively, the fresh oils under investigation are less peroxidized overall. According to Rudan Tasic and Klofutur<sup>31</sup> the four brands of sunflower oil in Slovenia had an average peroxide value of 2.090.

The refractive index (RI) is a measure used for identification and as a criterion of purity of oils. It is also used as an index of the degree of hydrogenation of the oil<sup>32, 33</sup>. The RI of oils depends on their molecular weight, their degree of unsaturation, the chain length of fatty acids, and the degree of conjugation<sup>34</sup>. The values of  $1.465 \pm 0.049$  and  $1.4648 \pm$

0.045 meq KOH/g for oil were obtained at 40 °C, for the oils

studied which are within the range established by ISO 6320, 2000 (1.474 - 1.477), the iodine value of "*Fleurial*" oil from "*Cevital*" displayed a high RI value of 1.467.

The saponification number of a fat presents a major advantage and allows the use of a fat without knowing its exact composition, which is always complex and variable in the case of natural fats. The values found for roasted and unroasted sesame oil were  $190.0 \pm 2.64$  and  $191 \pm 2$  respectively which are in accordance with the standards established by (Codex Alimentarius, 1983).

### 3.3 Study of the quality of the oil mixture during frying

#### 3.3.1 Free fatty acid (FFA)

The evolution of the acidity of the oil mixture is given in Figure 1.

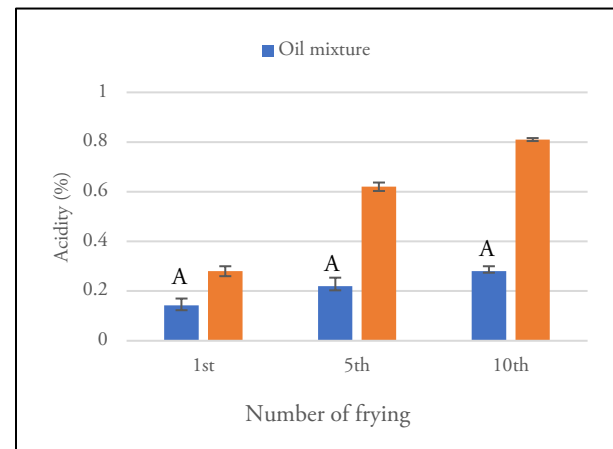
The results of the acid number of the obtained blend oil and the "*Elio*" oil are presented in Figure 1. The analysis of variance revealed a significant difference between the values of the acid number of the two oils after the different frying (1<sup>st</sup>, 5<sup>th</sup>, and 10<sup>th</sup> frying) under the simultaneous effect of the temperature and the frying time. According to Figure 1 data, the acid number increases significantly during frying process. The minimum AI was observed in the blend oil with a value of  $0.14 + 0.027\%$  compared to "*Elio*" which records a value of  $0.28 + 0.02\%$ ; the maximum was observed at the 10<sup>th</sup> frying of "*Elio*" with values of  $0.81 + 0.06\%$  while the blend oil presents a value of  $0.28 + 0.02\%$  at the 10<sup>th</sup> frying, the values of the 5<sup>th</sup> and 10<sup>th</sup> frying are higher than the Codex Alimentarius standard. According to Medina-Valtierra et al. <sup>34</sup>, acidity is proportional to the number of frying. The increase in the acidity of the oil is the source of the fatty acids released as a result of hydrolytic oxidation and the increase in the fatty acid content of an oil leads to the degradation of its properties.

#### 3.3.2 Free Peroxide value

The peroxide value is one of the most important indices for the oxidative rancidity of oils and fats. This measure of oxidation corresponds to the amount of oxygen peroxide per 1 kg of product and is a practical test with high sensitivity for assessing the early stages of oxidative damage <sup>36</sup>.

According to Figure 2 data, the value increases progressively with the number of frying to reach a maximum value above the norms, the Pv is started to increase to  $5.8 \text{ meq O}_2 / \text{kg}$  for *Elio* and  $2.2 \text{ meq O}_2 / \text{kg}$  for the blend oil on the 1<sup>st</sup> frying. A significant increase in peroxide value was recorded on the 5<sup>th</sup> and the 10<sup>th</sup> frying of both "*Elio*" and blend oil, the values presented were above the standards ( $33.83 \text{ meq O}_2 / \text{kg}$  and  $36.5 \text{ meq O}_2 / \text{kg}$ ). The increase in the peroxide value is due to primary oxidation during frying, resulting in the formation of primary peroxide compounds and free radicals, the peroxides

are colorless and odorless and have no particular taste <sup>37</sup>. Oxidation increases considerably with temperature. In general, the hotter the oil, the greater the risk of oxidation. The peroxides that develop are unstable and, at frying temperature, they break down to form a series of secondary oxidation products. Some of these by-products can develop pleasant frying tastes and odors, while others give rise to unpleasant odors, pungent and unpleasant flavors leading to rancidity of the product <sup>38</sup>.

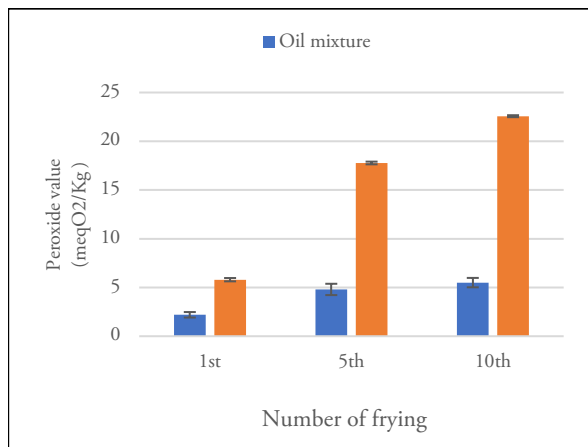


**Figure 1.** Evolution of acidity of frying oils as a function of the number of fries

#### 3.3.3 Polar compounds

The content of polar compounds in the oil mixture varies with the number of frying performed, as shown in Figure 3.

After the cooking (frying) of the, one can see very clearly in the figure a significant increase on both oils. *Elio's* oil results from the 1<sup>st</sup>, the 5<sup>th</sup>, and the 10<sup>th</sup> frying exceeded the 25% threshold (28.33 %, 33.83 %, 36.5 %), while only the results from the 10<sup>th</sup> frying exceeded the standard for the processed blend oil. The measurement of polar compounds is one of the most important indicators of the state of degradation of the oil, thus the level of polar compounds in the oil reflects its rate of degradation and dissociation of triglycerides <sup>39</sup>. According to Chen et al. <sup>40</sup> in European legislation, the maximum percentage of polar compounds allowed varies from 25 % to 27 % and it is beyond this that the oil is considered unfit for consumption or even toxic. Therefore, our frying oil can be considered to comply with the international standards.



**Figure 2.** Evolution of the peroxide value as a function of the number of fries

### 3.3.4 Accelerated oxidation test (Rancimat test)

The purpose of the Rancimat test is to predict the oxidative stability of fat. The induction period is calculated from the inflection point of the conductivity curve on a graph showing conductivity versus time<sup>41</sup>. The oil enriched with 2 % sesame has an induction time of 11.73 h, this value is higher than the oil "*Fleurial*" produced in "*Cevital*" which is composed only of soybean oil whose induction time is 8.27 h, while the "*Elio*" oil which is composed of sunflower oil and soybean has an induction time of 12.17.

### 3.3.5 Fry test

Table 5 summarizes the results obtained from the organoleptic tests of the oil mixture studied during frying. The observations were performed by laboratory experts in quality who are part of the "*Cevital*" agro-food company.

Observations showed that during the frying process, the oil mixture was remarkably stable with no smoke, no undesirable smell, and the oil color remained clear until the fourth frying. A slight coloration started to appear from the fifth frying; at the tenth (10<sup>th</sup>) frying the oil shows a dark coloration.

### 3.3.6 Incorporation of roasted sesame oil in the formulation of margarine

The results of the physicochemical analyses carried out on margarine previously enriched with roasted sesame oil are presented in Figure 6.

### 3.3.7 Solid Fat Content (SFC)

Solids content refers to the percentage of solid fat that is solid at different temperatures. SFC is an important indicator that shows several product characteristics, including its overall appearance, oil fluidity and organoleptic properties<sup>42</sup>. For

spreadable margarines, the solids content should not exceed 40 % at 5 °C, or 6 % at 35 °C or 32 % at 10 °C<sup>43</sup>. The SFC content of enriched margarine varied with temperature. Indeed, the SFC decreased with increasing temperature. The values were  $15.3 \pm 0.25$  at 20 °C,  $7.3 \pm 0.06$  at 30 °C, and  $1.8 \pm 0.098$  at 40 °C, these results are in accordance with ISO 8292 T60 - 250, 1995. The solid content was less than 6 % at 37 °C, which indicates that enriched margarine was melting, plastic and easy to spread and therefore to prepare according to good manufacturing practices.

In fact, each SFC value refers to the quality of the product; the range from 0.5 to 10 °C provides information on the spread-ability of the product at low temperatures; the range from 15 to 20 °C offers information on the hardness of the margarine as well as its oily exudation. The range from 20 to 25 °C shows a strong relationship with the stability of the margarine (oxidative stability). The range from 30 to 35 °C displays a relationship with the texture during certain uses such as tasting and the release of the flavor in the mouth.

### 3.3.8 Melting point

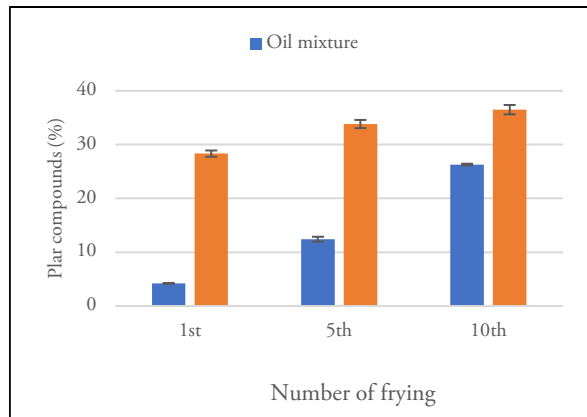
The melting point indicates the temperature at which the margarine should be smooth in the mouth. The international standard range for margarine melting points, ISO 6321 (2002), is 28 – 37 °C, which implies that margarine can melt quickly in the mouth and be firm at room temperature to resist mechanical work during spreading. The melting point of the developed margarine was  $36.00 \pm 0.09$  °C, in the range of the standard value. The statistical analysis revealed no significant difference between enriched and reference margarine.

A margarine that possesses a high saturated fatty acid content presents a higher melting point, which results in a harder texture with less plasticity and resulting in a harder texture with less plasticity; therefore, the spread ability will be more difficult at room temperature and its texture in the mouth will be less melting.

According to Silva et al.<sup>44</sup>, the melting point depends on several parameters attributed to the triglycerides structure, the number of double bonds; for the same length of the chain, the melting point decreases with the number of double bonds. The geometric form or the melting point of *cis* forms is lower than that of *trans* forms<sup>45</sup>.

The melting point is regularly correlated with the spread ability and plasticity of the margarine at room temperature and this where the need to keep the melting point temperature adequate for each type of margarine<sup>46,47</sup>.

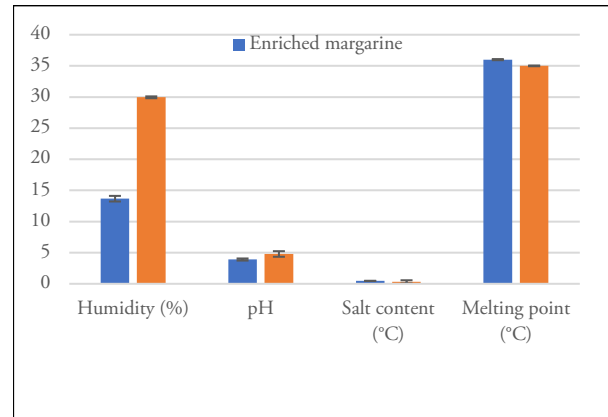




**Figure 3.** Evolution of polar compounds as a function of the number of fries

### 3.3.9 pH

The statistical analysis revealed a significant difference in pH between the enriched and the reference margarine but both values remain still within the established norms.



**Figure 4.** Quality indicators for a margarine enriched with roasted sesame oil

The pH is one of the quality indicators of margarine and is monitored at various stages of preparation and processing to ensure safety, improve production, and increase quality. It is one of the main obstacles that microbial flora must overcome to ensure its multiplication. According to Chougui et al. <sup>48</sup>, it is advisable to control the pH of the water phase since high values generally delay the growth of contaminating

**Table 5.** The observations made during the frying tests

Fry number	Colour of the bath	Odour	Colour of the fry	Nature of the smoke	State of the fry
1 <sup>st</sup>	Clear, limpid	The sesame touch	Walleye	None	Crunchy
2 <sup>nd</sup>	Clear, limpid	The sesame touch	Walleye	None	Crunchy
3 <sup>rd</sup>	Clear, limpid	The sesame touch	Walleye	None	Crunchy
4 <sup>th</sup>	Clear, limpid	The sesame touch	Walleye	None	Crunchy
5 <sup>th</sup>	Coloration (+)	Neutral	Walleye	None	Crunchy
6 <sup>th</sup>	Coloration (+)	Neutral	Walleye	None	Crunchy
7 <sup>th</sup>	Coloration (++)	Neutral	Walleye	None	Crunchy
8 <sup>th</sup>	Coloration (++)	Neutral	Walleye	None	Crunchy
9 <sup>th</sup>	Coloration (++)	Neutral	Walleye	None	Crunchy
10 <sup>th</sup>	Coloration (+++)	Neutral	Walleye	None	Crunchy

The pH value ( $3.9 \pm 0.2$ ) of the margarine enriched with 2 % sesame complies with the values recommended by standard NE.1.2.430/89. At this value, the stability of both emulsions was noticeable.

microorganisms and limit hydrolysis phenomena. While low pH values cause an unpleasant and acidic sensation that may be rejected by consumers <sup>49</sup>. However, the compliance of the results is linked to the effective monitoring of pH during production, the quality of water, preservatives, and pH correctors, as well as to the control of the quantities added.

### 3.3.10 Salt content

The salt content of the margarine formula was in the order of  $0.47 \pm 0.017$  % and is considered to be within the recommended standards. The statistical analysis revealed the presence of a significant difference between the two values, both of which still fall within the established norms.

According to Saillard<sup>50</sup>, the salt content varies according to the use of the margarine and its texture. It is in the order of 0.1 to 0.3 % for spreadable margarines. Salt plays a crucial role in the stabilization of the emulsion<sup>48</sup>.

### 3.3.11 Moisture

The reference margarine of "Cevital" had a moisture content of  $29.95 \pm 0.41$  %. The sesame-enriched margarine had a moisture content of  $13.67 \pm 0.45$  %, which was within the range determined by the standards ISO 662 (1998 – 90 - 1)<sup>51</sup>. The statistical analysis revealed the presence of a significant difference between the two values, both of which still fall within the established norms.

Due to the high-water content of the spread margarine, the vegetable oil in the spread rapidly oxidizes, creating a breeding ground for mold and the development of certain microorganisms such as Clostridium, Streptococcus, etc., as well as enzymatic hydrolysis. The moisture content varies according to the conditions and period of storage<sup>44</sup>. Margarine can oxidize even when refrigerated if it is not handled or stored correctly, or if it has passed its expiry date<sup>48</sup>.

## 4 Conclusions

The current study highlighted that the oil mixed with 2 % sesame resists up to the fifth (5<sup>th</sup>) frying and always remains in conformity with the standards and recommendations of the standardization body for a good use in frying and cooking contrary to *Elio's* oil which exceeds the standards after the fifth frying. This clearly confirms that the 2 % sesame oil has a better oxidative stability due to its richness in natural antioxidants. On the other hand, the enriched margarine complies with the criteria of the Codex Alimentarius and has a distinct taste, aroma, appearance, color and spread ability of the product with a sesame note. Our results support the interest in combining sesame oil with frying oil and margarine, as sesame oil is considered an alternative fat source that contributes to the diversification of the combined oils. This work is certainly called to be deepened because several points remain to be elucidated of which for example, to carry out a hedonic sensory analysis which would make it possible to better judge the acceptance of these mixtures by the consumers; to evaluate the antioxidant capacity and to define the real effects of the consumption of this oil on the human health because this oil recipe will be commonly used as vegetable oil in the domestic, restoration and industrial operations of frying.

**Author Contribution:** A.S. Practice and drafting the paper. B.D. Practice. F.G. framing and editing. D.R. English text correction. KM. RA. YR. DN. Orientation and guidance. S.H. Co-supervision. KM. Director.

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