International Invention of Scientific Journal
Available Online at http://www.iisj.in
eISSN: 2457-0958
Volume 03 | Issue 06 | June, 2019 |

Original Article

OPEN ACCESS

Sesame Seeds and Flax Food Oils: Extraction and Physicochemical Characterizations

Nabila BOUHADI[1,2*] , Boualem CHENNIT[1] and Lilya BOUDRICHE[1]
[1]Center for Technical and Scientific Research in Physical-Chemical Analysis, Bou-Ismail Tipaza, Algeria
[2]Research laboratory in food technology (LRTA) Faculty of Engineering Science University, M'hamed Bougara 35000 Boumerdes, Algeria

Received: June 10, 2019
Accepted: June 25, 2019
Published: June 30, 2019

***Corresponding Author:**
*** NABILA BOUHADI**
Center for Technical and Scientific Research in Physical-Chemical Analysis, Bou-Ismail Tipaza, Algeria

Abstract

The objective of this study is the extraction, characterization and analysis of seed oils of flax and sesame for a better appreciation of these oils. Extraction carried out by the Soxhlet method gave an oil yield of the order of 50.6% for sesame and 42% for flax. The obtained values for the different physical-chemical indices (refractive index, acid, peroxide and saponification) are in the standards except for flaxseed oil, which has an acid value of around 8 mg KOH /g of oil, reflecting the strong presence of free fatty acid. The profiles of the obtained FTIR spectra during the analysis of the two oils are similar, with relative intensities of the absorption bands slightly different. The analysis of the fatty acid profile by GC / MS shows the presence of unsaturated fatty acids (linoleic acid, oleic acid), saturated fatty acids; palmitic acid (C16) and stearic acid (C18) in the case of flax oil, and the presence of palmitic acid in sesame oil.

Key Words: Sesame, flax, Oil, Extraction, Physical-chemical characterization.

Introduction

Lipids, like other nutrients, occupy a very important place in the human diet. They are natural products widely present in nature, they include oils and fats of animal and vegetable origin. The oils represent, among the fatty substances, a vast family of foods exclusively lipidic, certainly very energetic (900 Kcal / 100g), but sources of very varied fatty acids [1]. A vegetable oil is a fatty substance extracted from an oleaginous plant (flax, sesame, sunflower, ..), in other words, from a plant that has the particularity of accumulating mainly oils in their seeds, nuts or fruits, its Nutritional value is based on its intake of essential fatty acids (essential) omega 3 and omega 6 or vitamins. Flax seeds are plant molecules with a high antioxidant capacity, which reduces the risk of developing high blood pressure and type 2 diabetes. Moreover, they are rich in alpha-linoleic acid, an omega 3 precursor. they allow a reduction of cholesterol and plasma triglycerides [2]. The therapeutic effect of flax oil on hypertriglyceridemia and hypercholesterolemia in older obese rats was also studied by Laissouf, Soulimane [3]. flax oil because of its fast-drying property, its oil is used for the preparation of paints, varnishes, ink, soap [4]. Sesame seeds contain two substances sesamine and sesamin known to lower cholesterol in humans and to prevent hypertension [5]. They are also consumed throughout the world in condiments and as an essential ingredient in different recipes, so sesame oil shows remarkable oxidative stability, this is attributed to the presence of lignans; [6-8]. Sesame oil has nutritional and dietary interests (confectionery products, bread ..) industrial and pharmaceutical (production of perfumes, treatment of the skin, hair ..) [6, 7, 9].

The national economy has a considerable deficit in oil, with 12 kg /person (less than 10% of olive oil), the consumption of vegetable oils in Algeria is at the level of Morocco and below Tunisia (17 kg). Oilseed crops are very limited (less than 20 000 ha, for a production of 50 000 Ton in 2012, mainly rapeseed), 90% of Algerian needs in edible oils (excluding olive oil) are covered by the import of crude oils (mainly soy) that are refined on site. As a result, the local production of oilcakes for animal feed is very low and also leads to massive imports [10]. The many applications and the therapeutic effects on the health of these edible oils had motivated our study, which has for first purpose, the analysis of the biochemical composition, the extraction and physicochemical characterization of flax oil. and sesame to deepen our knowledge of the composition of these oils.

Materials and methods

Sampling:

In this study, we used sesame seeds from India and flaxseed from Canada. These seeds were grinded using a knife mill (type IKA) to reduce this raw material, constituting the sample to be studied, into fine particles and to increase the contact surface with the solvent, thus facilitating obtaining the oil. The extraction of oils was carried out by two methods: the soxhlet extractor to determine the fat yield of the seeds and cold extraction to recover the oils to be characterized.

Physical and chemical characterizations of the obtained oils:

The physical and chemical characterization of the concerned oil had shown the following analyses Determination of the fat content by Soxhlet (NF EN ISO 734-1, 2000) using hexane; determination of the specific absorbance by ultraviolet radiation (ISO 3656); determination of acidity (ISO 660); determination of indices: refraction (ISO 6320), acid, peroxide (ISO 6320), saponification and ester (ISO 3657); Fourier Transform Infrared (FTIR) analysis, resistance to forced oxidation by the Rancimat test (ISO 6886) and determination of the fatty acid profile (GC / MS analysis).

Results and discussion

The results of the physical and chemical analyzes of the two oils are shown in Table 1.

Table 1: Characteristic parameters of the obtained oils

Physicochemical parameters	sesame	Codex Alimentary Standards	Flax	Codex Alimentary Standards	Probability threshold (P-VALUE)
Fat content (%)	50,6±3,4	35-57%	42,0±4,7	35- 47%	0,0629
Peroxide number (méq O_2/Kg)	7,2±0,4	<15	7,3±0,5	<10	0,9746
Acid number (mg KOH/g of oil)	1,9±0,09	< 4,5	8,3±0,1	4	<0,0001
Acidity(%)	0,9±0,03	1,0-3,0	4,1±0,07	1,0-2,5	<0,0001
Refractive index	1,471±0,01	1,474-1,477	1,481±0,01	1,479-1,484	0,0003
Saponification index (mg KOH/of oil)	184 ±10	187-195	185±5	189-196	0,9666
index of ester (mg KOH/g of oil)	182 ±10	183-191	176±5	185-192	0,4121
K_{232}	3,75±0,01	/	4,020±0,004	/	0,0001
K_{270}	1,304±0,004	/	1,033±0,004	/	0,0001
ΔK	0,037±0,0004	/	0,033±0,004	/	0,0001

Fat content:

The richness of the seeds in oil depends first of all on the species, their size, their shape, the state of maturity, the pedoclimatic conditions and the treatments during the harvest. According to the literature, the oil content of sesame seeds varies between 35 and 57% whereas flax seeds contain between 35 and 45% [11]. With regard to our results (50.6% for sesame and 42.0% for flax), the obtained values are in the standards which allows us to confirm that flax seeds and sesame can be classified in oilseeds. Several studies have confirmed the richness of these seeds in oil, for example Popa, Gruia [12] and Benmehdi, Amrouche [4] have recorded 30 and 40% of oil in flaxseed from Romania and Algeria respectively, Nzikou, Matos [9] obtained an oil content of 57% from sesame seeds. Mohammed and Hamza [13] used white and red sesame seeds and reported values between 48-50%.

Peroxide number:

The determination of the peroxide index is the most appropriate method for the measurement of peroxidized compounds (hydro peroxides, ketones, aldehydes, etc.) formed in the presence of oxygen and certain promoter factors. oxidation (UV, heat, traces of metals). The Codex Alimentarius (1999) sets the value of the peroxide value at a maximum of 15 meq O2 / kg of oil for virgin and cold pressed flax oils and 10 meq O2 / kg of oil for sesame oils. The results found (7.297 meq O2 / kg of sesame oil and 7.312 meq O2 / kg of linseed oil) are lower than the codex standard, proving that the oils remain relatively protected in their original matrix (the seed), despite the existence of certain enzymatic oxidation pathways, which are activated during natural physiological changes, such as germination. Also, the percentage of healthy seeds (free of cracks or breaks) contribute to the preservation of oxidation. Similar results to ours were recorded by Mohammed and Hamza [13] in two types of sesame seeds: white and red from Nigeria (between 7.45 and 8 meq KOH / g of oil). Benitez-Benitez and Ortega-Bonilla [14] used two methods of extraction (percolation and pressure) of sesame oil and obtained the values: 5,104 and 4,050 meq O2 / kg of oil. Benmehdi and Amrouche [4] recorded 4.74 meq O2 / kg for flaxseed oil.

Acid number:

The free fatty acid content of a fat is expressed in two ways: the acidity and the acid number which are experimentally determined in the same way and only their mode of expression differs. This is a measure that evaluates the hydrolytic alteration of fats. The presence of water in the seeds or in the medium can lead to hydrolysis phenomena. Oils, either by chemical or enzymatic action. The triglycerides are then partially hydrolyzed to free fatty acids. Higher than standard values were found for flaxseed oil (8.37 mg KOH / g oil), which is probably due to the presence of free fatty acid in quantity as a result of the reaction. enzymatic hydrolysis that goes into action when grinding seeds. The same case for the acidity, which represents the percentage of free fatty acids expressed conventionally according to the nature of the fatty substance. Ogbonna and Ukaan [6] analyzed 13 samples of sesame seeds from Negeria and gave values in the range of 0.68-2.78 mg / g of oil, while in oil Benitez-Benitez, Ortega-Bonilla [14] from Colombia recorded 4,444 and 4,196 mg / g of oil.

Refractive index:

A drying agent is a substance that acts as a catalyst by accelerating the drying or hardening of an oil Drying oils have refractive indices between 1.480 and 1.523, half-drying oils between 1.470 and 1.476, and non-drying oils between 1.468 and 1.470. The refractive index, closely related to the degree of unsaturation of the fatty acids constituting the fatty substance, is influenced by many other factors: free acidity, oxidation, polymerization, existence of secondary function on the molecules, etc. According to our results, flax oil (1,471) is a semi-drying oil while sesame oil (1,481) is drying. This will condition their behavior in the open and their fragility to oxidation too. Several authors have given values comparable to ours, 1,471 for sesame oil [8] and 1,469 for flaxseed oil [12]

Saponification index:

This is the number of milligrams of KOH needed to convert the free fatty acids and glycerides contained in 1g of fat into soap. The values found are slightly lower than the norm (184 mg KOH / g sesame oil and 185 mg KOH / g flaxseed oil) and lower than the values reported by Popa, Gruia [12] (190 mg KOH / g of linseed oil) and Mohammed and Hamza [13] (189-191 mg KOH / g of sesame oil). According to Benmehdi, Amrouche [4], the high values of the saponification index allow their use in the cosmetic industry (soap, champage ..). Several factors influence the chemical characteristics of oils: geographical origin and climatic factors as well as seed transport and storage. The same case for the index of ester, which represents the mass of potash necessary to saponify the esters contained in 1g of fat. That is, the amount of KOH needed to convert the fatty acids in the form of triglycerides contained in 1 g of fat into soap. In our study, it is 182 mg KOH / 1g for sesame oil and 176 mg KOH / 1g for flaxseed oil. Thus, it gives us a relevant indication as to the average lengths of the fatty acid chains that constitute them, those of the flax are a little longer than those of sesame.

Study of UV Alteration (Extinguishing Coefficient K232, K270):

The extinction at 232 nm and 270 nm of a crude fat can therefore be considered as an image of its state of oxidation. The greater the extinction at 232 nm, the more the oil is peroxidized. The greater the extinction at 270 nm, the more the oil is rich in secondary oxidation products. The obtained oils contain linoleic hydro peroxides and the conjugated dienes which absorb at around 232 nm and lower amounts of by-products of oxidation (diketones and unsaturated ketones) which absorb towards 270 nm. Among others because they are freshly extracted. Gharby, Harhar [7] worked on Moroccan sesame seeds and gave extinction values at 232 nm and 270 nm of 1.73 and 0.52 respectively. The statistical treatment by the Student's test of the results obtained showed that the variation is highly significant for the acidity, the acid number, the refractive index, and the coefficients of extinction, whereas it is not significant. for other indices, namely the peroxide index, the saponification index and the index of ester.

FTIR analysis:

Figure (1) shows the superposition of the (Bruker type FTIR-ATR) spectra obtained from oils extracted from sesame and flax seeds. The profiles of the FTIR spectra obtained during the analysis of the two oils are similar, with relative intensities of the absorption bands slightly different. These spectra show absorption bands corresponding to the extensional vibrations of the C-H groups (CH3 and CH2 fatty acids) at 3008 cm-1 and 2853 cm-1 [15-17] All organic compounds containing a carbonyl group C = O have an intense characteristic absorption around 1700 cm-1 [18, 19], which is the most intense and sharp band in the IR spectrum [19]. This same band can also move according to the compounds formed (ketones, aldehydes, carboxylic acids, etc.), witnesses of the degradation of the oil. In our case, an absorption band (C = O) associated with the presence of ester carbonyls, which represents the functional group of triglycerides [17-19], is observed around 1745.18 cm-1. Absorption at 1463.96 cm-1 is related to the deformation of CH (CH2) bonds [18].

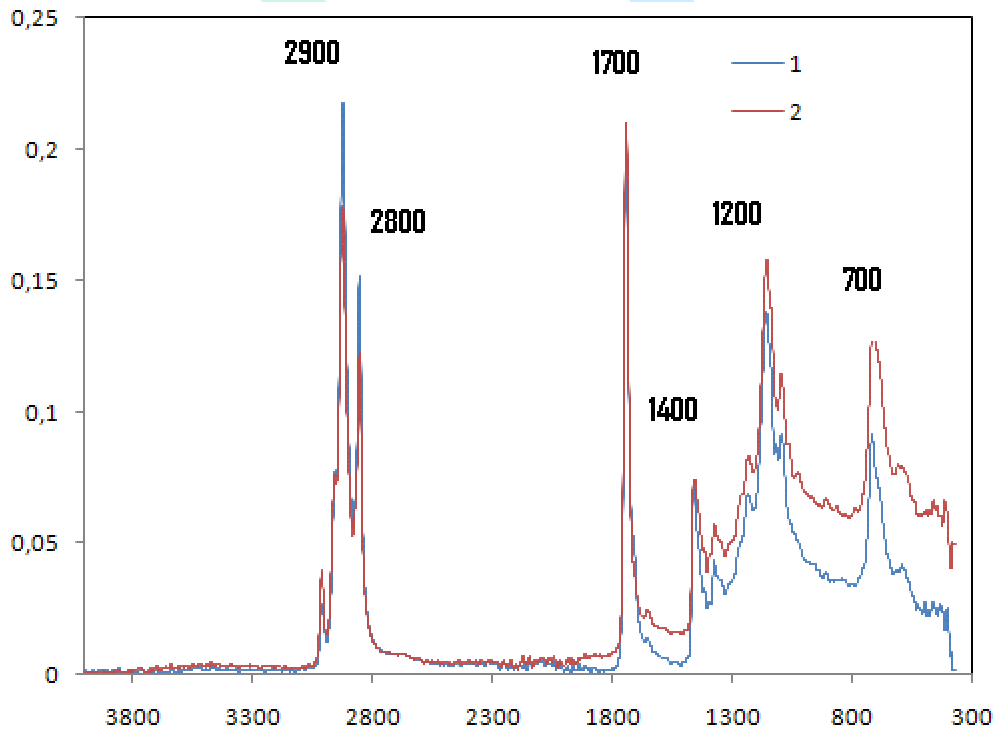

Figure 1: IR spectrum of oi, l (1) sesame, (2) flax.

Rancimat test:

This test can predict the oxidative stability of the oil and thus its shelf life. The results of the sample analysis (sesame oil and flaxseed oil) are shown in Figures (2 and 3). They are presented as graphs representing the induction time as a function of conductivity. The graph is in the form of a parabolic function. This trend is explained in the literature by the fact that volatile degradation products are trapped in distilled water, thus inducing the increase of the conductivity [20]. The induction period is determined from the inflection point of the conductivity curve. A shorter induction period means less oxidative stability. In our study, we observe that the induction periods vary from one sample to another. The shortest induction period was found for flaxseed oil (PI = 5h), while sesame oil has a 9-fold induction period (PI = 45h). Other authors have also found an induction time of 28.5 hours below our sesame oil [21]. According to the literature, the oil which contains a higher content of saturated fatty acids and a lower content of unsaturated fatty acids has a longer induction period. The richness of the oil in natural antioxidants (vit E) will also participate in the extension of the induction period, which is probably the case for sesame. A combination of several compounds is likely to be responsible for the good oxidation stability of sesame seed oil such as sesamol, sesamolin, sesamin, phenolic compounds and tocopherols [21].

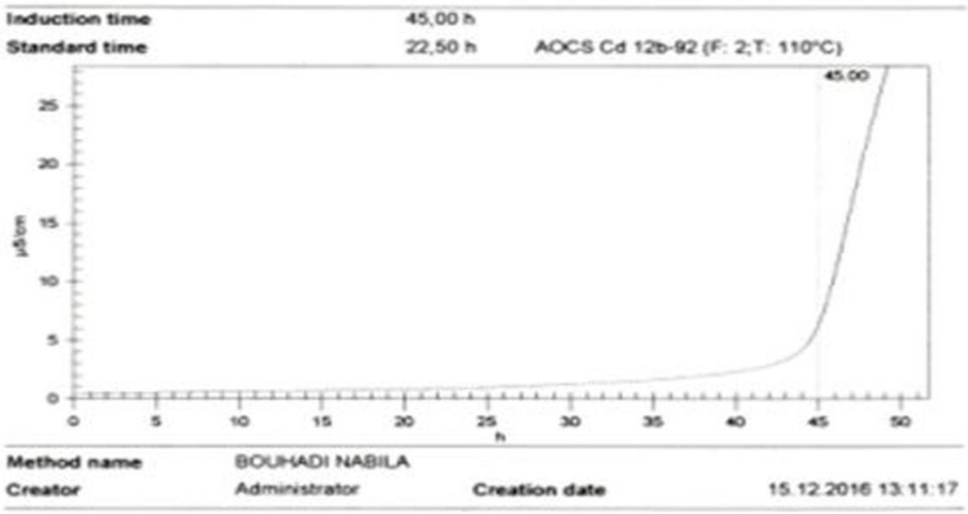

Figure 2: Oxidative stability curve in the Rancimat test of sesame oil

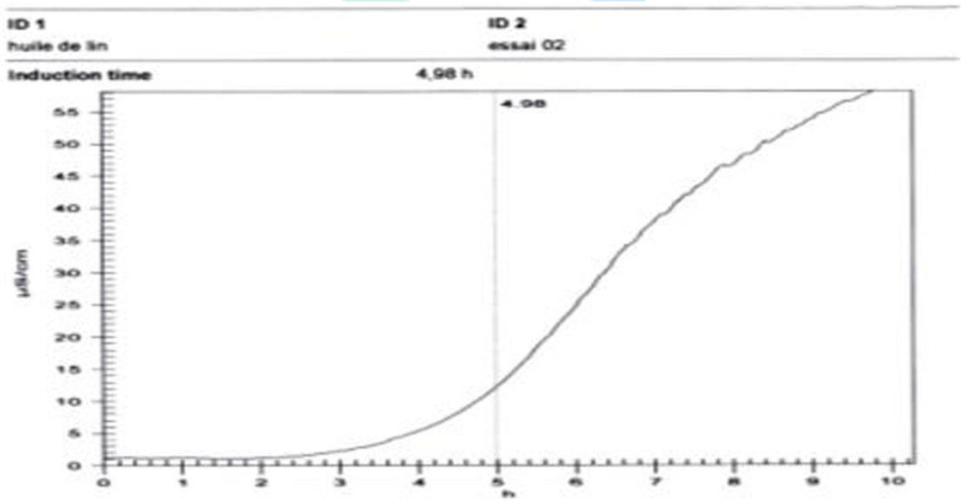

Figure 3: Oxidative Stability Curve in the Rancimat Test of Flax Oil

Fatty acid profile analysis (GC / MS):

Fatty acid analysis of two types of oil was performed using an Agilent quadrupole mass spectrometer coupled to a gas chromatograph equipped with a HP-5MS column. The operating conditions are as follows:

- Injector temperature (1:20 split mode): 250 ° C.
- Programming of the temperature: ° C for 2 min, 3 ° C / min up to 220 ° C., Isothermal for 15 min; 4 ° C / min up to 240 ° C., Isothermal for 5min.
- Vector gas used is Helium with a flow rate of 1.5 ml / min.
- Duration of analysis is 57 min.

The chemical composition of sesame and flaxseed oils is presented by the chromatograms (Figures 4 and 5) respectively. Chromatographic analysis allowed the identification of more than 13 compounds. In the obtained sesame oil, 5 fatty acids are identified: oleic acid is the main compound representing (33.44%) of the oil followed by linoleic acid (26.25%), stearic acid (9.59%) and palmitic acid (7.74%). These results are in agreement with the various studies showing that these fatty acids are the main compounds of sesame oil and oleic linoleic acid are the most important [7, 9].

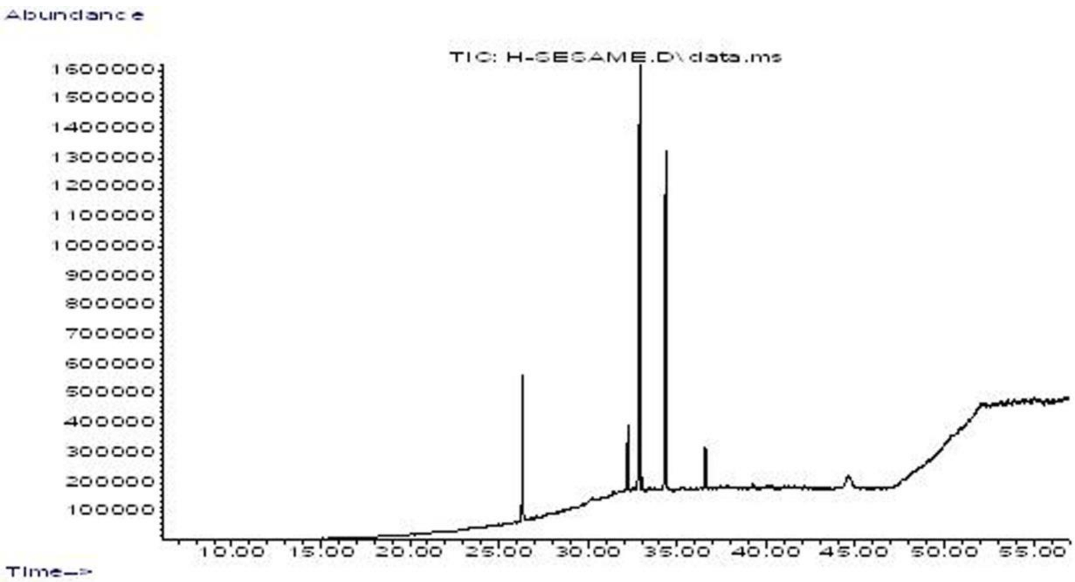

Figure 4: Chromatogram of sesame oil

The chemical composition of flaxseed oil is marked by the presence of linolenic acid (14.81%) as the major constituent, which is consistent with the literature [4], oleic acid (11.38%), linoleic acid (13.40%), lauric acid (0.15%), myristic acid (0.60%) and palmitic acid (0.21%). Popa, Gruia [12] found that the major fatty acids are linolenic, oleic and linoleic acid. On the other hand, other studies have identified linolenic acid and stearic acid as major constituents of flaxseed oil, and linoleic acid and palmitic acid as minor compounds with 15 acids. bold were detected sum trace components.

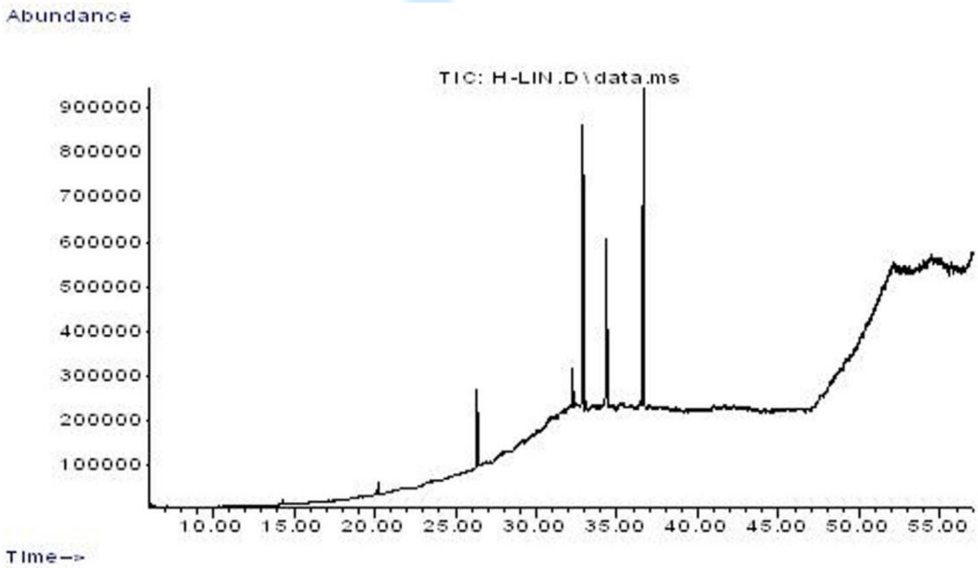

Figure 5: Chromatogram of linseed oil

Conclusion

Flaxseed and sesame seeds contain 42 and 50% oil respectively, which makes it possible to classify flax and sesame in the oilseed category. The sesame oil tested has on the one hand a relatively high acid number and on the other hand a low peroxide value. This index, whose values remain less than 10 meq O2 / kg of oil reflects a slight oxidation, which is confirmed by the low presence of conjugated dienes (K232) and secondary oxidation compounds (K270). On the other hand, flaxseed has a higher than standard acid number with the presence of secondary oxidation compounds (K270≈4). The FTIR profiles show that the two oils contain the same functional groups. The examination of the accelerated oxidative stability of linseed oil has a value 9 times lower than that of sesame oil. The fatty acid profile analysis identified 13 fatty acids in sesame oil and flaxseed oil. All the obtained results make it possible to affirm that the sesame oil extracted is of good quality whereas the linen oil requires a refining before being used.

References

1) Lecerf, J.-M., Les huiles végétales: particularités et utilités: Vegetable oils: Particularities and usefulness. Médecine des maladies Métaboliques, 2011. 5(3): p. 257-262.
2) Bekkouche. L , A.Y.D., La consommation de graines de lin associée à l'adoption du régime méditerranéen améliore l'hyperglycémie et la pression artérielle chez des patients de l'ouest algerien presentant un syndrome métabolique. . Annales de cardiologie et d'angéiologie, 35es Journées de l'Hypertension Artérielle 9th International Meeting of the French Society of Hypertension 2015. 64: p. S23-S36.
3) Laissouf, A., N.A.M. Soulimane, and H. Merzouk, L'effet thérapeutique de l'huile de lin «linumusitatissimum» sur l'hypertriglycéridémie et l'hypercholestérolémie chez des rats obèses âgés. Afrique Science: Revue Internationale des Sciences et Technologie, 2014. 10(2).
4) Benmehdi, H., et al., Physico-chemical screening of algerian linseed oil and characterization of their free acids methyl esters (Fames). 2014.
5) Anilakumar, K.R., et al., Nutritional, medicinal and industrial uses of sesame (Sesamum indicum L.) seeds-an overview. Agriculturae Conspectus Scientificus (ACS), 2010. 75(4): p. 159-168.
6) Ogbonna, P. and S. Ukaan, Chemical composition and oil quality of seeds of sesame accessions grown in the Nsukka plains of South Eastern Nigeria. African Journal of Agricultural Research, 2013. 8(9): p. 797-803.
7) Gharby, S., et al., Chemical characterization and oxidative stability of seeds and oil of sesame grown in Morocco. Journal of the Saudi Society of Agricultural Sciences, 2015.
8) Borchani, C., et al., Chemical characteristics and oxidative stability of sesame seed, sesame paste, and olive oils. Journal of Agricultural Science and Technology, 2010. 12: p. 585-596.
9) Nzikou, J., et al., Chemical composition on the seeds and oil of sesame (Sesamum indicum L.) grown in Congo-Brazzaville. Advance Journal of Food Science and Technology, 2009. 1(1): p. 6-11.
10) Belaid, D., La production des oleagineux en Algerie Collection dossiers agronomiques 2015.
11) Rombaut, N., Etude comparative de trois procédés d'extraction d'huile: aspects qualitatifs et quantitatifs: application aux graines de lin et aux pépins de raisin, 2013, Compiègne.
12) Popa, V.-M., et al., Fatty acids composition and oil characteristics of linseed (Linum Usitatissimum L.) from Romania. Journal of Agroalimentary Processes and Technologies, 2012. 18(2): p. 136-140.
13) Mohammed, M. and Z. Hamza, Physicochemical properties of oil extracts from Sesamum Indicum L. seeds grown in Jigawa state–Nigeria. Journal of Applied Sciences and Environmental Management, 2008. 12(2).
14) Benitez-Benitez, Ricardo, R.A. Ortega-Bonilla, and J. Martin -Franco, Comparison of two sesame oil extraction methods: percolation and pressed Biotecnologímethods:ector Agropecuario y Agroindustrial, 2016. 14(1): p. 10-18.
15) López Téllez, G., E. Vigueras-Santiago, and S. Hernández-López, Characterization of linseed oil epoxidized at different percentages. Superficies y vacío, 2009. 22(1): p. 05-10.
16) Kandhro, A.A., et al., Application of attenuated total reflectance Fourier transform infrared spectroscopy for determination of cefixime in oral pharmaceutical formulations. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 2013. 115: p. 51-56.
17) Elzey, B., D. Pollard, and S.O. Fakayode, Determination of adulterated neem and flaxseed oil compositions by FTIR spectroscopy and multivariate regression analysis. Food Control, 2016. 68: p. 303-309.

18) Rohman, A. and Y.C. Man, Fourier transform infrared (FTIR) spectroscopy for analysis of extra virgin olive oil adulterated with palm oil. Food Research International, 2010. 43(3): p. 886-892.
19) Gouvinhas, I., et al., Discrimination and characterisation of extra virgin olive oils from three cultivars in different maturation stages using Fourier transform infrared spectroscopy in tandem with chemometrics. Food chemistry, 2015. 174: p. 226-232.
20) de Mendoza, M.F., et al., Chemical composition of virgin olive oils according to the ripening in olives. Food chemistry, 2013. 141(3): p. 2575-2581.
21) Gharby, S., et al., Analyse chimique et sensorielle de l'huile d'argane. Technologies de Laboratoire, 2011. 6(22).