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CHARACTERIZATION AND COMPARISON OF THE THREE CULTIVARS SEED OIL OF *OPUNTIA FICUS-INDICA* IN TUNISIA

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Abstract:

The Barbary fig is a plant of the arid and semi-arid areas that is able to adapt to poor soils and high temperatures. The cactus has interesting properties that grow and develop the culture promote its byproducts. This study was conducted on three cultivars of Opuntia ficus-indica are those of Zelféne, Sbeïtla and Kairouan. The study was made in order to carry out a description and comparison between cultivars. In terms of recovery methods, extraction and characterization of the oil was made from the seeds. The oil yields vary from 1.35 to 1.9% for Sbeïtla and Kairouan cultivars and reached 2.7% for the cultivar Zelféne. The major fatty acids present in the oil of the seeds are palmitic acid (C16: 0), stearic acid (C18: 0), linoleic acid (C18: 2) and oleic acid (C18: 1). However, the most dominant fatty acid is linoleic acid with 61.79 of contents; 57.19 and 58.92% respectively for Zelféne, Sbeïtla and Kairouan. As for the sterol composition, siosterol Beta is the major component of this oil whose content varies from 67.14% for the cultivar Sbeïtla and 81.18% for that of Zelféne. Seed of oil is also rich in vitamins (tocopherols) which γ-tocopherol is the largest reaching 57.98 mg / 100 g of oil.

Keywords: *Opuntia ficus-indica -Oil seeds - fatty acids - tocopherol - sterols*

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INTRODUCTION:

The arid and semi-arid areas are deficient for conventional farming systems because of the low pluviometry, the poverty of soils and high temperatures. However, the productivity of these dry zones can be optimized by the introduction of such species adapted such as *Opuntia ficus-indica*. The average efficiency by hectare for Arid zones varies from 8 to 11 t / ha while a culture presenting with irrigation and proper fertilization can reach 22 t / ha [1]. In Tunisia, the efficiences are of the order of 4 t / ha this is probably due to the use of a plant material little performing further to the absence of varietal selection and poor control of specific production techniques to the species [2]. The cultivated surfaces are estimated at 600,000 hectares [3], including 400,000 and 200,000 hectares spineless cactus prickly cactus. The Barbary fig has interesting properties that lead to develop the culture and value in products of this plant. Moreover, this plant has been the subject of several studies showing the possibility of producing alcoholic drinks, nectars, juices and jams from the fruit pulp [4]. Besides, the chemical characterization of the Barbary fig fruit made by Piga ,2004 [5] allowed to accentuate its nutritional value and its technology function. Seeds have a means of recovery through production of oil. His characterization was the motive for the study carried by Ennouri et al [6], that are interested in the study of the fatty acids composition and physicochemical parameters of the oil from the seeds of *Opuntia*. Barbary fig seeds showed in recent years a lot of interest following the exemple of the other seeds especially those grapes and studies have multiplied to characterize their constituents to estimate their nutritional value. According to Uchoa *and al* [7], the protein reserves of the seed are albumin. However, the attention has focused especially on oils contained in the seeds. It is composed, from the fatty-acid point of view, mainly of the linoleic acid and of oleic acid and it presents point of view of composition a great similarity with the oil of maiis [8, 9].

MATERIAL AND METHODS:

1. Extraction and Oil Content

The extraction of oil from the seed is produced by cold pressing. The seeds once dried in the open air will be pressed by cold pressing through a press of the SMIR brand.

The mass percentage of the oil contained in the prickly pear seed is calculated using the following formula:

Oil % = m oil / m seeds x 100

With:

m_{oil} is the mass of oil the oil found

m_{seeds} is the mass of seeds in the sample of dry seed

2. Oil purity Analysis

2.1. Fatty acid composition

The fatty acid composition of the oils extracted was determined by chromatography in gas phase GPC of NT 118.04 (1986) standard.

• Preparation of methyl esters

Take a trial grip about 0.5 g and introduce it into a test tube, add 0.5 ml of a methanol solution of potassium hydroxide 2 N. After stirring, add N-heptane, shake and we decant. The methyl esters collect r in the heptane phase superior.

• Chromatographic analysis

The methyl esters were analyzed using a chromatographic system, gas chromatography (GC), with the following characteristics.

2.2. Sterol Composition

The seed oils of the sterol composition was determined by gas phase chromatography GPC according to ISO 6799 .1991 NT standard. We begin with Saponification of a test sample, extraction of the unsaponifiable and isolation of sterols from unsaponifiable by Thin Layer Chromatography (TLC). The analysis of sterols isolated by Gas Chromatography Capillary Column on (CPGCC).

2.3. Tocopherol Content

The determination of vitamins (tocopherols) of the prickly pear oil was made by liquid chromatography high performance HPLC-Fluorescence according to EN 12822 standard.

3. Oils identification Indices

3.1. Refractive Index

This is measured using an Abbe's refractometer, the index of refraction of a liquid sample to a specified temperature. Calibrating the device and then measure the sample refractive index for a test portion at a temperature of 20 ° C

Expression of results:

$$(I.R)_{Tréf} = (I.R)_{T1} + (T_1 - T_{réf}) \, x \, F$$

With:

T ref is the reference temperature 20C

T1 is the temperature measurement

F is equal to 0.00035 correlation factor for oils.

3.2. Criterion of Saponification

Boiling a sample reflux with an ethanolic solution of potassium hydroxide and titration of the excess of potassium hydroxide with a standard solution of hydrochloric acid. Insert 2g of fat into an erlenmeyer of 250ml, add it 25 ml of KOH. Adapt to the cooler and connect to the refrigerant flow of cold water. Heat for half an hour and then remove the erlenmeyer and cooled under a tap water stream. To add two drops of phenolphthalein and pull the potassium hydroxide in excess by 0.5N of

hydrochloric acid and note the volume of acid V. To make in parallel a blank test by replacing oil with distilled water or the V0 volume of acid used for the blank.

Saponification number expressed as milligrams of potassium hydroxide required to saponify 1g of fat is given by the following formula:

$$\mathbf{SN} = \frac{(V_0 - V)xCx51.6}{m}$$

With:

V0 is the volume in milliliters of the hydrochloric acid solution used for the blank

V is the volume in ml of hydrochloric acid solution used for determining

m is the mass in grams of the test sample (value presides 10-1 g meadows)

C is the concentration in moles / liter hydrochloric acid

4. Oils Quality Indices

4.1. Acid Number

Dissolving the sample in ethanol and then titration of free fatty acids with a ethanolic solution of sodium hydroxide.

Introducing a test sample of 5 g of oil in a flask. Dissolve taking fat test in 100 ml of the mixture of diethyl ether / ethanol (v / v) previously neutralized. Titrate while stirring with the standard solution of sodium hydroxide 0.1 mol / l until the indicator changes (the pink color of the phenolphthalein persisting for at least 10 s).

The acid number is equal to:

$$\mathbf{AN}=\frac{282xVxC}{10xm}$$

With:

V is the volume in milliliters of standard solution of sodium hydroxide used

C is the actual concentration in moles / liter of the solution of sodium hydroxide used

m is the mass in grams of the test sample

282 is the molar mass in g / mol of sodium hydroxide

4.2. Peroxide Index

A trial grip in solution in a mixture of acetic acid and some chloroform is treated with a potassium iodide solution. The liberated iodine is titrated with a standard solution of sodium thiosulfate. Introducing a trial grip of 2 g of oil in a Stoppared bottle. Add 10 ml of chloroform. Rapidly dissolve the fat with stirring. Add 15 ml of acetic acid and 1 ml of saturated potassium iodide solution. Block immediately the flask, shake it during a minute and then put it in the dark for 5min. Then add 75 ml of distilled water .Titrate with vigorous stirring and in the presence of starch as an indicator, the liberated iodine with sodium thiosulfate solution 0.01 N until the blue color disappears. Rate V the added sodium thiosulfate volume.

Perform parallel to the first test a blank test without the fat. Let V0 be the volume of thiosulfate added.

The peroxide index expressed in milliequivalents of active oxygen per kilogram of fat is given by the equation:

$$\text{P.i} = \frac{1000xCx(V-V_0)}{m}$$

With:

C is the concentration in moles / liter of the sodium thiosulphate solution

V0 is the volume in ml of the sodium thiosulphate solution used for the blank

V is the volume in ml of sodium thiosulphate solution used for determining

m is the mass in grams of the test sample

4.3. Specific Extinction

The oil was dissolved in cyclohexane (solvent) and then the absorbance is measured at the prescribed wavelength relative to the solvent. Introducing a test sample of 0.25 g of oil in a 25ml vial. Dissolve the sample in pure cyclohexane for spectrophotometry and make up to the mark and mix thoroughly and read the absorbance at 232 nm, 266 nm, 270 nm and 274 nm. The results are expressed as the specific absorbance of the solution of the fatty substance in a concentration of 1 g / 100 ml (1%) measured using an optical path of 1 cm; a ʎ wavelength is given by the formula:

$$E\ (\lambda) = \frac{A\ (\lambda)}{W}$$

With:

A (ʎ) is the absorbance at the wavelength ʎ

W is the concentration (g / 100ml) of the sample of the test solution

5. Statistical Data Processing

The various parameters studied were analyzed by means of the Statistica software (version 7). The set of measurements has been an analysis of variance using one factor ANOVA procedure with the comparison option medium LSD. The significance of the variance was verified by the Fisher test at the 5%. Excel software (2007 version) was used for the construction of the graphics.

RESULTS AND DISCUSSION:

1. Oil Content

The oil content of prickly pear seed is low. Indeed, the Zelféne cultivar present the highest yield (2.70%), followed by the cultivar Kairouan (1.90%) and the cultivar Sbeïtla (1.35%) (Table 1). The oil extraction work of prickly pear seed by the solvent method performed by Touil-Abdellaoui and Ennouri et al. [10, 6] showed higher values respectively 4.5 and 10.9%. This appears to be due in part to the extraction method, and secondly to the cultivar.

2. Analysis of purity of the oil

2.1. Fatty acid Composition

The fatty acid composition of oil samples of three seeds Barbary fig cultivars of regions Zelféne, Sbeïtla and Kairouan was determined by chromatography in gas phase. The results for identification of fatty acids and their proportions in the seed oils of the three cultivars are summarized in Table 2. The linoleic acid (C18 .2) appears to be the major fatty acid in the oil with a 61.79% proportion for the cultivar Zelféne and oleic acid (C18 .1) with 26.52% for the cultivar Sbeïtla, palmitic acid with 12.34% for the region Zelféne and stearic acid with a proportion greater than 3%. All other fatty acids may be minor constituents of the oil. The Zelféne cultivar presents the highest content in polyunsaturated fatty acids as well as PUFAs / AGS high ratio. These results are similar to those found in 2005 by Touil-Abdellaoui and Ennouri and al [10, 6]. who showed that linoleic acid, oleic acid, palmitic acid and stearic acid are the major fatty acids present in the oil of Opuntia ficus indica seeds with the following contents 60, 70.3; 20, 16.8; 13, 9.32; and 3, 3.11% respectively

2.2. Sterol Composition

The sterol composition of oil samples of seeds Barbary fig of three cultivars of regions Zelféne Sbeïtla and Kairouan was determined by chromatography in phase gas. The results relative to the identification of sterols and their proportions in the seed oils of the three cultivars are summarized in Table 3. All samples of the oils of seeds of the three cultivars studied have a content of Beta siosterol highest compared with that of other sterols. Indeed, a value of 81.18% was recorded for the cultivar Zelféne. Campesterol and Stigma sterol are present with lower contents are 13.15 and 3.27% for the cultivar Kairouan. These results agree with those of Ramadan and Morsel [11]. who showed that the Beta siosterol, campesterol and Stigmasterol are the major components of the oil from the seeds of Barbary fig tree.

2.3. Tocopherol Content

Tocopherols have a vitamin power and act as antioxidant. The tocopherol content of the seed oil is shown in Table 4. The oil of the three cultivars contain the however γ-tocopherol and δ-tocopherol is present only for the cultivar Kairouan. The α-tocopherol has the lowest content (0, 95-1,71mg / 100g), while γ-tocopherol is the dominant compound of this oil for three regions where it reached a value of 57.98 mg / 100g (for the cultivar Zelféne). The δ-tocopherol is present only for the cultivar Kairouan (0.26 mg / 100g). The results published by Matthäus and Özcan (2011) [12] show that the studied Barbary fig oils contained only the γ-tocopherol with grades ranging between 3.9 and 50 mg / 100 g. While the work of Feugang [13] and Ramadan and Morsel [11] show that α, β, γ and δ-tocopherol is present in the oil of the seeds with the dominance of the estimated γ tocopherol 33 mg / 100 g.

3. Oils identification Criteria

Identifying indicia of fats are the refractive index, the Saponification number.

3.1. Refractive Index

The refractive indices of the oil from the seeds of the three Barbary fig cultivars are studied for Zelféne 1.4744, 1.4742 and 1.4746 for Sbeïtla Kairouan (Table 5). These results are comparable to those found by Ennouri et al. (2005) showing that the refractive indices of the oil from *Opuntia ficus indica* seeds are in the range of 1.475 and 1.469 for *Opuntia ficus stricta.*

3.2. The Saponification

Saponification values of the oil from the seeds of the studied cultivars are to Zelféne 196.27, 187.07 and 179.80 of Sbeïtla and for Kairouan respectively (Table 5). Ennouri et al. [6] indicate that the saponification index of the oil of *Opuntia ficus indica* seeds and *Opuntia ficus stricta are* respectively in the order of 169 and 174.

4. Oils Quality Indices

Oil quality indices are the acid number, peroxide index and specific extinction.

4.1. Acid Number

The change in acid number of Barbary fig oil (Table 6) showed that this cultivar Zelféne the lowest acid value of 0.503, followed by the Kairouan cultivar 0.630 and that of Sbeïtla of 0.671. These acid number values are comparable to those of olive oil obtained by the Chemlali variety B'chir (2007) [13]. Which are of the order of 0.6%.

4.2. Peroxide Index

The lowest peroxide index recorded was 3.474 meq O2 / kg for the cultivar Sbeïtla, it is 4.136 meq O2 / kg for the cultivar Zelféne and the highest index of order of 5.588 meq O2 / kg for the cultivar Kairouan (Table 6). The peroxide index values of the Barbary fig oil obtained are much lower than those found by Mabrouk[16], where the index the lowest recorded peroxide is 8.30 meq O2 / kg in olive oil.

4.3. Specific Extinction

The Knowledge of the specific extinction allows to have an idea on the state of oxidation of the oil studied. The measurement of the specific extinction in the ultraviolet is two wavelengths in 232nm (K232) and 270nm (K270). Indeed, the specific extinction at 232 nm provides information on primary oxidation of the oil while that at 270 nm provides information on secondary oxidation. Table 6 shows that the oil which has the most oxidized state is the cultivar Sbeïtla whose specific

extinction values at 232 and 270nm respectively are of the order of 2.799 and 0.489 and of a Delta K ± 0.006 (tableau23). Salvo et al. (2002) [15], found the oil of Barbary fig seeds of K232 specific extinction values of the order of 3.13, and K270 in the range of 0.22 and Delta K ± 0, 01. These values are close to those found in this work.

CONCLUSION:

To characterize and value this culture, this study was realized on three Barbary fig cultivars to know Zelféne, Sbeïtla and Kairouan. The comparative photochemical study focused on the three Barbary fig cultivars allowed the development and characterization of the oil extracted from its seeds. The oil yields vary for Sbeïtla and Kairouan cultivars but cultivar Zelféne appeared the best. The major fatty acids present in the oil of the seeds are palmitic acid (C16: 0), stearic acid (C18: 0), linoleic acid (C18: 2) and oleic acid (C18: 1). However, the most dominant fatty acid is linoleic acid. As for the composition in sterols, beta siosterol is the major component of this oil. The oil of seeds is also rich in vitamins (tocopherols) which γ-tocopherol is the most important. It is also important for further research on this plant to take avantages of its virtues and direct them to the production of bioethanol as an energy source seen of the wealth of this fruit into fermentable sugars.

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Table 1: Oil content of Barbary fig seeds

region	Oil content
Zelféne	2,70%
Kairouan	1,90%
Sbeïtla	1,35%

Table 2: Composition of saturated fatty acids, mono- and polyunsaturated

Fatty acids	Zelfène (%)	Sbeitla (%)	Kairouan (%)
lauric acid (C12 .0)	-	-	-
Myristic acid (C14 .0)	-	-	-
Palmitic acid (C16 .0)	12.34	11.38	11.39
Palmitoleic acid (C16 .1)	0.65	0.62	0.64
Heptadecanoic acid (C17 .0)	-	-	-
Heptadecanoic acid (C17 .1)	-	-	-
Stearic acid (C18 .0)	3.28	3.68	4.17
Oleic acid (C18 .1)	21.45	26.52	24.31
Linoleic acid (C18 .2)	61.79	57.19	58.92
Linolenic acid (C18 .3)	0.22	0.20	0.23
Arachilique acid (C20 .0)	-	0.37	0.23
Total acides gras saturés AGS	15.87	15.43	15.79
Total acides gras monoinsaturés AGMIS	22.1	27.14	25
Total acides gras polyinsaturés AGPIS	62.01	57.39	59.15
AGPIS/ AGS	3.90	3.71	3.74

Table 3: Sterol Composition

Sterol	Zelfène (%)	Sbeitla (%)	Kairouan (%)
Cholesterol	-	-	-
Campesterol	11.74	6.93	13,15
Stigmasterol	2.14	1.17	3,27
Chlestosterol	1.90	22.53	-
Beta siosterol	81.18	67.14	80,55
Delta 5 avenasterol	-	-	-
Delta 7 stigmasterol	1.12	0.96	1,20
Delta 7 avenasterol	1.90	1.25	1,811
Erythrodiol	-	-	-
Uvaol	-	-	-

Table 4: Content of Tocopherols

Tocopherol (mg/100g d'huile)	Zelfène	Sbeïtla	Kairouan
α-tocopherol	0.95	1.71	1.23
β-tocopherol	-	-	-
γ-tocopherol	57.98	38.6	48.18
δ-tocopherol	-	-	0.26

Table 5: Identification Index Fats

Region	Refractive index	Saponification index
Zelféne	1,4744	196,27
Sbeïtla	1,4742	187,07
Kairouan	1,4746	179,80

Table 6: Quality Indices Oil of Barbary Fig Seeds

Region	Acid number	(méqd'o2/kg)	Peroxyde value k232	K270	ΔK
Zelféne	0,503	4,136	2,301	0,255	0,005
Sbeïtla	0,671	3,474	2,799	0,489	0,006
Kairouan	0,630	5,588	2,345	0,289	0,006