

CHEMICAL COMPOSITION AND ANTIOXIDANT ACTIVITY OF LIPIDS FROM *FICUS CARICA* L. FRUITS

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Abstract. Samples of oils from seven types of *Ficus carica* L. fruits from Algeria were investigated through determinations of their chemical characteristics, quantifying sterols and tocopherols, and analysis of the fatty acids profiles using gas chromatography, and evaluation of antioxidant activity by 1,1-diphenyl-2-picrylhydrazyl (DPPH) method and total antioxidant activity (TAA) using phosphomolybdenum methods. The results show that acid values ranged from 3.14 to 6.95 mg KOH/g indicating on the high amount of free fatty acids in the figs oils. Neutral lipids occupy a very important proportion of the crude figs oils (NL: 60.30–98.40%) compared to glycolipids (GL: 0.58–28.00%) and phospholipids (PL: 0.40–11.70%). Linoleic (11.70–34.74%) and linolenic (1.15–35.27%) were the major unsaturated fatty acids found in lipid fractions, while the main fraction of saturated fatty acid was palmitic. The tocopherols and sterols contents in fig oils ranged from 14.27 to 108.55 mg α -TE/g lipids and from 0.36 to 2.80 mg CE/g lipids respectively. The best inhibition concentration (IC_{50}) of DPPH antioxidant activity was marked by GL (0.23–1.06 g/L) and PL (0.67–1.23 g/L). The strongest TAA was also marked by GL (IC_{50} : 2.84 to 10.08 g/L) and PL (IC_{50} : 3.73–11.30 g/L). This finding demonstrated for the first time that the studied figs oils possessed good antioxidant activity which may be associated with their alleged health benefits.

Keywords: oil extract, fatty acid, tocopherol, sterol, antioxidant activity.

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Introduction

Ficus carica L. (family: Moraceae) is one of the oldest known trees in the world, native to Africa, Asia and Europe and today it is spread almost on all continents [1]. Different parts of the tree were traditionally used in the diet or as treatment for some illnesses [2]. The fruits occurring from this tree contain nutrient elements such as lipids, sugars, proteins, fibres, water, vitamins (ascorbic acid, thiamine, riboflavin, niacin, pyridoxine, pantothenic acid, folate, vitamin A, β -carotene, α -tocopherol and phylloquinone) and minerals (including Na, P, K, Ca, Mg, Zn, Fe, Cu and Mn ions) [3]. Due to these properties, recent research studies have focused on studying both primary and secondary metabolites of fig fruits. Some researchers investigated the influence of figs' nutrients by the changes of the physico-chemical characteristics, and the effect of drying of figs on the contents of sugars, organic acids, and phenolics [4,5]. Other

studies have focused on the optimization of ultrasound-microwave assisted extraction of pectin from figs and the optimization using response surface methodology to optimize recovery of total phenolic compounds and preservation of the antioxidant properties [6,7]. Recently, many studies of oil extraction and fatty acids composition of total lipids from fig fruits have been reported [8-11]. However, there is no information on the chemical composition of different lipids class (neutral lipids, glycolipids and phospholipids) of the fruits.

Thus, the present paper aims to investigate the chemical composition of the lipids class and antioxidant activity of oils from some figs (green and dark) from Algeria, which ranks fourth in the list of top ten global producers of figs [12]. The objectives were set to study the tocopherols and sterols contents of oils from figs fruits, to study their antioxidant activities and to identify the fatty acids using gas chromatography (GC)

analysis. This study, could help extending the biomedical applications of the oil extracts, including the use of fig's fatty acid in pure medicine instead of traditional uses or helping industries to produce fig oil that lead to more fig trees planting.

Experimental

Reagents

Vitamin C, hexane, ethanol, methanol, sodium sulphate, 1,10-phenanthroline, butylated hydroxyanisole (BHA), α -tocopherol (vitamin E), and butylated hydroxytoluene (BHT), chloroform, acetic anhydride, acetic acid, sulphuric acid, iron(III) chloride, hydroxide potassium, DPPH (2,2-diphenyl-1-picrylhydrazyl), sodium chloride, BF₃, phosphate sodium, cholesterol and molybdate ammonium were obtained from Sigma-Aldrich (St. Louis, M.O., U.S.A.), Riedel de Haën (Steinheim, Germany) and Prolabo (Leuven, Belgium), silica gel 60 Å 70-230 mesh, 63-200 µm for column chromatography was acquired from Fluka Analytical. Fatty acids pure standards (capric acid (C10:0), lauric acid (C12:0), myristic acid (C14:0), palmitic acid (C16:0), arachidic acid (C20:0) palmitoleic acid (C16:1), vaccenic acid (C18:1), linoleic acid (C18:2), linolenic acid (C18:3), and gondoic acid (C20:1)) were purchased from Sigma-Aldrich. The chemicals used were of analytical reagent grade.

Plant material

Seven samples of figs fruits were studied, which were chosen from different parts of Algeria; Blida and Tipazza in the North, Mostghanem in the west and from three different regions in Laghout (Tadjmout, Assafia, and Ksar-el-Hirane) situated in the south. Figs were collected between June and September and they were dried in the dark and at room temperature. For simplification, the following abbreviations have been adopted for the figs denomination: F1- fig of Ksar-el-Hirane, F2 - fig of Assafia, F3 - fig green of Tadjmout, F4 - black fig of Tadjmout, F5 - fig of Blida, F6 - fig of Tipazza, F7 - fig of Mostghanem.

Extraction procedure of oil from figs fruits

The oil extracts were obtained from seeds of figs fruits according to the Folch method [13], which consists in extracting the lipids with a mixture of chloroform:methanol (2:1, v/v), the mixture was dehydrated using anhydrous sodium sulphate and filtered. Afterwards, the solvents were evaporated under vacuum using a rotary evaporator, at 40°C. The resulting lipids were weighted and stored at 4°C until the analysis.

Separation of lipids fractions from figs oil

Total lipids (TL) were separated into neutral lipids (NL), glycolipids (GL) and phospholipids (PL). Column chromatography was used for the separation of lipids. At the beginning, 5 g of silica gel in chloroform was prepared, the mixture was introduced into the column and then an amount of 5 g of crude oil brought into contact with few millilitres of chloroform in the column. Different pure solvents were used for the elution of the lipids: chloroform which allowed eluting the neutral lipids, then the acetone which allowed eluting the glycolipids and finally the methanol to recover the phospholipids [14]. After drying the eluates with sodium sulphate anhydrous and filtration, the solvents were evaporated under reduced pressure at 40°C.

Determination of the chemical indices of the oil extracts

Acid value (AV), saponification value (SV) were determined according to Cd 3d-63 and Cd 3a-94 norms respectively as described by the procedure of American Oil Chemists' Society [15].

Quantification of total tocopherols

The total tocopherols were quantified according to the method of Emmerie-Engel [16]. Briefly, 1 mL of *o*-phenanthroline (0.4 %) was added to 1 mL of diluted oil (in ethanol) extract and then, 0.5 mL FeCl₃ (0.12 %) were added to the mixture. After 3 min of incubation in dark, the absorbance was measured at 510 nm on a UV-VIS-1800 Shimadzu spectrophotometer. The α -tocopherol was used as the standard for the calibration curve, and the results were expressed as mg α -tocopherol equivalent per gram of lipids (mg α -TE/g lipids).

Quantification of sterols

The sterols content was determined according to Lieberman-Burchard assay [17,18]. Briefly, a mixture made up of 1 mL of diluted oil solutions (in ethanol) with 2 mL of Lieberman-Burchard Reagent (60 mL anhydrous acetic + 10 mL concentrated sulphuric acid + 30 mL acetic acid). After 30 min of incubation in the dark, a green colour formed and the absorbance was measured at 550 nm. The calibration curve of cholesterol was drawn and the results were expressed as mg cholesterol equivalent per gram of lipids (mg CE/g lipids).

Gas chromatography analysis

The fatty acid composition was determined by GC after conversion to fatty acid methyl ester (FAME) by an acid-catalysed esterification method using a boron trifluoride-methanol complex 10% w/v [19]. A Delsi gas

chromatograph, equipped with a flame ionization detector and a Mega 10 column (25 m \times 0.25 mm *i.d.*, 0.25 μ m film thickness) was used for FAME determinations. The GC conditions were set as follows: initial oven temperature (150°C) heating rate 2°C/min, final temperature (200°C), injection port temperature (250°C), detector port temperature (250°C), hydrogen gas flow 30 mL/min, air flow 300 mL/min and helium gas carrier flow 1 mL/min. The injection volume was 0.1 μ L. The FA (fatty acids) were identified by comparing their retention times with those of pure standards.

Antioxidant activity evaluation

The DPPH radical scavenging activity assay

DPPH[•] antioxidant activity was determined spectrophotometrically according to the method of Brand Williams [20], in which 1 mL of oil extract diluted in butanol was mixed with 1 mL of 250 μ M methanolic DPPH[•] solution. This mixture was incubated for 30 min in the dark and the absorbance was measured at 517 nm.

The total antioxidant activity (TAA)

TAA was evaluated using the phosphomolybdate method that measures the reduction of Mo(VI) to Mo(V) using the reducing agent from plant materials [21]. The reaction solution was obtained by mixing 1 mL of tested samples dissolved in dimethyl sulphoxide, with 2 mL of a reagent constituted of 0.6 M sulphuric acid, 28 mM sodium phosphate and 4 mM ammonium molybdate. The test tubes were capped and incubated in a water bath at 70°C for 90 min. Afterwards, tubes were cooled down at room temperature and the absorbance was measured at 695 nm.

The result of DPPH antioxidant activity and the TAA were expressed as IC_{50} (concentration providing 50% inhibition) values that were calculated from the plotted graph of percentage of inhibition against the concentrations of the samples (g/L). Positive controls, ascorbic acid, BHA, BHT, and α -tocopherol, were also used in both tests.

Statistical analysis

All experiments were carried out in triplicate and the results are presented as means \pm SD (standard deviation) calculated using Microsoft Office Excel 2016. The significance of the differences among means was evaluated according to Tukey post hoc test ($p < 0.05$).

Results and discussion

Physicochemical characteristics of fig oil

Ficus carica oils had an olive-green colour, and were liquid at room temperature. The oils

yield obtained from the seven samples (F1, F2, F3, F4, F5, F6 and F7) ranged from 0.99 to 7.25%, where the highest yield of 7.25% was obtained for F3 whilst all figs of Laghouat region had the yield higher than $1.5 \pm 2\%$ (Table 1). The acid value (AV) ranged from 3.14 to 6.95 mg KOH/g. The AV measured the content of free fatty acids in our oils, the highest amount of AV indicates the high concentration of fatty acids, and should be noted that it reduces the shelf life of the fruit due to the high lipolytic activities [22]. In comparison to other fruit samples studied in the lab such as date fruit seeds ($AV \leq 1.38$) and *Pistacia atlantica* fruit ($AV = 4.8$) [23,24], figs oils have higher AV which led to a shorter period of storage. On the other hand, the saponification value (SV) ranged from 146.10 to 199.25 mg KOH/g, and figs F3, F4 and F5 had the highest SV (>190 mg KOH/g).

Table 1

Physicochemical properties of figs oil.			
Samples	Oil (%)	AV (mg KOH/g)	SV (mg KOH/g)
F1	2.51 \pm 0.01 ^{ae}	3.66 \pm 0.01 ^{abcd}	155.88 \pm 0.37 ^{abc}
F2	2.13 \pm 0.56 ^a	3.14 \pm 0.12 ^{abcde}	160.56 \pm 0.28 ^{ab}
F3	7.25 \pm 0.80 ^{abcde}	4.58 \pm 0.13 ^{abc}	193.61 \pm 0.50 ^c
F4	1.48 \pm 0.21 ^c	6.59 \pm 0.04 ^{ad}	199.01 \pm 0.55 ^{bde}
F5	0.99 \pm 0.01 ^{be}	6.15 \pm 0.13 ^c	199.25 \pm 1.26 ^{ae}
F6	2.82 \pm 0.10 ^{bd}	6.45 \pm 0.04 ^b	173.67 \pm 0.58 ^d
F7	1.20 \pm 0.11 ^d	5.37 \pm 0.02 ^d	146.10 \pm 0.40 ^e

*Abbreviations: AV -acid value; SV -saponification value.

Note: Values with different lowercase superscript letters are significantly different ($p < 0.05$) according to Tukey post hoc test

Lipid fractions separated from figs oil

The fig fruit was found to have high total lipid contents in comparison to other fruits such as apple, grapes and citrus [25]. This can be explained by the fact that fig fruit contains a massive number of small seeds rich in oil.

In fact, this oil contains a huge number of lipid substances [25]. The obtained oils in this study are separated into neutral lipids (NL), glycolipids (GL) and phospholipids (PL). Table 2 illustrates that the NL were quantitatively the largest fraction of lipids, their content ranged from 60.30% to 98.40% followed by GL and PL. F2 contained the highest amount of both PL (11.70%) and GL (28%) but afford the lowest NL. On the other hand, F7 contained increased amount of NL but small amount of both GL and PL. The obtained results were in line with those reported previously and it was found that among the total lipids the NL were the predominant class [19].

Generally, the PL are unsaturated lipids and their contents in fruits and vegetables were usually low in order to reduce oxidation and extend the shelf life of the fruits or vegetables [26].

Table 2

Samples	Lipid classes of fig oil (g/100 g oil).		
	NL	GL	PL
F1	82.00±2.00 ^e	9.50±1.01 ^{aej}	8.50±1.06 ^b
F2	60.30±1.08 ^{abc}	28.00±1.22 ^{abcdef}	11.70±1.20 ^a
F3	90.00±1.00 ^d	8.00±0.20 ^{df}	2.00±0.40 ^{abc}
F4	98.00±1.02 ^b	1.60±0.01 ^{be}	0.40±0.04 ^{abcd}
F5	81.25±2.00 ^f	10.40±1.11 ^{bf}	8.30±0.56 ^{cd}
F6	90.00±1.05 ^c	6.04±0.03 ^{ef}	3.96±0.38 ^{abd}
F7	98.40±3.12 ^a	0.58±0.01 ^f	1.02±0.01 ^b

*Abbreviations: NL -neutral lipids; GL -glycolipids; PL-phospholipids.

Note: Values with different lowercase superscript letters are significantly different ($p < 0.05$) according to Tukey post hoc test

Total tocopherols and sterols contents

The total tocopherols and sterols contents identified in the studied total lipids and neutral lipids are listed in the Table 3. First of all, the total tocopherols contents ranged from 5.97 to 108.55 mg α -TE/g lipids and the highest contents was in TL; the sterols contents ranged from 0.36 to 2.80 mg CE/g lipids in TL and from 0.16 to 0.5 mg CE/g lipids in NL. Both F1 and F2 had the highest contents of total tocopherols and sterols.

Table 3

The total content of tocopherols and sterols in figs oil.

Samples	The total content of tocopherols and sterols in figs oil.		
	Tocopherols (mg α -TE/g lipids)	Sterols (mg CE/g lipids)	
TL	F1	85.19±6.30 ^{ab}	2.40±0.05 ^{bd}
	F2	108.55±34.60 ^{abcdef}	2.80±0.06 ^{ad}
	F3	58.37±13.49 ^{bcef}	1.60±0.12 ^c
	F4	15.15±5.72 ^{bdf}	0.66±0.05 ^{ab}
	F5	14.27±1.18 ^{bd}	0.63±0.01 ^{ab}
	F6	26.50±3.08 ^{be}	0.36±0.03 ^{abc}
	F7	42.41±2.11 ^{bd}	0.50±0.04 ^d
NL	F1	32.40±23.47 ^a	0.22±0.02 ^b
	F2	28.15±6.64 ^b	0.50±0.04 ^a
	F3	17.35±14.80 ^{ad}	0.20±0.02 ^d
	F4	12.80±3.73 ^{abc}	0.20±0.04 ^e
	F5	12.35±18.40 ^{abc}	0.21±0.05 ^c
	F6	5.97±1.35 ^{abc}	0.16±0.04 ^a
	F7	21.12±1.24 ^c	0.19±0.02 ^a

*Abbreviations: TL -Total lipids; NL -Neutral lipids.

Note: The different letters in the same column of tocopherols and sterols contents show statistically significant differences among TL and NL samples according to Tukey post hoc test at $p < 0.05$.

The differences found among the samples may be due to regional differences and cultivar differences [19,27]. The registered values are much higher than those reported previously [28] with the total tocopherols and sterols contents in Dacula GA is 0.05 mg α -TE/g lipids and 13 mg CE/g lipids, respectively. Other researchers studied only the achenes of fig from Tunisia and they found a total sterols concentration equals to 10.61 mg CE/g lipids [8]. Another research on the Portuguese fig cultivars found that the major form of tocopherol (α , β , δ and γ) in the peel of figs was α -tocopherol (2.75 mg/100 g fruit weight). On the other hand, γ -tocopherol was the most abundant in the pulp (2.68 mg/100g fruit weight) and β -tocopherol was found in both samples but in low amount [29]. It has been proved that tocopherols have both powerful antioxidant activity and higher protective activity against cardiotoxicity [30]. Also, they can work as inhibitors of lipid peroxidation in different biological systems [30]. On the other hand, different studies found that sterols are important to verify authenticity, adulteration detection, varietal characterizations and as indicators of harvest time [31]. Moreover, studies suggest that a diet containing sterols can lower the risk of cancer by improving the immune system [32].

Fatty acids compositions figs oil

The identification of the FAME revealed that the GL contain a high sum of saturated fatty acids (Σ SFA) whereas both NL and PL contain a high sum of unsaturated fatty acids (Σ UFA), where the Σ UFA = Σ monounsaturated fatty acids (MUFA) + Σ polyunsaturated fatty acids (PUFA) (Tables 4, 5 and 6). The obtained results were in accordance with the previous research that studied the fatty acid composition of the main fractions of the lipids (NL, GL and PL) of two cultivars of *Ficus carica* L [14].

First, the SFA of neutral lipids (NL) ranged from 11.7 to 41.83%; that covered six SFAs: capric C10:0(0.03-0.12%), lauric C12:0 (0.05-0.52%), myristic C14:0(0.04-1.21), palmitic C16:0(8.17- 34.96%), stearic C18:0(1.43-3.75%) and arachidic C20:0(0.12-3.66%). The palmitic acid was the major saturated FA constituent in NL and both capric and lauric acids were presented in lower amounts in NL of figs. The capric acid was not detected in NL1, NL2, NL3 and NL5. On the other hand, NL consists of 56.94 to 88.05% of USFA. The five UFA were: palmitoleic C16:1(0.25-3.53%), oleic C18:1(17.41-24.27%), linoleic C18:2(18.23-34.74%), linolenic C18:3(12.54-35.27%) and gondoic C20:1

(0.02-0.91%). The PUFA (C18:2 and C18:3) were found in high percentage (31.96-70.01%) and in all samples in comparison with MUFA (C16:1, C18:1 and C20:1) (18.04- 28.14%). The gondoic acid (C20:1) was detected in the oil in very small amounts and it was absent in NL2, NL5 and NL7. Since the amount of UFA is larger than SFA, the ratio UFA/SFA for NL ranged from 1.36 to 7.52. The highest value of UFA/SFA was found in NL3 and NL4 since the amount of PUFA of both C18:2(NL3 34.74%, NL4 34%) and C18:3(NL3 35.27%, NL4 33.87%) were higher (Table 4). Previous research found that the addition of PUFA to children's' hypocaloric diet has a favourable effect on the immunologic

processes [33]. Moreover, PUFA were found to be essential to reduce cancer risk and to prevent both cerebrovascular and cardiovascular diseases [34].

Second, glycolipids are more polar than neutral lipids, thus the profiles of their FAME differentiate from the NLs' FAME profiles. Results from Table 5 revealed that FAME from GL contains 21.74 to 56.33% of SFA. Unlike in NL fractions, SFA (C10:0, C12:0, C14:0, C16:0, C18:0 and C20:0) were found in all GL fractions, with the absence of arachidic acid (C20:0) in GL5, and both capric and lauric acids are found in high amount and in all figs' GL fractions.

Table 4

Fatty acid compositions (%) of neutral lipids of figs oils.

<i>sdica yttaF</i>	NL1	NL2	NL3	NL4	NL5	NL6	NL7
C10:0	-	-	-	0.03	-	0.12	0.07
C12:0	0.37	0.52	0.05	0.12	0.46	0.37	0.50
C14:0	0.70	0.25	0.04	0.07	0.92	0.68	1.21
C16:0	28.78	33.45	8.17	9.12	34.50	24.69	34.96
C16:1 ω -7	3.53	0.25	0.35	0.40	2.57	3.34	0.66
C18:0	3.75	1.73	3.32	3.44	2.45	2.73	1.43
C18:1 ω -9	23.03	22.83	17.41	17.97	22.11	24.27	22.52
C18:2 ω -6	18.23	24.73	34.74	34.00	19.80	21.25	18.98
C18:3 ω -3	13.73	12.54	35.27	33.87	14.36	18.35	14.78
C20:0	0.46	1.29	0.12	0.29	1.95	1.72	3.66
C20:1 ω -9	0.91	-	0.28	0.02	-	0.53	-
Σ SFA	34.06	37.24	11.70	13.07	40.28	30.31	41.83
Σ MUFA	27.47	23.08	18.04	18.39	24.68	28.14	23.18
Σ PUFA	31.96	37.27	70.01	67.87	34.16	39.60	33.76
Σ UFA	59.43	60.35	88.05	86.26	58.84	67.74	56.94
UFA/SFA	1.74	1.62	7.52	6.59	1.46	2.23	1.36

Abbreviations: NL - Neutral lipids; SFA - saturated fatty acids; MUFA - monounsaturated fatty acids; PUFA - polyunsaturated fatty acids; UFA - unsaturated fatty acids.

Table 5

Fatty acid compositions (%) of glycolipids of figs oils.

<i>sdica yttaF</i>	GL1	GL2	GL3	GL4	GL5	GL6	GL7
C10:0	0.16	1.03	0.05	0.20	3.00	0.56	7.74
C12:0	0.66	1.36	0.36	0.35	10.20	0.13	4.73
C14:0	0.99	1.01	0.17	0.25	4.76	1.20	3.01
C16:0	37.71	33.02	18.44	16.68	35.72	31.42	28.82
C16:1 ω -7	1.75	8.16	1.47	1.53	-	1.82	1.43
C18:0	3.20	6.43	3.52	3.36	2.65	2.42	3.23
C18:1 ω -9	19.75	16.51	19.15	18.63	15.27	22.50	18.88
C18:2 ω -6	15.83	15.59	28.85	28.44	15.83	18.96	12.45
C18:3 ω -3	13.33	6.18	24.54	27.65	10.56	15.01	2.25
C20:0	0.57	1.42	0.88	0.90	-	3.29	7.46
C20:1 ω -9	2.18	0.38	0.16	0.48	-	-	-
Σ SFA	43.29	44.27	23.42	21.74	56.33	39.02	54.99
Σ MUFA	23.68	25.05	20.78	20.64	15.27	24.32	20.31
Σ PUFA	29.16	21.77	53.39	56.09	26.39	33.97	14.70
Σ UFA	52.84	46.82	74.17	76.73	41.66	58.29	35.01
UFA/SFA	1.22	1.05	3.16	3.52	0.73	1.49	0.63

Abbreviations: GL - glycolipids; SFA - saturated fatty acids; MUFA - monounsaturated fatty acids; PUFA - polyunsaturated fatty acids; UFA - unsaturated fatty acids.

Alternatively, UFA and PUFA fractions compositions showed low percentage in comparison to NL, and they were different, from UFA: 35.01-76.73% and PUFA: 14.7-56.09%. Moreover, the MUFA found in GL fractions varied from 15.27 to 25.05%. The gondoic acid (C20:1) were only found in GL1, GL2, GL3 and GL4 which belong to cultivars figs from Laghouat city in the south of Algeria. Some previous studies on the variability of fatty acids of Algerian *Pistacia lentiscus* L. leaves and date fruits, agreed that the differences found in the composition of fatty acids was due to developmental changes, geographical area and to the cultivars itself [27,35,36]. Others found that PUFA in GL fractions is related to seasonal changes [37]. The recent research found that glycolipids, which are the major constituent of the thylakoid membrane of different plants, have a potential therapeutic use for inflammatory vascular diseases [38]. Other studies found that after the addition of an auric acid solution the level of unsaturated glycolipids decreased significantly, these results proved that glycolipids play important roles in reducing Au(III) to Au(0) and could be used to develop a novel efficient method to produce gold nanoparticles [39].

Third, PL, which are recovered by methanol, proved to have a great role in cells by forming a permeability barrier [40], also they contribute in the synthesis of a proper endoplasmic reticulum function that have serious role in limiting obesity/diabetes risks [41]. In this research, the UFA of PL fractions varied from

30.85 to 68.28%. FAME resulting from PL contains the highest amount of MUFA ranged in the interval 18-40.84%. This percentage range was higher when comparing to MUFA of both GL and NL (Table 6). In contrast, the PUFA amount of PL varied from 12.85 to 36.22%. These values were lower than those found in GL and NL, with the exception of PUFA of PL2 and PL5 which were 30.07 and 31.12 times higher from PUFA of GL2 and GL5, respectively. Furthermore, the SFA of PL ranged from 28.77 to 56.63%. The SFA found in the FAME of PL were higher than those of NL (with the exception of PL5) and lower than those detected in GL with the exception of PL3, PL4 and PL7.

A very important remark considering both figs samples F3 and F4, is that they share almost the same percentage of MUFA. Also, they presented the highest value of UFA/SFA ratio since the PUFA and UFA were the highest in all fractions of NL, GL and PL. The only explanation to that, is that both fig cultivars belong to the same region (Tadjmout-Laghouat). Yet, they share the same geographical region *i.e.* same soil, irrigation and sunlight. The only difference between these two samples is that F4 corresponds to the black fig and F3 to green fig. Researchers found that the differences in figs colour basically related to phenolic composition, carotenoids and other genetic factors that modify the pigment of the fruits [42,40]. In this case, the UFA were not influenced by this genetic factor or secondary metabolites such as phenolics.

Table 6

Fatty acid compositions (%) of phospholipids of figs oils.

<i>sdica yttaF</i>	<i>PL1</i>	<i>PL2</i>	<i>PL3</i>	<i>PL4</i>	<i>PL5</i>	<i>PL6</i>	<i>PL7</i>
C10:0	0.79	0.85	0.58	0.51	-	0.74	4.17
C12:0	-	0.17	-	-	0.71	0.51	1.77
C14:0	0.68	0.63	Tr	0.56	0.32	2.44	20.70
C16:0	31.63	33.00	23.50	25.78	33.73	27.93	22.76
C16:1 ω -7	15.70	8.84	8.71	2.65	3.18	8.63	1.16
C18:0	1.84	2.48	4.32	5.49	1.64	2.46	1.31
C18:1 ω -9	23.03	19.24	23.10	23.16	24.38	24.25	15.55
C18:2 ω -6	15.89	23.81	27.34	25.14	20.62	17.08	11.70
C18:3 ω -3	6.72	6.26	8.88	11.03	10.50	8.660	1.15
C20:0	-	0.54	0.37	0.59	2.30	-	5.92
C20:1 ω -9	2.11	0.13	0.25	2.03	0.93	1.90	1.29
Σ SFA	34.94	37.67	28.77	32.93	38.70	34.08	56.63
Σ MUFA	40.84	28.21	32.06	27.84	28.49	34.78	18.00
Σ PUFA	22.61	30.07	36.22	36.17	31.12	25.74	12.85
Σ UFA	63.45	58.28	68.28	64.01	59.61	60.52	30.85
UFA/SFA	1.81	1.54	2.37	1.94	1.54	1.77	0.54

Abbreviations: PL -phospholipids; SFA -saturated fatty acids; MUFA -monounsaturated fatty acids; PUFA -polyunsaturated fatty acids; UFA -unsaturated fatty acids; Tr -trace.

Furthermore, F7 sample (figs of Mostaghanem) is characterized by a low ratio of UFA/SFA which is explained by their high amount of SFA and the low amount of PUFA and UFA. The tested cultivars were marked by the presence of the palmitic acid as the major fatty acid in all fractions (NL, GL, PL). Also, the two unsaturated fatty acids, oleic (C18:1) and linoleic (C18:2), were presented in high amount in comparison to other unsaturated fatty acids. From this outcome, the Algerian fig cultivars taken from north, south and west were characterized by a high amount of oleic and linoleic acids. Moreover, a research on the Portuguese fig cultivars found that linolenic acid (C18:3) was the abundant fatty acid in their samples followed by linoleic (C18:2), oleic (C18:1) and palmitic (C16:0) acids [29]. A recent study on Moroccan fig cultivars found that the malic acid (organic acid) was the abandoned fatty acid in 11 local and introduced fig cultivars cultivated in Moroccan climate. This was taken as a criterion to characterize the Moroccan fig cultivars [11].

Antioxidant activity of the oil extracts

The results of the antioxidant activity assessed by both DPPH assay (free radical scavenging activity) and TAA (total antioxidant activity) using the phosphomolybdate reagent are presented in Table 7. Starting with DPPH antioxidant activity, the GL showed the lowest IC_{50} ranging from 0.234 to 1.067 g/L in which F7 registered the lowest IC_{50} of 0.23 g/L, the lower IC_{50} value, the stronger the activity. In addition,

$IC_{50} < 5$ g/L of any extract make it active against free radicals [43].

The TL, NL and GL from F7 sample showed strongest DPPH antioxidant activity among the other tested figs; this cultivar was characterized with low UFA/SFA and high amount of SFA. The obtained result can be explained by the presence of phenolic compounds in figs oils that may interfere in the DPPH test. Furthermore, different researches suggested that oil from fig seeds contain a small quantity of phenolic compounds such as *o*-diphenols and flavonoids and they assumed the ability of these compounds to act as hydrogen donors [8]. From this outcome, and to better understand the antioxidant properties of lipids, it is necessary to quantify the phenol contents of any seeds. Moreover, a recent research of DPPH antioxidant activity of dark and light fig seed oils found the highest DPPH antioxidant value in the dark-coloured variety (45.25%), and the lowest DPPH antioxidant value in the light cultivar (28.33%). Additionally, it has been found that colours, and methods of assessment have different effects on antioxidant activity [9].

Furthermore, results of TAA of different lipids fractions showed a low antioxidant activity, varied from 2.84 to 28.67 g/L. These values were higher than values obtained from commercial antioxidant (vitamin C, vitamin E, BHT and BHA) except the GL obtained from F3, F6 and F7 which was characterized by lower IC_{50} than BHT 3.60 g/L.

Table 7

The TAA and DPPH radical scavenging activity of figs oil and its lipid fractions.

	IC_{50} (g/L)	TL	NL	GL	PL
F1		0.70±0.01 ^{abc}	3.95±0.17 ^{ac}	0.83±0.01 ^{ac}	0.76±0.10 ^{ab}
F2		0.66±0.01 ^{abc}	3.30±0.13 ^{ad}	0.46±0.09 ^{abd}	1.23±0.11 ^{adef}
F3		0.83±0.01 ^{abc}	2.30±0.02 ^{abf}	0.78±0.10 ^{ac}	0.67±0.04 ^{bf}
F4	DPPH	1.65±0.17 ^{acd}	3.28±1.14 ^{deg}	0.97±0.06 ^{bc}	1.06±0.71 ^{be}
F5	assay	1.91±0.01 ^{bd}	4.85±0.15 ^{abg}	1.06±0.01 ^{acd}	1.00±0.15 ^c
F6		2.39±0.12 ^{ad}	7.01±0.16 ^{ag}	0.69±0.03 ^{acd}	0.83±0.05 ^d
F7		0.44±0.05 ^d	1.09±0.01 ^{cg}	0.23±0.50 ^c	0.80±0.02 ^e
F1		3.68±0.07 ^{ab}	23.41±0.25 ^{ce}	3.83±0.43 ^{ab}	3.73±0.90 ^{ag}
F2		7.00±0.10 ^a	9.68±0.88 ^{abd}	10.08±0.30 ^{acde}	5.57±0.20 ^{ad}
F3		6.00±0.63 ^{abc}	12.82±0.20 ^{abcd}	2.97±0.34 ^b	5.56±0.87 ^{ae}
F4		6.80±0.23 ^a	19.53±1.19 ^{ade}	7.38±0.26 ^{cde}	6.63±0.51 ^b
F5	TAA	10.52±0.80 ^{bd}	25.76±0.05 ^{be}	7.91±0.31 ^{bde}	5.23±0.05 ^f
F6	assay	12.67±0.50 ^{acd}	28.67±0.50 ^{ae}	2.84±0.06 ^e	11.30±0.11 ^a
F7		5.86±0.23 ^d	11.46±1.93 ^{ce}	3.45±0.05 ^d	6.29±1.07 ^c
	IC_{50} (g/L)	Vitamin C	Vitamin E	BHT	BHA
	DPPH assay	0.009±0.001	0.010±0.002	0.006±0.001	0.006±0.002
	TAA assay	0.07±0.005	0.08±0.0001	3.60±0.0001	0.27±0.0001

*Abbreviations: TL -total lipids; NL -neutral lipids; GL -glycolipids; PL -phospholipids; BHT -butylated hydroxytoluene; BHA -butylated hydroxylanisole; TAA -total antioxidant activity.

Note: Values with different lowercase superscript letters in the same column are significantly different ($p < 0.05$) according to Tukey post-hoc test

In addition, TAA of TL, GL and PL of F1 presented moderate IC_{50} values equal to 3.684, 3.83 and 3.73 g/L respectively. A recent research study proposed that the antioxidant activity is mainly related to the presence of tocopherols in the lipids, also, linear correlations were found between %UFA (C18:1, C18:2, C18:3) and the total tocopherols contents [36]. Another research suggested that lipids, in vascular endothelial cells, with omega 3 fatty acid might act as indirect antioxidant rather than pro-oxidant, thus reducing inflammation and in turn, the risk of atherosclerosis and cardiovascular disease [44,45].

Conclusions

From the outcome of this research it can be concluded that the Algerian figs contained up to 7.25% of oil. The tocopherols and sterols contents in fig oils ranged from 14.27 to 108.55 mg α -TE/g lipids and from 0.36 to 2.80 mg CE/g lipids respectively. The neutral lipids formed the largest fraction of the fruit's lipids (NL: 60.30-98.40%) followed by glycolipids (GL: 0.58-28.00%) and phospholipids (PL: 0.40-11.70%). Based on the analyses of the resulting fatty acids, the amount of UFA ranged from 30.85% to 88.05% and both F3 and F4 (where, F3 is green figs of Tadjmout and F4 is black figs of Tadjmout) contain the highest UFA (64.01- 88.05%) in all their fractions.

The seven figs oil samples contained important unsaturated acids such as linoleic C18:2(25.14-34.74%), linolenic C18:3 (10.50-35.27%), which are widely known for their pharmaceutical and clinical benefits.

In this study, for the first time were analysed and compared the antioxidant activities of different lipids classes of the crude oils. The glycolipids and phospholipids fractions were marked with powerful antioxidant activity measured by DPPH, where the inhibition concentration, IC_{50} , ranged from 0.23 to 1.06 g/L and from 0.67 to 1.23 g/L from glycolipid and phospholipids respectively. Furthermore, the strongest TAA was marked by GL (IC_{50} : 2.84 to 10.08 g/L) and PL (IC_{50} : 3.73 to 11.30 g/L).

The provided results in this study could open new prospects for the valorisation of figs oil and may generate new edible products that can be used in different medicinal and industrial domains.

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