

EFFECT OF IMPREGNATION OF TWO VARIETIES OF DRY FIGS (ABRKANE AND TAAMRIOUTE) IN OLIVE OIL TO IMPROVE THEIR PHYSICO-CHEMICAL AND BIOLOGICAL PROPERTIES

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Abstract

Description of the subject: The dried fig and olive oil are consumed because of their high nutritional value in the essential fatty acids omega-3 and omega-6.

Objective: The objective of this study is to evaluate the nutritional and therapeutic intake of dried figs in their native state, extra virgin olive oil as is and figs soaked in olive oil after 90 days of maceration.

Methods: The methodology consists in studying the characterization of physicochemical parameters, the determination of phenolic compounds, the evaluation of the antioxidant activity and the anti-inflammatory activity of two varieties of dried figs (Abrkane and Taamrioute), and extra virgin olive oil.

Results: The results of the chemical indices allowed to classify the control oil and the two remaining macerate oils in the category "extra virgin" in relation to the standards of the International Olive Oil Council COI, 2018, an increase in the level of phenolic compounds in the macerates but on the other hand its decrease is remarkable in the remaining oils. Macerates have the best reducing power and the best antiradical activity with a highly significant decrease ($p < 0.001$) of the oedema of albino mice at the dose (200 mg/kg) compared to the control oil and figs only (74.62%) at the 4 th hour.

Conclusion: The results show that macerates have been nutritionally enriched including black fig macerates. They have anti-inflammatory and antioxidant properties that could justify their use against oxidative and inflammatory stress-related diseases.

Keywords: dried figs, olive oil, phenolic compound, antioxidant activity, and anti-inflammatory activity

EFFET DE L'IMPRÉGNATION DE DEUX VARIÉTÉS DE FIGES SÈCHES (ABRKANE ET TAAMRIOUTE) DANS L'HUILE D'OLIVE POUR AMÉLIORER LEURS PROPRIÉTÉS PHYSICOCHIMIQUES ET BIOLOGIQUES

Résumé

Description du sujet : La figue sèche, l'huile d'olive sont consommées par la haute valeur nutritionnelle grâce aux acides gras essentiels oméga-3 et oméga-6 que contiennent.

Objectifs : La présente étude a pour objectif d'évaluer l'apport nutritionnel et thérapeutique des figes sèches à l'état natif, l'huile d'olive extra vierge telle quelle et les figes imprégnées dans l'huile d'olive après 90 jours de macération

Méthodes : La méthodologie consiste à étudier la caractérisation des paramètres physicochimiques, la détermination des composés phénoliques, évaluation de l'activité anti-oxydante et l'activité antiinflammatoire de deux variétés des figes sèches (Abrkane et Taamrioute), de l'huile d'olive extra vierge.

Résultats : les résultats des indices chimiques ont permis de classer l'huile témoin et les deux huiles restantes de macérât dans la catégorie « extra vierge » par rapport aux normes du conseil oléicole international COI, 2018, une augmentation de taux en composés phénoliques des macérâtes par contre son diminution est remarquable dans les huiles restantes. Les macérâtes possèdent le meilleur pouvoir réducteur et la meilleure activité anti-radicalaires avec une diminution hautement significative ($p < 0,001$) de l'œdème des souris albinos a la dose (200 mg/kg) par rapport à l'huile témoin et les figes uniquement (74,62%) à la 4^{ème} heure

Conclusion : Les résultats montrent que les macérâtes ont étaient enrichies sur le plan nutritionnel notamment la macérâtes des figes noirs ont des propriétés anti-inflammatoires et antioxydantes qui pourraient justifier leur utilisation contre les maladies lies au stress oxydant et inflammatoires.

Mots clés : Figes sèches, huile d'olive, composés phénoliques, activité antioxydante, activité anti inflammatoire.

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INTRODUCTION

The Mediterranean diet is based on many plants including olive oil (*Olea Europaea*), Fig (*Ficus carica*). It is considered as the most suitable model of food for health. They are the heritage of an ancient tradition. Olive oil is the main source of fats in Mediterranean countries. It is widely appreciated for its nutritional benefits related to the optimal balance between fatty acids, it consists of 72% of monounsaturated fatty acids (MUFA), 14% of polyunsaturated fatty acids (PUFA) and 14% of saturated fatty acids (SFA) (AGS) [1], as well as the presence of antioxidants in abundance (up to 16g / kg) represented by actosides, hydroxytyrosol, tyrosol and phenylpropionic acids as well as other important compounds in the prevention and treatment of cancer, inflammatory and cardiovascular diseases. They are also used as additives in various industries: pharmaceutical and cosmetic [2]. It is one of the oldest vegetable oils and the only one that can be consumed in its raw form without prior treatment. The fig is a typical fruit of Mediterranean countries, consumed fresh or dry. It offers a range of substances that enhance human health as well as a high nutritional value according to its composition rich in various nutrients. It represents a high carbohydrate richness [3] low fat intake and no cholesterol. It is a very rich fruit in minerals that effectively restores the nutritional balance. The fig plays a laxative role due to its fiber content. Thanks to their antioxidant activities, the bioactive substances of the fig intervene in the protection of health against various pathologies such as cancer, cardiovascular and neurological diseases which are generally related to the oxidative stress. It is used as an antipyretic and relieves pain. The fruit of *Ficus carica* is used to treat anemia and hemorrhoids thanks to its high iron content. The leaves are boiled and used to treat painful or swollen hemorrhoids. The concentrate has an effect on diabetes and clarification of the kidneys and liver. They are also used to treat jaundice [4]. Fig tree latex is used as a tranquilizer and anthelmintic [5]. The roots are used in the treatment of leukoderma and fungal inflammation such as herpes. Many researchers around the world have already shown the beneficial effects of adding plant metabolites in olive oil, which could significantly contribute to the stabilization of the product itself and the health of consumers.

We have the works of Penalvo *et al.* [6] and Yilmazer *et al.* [7] who add herbs, herbs and spices, thyme the work of Benmoussa *et al.* [8], IOC [27] and Reboredo-Rodriguez *et al.* [28] on the stability and sensory characteristics of the flavored olive oil, the work of Sousa *et al.* [9] studied the effect of essential oils extracted from plants on the quality of olive oil. The work of Achat *et al.* [10] and Japon-Lujaan *et al.* [11] also tested the stabilization of olive oil by adding natural antioxidants from olive leaves. Reports on the topic little work has been done on the biological activities of adding dried figs to olive oil, a single study Debib *et al.* [12] which has shown its antibacterial and antioxidant effects by using two varieties of dried figs cut in half (Aberkan and Taamrioute), and Sigoise olive oil. However, to our knowledge very few data are present that highlight the anti-inflammatory, anti-oxidant activity and the physical-chemical characteristics of dried fig maceration and the remaining oil. To this end, a study has been undertaken on two varieties of dried figs (Abrkane of black colour, Taamrioute of white colour) traditionally dried and the extra virgin olive oil of the variety (Sigoise) obtained by artisanal extraction system and study the effect of their maceration using the whole fruit. A correlation between chemical composition and biological activities will be developed. It concerns the anti-oxidant and anti-inflammatory effect as well as the content of primary and secondary metabolites (polyphenols) of dried figs and olive oil alone and after 90 days of maceration.

MATERIAL AND METHODS

1. Chemical products

The Folin-Ciocalteu reagent comes from Sigma (USA). Polyphenolic standards (gallic acid, quercetin, catechol, β carotene and DPPH) come from Merck (Germany). Other reagents and solvents are obtained from Panreac, Cheminova, Prolabo, Aldrich, Organics and Janssen.

2. Plant material

The study on the extra virgin olive oil from the 2015/2016 olive cultivation season of the Sigoise variety cultivated in the region of western Algeria (Mascara), obtained from a unit equipped with 'a cold extraction system, preserved in clean dark glass bottles and dry, with a minimum volume of 250 ml and refrigerated in order to avoid the phenomenon of auto-oxidation.

Dry figs of black variety (Abrkane) grown in the central region of Algeria (Bouira) harvested and dried traditionally by farmers and dried white fig (Taamrioute) grown in the eastern region of Algeria (Sétif) harvested then traditionally dried by the farmers. Both samples were kept at 4°C in a refrigerator. The impregnation of dried figs in olive oil was prepared according to the traditional method in October 2015. The figs placed in hermetically sealed glass jars of 250ml, The oil is added to the figs until immersion with a ratio (150g / 80ml). The olive

oil jars were stored in the dark at room temperature (around 25-28°C) until the extraction and analysis stage (January). The number of samples to be studied are: three oils (Extra virgin olive oil before maceration (OO), remaining oil of black fig macerate (RMBO) and remaining oil of white fig macerate (RMWO) (Table 1) and on the four dry figs studied, Black dry figs (BF) before maceration, White dry figs before maceration (WF), black dry figs macerates (OFB) and white dry figs macerates (OFW) presented in (Table 1).

Table 1: The study samples

Samples	extra virgin olive oil	remaining oil of black fig macerate	remaining oil of white fig macerate	Black Figs without olive oil	White Dried figs without oliveoil	Macerate Black dried Fig	Macerate White dry Figs
Abréviation	OO	RMBO	RMWO	BF	WF	OFB	OFW

3. Animal material

Swiss albino mice (male and female) weighing between 20 and 30 g were used. These animals were raised at the Research and Development Center (CRD) center of the MEDIA SAIDAL antibiotic unit under standard lighting conditions (12 hours of white light, 12 hours of darkness) at a room temperature 25 ± 1°C. They received standard food and drank at will with tap water.

4. Physico-chemical parameters

Free acidity, which was determined in the cold, according to the EEC method No. 2568 (1991), and expressed in% of oleic acid [13], the peroxide index was determined according to EEC standards No. 2568 (1991) is expressed in milli equivalents of active oxygen per kilogram of fat [35]. The density is the ratio of the mass of a certain volume of oil to 20 °C, and the mass of an equal volume distilled water at the same temperature [14, 36], saponification index [13], water content [16], specific extinction coefficients in the ultraviolet at 232 nm (K232) and 270 nm (K270) which correspond to the maximum absorbance of hydro peroxides and secondary oxidation products were determined according to standard [13], To determine the carotenoid content of the oil we applied the protocol described by E.U.C.R.A.R. [14] The carotenoid contents are expressed in mg of β -carotene equivalent, with reference to a calibration curve made with β -carotene and the content carotenoids figs according to the protocol described by Isabel Mínguez-Mosquera *et al.* [15],

the determination of total sugars was made by the method of Dubois *et al.* [16], The results are expressed in mg equivalent glucose per 100 g of dry matter with reference to a curve calibrated with glucose. The lipid content according to standard [17]. The protein determination is carried out according to the Bradford method [18]. The protein concentration is determined by reference to a calibration curve obtained with bovine serum albumin (BSA).

5. Extraction of polyphenols

For the phenol compound extraction of olive oil we use the method of [19] 1g of oil is dissolved in 5 ml of hexane, then 5 ml of the mixture methanol / water (60:40, v/v) is added after vortexing. The methanolic phase is recovered, a second wash is carried out, to which 5 ml of hexane is added. Finally it is centrifuged at 3500 rpm for 10 minutes. The methanolic phase is recovered in which the various compounds are measured. Solid-liquid extraction [21], is applied to extract phenolic compounds from dried figs. An aliquot of fig crushed (200 mg) is introduced into a test tube and then 10 ml of solvent (methanol / water 80/20) are added. The tube is placed in a water bath equipped with an automatic stirrer at 40°C for 120 min. The extract is recovered by centrifugation at 5000 rpm / 10 min and then filtered and stored at 4°C in a refrigerator

6. Determination of phenolic compounds

The concentration of the phenol compounds is determined by the method of Folin-Ciocalteu (FC) [28], A volume of 200 μ l of extract is added with 750 μ l of the Folin-Ciocalteu reagent. After 5 min, 400 μ l of (7%) of sodium carbonate are added. The reaction mixture is left

in the dark for 60 min at room temperature. The absorbance of the blue color developed is measured at 750 nm. The results are expressed in mg gallic acid equivalent / g of dry vegetable material with reference to the calibration curve of gallic acid. The determination of total flavonoids was carried out according to method Singleton *et al.* [22], which consists of mixing 1 ml of extract and 1 ml of the 2% solution of aluminum chloride (AlCl₃). The mixture is left for 10 minutes in the dark and at room temperature. After incubation, the absorbance is measured at 430 nm. A control was prepared by replacing the extract with the same volume of the extraction solvent. The results are expressed in mg equivalent quercetin / g of dry vegetable material with reference to the quercetin calibration curve. The condensed tannins are determined by the method Djeridane *et al.* [23], A volume of 1 ml of the extract is added to 5 ml of the HCl-Vanillin reagent (2 g of vanillin in 100 ml+8 ml of HCl adjusted to 100 ml). The resulting mixture is allowed to react in the dark and at room temperature for 20 minutes. The absorbance is measured at 500 nm and the results are expressed in mg catechin equivalents / g of dry vegetable matter with reference to the catechin calibration curve.

7. Antioxidant activity

- *Measurement of the scavenging power of the DPPH radical:* The anti-radical activity of the extracts was determined using the free radical 2,2'-diphenyl-1-picrylhydrazyl (DPPH), the protocol described by Price *et al.* [24], a volume of 100 µl of the extract is mixed with 1 ml of the methanolic solution of DPPH (60 µM). After 30 min of incubation at room temperature, the absorbance is determined at 517 nm. The results are expressed as a reduction of the DPPH radical percentage relative to a control containing only the extraction solvent, according to the following formula:

$$PI\% = (Abs_{Ech} / Abs_T) \times 100.$$
 Abs T: absorbance of the test containing the extraction solvent., Abs Ech: absorbance of the solution containing the sample.

-*Reducing power measurement:* The protocol of Brand-Williams *et al.* [25], is used to evaluate the reducing power of the extracts. A volume of 250 µl of extract is added to 250 µl of phosphate buffer (0.2 M, pH=6.6) and 250 µl of potassium ferricyanide (1%).

After incubation at 50 ° C. for 30 minutes, 250 µl of (10%) trichloroacetic acid are added to the mixture. Then 1 ml of distilled water and 250 µl of (0.1%) ferric trichloride are added. The absorbance of the mixture is measured at 700 nm against a test whose sample is replaced by the volume of extraction solvent. The expression of the results is defined by following the calibration curve prepared with gallic acid.

8. Evaluation of anti-inflammatory activity

The carrageenin method inducing paw edema was used to evaluate anti-inflammatory activity. Paw diameter was measured using the micrometer before and after carrageenin injection to evaluate increasing and reducing the diameter [26], The 50 mice were divided into 10 lots of 5 mice each. 1 hour after tube feeding of different solutions, 0.1 ml of a suspension of 0.5% carrageen (dissolved in 0.9% saline solution) was injected subcutaneously in the plantar aponeurosis of the right hind paw of the mice. The edema was measured one (1) hour after injection of carrageenan and each hour for 6 hours using a micrometer. The percentage increase in edema was calculated each hour according to the following equation: % edema = ((Dn-D0)/D0)×100, D1: diameter of the paw each hour after injection of carrageenan. D0: diameter of the paw before the injection of carrageenan.

The percentage of inhibition was calculated based on the test according to the following formula: % inhibition = [(% edema (control) - % edema (extract)) / % edema (control)] × 100. Batch of control (negative control): received physiological saline (NaCl 0.9%) at a dose of 10 ml/kg. Standard lot (positive control): received Diclofenac as reference anti-inflammatory drug (nonsteroidal anti-inflammatory drug) at a dose of 10ml / Kg (50mg / kg). The powder of Diclofenac was dissolved in physiological saline (0.9%) with a ratio of 5mg / 10ml. Batch of control (negative control): received physiological saline (NaCl 0.9%) at a dose of 10 ml / kg. Standard lot (positive control): received Diclofenac as reference anti-inflammatory drug (nonsteroidal anti-inflammatory drug) at a dose of 10ml/Kg (50mg / kg). The powder of Diclofenac was dissolved in physiological saline (0.9%) with a ratio of 5mg/10ml. The batches of 3 to 10 are treated with the methanolic extracts of three studied oils and the e' extracts of white and black figs and the black and black fig macerate extract which have been prepared with physiological saline (NaCl 0.9%) at 200 mg/kg. A solution of 10 ml / kg was tube-fed for each mouse.

9. Statistical analyzes

All the data obtained represent the average of three three replicates. The anti-inflammatory activity data obtained represents the average of five trials. The statistical analysis was performed according to the standard methods of ANOVA 1 variance analysis followed by the Fischer LSD test using the XLSTAT 14 computer program. After prior verification of the homogeneity of the variances and the normality of the data to be analyzed, significant probability thresholds of 5 and 1% were used.

RESULTS

1. Physicochemical characterization

Table 2 : Physicochemical characterization of the three studied oils: olive oil (OO), remaining oil of black fig macerate (RMBO), and remaining oil of white fig macerate (RWBO).

Parametres	OO	RMBO	RMWO	COI 2018
Acidity	0.33±0.02 ^a	0.60±0.09 ^b	0.75±0.007 ^b	≤0,8
Density	0.910±0.13 ^a	0.915±0.04 ^a	0.916±0.06 ^a	0.910-0.916
I. peroxyde	0.58±0.06 ^a	0.60±0.36 ^a	0.61±0.084 ^a	<20.0
I. saponifications	186.46±0.56 ^a	184.50±0.05 ^b	189.57±0.028 ^b	184-196
K270	0.20±0.07 ^a	0.16±0.04 ^b	0.18±0.007 ^c	<0.22
K232	2.53±0.04 ^a	2.21±0.09 ^b	2.36±0.205 ^c	<2.50
Humidity	0.11±0.70 ^a	0.43±0.07 ^b	0.61±0.063 ^c	<0,2

The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a> b> c.

Table 3 : Physicochemical characterization of black dried figs FB, white dried figs FW, macerated black figs OFB and macerated dry white figs OFW

Parameters	FB	FW	OFB	OFW
weight	11.10±0.61 ^a	12.60±0.46 ^c	12.80±0.02 ^c	13.96±1.163 ^d
Length	37±0.04 ^a	39±0.17 ^c	37.6±2.034 ^b	39.5±2.586 ^c
Diameter	36±0.23 ^a	38±0.24 ^c	36.5±0.03 ^b	39.4±0.06 ^c
Acidity	0.41±0.02 ^a	0.62±0.04 ^b	0.28±0.07 ^c	0.51±0.04 ^d
Humidity	18.54±0.19 ^a	20.21 ±0.05 ^b	17.02±0.12 ^c	18.25±0.82 ^d
Proteins	3.88±0.02 ^a	2.92±0.1 ^b	3.42±0.02 ^c	2.52±0.03 ^d
Lipids	0,96±0.01 ^a	1.73±0.6 ^b	1.65±0.23 ^c	2.58±0.69 ^d
Sugar	72.74 ±0.70 ^a	88.61±0.41 ^b	64.63±0.70 ^c	72.12±0.41 ^d

The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a> b> c> d.

2. Content of bioactive compounds

The determination of the total polyphenols by the method of Folin ciocalteu for our phenolic extracts allowed us to obtain the results presented in (Table 4).

Table 4 : Quantification of phenolic compounds of black dried figs FB, white dried figs FW, macerated dark figs OFB, macerated dry white figs OFW, olive oil OO, remaining oil of white fig macerate (RMWO) remaining oil of black fig macerate (RMBO).

The highest levels are recorded in the oil before maceration with a rate of (616.52 mg / 100 g DM).

The results of physico-chemical analyzes carried out on the three studied oils (extra virgin olive oil (OO), remaining oil of black fig macerate (RMBO) and remaining oil of white fig macerate (RMWO) (Table 2 and on four dried figs studied, Black Figs (BF), White Dried Figs (WF), Macerate Black Dried Figs (OFB) and Macerate White Dry Figs (OFW) (Table 3) show that: All the quality parameters of the studied oils (Table 2), acidity, saponification number, peroxide index, density, K232 and K270 absorbances and moisture remained below the maximum value allowed for their classification as extra virgin oils in accordance to COI regulations, 2018 [27].

The flavonoid content of olive oil is 7.52 mg quercetin / kg (Table 4). A significant decrease ($p \leq 0.05$) is noted during the maceration, which reaches, after 90 days respectively, up to 4.75 and 4.84 mg / kg of the remaining oil of the white figs (RMWO) and black figs (RMBO) successively. The hydrosoluble tannin contents of the analyzed oil samples and the remaining oils mixture showed significant differences ($p < 0.05$) (Table 4).

They ranged from 173.91 mg E catechin / 100 g MS for the first time. control oil (OO) and 123.85 and 151.64 mg E catechin / 100 g MS for the remaining oil of white fig macerates (RMWO) and black (RMBO) and 28.68 mg EQ3G / 100g MS sequentially.

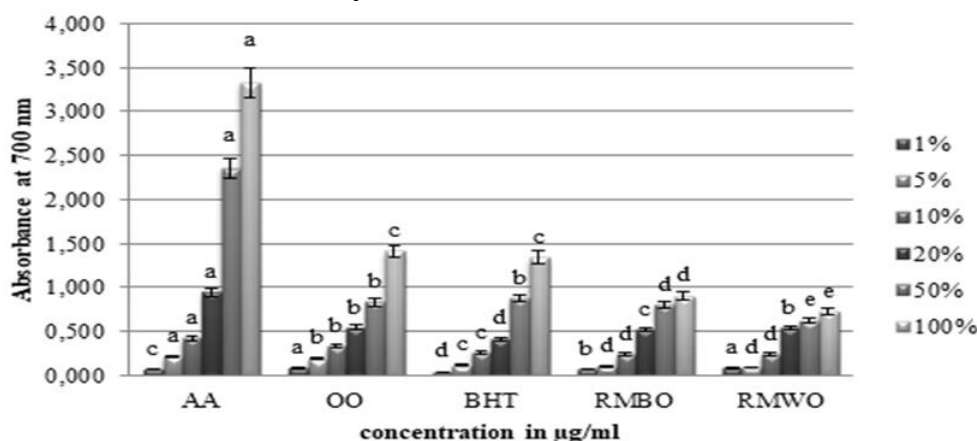
The carotenoid content of the oil (OO) before maceration is 40.25 mg / kg dry matter. After 90 days of maceration the oil has undergone a significant decrease in carotenoids with amounts of 38.45 and 38.64 mg / kg of dry matter for the remaining oil of the white (RMWO) and black (RMBO) macerate figs.

Samples	Total polyphenol (mgEAG/100g MS)	Flavonoïdes (mg Equerctin /100g MS)	TH (mg Ecatéchine /100 g MS)	Caroténoïdes mg/kg
OOA	616.52±0.61 ^a	7.52±0.01 ^a	173.91±0.01 ^a	40.25±0.03 ^a
RMWO	312.19±0.95 ^b	4.76±0.03 ^b	123.85±0.54 ^b	38.45±0.025 ^b
RMBO	410.92±0.71 ^c	4.84±0.02 ^c	151.64±0.58 ^c	38.64±0.015 ^c
FB	532.19±0.90 ^d	56.44±0.02 ^d	144.42±0.23 ^d	711.32±0.02 ^a
FW	305.17±0.09 ^e	42.46±0.04 ^e	51.23±0.09 ^e	623.02±0.01 ^b
MFB	605.63±0.42 ^f	58.47±0.27 ^f	169 ±0.01 ^f	928.14±0.01 ^c
MFV	479.29±0.68 ^g	43.16±0.05 ^f	58.98±0.02 ^g	745.12±0.05 ^d

The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e > f > g.

The highest levels are recorded in the oil before maceration with a rate of (616.52 mg / 100 g DM). The flavonoid content of olive oil is 7.52 mg quercetin / kg (Table 4). A significant decrease ($p \leq 0.05$) is noted during the maceration, which reaches, after 90 days respectively, up to 4.75 and 4.84 mg / kg of the remaining oil of the white figs (RMWO) and black figs (RMBO) successively. The hydrosoluble tannin contents of the analyzed oil samples and the remaining oils mixture showed significant differences ($p < 0.05$) (Table 4). They ranged from 173.91 mg E catechin / 100 g MS for the first time. control oil (OO) and 123.85 and 151.64 mg E catechin / 100 g MS for the remaining oil of white fig macerates (RMWO) and black (RMBO) and 28.68 mg EQ3G / 100g MS sequentially. The carotenoid content of the oil (OO) before maceration is 40.25 mg / kg dry matter. After 90 days of maceration the oil has undergone a significant decrease in carotenoids with amounts of 38.45 and 38.64 mg / kg of dry matter for the remaining oil of the white (RMWO) and black (RMBO) macerate figs.

3. Antioxidant activity



3.1. Reducing power test

The evolution of the reducing power (Fig. 1 and Fig. 2) of the different antioxidants according to the concentration, it is observed that the concentration has a highly significant effect on the reducing power ($p < 0.01$). (At low concentrations (2.5 and 10 $\mu\text{g} / \text{mL}$), ascorbic acid has a greater reducing power than that of BHA and the phenolic extracts studied and then at a concentration of 50 $\mu\text{g} / \text{mL}$. All the phenolic extracts of the studied figs show a greater reducing power (0.94; 0.9; 0.87 and 0.80) of black fig macerates (OBF), white fig macerates (OWF), black fig (FB), white fig (FW), successively by adding BHT (0.87). We recorded at the same concentration at 50 $\mu\text{g} / \text{mL}$ a reducing power of less BHA for all the studied oils (0.8; 0.80 and 0.62). For the oil before the maceration (OO) and the remaining oil of the black fig macerates (RMBO) and the remaining oil of the white fig macerates (RMWO) successively (Fig. 2),

Figure 1: Evolution of reducing power of the antioxidants and phenolic extracts of the studied oils as a function of concentration: BHT butylhydroxytoluene, AA: ascorbic acid, OO: olive oil before maceration, RMBO: remaining oil of black fig macerates, RMWO: remaining oil of white fig macerates. The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e.

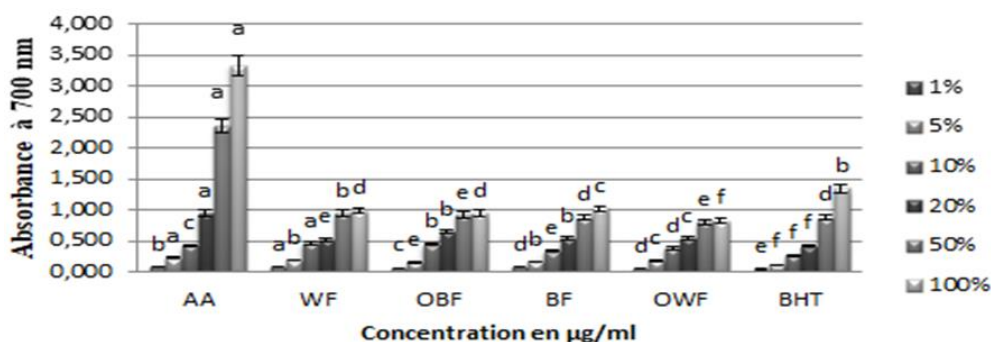


Figure 2: Evolution of reducing power of antioxidants and phenolic extracts of the figs studied as a function of concentration: BHT butylhydroxytoluene , AA: ascorbic acid, FB: black fig, FW: white fig, OBF: black fig macerate, OWF: fig macerate white. The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e > f.

3.2. The trapping power of the DPPH radical

The results of the anti-free radical power of the methanolic extracts expressed as percentage inhibition of the DPPH radical (Fig. 3 and Fig. 4) indicate that the ability to trap the DPPH radical of the extra virgin olive oil at concentrations of 5 10 and 20 µg / mL)) and less

than ascorbic acid (95.26%) and more effective than that of oil remaining black fig macerates (RMBO) and remaining oil of fig macerate white (RMWO) and BHT (48.68%) (Fig. 3 and Fig. 4), with a highly significant difference ($p < 0.001$).

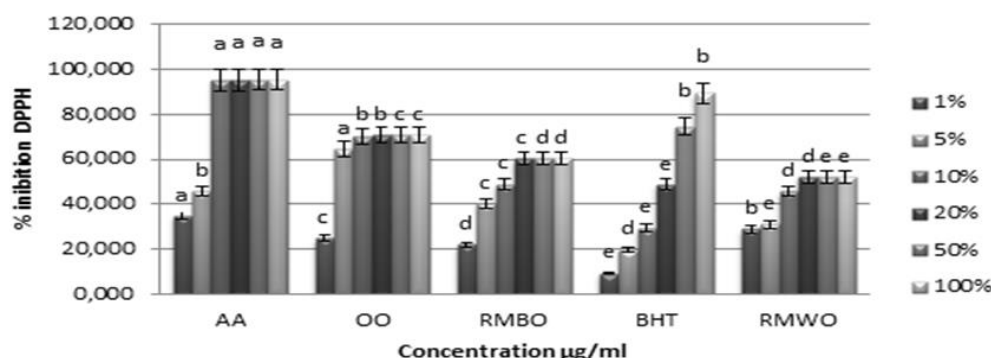


Figure 3: Evolution of percentage of DPPH inhibition of the antioxidants and phenol extracts of the studied oils as a function of the concentration: BHT butylhydroxytoluene , AA: ascorbic acid, OO: olive oil before maceration, RMBO: remaining oil of Fig macerates black RMWO: remaining oil of white fig macerates. The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e.

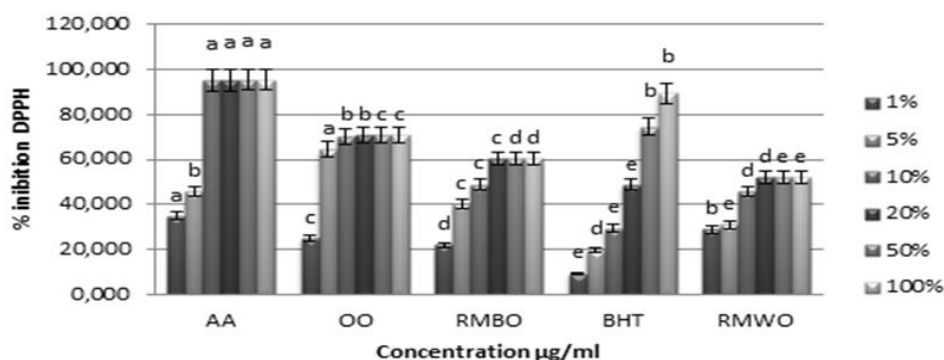


Figure 4: Evolution of DPPH inhibition percentage of antioxidants and phenolic extracts of the figs studied as a function of concentration: BHT butylhydroxytoluene , AA: ascorbic acid, OO: olive oil before maceration, RMBO: remaining oil of Fig macerate black RMWO: remaining oil of white fig macerates. The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e.

4. Anti-inflammatory activity

The anti-inflammatory activity of methanolic extracts was assessed by carrageenan method of inducing a paw edema in mice. The diameter of the lug measured by the micrometer yielded the results illustrated in (Table 5), showing the increase in the diameter of the paw as a function of time after injection of carrageenan. The increase is significant in the test group whereas it is significantly decreased in the groups treated with phenolic extracts and the standard. Note that the increase of edema is greater in the test

which reaches its maximum at the 4th hour (3.45 cm) followed by a regression phase. On the other hand, it is greater in all extracts compared to the standard. The results of % inhibition of edema are represented on (Fig. 5).

Table 5 : Evolution of the paw diameter as a function of the hours of the black dried figs FB, white dried figs FW, macerated dry black figs OFB, macerated dry white figs OFW, olive oil OO, remaining oil of white fig macerate (RMWO) remaining oil black fig macerate (RMBO), S: Standard, BI: Before injection.

Time	BI	1 hour	2 hours	3 hours	4 hours	5 hours	6 hours
Teste	2.88±0.23 ^{ab}	3.27±0.71 ^a	3.39±0.21 ^a	3.45±0.42 ^a	3.67±0.51 ^a	3.41±0.11 ^a	3.12±0.11 ^a
S	2.63±0.11 ^{ab}	2.88±0.31 ^a	2.80±0.11 ^a	2.78±0.12 ^a	2.75±0.11 ^a	2.74±0.12 ^a	2.68±0.13 ^a
OO	2.61±0.59 ^d	2.91±0.23 ^f	2.95±0.48 ^d	2.91±0.26 ^a	2.87±0.63 ^{de}	2.81±0.11 ^b	2.74±0.13 ^d
RMBO	2.88±0.26 ^a	3.18±0.11 ^c	3.15±0.36 ^c	3.12±0.63 ^a	3.10±0.51 ^{cd}	3.06±0.25 ^b	2.95±0.23 ^d
RMWO	2.68±0.71 ^d	2.96±0.64 ^b	2.95±0.45 ^b	2.93±0.73 ^a	2.91±0.27 ^b	2.86±0.82 ^b	2.76±0.85 ^a
FB	2.84±0.45 ^b	3.12±0.53 ^c	3.11±0.5 ^e	3.09±0.12 ^a	3.07±0.55 ^c	2.98±0.62 ^b	2.97±0.64 ^a
FW	2.88±0.19 ^c	3.20±0.15 ^d	3.18±0.73 ^e	3.15±0.18 ^a	3.13±0.50 ^d	3.12±0.23 ^b	2.99±0.25 ^a
OFB	2.64±0.14 ^e	2.92±0.66 ^g	2.99±0.49 ^e	2.95±0.39 ^a	2.84±0.33 ^f	2.80±0.97 ^d	2.68±0.96 ^a
OFW	2.66±0.56 ^{de}	2.99±0.17 ^e	3.01±0.93 ^f	2.99±0.54 ^a	2.89±0.29 ^e	2.86±0.17 ^c	2.75±0.16 ^a

The results are expressed as mean ± standard deviation (number of repetitions n = 3). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e >

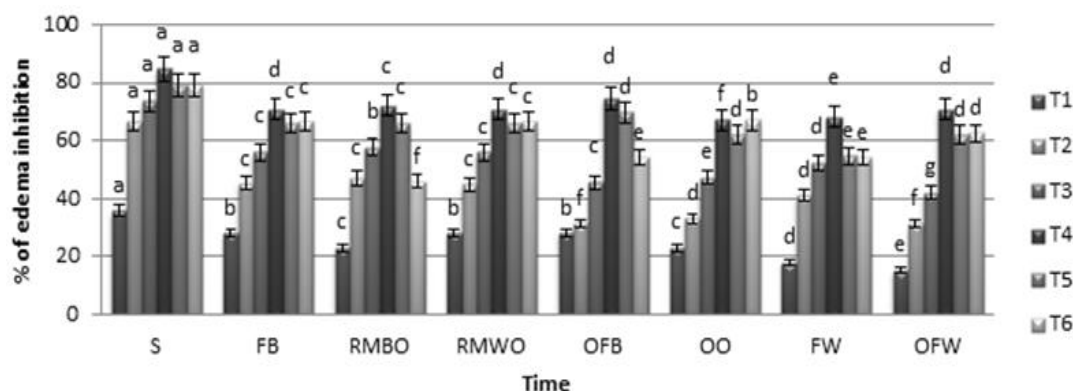


Figure 5: Evolution of edema inhibition percentage of dry black figs FB, dry white figs FW, macerated dark figs OFB, macerated dry white figs OFW, olive oil OO, remaining oil of white fig macerate (RMWO) oil remaining black fig macerate (RMBO). The results are expressed as mean ± standard deviation (number of repetitions n = 5). For each column, the values with different letters are significantly different at $p < 0.05$; a > b > c > d > e > f > g.

The anti-inflammatory activity of the figs is moderately correlated with the protein content, the total polyphenols and the antioxidant activity with ($r=0.34$, $r=0.23$ and $r=0.28$) (Table 6) respectively and strongly correlated with the hydrosoluble tannin content with ($r = 0.93$). The anti-inflammatory activity of olive oil and strongly correlated with the content of total polyphenols, the reducing power and the carotenoid with a ($r=0.97$, $r=0.97$ and $r=0.94$)

(Table 7) respectively and moderately correlates with the values of K232 and K270 and the antioxidant activity with ($r=0.21$, $r=0.52$ and $r=0.33$) respectively consistent with other work on the effect exerted by phenolic compounds such flavonoids [36, 45] and vitamin E. The latter in its γ -tocopherol form and its hydrosoluble metabolites inhibit the production of prostaglandin (pro-inflammatory mediator).

Table 6 : Correlation of the physico-chemical parameters of the studied figs L : width, D : diameter, H% : humidity, PPT : total polyphenols , TF : flavonoides , Caro : carotenoides, PR : reducing power and TH : hydro-soluble tannins, % I edema : percentage of inhibition of edema.

	Weight	L	D	Acidity	H%	Proteins	Lipids	Sugar	PPT	TF	TH	Carot	DPPH	PR
L	0,867***													
D	0,765***	0,817***												
Acidity	0,218*	0,579**	0,712***											
H%	-0,153	0,249*	0,392*	0,915***										
Proteins	-0,908	-0,953	-0,935	-0,606	-0,263									
Lipids	0,982***	0,905***	0,849***	0,335*	-0,055	-0,947								
Sugar	-0,072	0,308*	0,412*	0,909***	0,987***	-0,323	0,002*							
PPT	-0,535	-0,445	-0,243	-0,120	-0,070	0,467*	-0,405	-0,226						
TF	-0,847	-0,522	-0,343	0,332	0,637***	0,546*	-0,759	0,549*	0,492*					
TH	0,064*	-0,242	-0,264	-0,750	-0,880	0,254*	0,054*	-0,938	0,491*	-0,439				
Carot	-0,800	-0,455	-0,285	0,410	0,712***	0,477*	-0,716	0,637***	0,395*	0,993***	-0,543			
DPPH	-0,916	-0,885	-0,775	-0,439	-0,153	0,927***	-0,885	-0,264	0,759***	0,661***	0,329*	0,575***		
PR	-0,906	-0,743	-0,566	-0,119	0,147	0,779***	-0,822	0,016*	0,830***	0,830***	0,131*	0,759***	0,940***	
% I edema	0,048*	-0,331	-0,434	-0,918	-0,986	0,347*	-0,028	-1,000	0,234*	-0,530	0,936***	-0,619	0,285*	0,003*

Parameter measured only for the studied figs : * significant correlation ($p < 0.05$), ** Highly significant correlation ($p < 0.01$), *** Highly significant correlation ($p < 0.001$).

Table 7 : Correlation matrix of the physico-chemical parameters of the oils studied : H% : humidity, PPT : total polyphenols , TF : flavonoides , Caro : carotenoides, PR : reducing power and TH : hydro-soluble tannins, % I edema : edema inhibition percentage.

	Acidity	Density	IP	IS	K270	K232	H%	PPT	TF	TH	Caro	DPPH	PR
Density	0,802***												
IP	0,845***	0,953***											
IS	0,471*	0,156*	0,303*										
K270	-0,335	-0,311	-0,143	0,565**									
K232	-0,704	-0,738	-0,641	0,287*	0,85***								
H%	0,998***	0,788***	0,828***	0,498*	-0,324	-0,686							
PPT	-0,092	-0,314	-0,172	0,835***	0,857***	0,764***	-0,061						
TF	-0,924	-0,633	-0,723	-0,771	0,014*	0,387*	-0,936	-0,293					
TH	-0,125	-0,339	-0,199	0,816***	0,866***	0,786***	-0,094	0,999***	-0,261				
Caro	-0,881	-0,577	-0,679	-0,831	-0,076	0,293*	-0,896	-0,388	0,995***	-0,357			
DPPH	-0,489	-0,596	-0,487	0,539*	0,896***	0,958***	-0,462	0,913***	0,121*	0,927***	0,021*		
PR	-0,996	-0,79	-0,833	-0,413	0,393*	0,744***	-0,992	0,155*	0,897***	0,188*	0,849***	0,543**	
% I edema	-0,985	-0,734	-0,795	-0,609	0,215*	0,582**	-0,99	-0,073	0,975***	-0,039	0,947***	0,339*	0,972***

Parameter measured only for the studied oils : * significant correlation ($p < 0.05$), ** Highly significant correlation ($p < 0.01$), *** Highly significant correlation ($p < 0.001$).

DISCUSSION

1. Physicochemical characterization

The objective of this study is to evaluate the nutritional and therapeutic intake of dried figs in their native state, extra virgin olive oil as is and figs soaked in olive oil after 90 days of maceration. We have tried to contribute to its valuation by establishing a relationship between its chemical composition and its biological activities of dried figs and olive oil alone and after 90 days of maceration. In this study The acidity of olive oil (OO) before maceration corresponds to an extra virgin olive oil according to the standard set by the COI, 2018 [27]. As a direct result of hand harvesting and immediate extraction without stocking the olives, after 90 days of maceration we noticed a slight increase for the oil, remaining black fig (OFB) and the remaining fig oil (OFW) with a significant difference ($p < 0.05$), in this context, previous studies also showed that acidity values increased when aromatic plants were added to extra virgin olive oils [28]. On the other hand, we noticed a decrease in the acidity of macerated dried figs.

This value is within the range of results obtained by Simsek and Yildirim [29]. The variability of acidity may be due to genotypic characteristics, early or late harvest of fruits and the ecological conditions of fig growth [30], to reach after 90 days of maceration with a significant difference ($p < 0.05$). This may be due to the possible hydrolysis of triglycerides causing the release of fatty acid or a possible migration of organic acids from the figs to the oily phase.

The results reveal that there is an increase in moisture content during maceration of the studied oils (with a significant difference ($p < 0.05$)) and is very highly correlated with acidity, density and peroxide index. On the other hand, the results obtained from the water content of dried figs before maceration are within the range of values described by Su et al. [31]. After 90 days. The moisture content has decreased significantly ($p < 0.05$), this may be due to the migration of the water contained in the fruit to the oil. We also note that the values of the peroxide index of the analyzed oils during the whole maceration period (from the 1st day to the 90th day) are well below the limit established by the COI, 2018. So the oil has

undergone no significant change. It is highly correlated with density and acidity. This is due to the origin of the absence of peroxide and hydroperoxides in oils or the richness of powerful antioxidant extracts (polyphenols, tocopherols, carotenoids ...) that act against oxidation [32]. Note that olive oil before and after maceration has absorbance values of K232 and K270 that have a strong correlation, saponification number and density respecting the limit allowed by the COI 2018 standard for classification as extra virgin olive oil. We can therefore conclude that our studied oils are in an unoxidized state. The diameter and length of the studied figs are almost in the range found by [33]. Then after 90 days of maceration the diameter and length of the figs increased with a significant difference due to the membrane permeability of the figs, We have observed that the fig's weight is very highly correlated with the fruit's dimensions, lengths and diameters and even the acidity values of the fig is very highly correlated with the fruit dimensions, lengths and diameters. These results are consistent with those of Wang *et al.* [33]. The total sugar content of figs before maceration is in the range found by Wang *et al.* [33], with a content of about 85g/100g. After maceration, the sugar content underwent a highly significant decrease ($p < 0.05$) with a content of 64.63g/100g and 72.12 white and black fig macerates, respectively. This decrease is probably due to the dilution of the quantity. The sugar concentrations in the mixture were also highly correlated with the acidity and moisture of the figs. According to the results of our study, the total sugar level at FB level is higher than that of FW, OFB and OFW with a highly significant difference ($p < 0.001$). Our results are in perfect agreement with the work of Sreeramulu *et al.* [34] who found that the light skin variety has higher sugar content than the skin variety. However, the amount of sugars can still suffer considerable drops during storage in the skin. In our case, it essentially results from the phenomenon of diffusion, solubilization of the hydrolysable sugars of the figs in the oil. The percentage of proteins reaches a significant value in the black fig (FB) followed by the white fig (BW), our result is comparable to the work of Sreeramulu *et al.* [34]. With dry Calimyrana variety contains 3% MF and Dry Mission variety contains 2.82% MF.

After impregnation the protein content decreased for both varieties with a significant difference ($p < 0.05$). It results from the phenomenon of diffusion and solubilization of the proteins during the impregnation with the oil and no correlation is established between the contents of proteins and the other physicochemical parameters analyzed. The evaluation of the lipid content of figs reveals appreciable with a significant difference ($p < 0.05$), our results are in the range found [35]. Which yield 0.9 g/100g, it was found that the lipid content very highly correlated with the weight and dimensions of the and it is noted that the dry white skin fig contains more lipids than black fig.

2. Content of bioactive compounds

The total polyphenol contents recorded for our oil variety are higher than those of the 18 Italian varieties studied by Harzallah *et al.* [3]. for which the levels range between 115 and 377 mg/kg. After 90 days of maceration a significant decrease ($p \leq 0.05$) in total polyphenols with a rate of (410.19 and 283.92 mg/100 g of MS) remaining oil of black figs (RMBO) and white (RMWO) respectively. The total polyphenol content of the unmelted dried fig is 539, 19 and 305.17 mg/100g, black (FB) and white (FW) dried figs respectively this value is in the range found by Djeridane *et al.* [23]. With a content of 332 to 600 mg/100g for the dried fig, after maceration we noticed a highly significant increase ($p \leq 0.001$) up to 705.31 and 479.29 mg/100g macerates black figs (OFW) and white (OFW) respectively. This is consistent with the work done by Del Caro and Piga [37]. Who found a slight increase in polyphenol content from 254.5 to 262 mg / 100g when macerating flowers of prickly pears. This is probably due to the passage of phenolic compounds from olive oil to the dried. And the dark color variety (Abrkane) analyzed has higher grades than light-colored varieties (Taamrioute). These results are in agreement with data from the literature [23, 36].

The flavonoid content of the unmacerated dried fig is of an average of 56.44 and 42.26 mg of quercetin / 100g of dried figs material black (FB) and white (FW) successively. This result is lower than those reported by Darjazi [30], Ercisli *et al.* [45], which obtained concentrations varying according to the extraction solvent, between 79.9 to 105.6 mg/100g of dry matter in the black fig.

On the other hand, it is superior to that published by Harzallah *et al.* [38], who reported dry flavonoid contents of between 2.1 and 21.5 mg E.Q/100g. After 90 days of maceration there is a slight increase with a significant difference ($p < 0.05$). So the macerated fig has been enriched by this tiny contribution of flavonoids according to the evolution of time. The tannin content for the figs before the maceration is between 144.42 and 51.23 mg E catechin/100 g MS of the black (FB) and white (FW) dried figs successively to reach a rate of 169.42 and 58, 98 mg E catechin/100 g MS black fig macerates (FBO) and white fig macerates (FWO) successively. Our results are also consistent with those of Del Caro and Piga [37], who reported that some black and purple figs from Turkey, contained 2.5 times more condensed tannins higher than green and yellow. The same observation has been shown with Italian figs [38], fig fruits from Turkey [36], and figs from Tunisia [39]. The carotenoid content is in the range of those obtained by Zegane *et al.* [40], with amounts of 1-54.4 mg/kg. However, the content is higher than that recorded by [41], for extra virgin olive oil from Chemlal from four sites in Algeria, with amounts of 0.67 and 1.70 mg/kg and that of Thaipong *et al.* [42], For Croatian olive oils, the content varies from 1.89 to 2.06 mg / kg whereas the carotenoid contents for figs before maceration is between 711.32 and 623.02 $\mu\text{g}/100\text{g}$ of dry matter black (FB) and white (FW) dried figs successively. Our results are lower than that found by Darjazi [30], with around 11 mg E $\beta\text{C}/100\text{g}$ MS, to reach a level of 928.14 and 745.12 $\mu\text{g}/100\text{g}$ of dry matter black fig macerates (FBO) and white fig macerates (FWO) successively with a significant increase. This can be attributed to several factors such as; the varietal difference of the figs and the composition of the olive oil and also to their proportion in the mixture. According to Çalışkan and Aytakin Polat [36], the fig contains several carotenoids such as lutein, cryptoxanthin and lycopene which are the most abundant.

3. Antioxidant activity

3.1. Reducing power test

The results obtained show a strong significant correlation between the reducing power and the concentration of proteins, total polyphenols, flavonoids, carotenoids and antioxidant activity according to Jayaprakasha *et al.* [44] the reducing power is a simple, reproducible test and shows a significant correlation with the quantity of PPTs. All extracts showed a lower

reducing power than that of ascorbic acid, .However, black fig macerate extract (OBF) a high reducing power. The FRAP increases in the following order: AA> OBF> OWF> FB> FW> BHT> OO> RMBO> RMWO. This can be explained by the difference in the phenolic composition of the extracts. In the same context, Jayaprakasha *et al.* [44] demonstrated a positive correlation between the polyphenols, the reducing power and the antioxidant capacity measured by the DPPH test. The antioxidant power of a plant extract depends on its purity, nature, chemical structure and the bioactivity of its constituents. Several studies have attributed the reducing ability to other non-phenolic molecules such as non-enzymatic substances that can also intervene in antioxidant activity [44].

3.2. Radical scavenging evaluation

The results of the anti-free radical activity reveal that black fig (48.88%) and white fig (42.37%) before maceration and after 90 days of maceration figs underwent a significant increase ($p < 0.05$) of the percentage of the inhibitory activity of the DPPH radical of the order of 57.72% to 52.80%. This increase can be explained by the increase in the content of flavonoids and phenolic compounds of the two samples. Statistical analysis revealed linear correlations between the anti-radical activity of olive oil extracts and K232, K270, total polyphenols and hydrosoluble tannin content .We observed a linear correlation between the anti-radical activity of fig extracts with protein content, total polyphenols, flavonoids and carotenoid content. According to Del Caro and Piga [37], estimates that black figs have higher antioxidant activity than green figs, which is consistent with the results of our study, where the highest activities were recorded for the black variety (Aberkane). Positive relationships between levels of antioxidant compounds and antioxidant activities of the fig are also obtained by many authors [36].

4. Anti-inflammatory activity

The inhibition of the edema is more important in the standard (Diclofenac) with a percentage of 84%, compared to extracts from macerates black figs (74.62%) followed by the remaining oil macerates black figs (72.07%) of white figs macerates (70.84%) and figs black and remaining oil Macerates white figs white figs and oil before maceration (70.84%, 70.82%,

68.29% and 67.02%) successively with a significant difference ($p \leq 0.05$). This can be explained by the fact that Diclofenac is a pure molecule unlike the extracts used which are crude extracts. It was significant ($p \leq 0.05$) at the 3rd and 4th hour between the standard and the extract, which is explained by the difference in the chemical composition and the mode of action of the two samples. The inhibition of the standard (Diclofenac) reached its maximum at the 4th hour and at the same time for all the phenolic extracts studied.

CONCLUSION

In conclusion, the data obtained from this research show that white and black figs and extra virgin olive oil have high levels of micronutrients and secondary compounds in their native states and that the maceration of the two varieties of figs has given a strong correlation and synergy between their physico-chemical, biochemical and nutritional composition. The results of this study also show that these local products are an invaluable treasure that can be valorized as therapeutic products contributing to the production of medicines by exploiting their active ingredients mainly in cosmetology, pharmacy, human and animal nutrition. They can be considered as a very important natural source of phytopharmaceutical constituents used to eradicate the free radicals responsible for many pathologies. Similarly, it would be interesting to consider the use of these natural resources to replace synthetic antioxidants widely used in the food and pharmaceutical industries.

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