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Physico-chemical and antioxidant properties of oils and by-products obtained by cold press-extraction of Tunisian *Opuntia* spp. seeds

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ABSTRACT

Screw pressed prickly pear seed cakes from four Tunisian varieties were analyzed for their chemical and some functional and antioxidant properties, along with the physico-chemical characteristics of the extracted oils. This extraction yielded 40.14 to 62.95% of oil. Fatty acid (FA) composition revealed domination of linoleic and oleic acids and high content of polyunsaturated FA (~69-74 %) with presence of ɣ-linolenic, docosadienoic and eicosapentanoic acids. Characterization of the cake seeds showed high amounts of total dietary fibers (82.41-83.54 %), important polyphenol content (~113-180.81 mg GAE/100g dry matter (DM)) and scavenging activity (IC_{50}: 450-763 μg/mL), good oil holding capacity (4.31g/g for *Opuntia Stricta* variety), and light beige colors. Amino-acid composition revealed that major components were Glutamic acid, Arginine and Aspartic acid. The data indicate that cold pressed *Opuntia* seeds could be a source of fiber concentrates which could be used as functional ingredients in the food industry, as well as good quality oils.

1. Introduction

In Tunisia, prickly pear fruits grow spontaneously and are used exclusively for consumption as fresh fruit. The edible pulp is constituted by about 10-15 % seeds (Ramadan & Mörsel, 2003). These latter are generally discarded as waste, while their utilization as a by-product could lead to a new source of oil (Habibi et al., 2008) as well as functional ingredients, which could be used in the food industry (seed cake). In fact, prickly pear seed oil was found to be rich in polyunsaturated fatty acids, vitamins, and phenolic compounds (Chougui et al., 2013). Furthermore, solid waste residue obtained after oil extraction is rich in dietary fibers (Chahdoura et al., 2015a).

Some methods used for oil extraction may alter its quality. Screw pressing was found to be an alternative to solvent extraction (Martínez et al., 2013). Even though solvent method yields more quantity of oil than mechanical press, the latter is safer, simpler, less expensive, and ecologically friendly (Thanonkaew et al., 2012). In fact, cold press oils obtained by screw press are healthier since they are rich in bioactive compounds such as essential fatty acids, phenolics and tocopherol (Teh & Birch, 2013). In addition, the end products (oil and press cake) are free of chemicals and consequently, the obtained oils do not require refining procedures.

There is lack of scientific literature in studying extraction of oil from prickly pear seeds by screw press method. Therefore, the purpose of this investigation was to characterize two fractions obtained by cold pressing seeds of four prickly pear varieties: oils and press cakes. Thus, we studied physico-chemical properties of oils as well as chemical and some functional and antioxidant characteristics of the residual by-product.

The obtained results could be a useful tool for the valorization of prickly pear seed by-products as a source of natural ingredients in food and cosmetic industries.

2. Materials and methods

2.1. Plant material and sample preparation

In this study, mature fruits of prickly pears were used:

- *O. ficus indica* (spiny): yellow-orange (IS)

ADF, Acid detergent fiber; ADL, Acid detergent lignin; DM, Dry matter; DPPH, 1,1-diphenyl-2-picrylhydrazyl; FA, Fatty acid; GAE, Gallic acid equivalent; GC, Gas-liquid chromatography; HPLC, High performance liquid chromatography; IV, Iodine value; NDF, Neutral detergent fiber; OHC, Oil holding capacity; QE, Quercetin equivalent; SDF, Soluble dietary fibers; TDF, Total dietary fibers; TP, Total phenolic; WHC, Water holding capacity.

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- *O. ficus indica* (thornless): yellow-orange (ITy) and pink-purple (ITp) ecotypes.
- *O. stricta* (OS): red-purple.

All samples were collected between August and November. The spiny form (IS) was from the region of Mahdia (centre of Tunisia), whereas the thornless forms (ITy and ITp) were from the regions of Kasserine (west-centre of Tunisia) and Nabeul (northeast of Tunisia), respectively. Finally, O. *stricta* (OS) came from the region of Mahdia.

Fruits were processed in a fruit beverage industry (Cindrella, Mahdia) as described: Fruits were brushed under tap water, peeled manually and crushed. Then, automatic sieves (C120 Robot-Coupe, France) were used for pulp and seeds separation.

Seeds were cleaned, air dried at ambient temperature and weighted. Extraction of seed oil was carried out in Nopal Tunisie society (Kasserine, Tunisia) as follows: Firstly, seeds were dried at 50°C for 30 min in order to improve extraction yield. Then, oil was extracted by cold pressing using a screw press. Two fractions were analyzed in this study: The solid residues (i.e. press cakes) and seed oils. Solid residues were transformed into fine powders using a grinder (Fritsh, pulverisette 19). Finally, the two fractions were stored at -20°C until analysis.

2.2. Analytical methods

2.2.1. Physical properties of prickly pear fruit cultivars

The physical properties were determined on 15 randomly selected prickly pear fruits: length (cm), width (cm), total weight (g) and percentage of seeds in the total fruit (%).

For total weight determination, the fruit was weighted entirely with its skin using a balance with a sensitivity of 0.001 g.

2.2.2. Oil extraction yield

The oil extraction yield was calculated using the following equation (Eqn 1):

$$\text{Yield (\%)} = \text{quantity of the recovered oil (g)} \times 100/\text{quantity of total oil originally present in the seeds (g)} \quad (1)$$

2.2.3. Seed oil analysis

The density was measured using a Mettler PM 200 balance. The refractive index was determined using an Abbe refractometer (Optech, Germany) at 20°C.

Iodine value (IV) was calculated from fatty acid percentages according to the method of Kyriakidis and Katsiloulis (2000).

The fatty acid composition was analyzed by gas-liquid chromatography (GC) after derivatization at room temperature of fatty acid to methyl esters (FAMEs) with a mixture BF3 methanol (IUPAC, 1992). GC analyses were achieved according to the method described by Yaich et al. (2011).

The CieLab coordinates (L*, a*, b*) of seed oils were directly read with a spectrophotocolorimeter (Tintometre, Lovibond PFX 195 V 3.2, Amesbury, UK).

L^*: is a measure of lightness, ranging from 0 (black) to 100 (white)
a^*: ranges from -100 (greenness) to +100 (redness).
b^*: ranges from -100 (blueness) to +100 (yellowness).

The hue angle (h*) and chroma or intensity (C*) were calculated according to the following equations (Eqn 2 and 3):

$$h* = \arctan\ (b* / a*) \quad (2)$$

$$C* = \left(a*^2 + b*^2\right)^{1/2} \quad (3)$$

2.2.4. Seed Cakes analyses

2.2.4.1. Chemical analyses. Dry matter was determined by drying seed cake at 105°C to constant weights (AOAC, 1995). Ash content was determined by sample incineration at 550°C using a muffle furnace (NABER, Germany) for 8 h. The total ash was expressed as percent of dry weight (AOAC, 1995).

Protein (N × 6.25) was analyzed according to the Kjeldhal procedure (AOAC, 1995). Fat content was determined by extraction with hexane using a Soxhlet apparatus (AOAC, 1995).

Total soluble solids content was determined according to phenol-sulfuric method as described by Dubois et al. (1956). Pectin content was determined following the colorimetric method of Englyst et al. (1994), using galacturonic acid as a standard.

Total dietary fibers (TDF) were determined by a modified method of Englyst et al. (1992). To 1 g of *Opuntia* seed cake powder, 10 ml of distilled water was added and the mixture was maintained at 100°C for 10 min. 40 ml of absolute ethanol was added and the mixture was agitated, and then left in ice water for 30 min. Then, the mixture was centrifuged at 1500 g for 10 min and the residue was added by 50 ml of 85% ethanol, mixed and centrifuged at 1500 g for 10 min. This step was repeated using 50 ml of absolute ethanol. The obtained mixture was centrifuged and the supernatant was removed. Finally, the residue was dried at 80°C during 24 h. Results were expressed as g/100 g dry samples.

Dietary fiber fractions were determined according to Van Soest (1963) and Van Soest and Wine (1967). Neutral detergent fiber (NDF) is defined as the insoluble part of a neutral detergent solution. Acid detergent fiber (ADF) is the fraction of insoluble components in the acid detergent solution. Acid detergent lignin (ADL) is defined as the insoluble lignin fraction in 72% H_2SO_4. The amount of cellulose in samples was calculated by difference between ADF and ADL, whereas that of hemicellulose was calculated by difference between NDF and ADF amounts.

Amino acids were analyzed using high performance liquid chromatography (HPLC) following the OJEC standard method (OJEC, 1998). Seed cakes powders were hydrolyzed with concentrated HCl (6N) at 110°C for 24 h. Then, 30 ml of citrate buffer (pH 2.2) were added, and the pH was adjusted between 0.5 and 1, with 7.5 N NaOH and pH 2.2 with 1 N NaOH. The obtained sample was diluted to 100 ml with citrate buffer after adding 1 ml of a norleucine solution 50 μM (internal standard). The sample was filtered through a 0.2 μm nylon filter before being analyzed by HPLC (Yaich et al., 2011). Sulphur-containing amino acids, cystine and methionine were analyzed using the OJEC method (1998), after a pre-hydrolysis oxidation with performic acids. The HPLC system (Biochrom 20 Plus) was equipped with an UV–vis detector with two wavelengths, 440 nm (proline) and 570 nm (other amino acids), and a cation exchange column (200 × 4.6 mm). The contents of the different recovered amino acids were presented as g/100 g dry matter.

Water activity (a_w) was measured at 25°C using a Novasina (Aw Sprint TH-500, Switzerland) apparatus.

Extraction of phenolic compounds from seed cake powders was carried out following the method described by Chougui et al. (2013) with small modifications. 2 g of sample were extracted in the dark with 20 ml of methanol 80 % (v/v) containing 0.01% HCl for 2 h of continuous agitation at room temperature. The mixture was centrifuged at 5000 g for 15 min. The pellet was subjected to a second extraction following the same conditions. After centrifugation, the supernatants were collected, concentrated by evaporation of the solvent, using a rotary evaporator. Finally, the obtained extract was reconstituted in 10 ml of pure methanol and stored at -20°C. This extract was used for determination of phenolic and flavonoid contents, as well as antioxidant properties.

Total phenolic (TP) content was determined by the Folin-Ciocalteu method according to Singleton et al. (1999). Results were expressed as gallic acid equivalent (GAE) per gram of dry matter.

Total flavonoid content was examined following the slightly modified method of Jia et al. (1999). In brief, 1 ml of distilled water and 150

μl of Na NO_2 (15%) were added to 250 μl of extract. The mixture was kept at room temperature for 6 min. 75 μl of Al Cl_3 (10 %, w/v) were added to the mixture and incubated for 5 min, followed by addition of 1 ml NaOH (40 g/l). Mixture was made up to 2.5 ml with distilled water. Then, absorbance was measured at 510 nm, after incubation for 15 min at room temperature. The total flavonoid content was calculated based on the standard curve for quercetin solutions and expressed as quercetin equivalents (mg quercetin/ 100 g of dry weight).

Free radical scavenging activity was evaluated with the DPPH· (1,1-diphenyl-2-picrylhydrazyl) radical assay. The antiradical capacity of the sample extracts was determined according to Bersuder et al. (1998). A volume of 500 μl of each sample at different concentrations was added to 375 μl of 99% ethanol and 125 μl of DPPH solution (0.02% in ethanol) as free radical source. The mixture was then vortexed vigorously and then incubated for 60 min in the dark at room temperature.

The absorbance was measured at 517 nm. Antioxidant activity was expressed as percentage DPPH-scavenging activity relative to the control, using the following equation (Eqn 4):

$$\% \text{ Radical scavenging activity} = \left[A_{control} - A_{sample}/A_{control}\right] \times 100 \quad (4)$$

Where $A_{control}$ is the absorbance of the control and A_{sample} is the absorbance of the sample extracts.

The reducing power of samples was determined using the method of Oyaizu (1986). Each extract (1 ml) was mixed with 2.5 ml of 0.2 M phosphate buffer (pH 6.6) and 2.5 ml of 1 % potassium ferricyanide [$K_3Fe(CN)_6$]. After incubation at 50°C for 20 min, 2.5 ml of 10 % trichloroacetic acid were added to the mixture followed by centrifugation at 3000 g for 10 min at room temperature. 2.5 ml of the supernatant of solution was mixed with 2.5 ml distilled water and 0.5 ml of 0.1 % ferric chloride ($FeCl_3$) and the absorbance was measured at 700 nm. Increased reaction mixture absorbance indicated increased reducing power.

The antioxidant activity was expressed as IC_{50} value (μg/mL) which is the extract concentration corresponding to 50% of antioxidant activity, or 0.5 of absorbance in the reducing power assay.

2.2.4.2. Functional properties

Water holding capacity (WHC) of seed cakes powders was determined using the method described by MacConnell et al. (1974). WHC was expressed as g of water fixed/g sample.

OHC was measured using the method described by Lin et al. (1974). It was expressed as g oil *held/g sample.*

2.2.4.3. Color measurement. Color measurement of seed cakes was carried out using a spectrophotocolorimeter Mini Scan XE^{TM} (Hunter Lab, In., Reston, VA, USA).

2.2.5. Statistical analysis

All analytical determinations were carried out at least in triplicate. Values of different parameters were expressed as the mean ± standard deviation ($\bar{x} \pm$ S.D.). Statistical analyses were assessed using a statistical software program (STATISTICA, Release 5.0 Stat Soft Inc. Talsa, OK). Duncan's test was performed to evaluate the significance of differences between mean values at the level of $p < 0.05$.

3. Results and discussion

3.1. Physical properties of prickly pear studied cultivars

A specific knowledge of the cactus pear physical properties is necessary to design of fruit processing equipment, since the functioning of some machines is influenced by the size and the shape of the fruit (Kabas et al., 2006). Physical properties of prickly pear samples are given in Table 1. The studied cultivars exhibited significant morphological variations ($p < 0.05$). *Opuntia stricta* (OS) was the smallest in shape and weight ($p < 0.05$), whereas ITp cultivar had the highest value for weight ($p < 0.05$). According to Felker et *al.* (2002), fruit characteristics of size and weight are determined by genetic factors. These play important role in consumer selection (De Wit et al., 2010).

Finally, OS had significantly ($p < 0.05$) higher percentage of seeds (6.6 %) than samples from *indica* variety (IS, ITp and ITy), with values ranging from 2.76 to 3.8 %. This may affect the seed by-product yield.

3.2. Seed oils characteristics

3.2.1. Seed oil recovery

The oil content of the studied prickly pear seeds ranged from 7.1 to 11.4 % MS, respectively for ITy and OS (data not shown).

The oil yields, obtained from *Opuntia* seeds are presented in Table 2. Values ranged from 40.14 to 62.95 %, respectively for IS and ITy. Oil yield differences could be explained mainly by intrinsic characteristics of seeds such as tissue porosity and structure, as well as moisture content. In fact, according to Singh and Bargale (1990), high moisture content can increase plasticity which consequently reduce the level of compression, and give lower oil recovery. In our case, the seeds had initial moisture contents ranging from 9.33 to 12.1, respectively for IS and ITp (data not shown). Compared with the other cultivars, OS and ITy had significantly the highest yields ($p < 0.05$) (60.96 and 62.95%, respectively), corresponding to a seed moisture content in the average of 10 % (data not shown). A similar tendency was observed by Singh and Bargale (1990) for flaxseed screw pressing, who reported that oil recovery increased, as moisture content increased from 5 to 7 %, but decreased at 9 % moisture content.

3.2.2. Physico-chemical characteristics of seed oils

Physico-chemical properties of *Opuntia* seed oils extracted by screw press are illustrated in Table 2. Significant differences ($p < 0.05$) between varieties were observed for oil densities, with values ranging from 0.911 to 0.941, respectively for yellow thornless (ITy) and spiny (IS) *indica* varieties. These values were comparable to those reported by Noureddini et al. (1992) for some vegetable oils (0.907 and 0.939, respectively for rapeseed and lesquerella oils).

Refractive indices were significantly different ($p < 0.05$) (1.472-1.475). These values were comparable to those found by other authors (Ennouri et al., 2005; El Mannoubi et al., 2009, Kiralan et al., 2014) for prickly pear seeds and black cumin oils.

ITp showed the highest ($p < 0.05$) iodine value (IV) (131.78), indicating higher content of unsaturated fatty acids. No significant differences ($p > 0.05$) were observed between IS and ITy cultivars. Ennouri et al. (2005) reported lower IV (101.5 and 91.6, respectively for *O. ficus indica* and *O. stricta*). This could be due to differences in the oil extraction methods. In fact, in their study, extraction was carried out with hexane in a Soxhlet system. Thanonkaew et al. (2012) reported that cold pressed rice bran oil had higher iodine number than that of solvent extracted oil.

Color is an important parameter for visual acceptance of oils (Thanonkaew et al., 2012). Analysis of oil colors showed significant differences ($p < 0.05$) between the analyzed samples. The thornless form of *indica* variety (ITy) had the lightest and the most ($p < 0.05$) yellow-colored seed oil (L^*=82.72; b^*=66.79). This latter had higher b^* value than other vegetable oils such as olive (Chtourou et al., 2013) and date seed (Besbes et al., 2004) oils, with respectively 10.58 and ~ 59. This may be due to the presence of more yellow pigments.

Table 3 illustrates fatty acid composition of prickly pear seed oils. For the four cultivars, linoleic acid was the major fatty acid (from 67.6 to 70.3 %), followed by oleic (15.36-19.96 %) and palmitic (4.7-6.04 %). This was also reported by Ennouri et al. (2005) and El Mannoubi et al. (2009) for Tunisian *Opuntia* seed oils.

However, some fatty acids found in this study were not reported by these authors, particularly, ɣ-linolenic acid (from 1.19 to 1.98 %, respectively for ITy and ITp) and docosadienoic acid (C22 :2), found in

Table 1
Morphological characteristics of prickly pear from different *Opuntia* cultivars*.

	IS	ITp	ITy	OS
Length (cm)	9.12 ± 0.84^{a}	11.11± 1.5^{b}	8.5 ± 0.93^{ab}	4.28 ± 0.44^{c}
Width (cm)	5.56 ± 0.5^{a}	6.2 ± 0.7^{a}	4.96 ± 0.33^{a}	3.79 ± 0.22^{b}
Total weight (g)	106.83 ± 13.34^{a}	160.08 ± 39.15^{b}	93.28 ± 13.68^{a}	27.08 ± 3.27^{c}
Pourcentage of seeds (%)	3.6 ± 0.9^{a}	2.76 ± 1.2^{a}	3.8 ± 0.6^{a}	6.6 ± 0.8^{b}

* mean of 15 samplesIS: O. *ficus indica* « spiny » (yellow-orange) ; ITp : *O. ficus indica* « thornless » (pink-purple) ; ITy : *O. ficus indica* « thornless » (yellow-orange) ; OS : *O. stricta*.Results are expressed as mean values of 15 determinations ± SD; Means within the same row with different letters are significantly different ($p < 0.05$).

Table 2
Physico-chemical characteristics of *Opuntia* seed oils.

Parameters	IS	ITp	ITy	OS
Oil yield (%)	40.14 ± 1.40^{a}	46.78 ± 1.30^{b}	62.95 ± 1.50^{c}	60.96 ± 1.10^{c}
Density	0.941± 0.002^{a}	0.916± 0.002^{b}	0.911 ±0.002^{d}	0.936 ± 0.003^{c}
Refractive index	1.473 ± 0^{a}	1.474 ± 0^{b}	1.472 ± 0^{d}	1.475 ± 0.001^{c}
IV (g of I_2/100 g oil)	129.2 ± 0.21^{a}	131.78 ±0.25^{b}	129.3 ± 0.1^{a}	129.85 ± 0.05^{c}
Color:				
L^*	25.22 ± 0.05^{a}	43.14 ± 0.01^{b}	82.72 ± 0.02^{d}	31.86 ± 0.01^{c}
a^*	2.22 ± 0.06^{a}	0.70 ± 0.02^{b}	0.35 ± 0.01^{d}	0.74 ± 0.02^{b}
b^*	35.21 ± 0.09^{a}	37.83 ± 0.03^{b}	66.79 ± 0.06^{d}	23.47 ± 0.06^{c}
C^*	35.28 ± 0.09^{a}	37.83 ± 0.03^{b}	66.79 ± 0.06^{d}	23.48 ± 0.06^{c}
h^*	86.39 ± 0.09^{a}	88.93 ± 0.02^{b}	89.70 ± 0.01^{d}	88.19 ± 0.05^{c}

IV : Iodine value
IS : *Opuntia ficus indica* « spiny » (yellow-orange) ; ITp : *Opuntia ficus indica* « thornless » (pink-purple) ; ITy : *Opuntia ficus indica* « thornless » (yellow-orange) ; OS : *Opuntia stricta.*
Results are expressed as mean values of three determinations ± SD; Means within the same row with different letters are significantly different ($p < 0.05$).

Table 3
Fatty acid composition of *Opuntia* seed oils (g/100g of total fatty acid).

Fatty acid	IS	ITp	ITy	OS
Palmitic C16:0	6.04 ± 0.13^{a}	5.04 ± 0.08^{b}	4.70 ± 0.01^{c}	4.72 ± 0.01^{c}
Palmitoleic C16 :1	3,15 ± 0.08^{a}	3,06 ± 0.03^{b}	2.86 ± 0.02^{d}	2.62 ± 0.02^{c}
Stearic C18:0	2.78 ± 0.03^{a}	1.67 ± 0.04^{b}	2.24 ± 0.03^{d}	1.96 ± 0.02^{c}
Oleic C18:1	19.96 ± 0.13^{a}	15.36 ± 0.09^{b}	19.54 ± 0.04^{d}	17.42 ± 0.02^{c}
Linoleic C18:2	67.60 ± 0.16^{a}	70.30 ± 0.06^{b}	68.06 ± 0.06^{c}	67.68 ± 0.14^{a}
ɣ- Linolenic C18 :3	1.31 ± 0.05^{a}	1.98 ± 0.02^{b}	1.19 ± 0.04^{c}	1.50 ± 0.04^{d}
Eicosenoic C20:1	0.58 ± 0.02^{a}	1.28 ± 0.04^{b}	0.82 ± 0.03^{c}	0.66 ± 0.03^{d}
Docosadienoic C22:2	0.41 ± 0.02^{a}	0.96 ± 0.04^{b}	0.60 ± 0.05^{c}	1.31 ± 0.01^{d}
Eicosapentanoic C20:5	ND	1.00 ± 0.03^{a}	ND	0.92 ± 0.01^{b}
Nervonic C24:1	ND	ND	ND	1.74 ±0.02
MUFA	23.69 ± 0.23^{a}	19.70 ± 0.16^{b}	23.22 ± 0.09^{c}	22.44 ± 0.09^{d}
PUFA	69.32 ± 0.23^{a}	74.24 ± 0.15^{b}	69.85 ± 0.15^{c}	71.40 ± 0.2^{d}
UFA/SFA	10.54 ± 0.19^{a}	14.00 ± 0.28^{bd}	13.4 ± 0.06^{c}	14.05 ± 0.03^{b}

ISy : *Opuntia ficus indica* « spiny » (yellow-orange) ; ITp : *Opuntia ficus indica* « thornless » (pink-purple) ; ITy : *Opuntia ficus indica* « thornless » (yellow-orange) ; OS : *Opuntia stricta.*
ND : not detected ; UFA/SFA : unsaturated/saturated fatty acids ratio ; MUFA : monounsaturated fatty acids ; PUFA : polyunsaturated fatty acids.
Results are expressed as mean values of three determinations ± SD; Means within the same row with different letters are significantly different ($p < 0.05$).

small amounts, with a highest content ($p < 0.05$), detected in OS cultivar (1.31 %). Eicosapentanoic acid (C20 :5) has an important role in the prevention of many diseases (Nonviho et al., 2015). This fatty acid was only present in ITp and OS (1 and 0.92 %, respectively). Significant differences were observed between these two cultivars ($p < 0.05$). However, nervonic acid C24:1 was detected only in OS variety (1.74 %).

Difference in fatty acid composition among cultivars was previously reported in many studies for many vegetable oils such as date (Besbes et al., 2004) and *Opuntia* (Ennouri et al., 2005; Chougui et al., 2013) seed oils. IS was the richest ($p < 0.05$) in palmitic, stearic, palmitoleic and oleic acids, whereas ITp caltivar had the highest content ($p < 0.05$) in eicosenoic, linoleic and ɣ- linolenic acids.

In addition, the studied seed oils were found to be highly unsaturated. OS and ITp cultivars had significantly the highest (UFA/SFA) ratios ($p < 0.05$) (14.05 and 14, respectively). The highest levels ($p < 0.05$) in polyinsaturated and monounsaturated fatty acids were recorded respectively for ITp and IS cultivars (74.24 and 23.69 and IS, respectively). These levels were higher, compared to other studies for Tunisian *Opuntia* seed oils (Ennouri et al., 2005; El Mannoubi et al., 2009). This difference could be due to degree of maturation, climatic conditions or to cold pressed extraction. In fact, according to Simopoulos et al. (2000), cold pressed oils are characterized by high content in polyunsaturated fatty acids such as α-linolenic and linoleic acid. These results raise a great interest since *Opuntia* seed oils are natural source of healthy fatty acids.

3.3. Seed cakes characteristics

3.3.1. Chemical composition

Table 4 shows that seed cakes had dry matter contents ranging from 96.23 to 98.77 %). The highest value ($p < 0.05$) was recorded for ITp va-

Table 4
Chemical composition (g/100g DM) and water activities of seed cakes of *Opuntia* cultivars.

Parameters	IS	ITp	ITy	OS
Dry matter (%)	98.7 ± 0.01^{a}	98.77 ± 0.02^{b}	96.23 ± 0.01^{d}	98.44 ± 0.02^{c}
Ash	1.58 ± 0.02^{a}	1.39 ± 0.03^{b}	1.29 ± 0.02^{d}	1.71 ± 0.02^{c}
Fat	4.28 ± 0.06^{a}	3.98 ± 0.03^{b}	2.63 ± 0.03^{d}	4.45 ± 0.04^{c}
Total soluble sugars	0.12 ± 0.01^{a}	0.13 ± 0.01^{a}	0.23 ± 0.01^{b}	0.12 ± 0.01^{a}
Proteins	6.5 ± 0.2^{a}	6.97 ± 0.2^{ac}	7.52 ± 0.42^{c}	7.65 ± 0.5^{c}
TDF	82.83 ±0.42^{ab}	82.41 ± 0.37^{a}	83.27 ± 0.21^{b}	83.54±0.18^{b}
Pectins	4.52 ± 0.03^{a}	4.26 ± 0.02^{b}	4.58 ± 0.02^{d}	4.34 ± 0.02^{c}
NDF	75.88 ± 0.01^{a}	75.25 ± 0.04^{b}	77.60 ± 0.03^{d}	73.12 ± 0.03^{c}
ADF	49.82 ± 0.05^{a}	51.5 ± 0.03^{b}	53.92 ± 0.03^{d}	47.92 ± 0.06^{c}
ADL	18.73 ± 0.02^{a}	19.27 ± 0.04^{b}	18.11 ± 0.02^{d}	14.82 ± 0.03^{c}
Cellulose	31.09 ± 0.07^{a}	32.22 ± 0.04^{b}	35.81 ± 0.04^{d}	33.1 ± 0.09^{c}
Hemicellulose	26.05 ± 0.04^{a}	23.75 ± 0.07^{b}	23.68 ± 0.04^{b}	25.21 ± 0.08^{c}
aw	0.150 ± 0.001^{a}	0.187±0.011^{b}	0.328±0.002^{d}	0.167 ± 0.003^{c}

ISy : *Opuntia ficus indica* « spiny » (yellow-orange) ; ITp : *Opuntia ficus indica* « thornless » (pink-purple) ; ITy : *Opuntia ficus indica* « thornless » (yellow-orange); OS : *Opuntia stricta* ; TDF : total dietary fibers ; NDF : neutral detergent fiber; ADF : acid detergent fiber; ADL : acid detergent lignin.
Results are expressed as mean values of three determinations ± SD; Means within the same row with different letters are significantly different ($p < 0.05$).

riety. Such low moisture content, as well as low water activities (0.150-0.328) allows these by-products to have an easy conservation, particularly IS cultivar, which had significantly the lowest a_w value ($p < 0.05$).

The approximate composition is dominated by dietary fibers (82.41-83.54 %). No significant differences were observed between IS, ITy and OS samples ($p > 0.05$). Chahdoura et al. (2015a) analyzed prickly pear seeds and found a TDF content of 78.87 g/100 DM for *O. microdasys* variety.

TDF were dominated by the insoluble fraction. In fact, seed cakes contained lower content in soluble dietary fibers (SDF). The highest value of pectin ($p < 0.05$) was recorded for ITy cultivar (4.58 g/100g DM).

Composition of cell wall polysaccharides (insoluble dietary fibers) of seed cakes showed significant differences ($p < 0.05$) between the studied samples for NDF, ADF and ADL. High amounts of NDF (73.12-77.60) were detected, particularly for ITy variety, which had significantly the highest content (77.6 %MS) ($p < 0.05$). These values were near those reported by Bouaziz et al. (2010) for defatted date seeds (70.81 %MS) but much higher than those found by Boudouma (2009) for Algerian durum wheat bran (41.7 %MS).

Such high content in fiber allow *Opuntia* seed cakes to be considered as fiber concentrates which could be used as ingredient in some dietetic food formulations.

Insoluble dietary fibers were represented mainly by cellulose (31.09-35.8 %), followed by hemicellulose (23.68-26.05 %) and lignin (ADL) (14.82-19.27%). ITy and IS cultivars had respectively the highest contents in cellulose and hemicellulose ($p < 0.05$).

Residual fat contents were significantly different for the studied seed cakes, with values ranging from 2.63 and 4.45 % DM (Table 4). These values could be explained by seed oil extraction method (screw press) which gives lower oil extraction yield, compared with solvent methods, and consequently higher retained oil in the seed by-products (Labuckas et al., 2014).

Significant differences were also recorded between the studied varieties for ash content ($p < 0.05$). Ash values ranged from 1.29 to 1.71, respectively for ITy and OS varieties. These values were lower than those found by Chahdoura et al. (2015a) for *O. macrorhiza* variety (2.5 g/100 g FM).

Total sugar content ranged from 0.12 to 0.23, respectively for IS and ITy. No significant differences were observed between IS, ITp and OS samples ($p > 0.05$). Similar results were reported by Chahdoura et al. (2015a) for *O. macrorhiza* seeds (0.48 % DM). Protein content (Kjeldahl method) varied between 6.5 and 7.65 %, respectively for IS and OS varieties (Table 4). These values were higher than those reported by Chahdoura et al. (2015a), but lower than those of Nassar (2008) for other *Opuntia* varieties. No significant differences were observed between ITp, ITy and OS samples ($p > 0.05$).

Amino-acid composition of seed cakes (Table 5) showed that Glutamic acid was the major amino-acid found (1.27-1.51 %), followed by Arginine (0.76-0.95 %) and Aspartic acid (0.53-0.64%). The majority of essential amino-acids were detected in seed cakes but in fewer amounts. However, Tryptophan was probably destroyed during acid hydrolysis. The highest values were observed for OS variety, for most of the amino-acids ($p < 0.05$). However, no significant differences were observed between samples ($p > 0.05$) for Pro, Cys-Cys, Val, Met, and Ile. Similar results were found by Bouaziz et al. (2008) for defatted date seeds. In their study, they reported the same dominant amino-acids (Glu, Arg and Asp).

According to these results, the analyzed by-products could be considered as having interesting biological value.

3.3.2. *Polyphenols, flavonoids and antioxidant properties*

Phenolics are secondary metabolic products, mainly composed by flavonoids (such as flavanones, flavanols and anthocyanidins), phenolic acids, stilbenes, coumarins and tannins (Lin et al., 1974). They could be used as a pertinent indicator of antioxidant capacity and can be used to screen the products intended to be used as natural sources of antioxidants in many functional foods (Viuda-Martos et al., 2011).

Total polyphenols and flavonoids levels are presented in Table 6. TP compounds ranged from 113.8 to 180.81 mg GAE/100g DM. The highest value was recorded for ITy seed by-products ($p < 0.05$). Similar results were obtained by Saïdani-Tounsi et al. (2011) for entire prickly pear seeds (172 mg GAE/100 g DM), and by Alu'datt et al. (2013) for olive cake (185 mg GAE/100 g DM), but higher than those reported for some seed by-products, such as defatted flaxseed (120 mg GAE/100 g DM) (Alu'datt et al., 2013) and wheat bran (100 mg GAE/100 g DM) (Kahkonen et al., 1999). Thus, *Opuntia* seed cakes could be considered as natural source of polyphenols.

Total flavonoids contents were significantly different between cultivars ($p < 0.05$), ranging from 40.96 to 59.47 mg QE/100 g DM, respectively for ITp and OS variety. These values were lower than those reported by Saïdani-Tounsi et al. (2011) which analyzed non-defatted seeds.

Antioxidant activity was measured by evaluating DPPH scavenging activity and reducing power (IC_{50} values). The highest values ($p < 0.05$) were recorded for the pink thornless variety (763.5 and 204.5 μg/mL, respectively for DPPH scavenging activity and reducing power), indicat-

Table 5
Amino acid composition of *Opuntia* seed cakes (g/100g of dry matter).

Amino acid	IS	ITp	ITy	OS
Asp	0.54 ± 0.04^{a}	0.53 ± 0.02^{ac}	0.56 ±0.03^{a}	0.64 ± 0.05^{bc}
Thr	0.24 ± 0.02^{a}	0.23 ±0.01^{a}	0.25 ±0.02^{a}	0.29 ± 0.02^{b}
Ser	0.28 ± 0.01^{a}	0.27 ± 0.01^{a}	0.29 ±0.03^{a}	0.34 ±0.02^{b}
Glu	1.31 ± 0.07^{a}	1.27 ± 0.05^{a}	1.33 ± 0.06^{a}	1.51 ± 0.06^{b}
Pro	0.26 ± 0.03^{a}	0.29 ± 0.01^{a}	0.26 ±0.03^{a}	0.31 ±0.01^{a}
Gly	0.44 ±0.04^{a}	0.44 ± 0.02^{a}	0.46 ±0.04^{a}	0.59 ±0.03^{b}
Ala	0.27 ±0.02^{a}	0.28 ± 0.01^{a}	0.28 ± 0.02^{a}	0.33 ± 0.02^{b}
Cys-Cys	0.11± 0.02^{a}	0.10 ± 0.01^{a}	0.10 ± 0.01^{a}	0.13 ±0.01^{a}
Val	0.37 ± 0.04^{a}	0.35 ±0.02^{a}	0.36 ± 0.02^{a}	0.42 ±0.03^{a}
Met	0.06 ± 0.01^{a}	0.06 ± 0.01^{a}	0.08 ± 0.01^{a}	0.07 ±0.01^{a}
Ile	0.23 ± 0.02^{a}	0.22 ±0.01^{a}	0.24 ± 0.03^{a}	0.28 ±0.01^{a}
Leu	0.45 ± 0.03^{a}	0.43 ±0.03^{a}	0.47 ±0.03^{a}	0.55 ±0.04^{b}
Tyr	0.32 ± 0.01^{a}	0.32 ± 0.02^{a}	0.32 ± 0.02^{a}	0.41 ± 0.02^{b}
Phe	0.14 ± 0.01^{a}	0.13 ±0.01^{a}	0.14 ± 0.01^{a}	0.18 ±0.01^{b}
His	0.26 ± 0.02^{a}	0.26 ± 0.01^{a}	0.26 ± 0.02^{a}	0.32 ±0.01^{b}
Lys	0.19 ± 0.01^{a}	0.15 ± 0.01^{b}	0.16 ± 0.02^{db}	0.23 ± 0.01^{c}
Arg	0.78 ± 0.05^{a}	0.76 ± 0.05^{a}	0.79 ± 0.06^{a}	0.95 ± 0.07^{b}
Total	6.25 ± 0.45^{a}	6.09 ± 0.3^{a}	6.35 ± 0.46^{a}	7.55 ± 0.43^{b}

ISy : *Opuntia ficus indica* « spiny » (yellow-orange) ; ITp : *Opuntia ficus indica* « thornless » (pink-purple) ; ITy : *Opuntia ficus indica* « thornless » (yellow-orange); OS : *Opuntia stricta*
Results are expressed as mean values of three determinations ± SD; Means within the same row with different letters are significantly different ($p < 0.05$).

Table 6
Phenolic content, antioxidant, functional properties and color parameters of seed cakes.

Parameters	IS	ITp	ITy	OS
Total phenolics (mg GAE/100g DM)	148.43±4.95^{a}	113.80± 2.26^{b}	180.81 ± 9.89^{c}	143.74 ± 9.19^{a}
Total flavonoids (mg QE/100g DM)	53.15 ± 0.39^{a}	40.96 ± 0.79^{b}	56.48 ± 0.31^{c}	59.47 ± 0.81^{d}
DPPH scavenging activity (μg/mL)*	600.4 ± 0.5^{a}	763.5 ± 0.74^{b}	450 ± 0.78^{c}	650 ± 0.81^{d}
Reducing power (μg/mL)*	159 ± 0.41^{a}	204.5 ± 0.81^{b}	124.5 ± 0.35^{c}	177.4 ± 0.74^{d}
WHC (g/g)	1.8 ± 0.15^{a}	2 ± 0.11^{b}	2.08 ± 0.04^{ab}	2.06 ± 0.03^{ab}
OHC (g/g)	1.2 ± 0.2^{a}	2.07 ± 0.09^{b}	1.6 ± 0.09^{c}	4.31 ± 0.16^{d}
L^*	60.26 ± 0.04^{a}	63.31 ± 0.03^{b}	60.65 ± 0.05^{d}	62.15 ± 0.02^{c}
a^*	7.65 ± 0.03^{a}	6.99 ± 0.03^{b}	7.27 ± 0.02^{d}	6.38 ± 0.03^{c}
b^*	23.06 ± 0.02^{a}	22.69 ± 0.04^{b}	24.44 ± 0.05^{d}	25.56 ± 0.04^{c}
C^*	24.29 ± 0.03^{a}	23.74 ± 0.04^{b}	25.5 ± 0.05^{d}	21.25 ± 0.04^{c}
h^*	71.65 ± 0.05^{a}	72.87 ± 0.05^{b}	73.43 ± 0.04^{d}	72.53 ± 0.05^{c}

IS : *Opuntia ficus indica* « spiny » (yellow-orange) ; ITp : *Opuntia ficus indica* « thornless » (pink-purple) ; ITy : *Opuntia ficus indica* « thornless » (yellow-orange); OS : *Opuntia stricta* ; GAE: Gallic acid equivalent ; QE: quercetin equivalent.
*IC_{50} values
Results are expressed as mean values of three determinations ± SD; Means within the same row with different letters are significantly different ($p < 0.05$).

ing lower antioxidant activity. These values were near those found by Chahdoura et al. (2015b) for other Tunisian varieties.

These antioxidant properties could be explained by the presence of bioactive compounds, particularly polyphenolic compounds. Indeed, Martínez et al. (2012) reported that these latter are the main phytochemicals responsible for the antioxidant activity of fruits and vegetables. Chahdoura et al. (2015b) compared antioxidant properties of two varieties of prickly pears (*O. macrorhiza* and O. *microdasys*) and found higher values for the first variety. They explained this result by its higher levels of phenolic compounds.

Other compounds could also contribute to the antioxidant potential of some agri-food products, such as ascorbic acid, pigments and proteins. Indeed, in other studies, Chaalal et al. (2013) reported that antioxidant activities of Algerian *Opuntia* seeds were highly correlated with total phenolic and flavonoids, and also with carotenoids and ascorbic acid contents. However, Fernández-López et al. (2010) reported positive correlation of free radical scavenging capacity with TP and flavonoid content, but negative correlation with carotenoids content of the entire cactus pear fruit.

In a more recent study, Borchani et al. (2021) studied some antioxidant properties of two protein fractions extracted from the seed press cake of *Opuntia ficus indica* L. var. *inermis,* and reported that they exhibited good DPPH and metal scavenging activities, and a moderate reductive ability.

3.3.3. *Functional properties*

Water holding capacities were in the range of 2 g/g (Table 6). No significant differences were observed between ITp, ITy and OS ($p > 0.05$). These values were near those found by Larrauri (1999) for pineapple fiber concentrate (2.1 g/g dry matter) but lower than those reported by Besbes et al. (2010) for commercial pea fibers (3.08 g/g). This could be explained by the low content of *Opuntia* seed by-products in SDF fraction. In fact, according to Fuentes-Alventosa et al. (2009), WHC can be related to SDF content due to its dispersible quality in water.

Oil holding capacity (OHC) is related with the chemical structure of plant polysaccharides. It depends on surface properties, thickness, hydrophobic nature of fiber particles and overall charge density (Fernández-López et al., 2009).

Values were significantly different for OHC ($p < 0.05$). OS cultivar had the highest value (4.31%). This latter was much higher than that reported by Besbes et al. (2010) for a commercial pea fiber concentrate (1.74 g/g).

Thus, *Opuntia* seed by-products, particularly those of *O. Stricta* could be exploited in many foods to enhance their oil and flavor retention during industrial processing and storage (Besbes et al., 2010).

3.3.4. Color

Color is an important sensory attribute for determining visual acceptance of food ingredients. Table 6 illustrates CieLab coordinates for the dried seed cakes. The studied powders were light beige colored, with moderate color intensities (C^*). However, significant differences were observed for all measured parameters ($p < 0.05$). ITp sample had the lightest color ($L^* = 63.31$), and the lowest b^* value. Such color allows these by-products to be used in many light-colored food formulations without affecting considerably their initial appearance.

4. Conclusion

This investigation revealed that cold pressed *Opuntia* seeds generated two valuable fractions: oils and press-cakes. Analysis of the first fraction for four Tunisian cultivars showed high content of polyunsaturated fatty acids, and domination of linoleic and oleic acids, with presence of ɣ- Linolenic, docosadienoic (C22:2) and eicosapentanoic (C20:5) acids. The oil samples showed differences in color, with lighter and more yellowish color for the thornless yellow variety (ITy). Furthermore, press-cakes were characterized by high amounts of dietary fibers, with domination of the insoluble fraction. These by-products had also healthy composition of amino-acids, good antioxidant and functional properties, particularly for *O. Stricta* variety, having interesting OHC. Finally, press-cakes had moderate color intensities.

Thus, all these data suggest that the studied by-products could be considered as a cheap source of fibers and natural polyphenols, which could be used as ingredients, resulting in adding value to many food formulations.

Declaration of Competing Interest

All authors have no conflicts of interest.

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Manel Masmoudi: Conceptualization, Methodology, Writing – original draft, Writing – review & editing. **Arwa Baccouche:** Investigation, Formal analysis. **Maha Borchani:** Investigation. **Souhail Besbes:** Validation, Writing – review & editing. **Christophe Blecker:** Resources. **Hamadi Attia:** Resources, Supervision.

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References

Alu'datt, M. H., Rababah, T., Ereifej, K., & Alli, I. (2013). Distribution, antioxidant and characterization of phenolic compounds in soybeans, flaxseed and olives. *Food Chemistry, 139*, 93–99.

AOAC. (1995). *Official Methods of Analysis* (15th ed.). Washington, DC: Association of Official Analytical Chemists.

Bersuder, P., Hole, M., & Smith, G. (1998). Antioxidants from a heated histidine glucose model system. I: Investigation of the antioxidant role of histidine and isolation of antioxidants by high performance liquid chromatography. *Journal of the American Oil Chemists' Society, 75*, 181–187.

Besbes, S., Blecker, C., Deroanne, C., Drirac, N., & Attia, H. (2004). Date seeds: chemical composition and characteristic profiles of the lipid fraction. *Food Chemistry, 84*, 577–584.

Besbes, S., Ghorbel, R., Ben Salah, R., Masmoudi, M., Jedidi, F., Attia, H., & Blecker, C. (2010). Date fiber concentrate: Chemical compositions, functional properties and effect on quality characteristics of beef burgers. *Journal of Food and Drug Analysis, 18*(1), 8–14.

Borchani, M., Yaich, H., Abbès, F., Blecker, C., Besbes, S., Attia, H., & Masmoudi, M. (2021). Physicochemical, Functional and Antioxidant Properties of the Major Protein Fractions Extracted from Prickly Pear (*Opuntia ficus indica* L.) Seed Cake. *Waste and Biomass Valorization, 12*, 1749–1760.

Bouaziz, M. A., Besbes, S., Blecker, C., Wathlet, B., Deroanne, C., & Attia, H. (2008). Protein and amino acid profiles of Tunisian Deglet Nour and Allig date palm fruit seeds. *Fruits, 63*, 37–43.

Bouaziz, M. A., Ben Amara, W., Attia, H., Blecker, C., & Besbes, S. (2010). Effect of the addition of defatted date seeds on wheat dough performance and bread quality. *Journal of Texture Studies, 41*, 511–531.

Boudouma, D. (2009). Chemical composition of the durum wheat bran produced by industrial algerian mills. *Livestock Research for Rural Development, 21*(10), Article 167.

Chaalal, M, Louaileche, H., Touati, N., & Bey, M. B. (2013). Phytochemicals, in vitro antioxidant capacity and antiradical potential of whole and ground seeds of three prickly pear varieties: A comparative study. *Industrial Crops and Products, 49*, 386–391.

Chahdoura, H., Morales, P., Barreira, J. C. M., Barros, L., Fernandez-Ruiz, V., Ferreira, I. C. F. R., & Achour, L. (2015a). Dietary fiber, mineral elements profile and macronutrients composition in different edible parts of *Opuntia microdasys* (Lehm.) Pfeiff and *Opuntia macrorhiza* (Engelm.). *LWT-Food Science and Technology, 64*(1), 446–451.

Chahdoura, H., Barreira, J. C. M., Barros, L., Santos-Buelga, C., Ferreira, I. C. F. R., & Achour, L. (2015b). Seeds of *Opuntia* spp. as a novel high potential by-product: phytochemical characterization and antioxidant activity. *Industrial Crops and Products, 65*, 383–389.

Chougui, N., Tamendjari, A., Hamidj, W., Hallal, S., Barras, A., Richard, T., & Larbat, R. (2013). Oil composition and characterization of phenolic compounds of *Opuntia ficus-indica* seeds. *Food chemistry, 139*, 796–803.

Chtourou, M., Gargouri, B., Jaber, H., Abdelhedi, R., & Bouaziz, M. (2013). Comparative study of olive oil quality from *Chemlali* Sfax versus *Arbequina* cultivated in Tunisia. *European Journal of Lipid Science and Technology, 115*, 631–640.

De Wit, M., Nel, P., Osthoff, G., & Labuschagne, M. T. (2010). The effect of variety and location on cactus pear (*Opuntia ficus-indica*) fruit quality. *Plant Foods Human Nutrition, 65*(2), 136–145.

Dubois, M., Giles, K. A., Hamilton, F. K., Rebers, P. A., & Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry, 28*, 350–356.

El Mannoubi, I., Barrek, S., Skanji, T., Casabianca, H., & Zarrouk, H. (2009). Characterization of *Opuntia ficus indica* seed oil from Tunisia. *Chemistry of Natural Compounds, 45*(5), 616–620.

Englyst, H. N., Quigley, M. E., Hudson, G. J., & Cummings, J. H. (1992). Determination of dietary fiber as non-starch polysaccharides by gas liquid chromatography. *Analyst, 117*, 1707–1714.

Englyst, H. N., Quigley, M. E., & Hudson, G. J. (1994). Determination of dietary fiber as non starch polysaccharides with gaz-liquid chromatographic or spectrometric measurement of constituent sugars. *Analyst, 119*, 1497–1509.

Ennouri, M., Bourret, E., Mondolot, L., & Attia, H. (2005). Fatty acid composition and rheological behavior of prickly pear seed oils. *Food Chemistry, 93*, 431–437.

Felker, P., Soulier, C., Leguizamon, G., & Ochoa, J. (2002). A comparison of the fruit parameters of 12 Opuntia clones grown in Argentina and the United States. *Journal of Arid Environment, 52*(3), 361–370.

Fernández-López, J., Sendra-Nadal, E., Navarro, C., Sayas, E., Viuda-Martos, M., & Pérez-Alvarez, J. A. (2009). Storage stability of a high dietary fiber powder from orange by-products. *International Journal of Food Science and Technology, 44*, 748–756.

Fernández-López, J. A., Almela, L., Obón, J. M., & Castellar, R. (2010). Determination of anti-oxidant constituents in cactus pear fruits. *Plant Foods for Human Nutrition, 65*, 253–259.

Fuentes-Alventosa, J. M., Rodríguez-Gutiérrez, G., Jaramillo-Carmona, S., Espejo-Calvo, J. A., Rodríguez-Arcos, R., Fernández-Bolaños, J., Guillen, R., & Jiménez, A. (2009). Effect of extraction method on chemical composition and functional characteristics of high dietary fiber powders obtained from asparagus by-products. *Food Chemistry, 113*, 665–671.

Habibi, Y., Heux, L., Mahrouz, M., & Vignon, M. R. (2008). Morphological and structural study of seed pericarp of *Opuntia ficus-indica* prickly pear fruits. *Carbohydrate Polymers, 72*, 102–112.

International Union of Pure and Applied Chemistry (IUPAC). (1992). In C. Paquot, & A. Hautfenne (Eds.), *Standard method for the analysis of oils, fats and derivatives* (7th ed.). London: Blackwell Scientific Publications.

Jia, Z., Tang, M., & Wu, J. (1999). The determination of flavonoid contents in mulberry and their scavenging effects on superoxide radicals. *Food Chemistry, 64*, 555–559.

Kabas, O., Ozmerzi, A., & Akinci, I. (2006). Physical properties of cactus pear (*Opuntia ficus indica* L.) grown wild in Turkey. *Journal of Food Engineering, 73*, 198–202.

Kahkonen, M. P., Hopia, A. I., Vuorela, H. J., Rauha, J.-P., Pihlaja, K., Kujala, T. S., & Heinonen, M. (1999). Antioxidant Activity of Plant Extracts Containing Phenolic Compounds. *Journal of Agricultural Food Chemistry, 47*, 3954–3962.

Kiralan, M., Özkan, G., Bayrak, A., & Ramadan, M. F. (2014). Physicochemical properties and stability of black cumin (*Nigella sativa*) seed oil as affected by different extraction methods. *Industrial Crops and Products, 57*, 52–58.

Kyriakidis, N. B., & Katsiloulis, T. (2000). Calculation of iodine value from measurements of fatty acid methyl esters of some oils: comparison with the relevant American Oil Chemists Society method. *JAOCS, 77*(12), 1235–1238.
Labuckas, D., Maestri, D., & Lamarque, A. (2014). Effect of different oil extraction methods on proximate composition and protein characteristics of walnut (*Juglans regia L.)* flour. *LWT- Food Science and Technology, 59*, 794–799.
Larrauri, J. A. (1999). New approaches in the preparation of high dietary fibre powders from fruits by-products. *Trends in Food Science and Technology, 10*, 3–8.
Lin, M. J. Y., Humbert, E. S., & Sousulki, F. W. (1974). Certain functional properties of sunflower meal product. *Journal of Food Science, 39*, 368–370.
MacConnell, A. A., Eastwood, A., & Mitchell, W. D. (1974). Physical characterization of vegetable foodstuffs that could influence bowel function. *Journal of the Science of Food and Agriculture, 25*, 1457–1464.
Martínez, R., Torres, P., Meneses, M. A., Figuerola, J. G., Pérez-Alvarez, J. A., & Viuda-Martos, M. (2012). Chemical, technological and *in vitro* antioxidant properties of mango, guava, pineapple and passion fruit dietary fiber concentrate. *Food Chemistry, 135*, 1520–1526.
Martínez, M., Penci, M. C., Marin, M. A., Ribotta, P. D., & Maestri, D. M. (2013). Screw press extraction of almond (*Prunus dulcis* (Miller) D.A. Webb): Oil recovery and oxidative stability. *Journal of Food Engineering, 119*, 40–45.
Nassar, A. G. (2008). Chemical Composition and Functional Properties of Prickly Pear (*Opuntia ficus indica*) Seeds Flour and Protein Concentrate. *World Journal of Dairy & Food Sciences, 3*(1), 11–16.
Nonviho, G., Paris, C., Muniglia, L., Sohounhloué, D., & Brosse, N. (2015). *Lophira lanceolata* seed oil extraction method (ancestral or modern) modifies the properties of the oil. *Industrial Crops and Products, 67*, 49–54.
Noureddini, H., Teoh, B. C., & Davis Clements, L. (1992). Densities of vegetable oils and fatty acids. *Journal of American Oil Chemistry Society, 69*(12), 1184–1188.
Official Journal of the European Communities (OJEC) (1998). Determination of amino acid in feed by HPLC-Development of an Optimal Hydrolysis and Extraction Procedure by the EU Commission DGXII in three International Collaborative Studies, 257, 14–28.
Oyaizu, M. (1986). Studies on product of browning reaction prepared from glucose amine. *Japanese Journal of Nutrition, 44*, 307–315.
Ramadan, M. F., & Mörsel, J-T. (2003). Oil cactus pear (*Opuntia ficus* L.). *Food Chemistry, 82*, 339–345.
Saïdani-Tounsi, M., Ouerghemmi, I., Ksouri, R., Wannes, W. A., Hammrouni, I., & Marzouk, B. (2011). HPLC-determination of phenolic compounds and antioxidant capacity of Cactus Prickly Pear Seeds. *Asian Journal of Chemistry, 23*(3), 1006–1010.
Simopoulos, A. P., Leaf, A., & Salem Jr„ N. (2000). Workshop statement on the essentiality of and recommended dietary intakes for omega-6 and omega-3 fatty acids. *Prostaglandins Leukotrienes and Essential Fatty Acids, 63*(3), 119–121.
Singh, J., & Bargale, P. (1990). Mechanical expression of oil from linseed (*Linum usitatissimum* L.). *Journal of Oilseeds Research, 7*(1), 106–110.
Singleton, V. L., Orthofer, R., & Lamuela-Faventós, R. M. (1999). Analysis of total phenols and other oxidation substrates and antioxidants by means of Folin-Ciocalteau reagent. *Methods Enzymology, 299*, 152–178.
Teh, S-S., & Birch, J. (2013). Physico-chemical and quality characteristics of cold-pressed hemp, flax and canola seed oils. *Journal of Food Composition and Analysis, 30*, 26–31.
Thanonkaew, A., Wongyai, S., McClements, D., & Decker, E. A. (2012). Effect of stabilization of rice bran by domestic heating on mechanical extraction yield, quality, and antioxidant properties of cold-pressed rice bran oil (*Oryza saltiva* L.). *LWT-Food Science and Technology, 48*, 231–236.
Van Soest, P. J. (1963). Use of detergents in the analysis of fibrous feeds. I. Preparation of fiber residues of low nitrogen content. *Journal of Association Official Agricultural Chemists, 46*, 825–829.
Van Soest, P. J., & Wine, R. H. (1967). Use of detergents in the analysis of fibrous feeds. IV Determination of plant cell wall constituents. *Journal of Association Official Analytical Chemists, 50*, 50–55.
Viuda-Martos, M., Ruiz-Navajas, Y., Fernández-López, J., Sendra, E., Sayas-Barberá, E., & Pérez-Álvarez, J. A. (2011). Antioxidant properties of pomegranate (*Punica granatum* L.) bagasses obtained as co-product in the juice extraction. *Food Research International, 44*, 1217–1223.
Yaich, H., Garna, H., Besbes, S., Paquot, M., Blecker, C., & Attia, H. (2011). Chemical composition and functional properties of *Ulva lactuca* seaweed collected in Tunisia. *Food Chemistry, 128*, 895–901.