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ORIGINAL ARTICLE

Effect of seeds roasting time on physicochemical properties, oxidative stability, and antioxidant activity of cactus (*Opuntia ficus-indica* L.) seed oil

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Abstract

Cactus (*Opuntia ficus-indica* L.) seed oil, which is extracted from prickly pear fruit seeds, might constitute an alimentary source of substances of nutraceutical value. Physicochemical properties, chemical composition, antioxidant activity, and oxidative stability of prickly pear seed oil prepared from unroasted seeds were evaluated and compared with those of oil that was prepared from seeds roasted for different times. Prolonged roasting time had no significant influence on the protein content of the seeds. However, an increase in total phenolic compounds was observed from 225.9 mg GAE/100 g oil to 362.7 mg GAE/100 g oil after 40 min of roasting at 110°C. Consequently, an increase in antioxidant power which will induce a better oxidation resistance was found by the 1.1-diphenyl-2-picrylhydrazyl method expressed as EC_{50} value that was reduced from 0.6 to 0.1 mg/ml. No significant change was observed in triacylglyceride and fatty acid composition, whereas tocopherol (512.8 to 542.1 mg/kg after 40 min of roasting) and sterol (8,292 to 8,629 mg/kg after 40 min of roasting) levels increased. Oxidative stability increased remarkably from 3.1 to 7.6 hr after 40 min of roasting with the increase in roasting time. The current study revealed that prickly pear seeds and the resulting oil have excellent nutritional qualities that were significantly elevated after roasting.

Practical applications

Cactus seed oil is one of the most expensive vegetable oils and roasting of the seeds before pressing can help to improve not only the sensory quality of the oil but also the oxidative stability. Therefore, it is important to know more about the influence of roasting time on the quality of the oil. With this information, it is possible to optimize the roasting process regarding oil stability but also the release of bio-active compounds such as tocopherols or phenolic compounds. Overall, this will significantly improve the acceptance of this oil.

1 | INTRODUCTION

The prickly pear is a plant known under the scientific name *Opuntia ficus indica* L., native to Mexico, belonging to the Cactaceae family (Mulas & Mulas, 2004). It is economically important, but remains very little exploited. The plant can be used in agri-food, cosmetic, and pharmaceutical products. These products will play an important socio-economic role for farmers and rural populations and will contribute to sustainable development in rural areas (Arba, 2009).

Prickly pear seed oil contains several kinds of bioactive molecules which make the oil a nutritionally interesting product. In terms of fatty acids, prickly pear seed oil contains a very high content of linoleic acid, ranging from 49.7% to 62.1% (Chougui et al., 2013; Ennouri et al., 2005; Ennouri, Fetoui, Bourret, Zeghal, & Attia, 2006; Labuschagne & Hugo, 2010; Matthäus & Özcan, 2011; Ramadan & Moersel, 2003; Yeddes et al., 2012). The composition of phenolic compounds is found to be very rich in feruloyl derivatives. Other phenolic compounds have been cited in the literature such as flavonoids and tannins (Chougui et al., 2013) but also colored compounds such as betalain pigments that give prickly pear fruits its color (Butera et al., 2002). Tocopherols represent 0.403 g/kg and in particular γ-tocopherol with 0.330 g/kg (Matthäus & Özcan, 2011; Ramadan & Moersel, 2003). In terms of phytosterols, Ramadan and Morsel (2003) have shown the presence of very high contents of β-sitosterol and campesterol with 6.75 and 1.66 g/kg, respectively.

The composition of prickly pear seed oil shows several interesting properties due to large amounts of polyunsaturated fatty acids (Ennouri, Bourret, et al., 2006). It is very useful in the management of diabetes mellitus (Chougui et al., 2013). Prickly pear seed oil has been reported to have cholesterol-lowering and hypoglycemic effects (Ennouri, Fetoui, Bourret, Zeghal, & Attia, 2006; Ennouri, Fetoui, Bourret, Zeghal, Guermazi, et al., 2006). All these qualities encourage the exploitation and the use of prickly pear seed oil in the food and cosmetic industry (Piga, 2004).

Roasting is a heat treatment that has a strong impact on the quality of food with regard to flavor, color, texture, appearance, and also the composition. On one side, it destroys unwanted microorganisms and inactives enzymes that promote product deterioration during storage, but it is also responsible for the formation of antioxidant compounds formed during non-enzymatic reactions (Coghe et al., 2006).

The influence of roasting on the quality of oils is very controversially discussed, some researchers have reported that the chemical composition (fiber, ash, protein, and fatty acids) of the oil is independent of the roasting temperature (Anjum et al., 2006), while others have concluded that roasting influences the composition of the oil (Kim et al., 2002).

To the best of our knowledge, no study has been performed on the effect of roasting on the physicochemical properties and oxidative stability of prickly pear seed oil. The aim of this work was to study the influence of roasting time on the chemical composition and oxidative stability of the oil prepared from these seeds.

2 | MATERIALS AND METHODS

2.1 | Plant material and chemicals

The *Opuntia ficus-indica* fruits were harvested in June 2017 from Sidi Ifni, Morocco (29°22′45″ N, 10°10′17.6″ W; elevation: 0 m). It was identified by Professor Fennane from the Scientific Institute, Mohammed V University in Rabat. The seeds were separated from the fruits and dried. Roasting of seeds was carried out at 110 ± 5°C for 10, 20, 30 or 40 min using a roaster with continuous mixing of the material. The temperature was monitored using a Testo 945 thermometer sensor (Testo, Casablanca, Morocco; Nounah et al., 2020). The prickly pear seed oil was extracted by a KOMET D85 type screw press (Harhar et al., 2011). The yield of oil was determined gravimetrically (Annegowda et al., 2012). The oils were filtered and stored in brown glass bottles at 4°C until analysis (Chbani et al., 2020).

The procedure for extracting phenolic compounds from cactus seeds was: 1 g ground seeds of each sample was soaked in 10 ml of 80% methanol. Samples were sonicated for 30 min in a bath-type sonicator. The extract was centrifuged at 3,000 rpm for 5 min. The resulting insoluble residue was treated twice. After discarding the insoluble residue, the combined supernatants were collected and concentrated by a rotary evaporator at 40°C then stored in the fridge until use.

All reagents and solvents used were of analytical grade. The mobile phase was chromatographic grade and purchased from VWR international (Darmstadt, Germany).

2.2 | Moisture content, specific extinction, and free fatty acids (acidity)

Moisture content (ground seeds), extinction coefficients (K_{232} and K_{270}), and free fatty acids (acidity) (seed oil) were determined according to the official analytical methods (ISO 662:2016; ISO 3,656 2011; ISO 660 2009, respectively).

Moisture content was expressed as the percentage by mass. In brief, 10 g of seeds was placed in an oven regulated at 103°C for 3 hr until reaching a constant weight. The difference between the results of the last two determinations was 0.01 g of moisture per 100 g of sample.

The extinction coefficients K_{232} and K_{270} were expressed as the specific extinctions of a 1% (wt/vol) solution of oil in cyclohexane measured in a 10 mm cuvette, using a SCILOGEX SP- UV1100 spectrometer.

Free fatty acids were expressed as the weight percent of oleic acid and determined by the titration of a solution of oil in ethanol with NaOH (0.1 N).

2.3 | Protein content

Crude protein content was calculated from the nitrogen content of the seeds using a factor of 6.2. The nitrogen content of the ground

seeds was measured by the Kjeldahl procedure with a Gerhardt model Vapodest 20 instrument (Jadouali et al., 2018; Lee, 1995).

2.4 | Tocopherols composition

Method DGF-F-II 4a (DGF, 2013) was used to determine the tocopherol composition. One hundred and fifty mg of oil was dissolved in 10 ml of n-heptane. After filtration, 20 µl of the sample was directly injected by a Marathon Basic autosampler (Spark, Ajemmen, The Netherlands) onto a Diol phase HPLC column 25 cm ×4.6 mm ID (Merck, Darmstadt, Germany) used with a flow rate of 1.3 ml/min. The HPLC analysis was conducted using a Merck-Hitachi low-pressure gradient system, fitted with an L-6000 pump, a Merck-Hitachi F-1000 fluorescence spectrophotometer (detector wavelengths for excitation 295 nm, for emission 330 nm), and a ChemStation integration system. The mobile phase used was a 99:1 n-heptane/tert-butyl methyl ether (vol/vol).

2.5 | Triacylglyceride composition

Triacylglyceride composition was determined by gas chromatography analysis according to the method DGF-C-VI 14 (08) (DGF, 2013). An Agilent 6,890 GC-System combined with an Agilent 7683B injector (Agilent, Technologies Deutschland GmbH & Co., KG, Waldbronn, Germany), and flame ionization detector (FID) was used. Fifty milligrams of the oil were dissolved in 10 ml of isooctane and injected by an autosampler onto an RTX-65 column (30 m length, 0.32 mm ID, 0.1 µm film thickness) using hydrogen (flow rate 1 ml/min) as carrier gas. The initial oven temperature was 300°C, the final temperature was 360°C, and the temperature gradient was 2°C/min. Injector and detector temperature were set at 380°C. The injection volume of the samples was 1 µl in a split mode (split ratio 1:40). Triacylglycerides were identified by comparing their retention time to that of other oils, under the same analytical conditions, and data from the literature.

2.6 | Fatty acid composition

The fatty acid profile of the prickly pear seed oil was analyzed following the method DGF-C-VI 10 (13) (DGF, 2013). The fatty acid composition was determined as corresponding methyl esters by gas chromatography (HP5890, Agilent Tech. GmbH & Co., KG, Waldbronn, Germany) coupled with a flame ionization detector (GC-FID). The capillary column CP-Sil 88 (100 m × 250 µm i.d., 0.2 µm film thickness) was used. The carrier gas was hydrogen and the total gas flow rate was 1 ml/min. The initial oven temperature was 155°C, the final temperature was 220°C, and the temperature gradient was 1.5°C/min. Detector and injector temperature were set at 250°C. The injection volume of the samples was 1 µl in a split mode (split ratio 1:50). The standard mixture of fatty acids (Sigma Chemical Co.,) was used for the identification of peaks.

2.7 | Sterol composition

The sterol composition of the oils was determined following ISO 12,228:1999 (ISO, 1999). After the addition of betulin as internal standard, the oil (250 mg) was refluxed with 5 ml of ethanolic KOH solution for 15 min. The unsaponifiable matter was isolated by solid-phase extraction on an aluminum oxide column on which fatty acid anions were retained and unsaponifiable matter passed through. The sterol fraction was separated from unsaponifiable matter by thin-layer chromatography using as mobile phase a mixture of n-heptane and distilled diethyl ether (50:50 vol/vol). After separation, the sterol fraction was converted into silylated derivatives (TMS) using a silylating agent (MSHFBA, with 50 µl of methylimidazole solved in the MSHFBA-vail). The composition of the sterol fraction was determined by GC and the compounds were separated on a SE 54 CB (Macherey-Nagel, Düren, Germany; 50 m long, 0.32 mm ID, 0.25 µm film thickness). Further parameters were as follows: hydrogen as a carrier gas, injection, and detection temperature adjusted to 320°C, temperature program, 245–260°C at 5°C/min. Peaks were identified either by standard compounds (β-sitosterol, campesterol, stigmasterol), by a mixture of sterols isolated from rapeseed oil (brassicasterol) or by a mixture of sterols isolated from sunflower oil (Δ7-avenasterol, Δ7-stigmasterol, and Δ7-campesterol). The results were expressed as mg/kg of each individual sterol peak.

2.8 | Oxidative stability

The oxidative stability of the extracted oil was determined by the Rancimat method (743 Rancimat, Metrohm AG, Herisau, Switzerland). About 3 g oil was weighted into a test tube that was placed into the heating block kept at 120°C with an airflow of 20 L/h. Degradation products (volatile compounds) developed as a result of oxidation were transferred by the constant air flow into a measuring cell filled with distilled water. The change in conductivity resulting from the volatile compounds formed during oxidation was monitored constantly. The point of inflection of the curve recorded when the oil lost the ability to resist oxidation was proposed as induction time in hours (Matthäus, 2006).

2.9 | Amount of total phenolic compounds and antioxidant activity

The content of total phenolic compounds of extracts obtained from the seeds was determined by using Folin–Ciocalteu (FC) reagent as described by Nounah et al. (2017). The results are given as mg gallic acid equivalent/100 g of extract (mg GAE/100 g).

The free radical scavenging activity of the extracts was determined using 1.1-diphenyl-2-picrylhydrazyl (DPPH) according to Scherer and Godoy (2009) with some modifications. For each extract, different concentrations were tested. An aliquot (0.5 ml) of the DPPH solution (about 50 mg/100 ml) was diluted in 4.5 ml of methanol, and 0.1 ml of a methanolic solution of the extract was added. The mixture was shaken vigorously and allowed to stand for 45 min in the dark. The decrease in absorbance was measured at 515 nm against a blank (without extract) with a spectrophotometer. From a calibration curve obtained with different amounts of extract, the ED_{50} was calculated.

2.10 | Statistical analysis

Analysis of variance (ANOVA) was calculated by the software IBM SPSS Statistics 21 for checking the statistical significance by Tukey tests at a confidence level of 95.0%, and the results were presented as means ± standard deviation of the mean. The Pearson correlation calculation was performed using Microsoft Excel 2010 software.

3 | RESULTS AND DISCUSSION

3.1 | Moisture content

Moisture content is an important factor in giving some information about the stability of seeds, especially during storage. It is also one of the interesting factors influencing the quality of the oil extracted, as high water content causes the deterioration of the seeds resulting in poor quality rancid oil (Harhar et al., 2010). For a good preservation of oilseeds, the moisture content must not exceed 8%, as proposed by Brooker et al. (1992) and Patterson (1989).

Based on the recorded data (Table 1), it was noted that the amount of moisture in the seeds exceeded 8% only for unroasted seeds; indeed, the amount of water decreased from 8.1% for unroasted seeds to 3.0% after 40 min of roasting. The decrease in water content can be a factor that reduces the rancidity and oxidation of oils (De Wit et al., 2017). Several previous researches have mentioned that the roasting of oilseeds affects the content of moisture (Ermis & Yanmaz, 2012; Gharby et al., 2018).

3.2 | Protein content

Protein is the main requirement for the growth and development of all organisms (Friedman & Brandon, 2001). The protein content of prickly pear seeds is about 7 g/100 g DM; these proteins could be used as healthy ingredients in nutraceutical foods and can constitute a new food source for different population sectors. The protein content of unroasted prickly pear seeds was 7.0 g/100 g DM (Table 1) close to the value found by Tlili et al. (2011) for the prickly pear seeds of Tunisia (6%). After 40 min of roasting, the protein content decreased to 6.5%, but this change was not significant ($p < .05$). This small loss may be due to the Maillard-type non-enzymatic reactions when proteins react with reducing sugars during roasting (Coghe et al., 2006). Those substances possess preservation properties (Yen & Shyu, 1989; Yoshida, 1994) and are partially responsible for the better stability of edible argan oil, compared to beauty oil (Gharby, Harhar, Guillaume, et al., 2012). It also may be responsible for the high stability of prickly pear seed oil after 40 min of roasting.

3.3 | Oil yield

Initial oil yield in prickly pear seeds extracted by a screw press was 4.1%, this value is close to the value reported by Taoufik et al. (2015) with 5.4% for prickly pear seeds from Skhour Rhamna and it is lower than the value reported by Gharby et al. (2015) for prickly pear seeds from Sidi Ifni (6.5%). An oil yield ranging from 3.5% to 4.3% was obtained by roasting treatment (Table 1).

Roasting of seeds did not induce any significant modification of the extraction yield ($p > .05$), while Anjum et al. (2006) showed a significant difference in oil yield after the roasting of the seeds. Moreover, an increase in the oil yield of roasted lupine seeds has been reported by Yanez et al. (1983).

3.4 | Phenolic compounds and antioxidant activity

Phenolic compounds constitute a family of organic molecules widely present in the plant kingdom (Cartea et al., 2011). They are found in plants, from roots to fruits, and they are therefore an integral part

TABLE 1 Effect of roasting time on moisture content, total protein, yield of oil, total phenolic compounds, and antioxidant power

	Roasting time (min)				
	0	10	20	30	40
Moisture (%)	8.1 ± 0.3^a	4.5 ± 0.1^b	3.8 ± 0.1^c	3.6 ± 0.1^c	3.0 ± 0.1^d
Total protein (g/100 g DM)	7.0 ± 0.2^a	7.5 ± 0.1^a	6.7 ± 0.8^a	7.5 ± 0.10^a	6.5 ± 0.4^a
Oil yield (%)	4.1 ± 0.2^a	4.3 ± 0.1^a	3.5 ± 0.1^b	4.2 ± 0.2^a	3.5 ± 0.1^b
Total polyphenol content (mg GAE/100 g of extract)	226 ± 5^a	$2{,}145 \pm 7^b$	188 ± 6^c	262 ± 9^d	363 ± 11^e
EC_{50} value (mg/ml)	0.60 ± 0.01^a	0.60 ± 0.01^a	0.60 ± 0.01^a	0.50 ± 0.01^b	0.10 ± 0.01^c

Note: Mean values ± *SD* of determination for triplicate samples; means followed by similar letters superscript in the same column are not different ($p < .05$).

Abbreviation: DM, dry matter.

of our diet (Nounah et al., 2019). Phenolic compounds have antioxidant properties due to their ability to trap free radicals and reactive oxygen species which can initiate the radical process (Sökmen et al., 2012) and these compounds interfere with the oxidation of lipids and other molecules by the rapid donation of a hydrogen atom to free radicals (Rice-Evans et al., 1997). Butera et al. (2002) showed that aqueous extracts from prickly pear fruit have a marked antioxidant activity due to the content of betalain pigments as important antioxidant compounds. Du Toi et al. (2018) investigated the antioxidant content and activity in fresh and processed cactus pear fruits from different colored cultivars (purple, green, orange, and pink). The authors found the highest antioxidant content and potential in purple (*Opuntia robusta* cv Robusta) fruit products due to high amounts of betalains (1,140.4 mg/kg), while the second highest antioxidant activity was found for orange fruits (*O. ficus-indica*) as a result of higher amounts of ascorbic acid and phenolic compounds. During processing, betalains were highly retained in all processed products, while ascorbic acid was mainly found in processed products less treated by heat. De Wit et al. (2019) showed the appearance of antioxidant compounds in seeds, with the lowest antioxidant potential in the DPPH scavenging effect in comparison to fruit pulp, peel, and cladodes.

The total content of phenolic compounds was determined from the regression equation of the standard gallic acid curve ($y = 3.2273x - 0.1151$) and expressed in mg GAE/100 g of extract. Our results showed that the total content of phenolic compounds changed with the roasting time. The concentration of total phenolic compounds decreased from 225.9 mg GAE/100 g to 188 mg GAE/100 g during 20 min of roasting. This decrease may be due to the degradation of natural phenolic compounds by the heat treatment.

A roasting time of between 20 and 40 min led to an increase of the content of phenolic compounds. The latter reached a value of 362.7 mg GAE/100 g during 40 min of roasting at 110°C. The increase may be due to the release of phenolic compounds associated with the disruption of the cell walls during roasting.

Consistent with our current findings, some earlier studies have also found that roasting helps to increase the content of phenolic compounds in different foods. Win et al. (2011) showed that the amount of total phenolic compounds in skinless roasted peanut kernels increased from 94 mg GAE/100 g in the unroasted kernels to 204 mg GAE/100 g in kernels roasted at 160°C for 50 min. Similar results have been reported for dry beans (Boateng et al., 2008), carob powder vegetables (Sultana et al., 2008), and grape seeds (Kim et al., 2006).

In this context, it should be considered that the determination of the content of total phenolic compounds measures all compounds that react with the redox reagent Folin-Ciocalteu to form a blue complex, not only phenolic compounds. Therefore, the increase of total phenolic compounds as a result of roasting is mainly driven by the release of phenolic compounds or other antioxidant active components but could also be due to the formation of tyrosine, formic acid, and acetic acid that strongly interfered with the results (Bastola et al., 2017). Other compounds also interfere with the Folin–Ciocalteu reagent resulting in a higher measured amount of total phenolic compounds, but since phenolic compounds are the most abundant antioxidants in most plants, it gives a rough approximation of total phenolic compound content in most cases (Everette et al., 2010).

Phenolic compounds have significant antioxidant power (Nounah et al., 2017). In the present work, the DPPH test (1,1-diphenyl-1-picryl-hydrazyl) has been used. DPPH is a stable free radical capable of accepting an electron or hydrogen from antioxidant compounds and to be converted to a stable molecule of DPPH-H. The loss of the DPPH radicals can be monitored by the decrease in the absorbance of the test solution at 517 nm after the addition of the antioxidant extract. The results are expressed in EC_{50} (Table 1). The EC_{50} is the antioxidant concentration required to inhibit 50% of the radicals in the test solution.

The results of the present study showed that the roasting time resulted in a decrease in the EC_{50} value of water/methanol extracts (20/80, vol/vol) from prickly pear seeds, which revealed an increase of their antioxidant activity, since less extract is necessary to reach the same reduction of radical concentration. However, the EC_{50} is not affected during the initial (20 min) stage of roasting. A roasting time between 20 and 40 min reduced the EC_{50} value from 0.56 to 0.10 mg/ml showing a remarkable increase of the antioxidant power of the extracts due to the roasting time. Ali et al. (2016) reported that the antioxidant activity of seeds from groundnut increased with increasing heating time.

This shows that increasing the roasting time improves antioxidant activity. This improvement is correlated with the level of phenolic compounds and the ability of the seeds to release phenolic compounds to act as scavengers for free radicals in the cell matrix (Dewanto et al., 2002).

3.5 | Free fatty acid

Free fatty acid value in edible oils is one of the important parameters describing the quality and it classifies the grade of virgin oils such as argan and olive oils (Rao et al., 2009). As shown in Table 2, the amount of free fatty acids was equal ($p > .05$) for all oils obtained from roasted seeds. That indicates that the oil samples maintained a good quality during roasting as their acid values did not exceed the maximum limit of 4.0 mg KOH/g of oil according to the Codex Alimentarius (1999) (which is equivalent to 2% oleic acid when a conversion factor of 1.99 is used).

Several studies have reported that acidity increases with roasting time. Fukuda (1990) found that sesame oils from roasted seeds have higher acidity than oils from unroasted seeds. Similar results have also been reported by Harhar et al. (2011) for argan oil.

3.6 | UV absorption at 232 and 270 nm

Specific extinctions at 232 and 270 nm are useful to reflect the oxidative deterioration and purity of prickly pear seed oil through the

TABLE 2 Effect of roasting time on free fatty acids and specific extinction (232 and 270 nm)

	Roasting time (min)				
	0	10	20	30	40
FFA (%)	1.23 ± 0.03^{a}	1.28 ± 0.04^{a}	1.28 ± 0.05^{a}	1.28 ± 0.02^{a}	1.28 ± 0.04^{a}
K_{232}	3.3 ± 0.1^{a}	3.3 ± 0.2^{a}	3.7 ± 0.1^{a}	4.7 ± 0.1^{b}	9.1 ± 0.1^{c}
K_{270}	0.55 ± 0.01^{a}	0.56 ± 0.01^{a}	0.94 ± 0.03^{b}	1.28 ± 0.07^{c}	2.73 ± 0.25^{d}

Note: Mean values ± *SD* of determination for triplicate samples; means followed by similar letters superscript in the same column are not different ($p < .05$).

detection of peroxides (232 nm) and secondary oxidation products (270 nm) (Gharby et al., 2011). Specific extinctions in prickly pear seed oil prepared from unroasted kernels were 3.31 and 0.55 at 232 and 270 nm, respectively (Table 2). When prickly pear seeds were roasted at 110°C, the resulting oils did not show any significant change in the specific extinctions after 10 min of roasting, demonstrating the absence of formation of large quantities of primary and secondary oxidation products. However, the results suggest that primary and secondary oxidation products increased rapidly after 20 min of roasting. The influence of these products on the deterioration of prickly pear seed oil could be important because they could promote the formation of many non-fatty oxidizing products, rapidly decreasing the level of antioxidant molecules and promoting the formation of off-taste (Gharby et al., 2011; Matthäus et al., 2010). This finding was in agreement with those of Harhar et al. (2011) for argan oil.

3.7 | Triacylglyceride composition

Table 3 shows the triacylglyceride composition of oil from unroasted prickly pear seeds and prickly pear seeds roasted at 110°C for 10, 20, 30, and 40 min. The main triacylglycerides of prickly pear seed oils are PLL (19.4 ± 0.8 to 20.8 ± 0.4 g/100 g), LLO (18.5 ± 0.2 to 19.0 ± 0.7 g/100 g), and LLL (16.3 ± 0.6 to 16.6 ± 0.5 g/100 g) (O, oleic acid; L, linoleic acid; P, palmitic acid). No significant variations were observed in the triacylglyceride composition of prickly pear seed oil prepared from unroasted and roasted prickly pear seeds. Similar results have been reported in the literature for oils from sunflower, sesame, and pumpkin seeds (Yoshida et al., 2000, 2001).

3.8 | Fatty acids composition

The fatty acids composition of oil reflects its stability, but in the case of edible oils, it is also an essential indicator of its nutritional value (Gharby, Harhar, El Monfalouti, et al., 2012; Hajib et al., 2018; Harhar et al., 2011). The fatty acids composition of olive oil is very well documented and it is particularly rich in mono- and polyunsaturated fatty acids (Taoufik et al., 2015). Table 4 shows the results of the relative percentage of fatty acids of oils from unroasted and roasted prickly pear seeds. No significant differences in the fatty acid composition of prickly pear seed oils prepared with different roasting times were observed. Indeed, roasting of seeds did not induce any significant modification of the fatty acid distribution after 40 min at 110°C and was found to be in the range of published values for prickly pear seed oil from Morocco (Ennouri, Bourret, et al., 2006; Taoufik et al., 2015; Zine et al., 2013), or of geographically close origin (Matthäus & Özcan, 2011; Özcan and Al Juhaimi, 2011). Ghazi et al. (2013) also found a similar fatty acid composition for the oil extracted from prickly pear but much higher amounts of linoleic acid in *O. dilleniid* from Morocco. An investigation of Belviranli et al. (2019) on the fatty acid composition of prickly pear seeds from different locations in Turkey showed a high content of linoleic acid between 60.9% and 63.4% of the total fatty acids with significant differences between seeds from different locations. The authors assumed that these differences resulted from different locations of growth, climate, environmental conditions, and degree of ripeness. De Wit et al. (2017) investigated the fatty acid composition of seed oil from seven different South African cactus pear varieties (*O. ficus-indica* and *O. robusta*) with linoleic acid ranging from 55.8% to 67.3% in the seed oil as the main fatty acid. Oleic acid was the second important fatty acid with amounts between 15.2% and 21.8%. The results of the present work were also confirmed by an investigation of cactus pear seeds from 42 South African cultivars that found linoleic acids as the most dominant fatty acid (56.9%–65.2%), followed by oleic acid (16.4% and 22.5%) (de Wit et al., 2018). The content of palmitic (12.7%–16.1%) was a little lower in the present work while stearic acid (2.2%–3.4%) was found in higher amounts, probably due to differences in climatic conditions. Al Juhaimi et al. (2020) showed that the composition of the seed oil from *O. ficus-barbarica* was strongly affected by the stage of maturation. While at early harvest mid of June, highest values for oleic (28.5%), palmitic (22.6%), and stearic (9.2%) acid were found, at the latest stage of maturation (15 August) the content of linoleic acid (57.5%) was highest. The results for the fatty acid of prickly pear seeds were similar to the results mentioned by Harhar et al. (2011) for argan oil and by Lee et al. (2004) for safflower oil.

3.9 | Sterols composition

Prickly pear seed is rich in sterols, also known as phytosterols, and the major sterols in prickly pear seed oil are β-sitosterol, campesterol, Δ5-avenasterol, and stigmastanol (El Mannoubi et al., 2009; Taoufik et al., 2015). These compounds can be used to verify the

TABLE 3 Effect of roasting time on triacylglycerides (%) composition of prickly pear seed oil

Roast-ing time (min)	PLP	POO	PLS	PLO	PLL	OOO	SLO	OLO	LLO	LLL
0	4.6 ± 0.1^{a}	2.7 ± 0.1^{a}	3.2 ± 0.3^{a}	11.4 ± 0.2^{a}	20.8 ± 0.4^{a}	1.1 ± 0.1^{a}	3.4 ± 0.1^{a}	5.6 ± 0.2^{a}	18.7 ± 0.3^{a}	16.6 ± 0.5^{a}
10	4.5 ± 0.3^{a}	2.7 ± 0.2^{a}	3.1 ± 0.7^{a}	11.1 ± 0.3^{a}	20.3 ± 0.6^{a}	1.9 ± 0.1^{b}	3.8 ± 0.2^{b}	5.7 ± 0.1^{a}	18.5 ± 0.2^{a}	16.3 ± 0.6^{a}
20	4.3 ± 0.4^{a}	2.6 ± 0.1^{a}	3.0 ± 0.6^{a}	10.6 ± 0.4^{b}	19.4 ± 0.8^{a}	1.9 ± 0.5^{ab}	3.9 ± 0.3^{b}	5.7 ± 0.9^{a}	18.8 ± 0.4^{a}	16.5 ± 0.8^{a}
30	4.3 ± 0.2^{a}	2.5 ± 0.4^{a}	3.0 ± 0.7^{a}	10.6 ± 0.3^{b}	19.6 ± 0.1^{a}	1.3 ± 0.1^{a}	3.9 ± 0.4^{b}	5.7 ± 0.3^{a}	18.9 ± 0.9^{a}	16.6 ± 0.4^{a}
40	4.3 ± 0.3^{a}	2.5 ± 0.1^{a}	3.0 ± 0.2^{a}	10.6 ± 0.2^{b}	19.5 ± 0.9^{a}	1.8 ± 0.1^{b}	3.9 ± 0.4^{b}	5.7 ± 0.3^{a}	19.0 ± 0.7^{a}	16.5 ± 0.5^{a}

Note: Mean values ± *SD* of determination for triplicate samples; means followed by similar letters superscript in the same column are not different ($p < .05$).
Abbreviations: L, linoleic acid; O, oleic acid; P, palmitic acid; S, stearic acid.

authenticity of edible oils (Lucci et al., 2020). They are also important molecules endowed with potent biological properties and their determination is of major interest due to their antioxidant activity and impact on health (Gharby et al., 2016; Hajib et al., 2018; Taoufik et al., 2015). The results for individual and total phytosterols are listed in Table 5. The total sterols of prickly pear oil exceeded those of sunflower oil (4,510 mg/kg) and olive oil (2,560 mg/kg) (Schwartz et al., 2008). The richness of prickly pear oil in total sterols encourages the use of this oil because sterols play a role in cell differentiation and proliferation (Ifere et al., 2009).

As observed for fatty acids, no significant variations in individual sterol content were found for prickly pear seed oils prepared with different roasting times. However a slight increase was observed in total phytosterols of oils from roasted samples, varying between 8,292 mg/kg in oil from unroasted and 8,629 mg/kg in oil from prickly pear seeds roasted for 40 min, which is in agreement with that previously found by Matthäus et al. (2010) in sesame seeds roasted at 180°C for 30 min.

3.10 | Tocopherol composition

Tocopherols are natural lipophilic antioxidants present in extracted oils. This class of compounds has several benefits on overall human health (Azhari et al., 2014). They participate in the conservation of oil and also have certain therapeutic and antioxidant properties owed to their ability to scavenge free radicals (Matthäus et al., 2010; Monfalouti et al., 2010). Table 6 presents the individual and total tocopherol contents of oil from unroasted and roasted prickly pear seeds.

The main tocopherol of prickly pear seed oil is γ-tocopherol (473 mg/kg), while α- and δ-tocopherols represent 17.6 and 5.9 mg/kg, respectively, in the oil of unroasted seeds. In this study, as the roasting time increased, the content of the α-and δ-tocopherol increased significantly, while the content of γ-tocopherol as the main tocopherol decreased. One reason for this opposite development of the tocopherols could be that γ-tocopherol is more susceptible to oxidation that results in a decrease of the amount on one side, but also in the protection of the other tocopherols on the other side. This hypothesis is confirmed by results from frying experiments at higher temperature when the decomposition rate of γ-tocopherol also was greater than for α-tocopherol (Aggelousis & Lalas, 1997; Carlson & Tabacchi, 1986). In addition, heating during roasting improves the release of tocopherols from the cells resulting in an increase of α-and δ-tocopherol in the oil.

Our results showed that the γ-tocopherol content decreased slightly, reaching the lowest content when the seeds were roasted for 20–30 min. This can be interpreted as an effect of the thermal degradation of this tocopherol induced by high temperatures.

The content of α- and δ-tocopherol in prickly pear seed oil gradually increased as the roasting time increased.

Our results are consistent with those described in the literature for argan oil (Harhar et al., 2011). However, Lee et al. (2004)

TABLE 4 Effect of roasting time on fatty acids (%) composition of prickly pear seed oil

	Roasting time (min)				
	0	10	20	30	40
Palmitic acid C16:0	10.9 ± 0.1[a]	11.0 ± 0.1[a]	10.9 ± 0.1[a]	11.1 ± 0.2[a]	10.6 ± 0.5[a]
Stearic acid C18:0	3.8 ± 0.01[a]	3.8 ± 0.02[a]	3.9 ± 0.01[a]	3.8 ± 0.01[a]	3.9 ± 0.05[a]
Oleic acid C18:1	18.7 ± 0.01[a]	18.8 ± 0.03[a]	19.0 ± 0.01[a]	18.6 ± 0.02[a]	18.6 ± 0.05[a]
Linoleic acid C18:2	57.1 ± 0.11[a]	56.9 ± 0.1[a]	56.9 ± 0.1[a]	56.9 ± 0.1[a]	56.1 ± 0.4[b]
Linolenic acid C18:3	0.2 ± 0.1[a]	0.2 ± 0. 1[a]	0.2 ± 0. 1[a]	0.2 ± 0. 1[a]	0.2 ± 0. 1[a]
SFA	15.7 ± 0.2	15.7 ± 0.1	16.5 ± 0.1	15.8 ± 0.2	15.5 ± 0.4
UFA	82.4 ± 0.5	82.4 ± 0.6	82.5 ± 0.2	82.3 ± 0.9	81.9 ± 1.2
MUFA	24.7 ± 0.3	24.9 ± 0.7	24.9 ± 0.5	24.7 ± 0.3	24.7 ± 0.7

Note: Mean values ± *SD* of determination for triplicate samples; means followed by similar letters superscript in the same column are not different ($p < .05$).

Abbreviations: MUFA, monounsaturated fatty acids; SFA, saturated fatty acids; UFA, unsaturated fatty acids.

TABLE 5 Effect of roasting time on individual and total sterol composition of prickly pear seed oil

	Roasting time (min)				
	0	10	20	30	40
Total (mg/kg)	8,292±11[a]	8,422±10[b]	8,443±14[c]	8,582±14[d]	8,629±13[e]
campesterol (mg/kg)	837.5 ± 0.1[a]	850.6 ± 0.1[a]	852.7 ± 0.3[a]	866.8 ± 0.1[a]	871.5 ± 0.4[a]
β-sitosterol (mg/kg)	5,970±4[a]	6,047±3[a]	6,003±1[a]	6,136±2[a]	6,135±2[a]
stigmastanol (mg/kg)	281.9 ± 0.4[a]	294.7 ± 0.5[a]	295.5 ± 0.2[a]	300.3 ± 0.1[a]	302.0 ± 0.8[a]
Δ5-avenasterol (mg/kg)	339.9 ± 0.2[a]	345.3 ± 0.1[a]	354.6 ± 0.6[a]	360.4 ± 0.5[a]	362.4 ± 0.3a

Note: Mean values ± *SD* of determination for triplicate samples; means followed by similar letters superscript in the same column are not different ($p < .05$).

TABLE 6 Effect of roasting time on tocopherol composition (mg/kg) and oxidative induction time (Rancimat) of prickly pear seed oil

	Roasting time (min)				
	0	10	20	30	40
Total tocopherol	513 ±3[a]	514 ±2[a]	510 ±3[b]	499 ±3[c]	542 ±4[d]
α- tocopherol (mg/kg)	17.6 ± 0.5[a]	19.3 ± 0.5[b]	16.2 ± 0. 3[c]	19.4 ± 0. 5[b]	26.7 ± 0.4[d]
γ- tocopherol (mg/kg)	474 ±3[a]	468 ±4[b]	448 ±3[c]	448 ±2[c]	464 ±3[d]
δ- tocopherol (mg/kg)	5.9 ± 0.2[a]	5.08 ± 0.6[a]	7.8 ± 0.3[b]	9.0 ± 0. 6[c]	12.01 ± 0.9[d]
Rancimat (h at 120°C)	3.1 ± 0.2[a]	3.3 ± 0.1[a]	3.7 ± 0.3[a]	5.1 ± 0.4[b]	7.6 ± 0.8[c]

Note: Mean values ± *SD* of determination for triplicate samples; means followed by similar letters superscript in the same column are not different ($p < .05$).

reported that the levels of α-tocopherol in safflower oil gradually increased as the roasting temperature increased to 160°C. It has also been reported that the total amount of tocopherols in oil from milk thistle seeds was significantly increased ($p < .05$) with microwave heating (Fathi-Achachlouei et al., 2019), while Yoshida and Takagi (1996) reported that the tocopherol content gradually decreased in oils prepared from soybeans roasted in the microwave oven and that the percentage of losses increased considerably after 12 min of exposure time. These results suggest that temperature and roasting time influence the tocopherol content in oils (Anjum et al., 2006).

3.11 | Oxidation induction time (OIT)

Oxidative stability of prickly pear seed oils was evaluated using the Rancimat method. The Rancimat method makes it possible to

TABLE 7 Pearson's correlation matrix coefficient between phenolic content, K232, K270, phytosterols, total tocopherols, and induction time

	Phenolic content	K232	K270	Phytosterols	Total tocopherol	Induction time
Phenolic content	1					
K232	0.958	1				
K270	0.929	0.990	1			
Phytosterols	0.733	0.781	0.835	1		
Total tocopherol	0.747	0.817	0.751	0.353	1	
Induction time	0.946	0.978	0.989	0.883	0.685	1

determine the induction time of oil subjected to accelerated heating conditions (120°C) and air supply of 20 L/h. The induction time mainly measures the formation of secondary oxidation products and it depends on the composition of the oil and the amount of antioxidants present in oils (Pereira et al., 2019). The induction time of prickly pear seed oil prepared from unroasted seeds was 3.1 hr, the shortest of all studied samples (Table 6). The induction time increased gradually with increasing roasting time. Prickly pear seed oil obtained after a roasting time of 40 min showed a remarkably higher induction time (7.6 hr). The roasting time significantly increased the induction time of the oils ($p < .05$). Similar results were reported for oils from roasted pistachios (Rabadán et al., 2018), argan (Harhar et al., 2011), and rapeseeds (Rękas et al., 2017).

The induction time exhibited a highly significant positive correlation with the content of phenolic compounds, phytosterols, and tocopherols ($r^2 = 0.946$, 0.883, and 0.685, respectively, $p \leq .05$) indicating that an increase in the amount of the antioxidant components increases the oxidative stability of oils (Table 7). The antioxidant nature of phenolic compounds, phytosterols, and tocopherols plays an important role in the oxidative stability of edible oils (Gharby et al., 2014). The highly significant negative correlation of the induction time with K_{232} and K_{270} ($r^2 = -0.978$ and -0.989, respectively, $p \leq .005$) indicated that higher amounts of primary and secondary oxidation products were formed during the roasting processes, but on the other side, the oxidative stability of the oils increased with increasing roasting time.

4 | CONCLUSION

The results presented in this study showed that the duration of roasting of prickly pear seeds decreased the water and protein content of the seeds. Moreover, the content of total phenolic compounds increased with the duration of roasting and therefore the antioxidant power of seeds. Regarding the physicochemical parameters and the oxidation stability of prickly pear seed oil prepared from seeds roasted at 110°C for 10 to 40 min, our results showed no significant change in FFA; however, the resistance to oxidation (Rancimat) had been improved. The amount and composition of phytosterols, fatty acids, and triacylglycerides were not modified in the roasted samples, while minor modifications have been observed in the composition of the tocopherols.

From these results, roasting seems to be an essential treatment to preserve the immediate and long-term nutritional and physicochemical properties of prickly pear seed oil.

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CONFLICTS OF INTEREST

The authors have declared no conflicts of interest for this article.

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